Thermo Scientific

Dionex IonPac AS9-HC

Column Product Manual

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Thermo
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Product Manual

for

Dionex IonPac AG9-HC Guard Column

(4 × 50 mm, P/N 051791)
(2 × 50 mm, P/N 052248)

Dionex IonPac AG9-HC Capillary Guard Column

(0.4 × 50 mm, P/N 088296)

Dionex IonPac AS9-HC Analytical Column

(4 × 250 mm, P/N 051786)
(2 × 250 mm, P/N 052244)

Dionex IonPac AS9-HC Capillary Column

(0.4 × 250 mm, P/N 082319)

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Revision History:

Revision 11, May, 2014: Rebranded for Thermo Scientific. Added capillary column information.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

SAFETY	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
WARNING	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
CAUTION	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
NOTE	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

Contents

1. Introduction

The Thermo Scientific™ Dionex™ IonPac™ AS9-HC Analytical/Capillary Column in combination with the Thermo Scientific Dionex IonPac AG9-HC Guard/Capillary Guard Column is designed for the analysis of inorganic anions and oxyhalides including bromate, chlorite, and chlorate. The selectivity of the Dionex IonPac AS9-HC Guard plus Analytical Column set has been designed to retain fluoride well out of the water dip (system dip) and to isocratically separate common anions. The Dionex IonPac AS9-HC column is compatible with pH 0-12 eluents and eluents containing organic solvents from 0 - 100% in concentration. The Dionex IonPac AS9-HC column can be used with any suppressible ionic eluent that does not exceed the capacity of the suppressor. The Dionex IonPac AS9-HC column has nominal efficiency of at least 6,000 plates/column for sulfate using standard operating conditions.

The Dionex IonPac AS9-HC Capillary Column (0.4 × 250 mm) is packed with the same material as the equivalent standard bore version (producing the same performance as a 4 mm column), but requires only 1/100th the eluent flow rate. The capillary format offers the advantage of less eluent consumption providing reduced costs.

Table 1 Packing Specifications

Column	Nominal Particle Diameter, µm	Substrate X-linking, %	Column Capacity, µeq/column	Functional Group	Hydrophobicity
Dionex IonPac AS9-HC 4 × 250 mm	7	55	190	Alkyl/Alkanol quaternary ammonium	Medium-low
Dionex IonPac AG9-HC 4 × 50 mm	11	55	6	Alkyl/Alkanol quaternary ammonium	Medium-low
Dionex IonPac AS9-HC 2 × 250 mm	7	55	47.5	Alkyl/Alkanol quaternary ammonium	Medium-low
Dionex IonPac AG9-HC 2 × 50 mm	11	55	1.5	Alkyl/Alkanol quaternary ammonium	Medium-low
Dionex IonPac AS9-HC 0.4 × 250 mm	7	55	1.9	Alkyl/Alkanol quaternary ammonium	Medium-low
Dionex IonPac AG9-HC 0.4 × 50 mm	11	55	0.06	Alkyl/Alkanol quaternary ammonium	Medium-low

Analytical Column resin composition: supermacroporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene.

Guard Column resin composition: microporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene.

Table 2 Operating Parameters

Column	Typical Back Pressure psi (MPa[a]), 30°C[b]	Standard Flow Rate, mL/min	Maximum Flow Rate, mL/min[c]
Dionex IonPac AS9-HC 4 mm Analytical	≤ 2000 (13.78)	1.0	3.0
Dionex IonPac AG9-HC 4 mm Guard	≤ 250 (1.72)	1.0	3.0
Dionex IonPac AS9-HC + Dionex IonPac AG9-HC 4 mm columns	≤ 2250 (15.5)	1.0	3.0
Dionex IonPac AS9-HC 2 mm Analytical	≤ 2000 (13.78)	0.25	0.75
Dionex IonPac AG9-HC 2 mm Guard	≤ 250 (1.72)	0.25	0.75
Dionex IonPac AS9-HC + Dionex IonPac AG9-HC 2 mm columns	≤ 2250 (15.5)	0.25	0.75
Dionex IonPac AS9-HC 0.4 mm Capillary	≤ 2000 (13.78)	0.010	0.030
Dionex IonPac AG9-HC 0.4 mm Capillary Guard	≤ 250 (1.72)	0.010	0.030
Dionex IonPac AS9-HC + Dionex IonPac AG9-HC 0.4 mm columns	≤ 2250 (15.5)	0.010	0.030

[a] **Note: 1 MPa = 145.04psi**
[b] **Total backpressure at standard flow rates.**
[c] **In all cases, flow rate should not result in pressure over 3000 psi.**

WARNING

Exceeding the maximum flow rates listed in the above table, can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column.

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

2. Ion Chromatography Systems

The proper configuration of an Ion Chromatography System (ICS) in 2 mm or 4 mm format is based on the ratio of the 2 mm to 4 mm column cross-sectional area (a factor of 1/4). The selected format will affect the type of pump recommended. A gradient pump is designed to blend and pump isocratic, linear, or gradient mixtures of up to four mobile phase components at precisely controlled flow rates. An isocratic pump is for applications not requiring gradient and multi-eluent proportioning capabilities. Both are offered in either standard bore or microbore options.

- For an ICS in 2 mm format, a microbore isocratic pump, standard bore isocratic pump, microbore gradient pump, or standard bore gradient pump is recommended.
- For an ICS in 4 mm format, a standard bore isocratic pump or standard bore gradient pump is recommended.
- For an ICS in 0.4 mm format, a Capillary IC system such as the Thermo Scientific Dionex ICS-5000^{+} HPIC system or Thermo Scientific Dionex ICS-4000 capillary system is recommended.

See Appendix B, ”System Configuration” for specific recommended settings and parts including pumps, eluent flow rate, Thermo Scientific Dionex Electrolytically Regenerated Suppressor (ERS), Thermo Scientific Dionex MicroMembrane Suppressor (MMS), Thermo Scientific Dionex Capillary Electrolytic Suppressor (CES), injection loop, system void volume, detectors, and tubing back pressure.

3. Installation

3.1 Column Start-Up

The column is shipped using 100 mM Sodium bicarbonate as the storage solution.

Prepare the eluent shown on the Quality Assurance Report (QAR), install the column in the chromatography module and direct the column effluent to waste for 30 minutes, and then connect to the suppressor. Test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

If peak efficiencies or resolution on the capillary column are poorer than the QAR, see Section 3.14 “Installation of the Capillary Column” and 6.3.5 “Poor Efficiency using Capillary Columns” for information regarding proper connections.

3.2 Column Storage

For short-term storage (< 1 week), use eluent, for long-term storage, use 100 mM Sodium Bicarbonate for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

3.3 System Requirements

3.3.1 System Requirements for 0.4 mm Operation

The Dionex IonPac AS9-HC 0.4 mm Capillary Guard and Capillary Columns are designed to be run on a capillary ion chromatography system equipped with suppressed conductivity detection. It is recommended to run the capillary column only on a capillary IC system such as the Dionex ICS-5000^{+} HPIC system or Dionex ICS-4000 capillary system for best performance.

3.3.2 System Requirements for 2 mm Operation

The Dionex IonPac AS9-HC 2 mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a pump with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a microbore pump (1/16" pistons) is recommended.

3.3.3 System Requirements for 4 mm Operation

The Dionex IonPacAS9-HC 4 mm Guard and Analytical Columns are designed to run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a pump with a standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons).

3.3.4 System Void Volume

When using 2 mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4 mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" i.d. PEEK tubing (P/N 044221). PEEK tubing of 0.010" i.d. (P/N 042260) may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

3.4 The Sample Concentrator

The function of a concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the concentrator column, lowering detection limits by 2- 5 orders of magnitude. The concentrator column is used in lieu of the sample loop.

The Dionex IonPac Trace Anion Concentrator Low Pressure Column (Dionex IonPac TAC-LP1, P/N 046026), the Dionex IonPac Trace Anion Concentrator Ultra Low Pressure Column (Dionex IonPac TAC-ULP1, P/N 061400), the Dionex IonPac Ultra Trace Anion Concentrator Low Pressure Column (Dionex IonPac UTAC-LP1, P/N 063079) or (Dionex IonPac UTAC-LP2, P/N 079917), the Dionex IonPac Ultra Trace Anion Concentrator Ultra Low Pressure Column (Dionex IonPac UTAC-ULP1, P/N 063475) or (Dionex IonPac UTAC-ULP2, P/N 079918), the Dionex IonPac Ultra Trace Anion Concentrator Extremely Low Pressure Column (Dionex IonPac UTAC-XLP1, P/N 063459) or (Dionex IonPac UTAC-XLP2, P/N 072781), or the Dionex IonPac AG9-HC Guard Column can be used for trace anion concentration work with the 2 mm and 4 mm Dionex IonPac AS9-HC column. For trace anion concentration work with the 0.4 mm Dionex IonPac AS9-HC capillary column, use the Dionex IonSwift Monolith Anion Concentrator Column (Dionex IonSwift MAC-100, P/N 074702).

Pump the sample onto the concentrator column in the OPPOSITE direction of the eluent flow. When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column. For more detailed information on sample concentration techniques for high sensitivity work and a detailed discussion of anion concentration techniques refer to:

- Section 4, "Operation," of the Dionex IonPac Trace Anion Concentrator Low Pressure (Dionex IonPac TAC-LP1) and Dionex IonPac Ultra Low Pressure (Dionex IonPac TAC-ULP1) Column Product Manual (Document No. 034972).
- Section 4, "Operation," of the Dionex IonPac Ultra Trace Anion Concentrator Low Pressure (Dionex IonPac UTAC-LP1), Dionex IonPac Ultra Low Pressure (Dionex IonPac UTAC-ULP1), and Dionex IonPac Extremely Low Pressure (Dionex IonPac UTAC-XLP1) Column Product Manual (Document No. 065091.)
- Section 4, "Operation," of the Dionex IonPac Ultra Trace Anion Concentrator 2 Low Pressure (Dionex IonPac UTAC-LP2), Dionex IonPac Ultra Low Pressure (Dionex IonPac UTAC-ULP2), and Dionex IonPac Extremely Low Pressure (Dionex IonPac UTAC-XLP2) Column Product Manual (Document No. 065376.)
- Section 4, "Operation" of the Dionex IonSwift Monolith Anion Concentrator (Dionex IonSwift MAC-100) Column Product Manual (Document No. 065387).

3.5 The Injection Loop

3.5.1 The 0.4 mm System Injection Loop, 0.4 µL Internal Loop and 2 µL External Loop

For most applications on a 0.4 mm capillary system, a 0.4 µL injection loop is sufficient. Generally, do not inject more than 0.5 nanomoles of any one analyte into a 0.4 mm capillary column. Injecting larger numbers of moles of a sample can result in overloading the column, which can affect the detection linearity. For samples containing low concentrations of analytes, larger injection loops can be used to increase sensitivity.

3.5.2 The 2 mm System Injection Loop, 2 - 15 µL

For most applications on a 2 mm analytical system, a 2 - 15 µL injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. The Dionex IonPac AS9-HC 2 mm column requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4 mm analytical system (Appendix B, "System Configuration").

3.5.3 The 4 mm System Injection Loop, 10 - 50 µL

For most applications on a 4 mm analytical system, a 10 - 50 µL injection loop is sufficient. Generally, you should not inject more than 50 nanomoles of any one analyte onto the 4 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. For typical drinking water samples, you can inject up to 200 µL.

3.6 The Dionex IonPac AG9-HC Guard Column

The Dionex IonPac AG9-HC Guard/Capillary Guard Column is normally used with the Dionex IonPac AS9-HC Analytical/Capillary Column. Retention times will increase by approximately 2-3% when a guard column is placed in-line prior to the analytical column. A guard/capillary guard is placed prior to the analytical/capillary column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical/capillary column. Replacing the Dionex IonPac AG9-HC Guard/Capillary Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the Dionex IonPac AS9-HC Analytical/Capillary Column.

3.7 Installing the Thermo Scientific Dionex IonPac Anion Trap Column, Dionex IonPac ATC-3 Column

When performing a gradient anion exchange application, a borate eluent system should be used instead of a carbonate system because of its low background conductivity. A Dionex IonPac Anion Trap Column (Dionex IonPac ATC-3 (2 mm), P/N 059661 or Dionex IonPac ATC-3 (4 mm), P/N 059660) trap column should be installed between the gradient pump and the injection valve. Remove the high pressure Gradient Mixer if present. The Dionex IonPac ATC column is filled with high capacity anion exchange resin which helps to minimize the baseline shift caused by increasing anionic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis.

To install the Dionex IonPac ATC-3 (2 mm) or Dionex IonPac ATC-3 (4 mm) column, complete the following steps:

A. Remove the Gradient Mixer, if installed between the gradient pump pressure transducer and the injection valve.

B. Connect the gradient pump directly to the Dionex IonPac ATC column. Connect a waste line to the Dionex IonPac ATC column outlet and direct the line to a waste container.

C. Flush the Dionex IonPac ATC column with 200 mL of 70 mM $Na_2B_4O_7$ at a flow rate of 0.5 mL/min when using the Dionex IonPac ATC-3 (2 mm) column or 2.0 mL/min when using the Dionex IonPac ATC-3 (4 mm) column.

D. Rinse the Dionex IonPac ATC column with the strongest eluent that will be used during the gradient analysis.

E. After flushing the Dionex IonPac ATC column with eluent, connect the Dionex IonPac ATC column to the eluent line that is connected to the injection valve.

The background conductivity of your system should be less than 7 µS when $Na_2B_4O_7$ is being pumped through the chromatographic system with the Dionex AERS suppressor in-line and properly functioning. The baseline shift should be no greater than 10 µS during a borate gradient eluent concentration ramp from 0 to 70 mM $Na_2B_4O_7$. If the baseline shifts are greater than 10 µS, the Dionex IonPac ATC column should be cleaned using steps A - E above.

The Dionex IonPac ATC column can be flushed, at the end of each operating day, to remove any impurities that may have accumulated on it. This will minimize periodic maintenance and lost data.

A. Flush the Dionex IonPac ATC column with 30 mL of 70 mM $Na_2B_4O_7$.

B. Prior to next day use of the chromatographic system, flush the Dionex IonPac ATC column with 30 mL of the strongest eluent used in the gradient program.

See the Product Manual for the Dionex IonPac ATC-3 column (Document Number 032697) for instructions on cleaning a contaminated Dionex IonPac Anion Trap Column.

3.8 Eluent Storage

Dionex IonPac AS9-HC columns are designed to be used with bicarbonate/carbonate eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

NOTE

It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of CO_2 will affect retention times and selectivity. This is particularly important for Capillary IC as a single batch of eluent can last up to 3 months.

3.9 Dionex Anion Electrolytically Regenerated Suppressor (Dionex AERS 500) Requirements

A Dionex Anion Electrolytically Regenerated Suppressor should be used for applications that require suppressed conductivity detection. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all Dionex AERS 500 Anion Electrolytically Regenerated Suppressor modes of operation.

NOTE

The Dionex AERS 500 suppressor does not support the chemical regeneration mode. The Dionex MicroMembrane MMS 300 suppressor is recommended for chemical regeneration mode.

NOTE

Solvent containing eluents should be used in the AutoSuppression External Water Mode.

For Dionex IonPac AS9-HC 4 mm Analytical Column, use a Dionex AERS 500 (4 mm, P/N 082540).

For Dionex IonPac AS9-HC 2 mm Analytical Column, use a Dionex AERS 500 (2 mm, P/N 082541).

For Dionex IonPac AS9-HC 0.4 mm Capillary Column, use a Dionex ACES 300 (0.4 mm, P/N 072052).

For detailed information on the operation of the *Dionex Anion Electrolytically Regenerated Suppressor*, see Document No. 031956, the “Thermo Scientific Dionex ERS 500 Suppressor Product Manual”.

For detailed information on the operation of the *Dionex Anion Capillary Electrolytic Suppressor* 300, see Document No. 065386, the “Thermo Scientific Dionex CES 300 Product Manual”.

3.10 Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300) Requirements

A Dionex AMMS 300 suppressor may be used instead of a Dionex AERS 500 suppressor (4 mm) for applications that require suppressed conductivity detection. Use a Dionex AMMS 300 (4 mm) (P/N 064558) with the Dionex IonPac AS9-HC 4 mm Analytical column. It is compatible with all solvents and concentrations with which the systems and columns are compatible. For 2 mm operation, use the Dionex AMMS 300 2 mm (P/N 064559).

For detailed information on the operation of the Dionex Anion MicroMembrane Suppressor, see Document No. 031727, the "Product Manual for the Dionex Anion MicroMembrane Suppressor 300, the Dionex AMMS 300".

3.11 Using Dionex AutoRegen with the Dionex AMMS 300 in the Chemical Suppression Mode

To save regenerant preparation time and reduce regenerant consumption and waste, it is recommended to use an AutoRegen Accessory (P/N 039594). For more detailed information on the use of the Dionex AutoRegen Accessory see the Dionex AutoRegen Accessory manual (Document No. 032853). For more detailed information on the use of Dionex AutoRegen Regenerant Cartridges, see the "Product Manual for the Dionex AutoRegen Regenerant Cartridge Refills" (Document No. 032852).

3.12 Using Dionex Displacement Chemical Regeneration (DCR) with the Chemical Suppression Mode

The Dionex Displacement Chemical Regeneration (Dionex DCR) Mode is recommended for chemical suppression using sulfuric acid and the Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300). See the Dionex DCR kit manual, Document P/N 031664, for details.

SAFETY

Use proper safety precautions in handling acids and bases.

3.13 Using the Dionex EGC-Carbonate Eluent Generator

The Dionex IonPac AS9-HC column is recommended for use with Dionex systems equipped with a Dionex Eluent Generator. The Dionex Eluent Generator is used to automatically produce carbonate and bicarbonate eluents from deionized water. For more information, see the manual for the Dionex Eluent Generator Cartridges Product Manual, Document No. 065018.

3.14 Detector Requirements

See Section 2, "Ion Chromatography Systems", for standard bore, microbore and capillary system detector, cell and thermal stabilizer requirements.

3.15 Installation of the Capillary Column

1. Before installing the new separator column, cut off the column label and slide it into the holder on the front of the cartridge (see Figure 6).
2. For reference, Figure 1 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 2 shows the column cartridge after installation of only a capillary separator column.

Figure 1 Separator and Guard Columns Installed in Column Cartridge

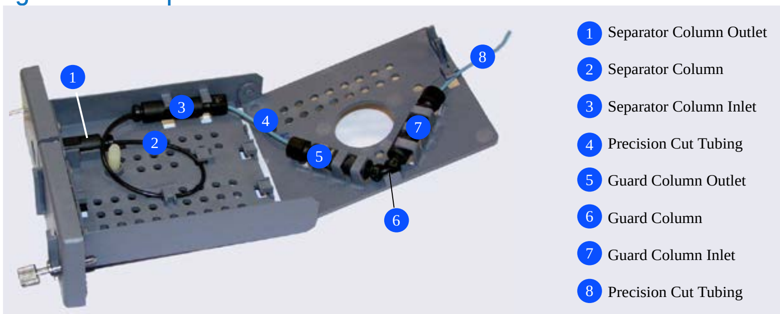
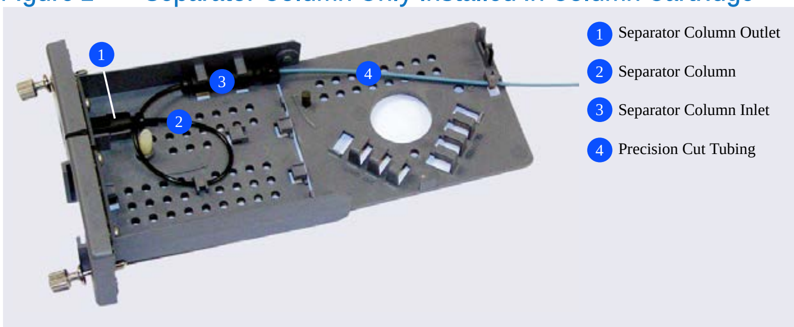


Figure 2 Separator Column Only Installed in Column Cartridge



3. Locate the Dionex IC Cube Tubing Kit (P/N 072186) that is shipped with the Dionex IC Cube. The tubing kit includes the following items:

Table 3 Contents of the Dionex IC Cube Tubing Kit (P/N 072186)

Part	Length / Quantity	Part Number	Used To Connect
Precision cut 0.062 mm (0.0025-in) ID PEEK tubing, blue	65 mm (2.56 in)	072188	50 mm guard column outlet to 250 mm separator column inlet
Precision cut 0.062 mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	115 mm (4.53 in)	072189	Guard column inlet to injection valve
Precision cut 0.062 mm (0.0025-in) ID PEEK tubing, blue	75 mm (2.93 in)	074603	35 mm guard column outlet to 150 mm separator column inlet
Precision cut 0.062 mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	210 mm (8.27 in)	072187	Separator column inlet to injection valve (if a guard column is not present)
0.25 mm (0.010-in) ID PEEK tubing, black	610 mm (24 in)	042690	EG degas cartridge REGEN OUT to waste (if an EG is not present)
Fitting bolt, 10-32 hex double-cone (smaller), black	3	072949	Connect precision cut 0.062 mm (0.0025-in) ID PEEK tubing
Fitting bolt, 10-32 double-cone (larger), black	1	043275	Connect 0.25 mm (0.010-in) ID PEEK tubing (black)
Ferrule fitting, 10-32 double-cone, tan	4	043276	Use with both sizes of fitting bolts

5. Refer to the following figures for the precision cut tubing required for your configuration:

Figure 3 Tubing Connections for 250 mm Separator Column and 50 mm Guard Column

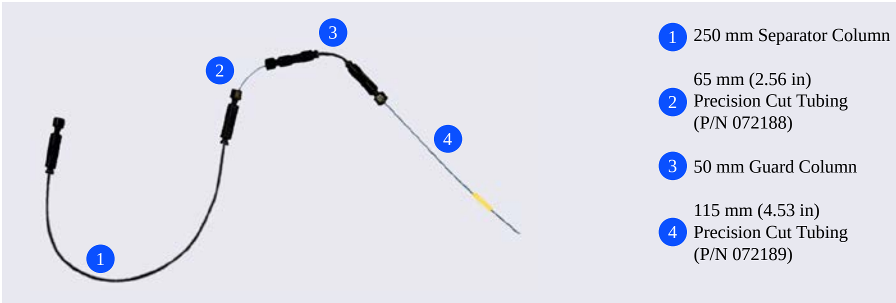
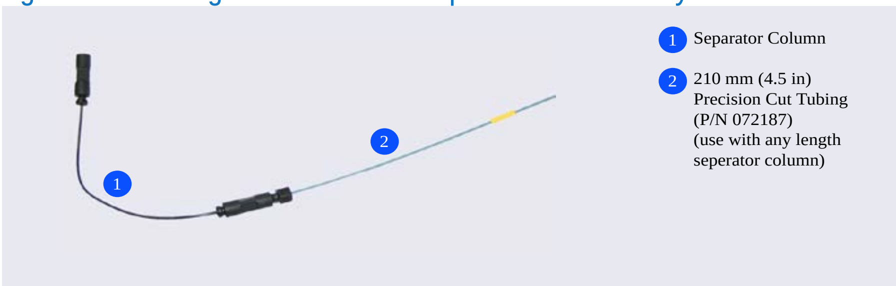
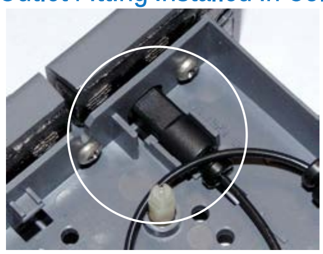


Figure 4 Tubing Connections for Separator Column Only



6. Lift up the lid of the column cartridge to open it.
7. Remove the fitting plug from the outlet fitting on the separator column. Orient the fitting with a flat side up (see Figure 5) and push the fitting into the opening at the front of the column cartridge until it stops.

Figure 5 Column Outlet Fitting Installed in Column Cartridge



8. Coil the separator column tubing inside the cartridge as shown in Figure 1 or Figure 2. Secure the column tubing and the inlet fitting in the clips on the column cartridge.
9. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
10. Route the guard column inlet tubing (if used) or the separator column inlet tubing through the clip on the top edge of the column cartridge lid.
11. Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see Figure 6).

NOTE

If the columns are installed correctly, the cartridge lid snaps closed easily. If the lid does not close easily, do not force it. Open the lid and verify that the columns and tubing are installed correctly and secured in the clips.

Figure 6 Column Cartridge Closed

4. Operation

4.1 General Operating Conditions

Sample Volume:	0.4 mm: 0.4 µL loop 2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume 4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	0.4 mm: Dionex IonPac AS9-HC 0.4 mm Capillary Column + Dionex IonPac AG9-HC 0.4 mm Capillary Guard column 2 mm: Dionex IonPac AS9-HC 2 mm Analytical Column + Dionex IonPac AG9-HC 2 mm Guard Column Analytical Column 4 mm: Dionex IonPac AS9-HC 4 mm Analytical Column + Dionex IonPac AG9-HC 4 mm Guard Column Analytical Column
Eluent:	9 mM Na_2CO_3
Temperature:	30 °C
Eluent Flow Rate:	0.4 mm: 10 µL/min 2 mm: 0.25 mL/min 4 mm: 1.0 mL/min
ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor, Dionex AERS 500 (2 or 4 mm)
CES Suppressor:	Dionex Anion Capillary Electrolytic Suppressor, ACES 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS 300 (2 mm or 4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	24-30 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

4.2 Dionex IonPac AS9-HC Column Operation Precautions

CAUTION

Filter and Degas Eluents
Filter Samples
Eluent pH between 0 and 12
Sample pH between 0 and 14
0.75 mL/min Maximum Flow Rate for 2 mm Columns
3.0 mL/min Maximum Flow Rate for 4 mm Columns
30 µL/min Maximum Flow Rate for 0.4 mm Columns
Maximum Operating Pressure = 4,000 psi (27.57 MPa)

4.3 Chemical Purity Requirements

Obtaining reliable, consistent and accurate results requires eluents that are free of ionic impurities. Chemicals, solvents and deionized water used to prepare eluents must be of the highest purity available. Low trace impurities and low particle levels in eluents also help to protect your ion exchange columns and system components. Thermo Fisher Scientific cannot guarantee proper column performance when the quality of the chemicals, solvents and water used to prepare eluents has been compromised.

4.3.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label. Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex IonPac AS9-HC Eluent Concentrate (P/N 064161) is recommended in order to avoid this problem. Otherwise, use of a high purity grade of sodium carbonate to prepare eluents will generally prevent the problem.

4.3.2 Deionized Water

The deionized water used to prepare eluents should be Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 µm. Bottled HPLC-Grade Water (with the exception of Burdick & Jackson) should not be used since most bottled water contains an unacceptable level of ionic impurities.

4.3.3 Solvents

Solvents can be added to the ionic eluents used with Dionex IonPac AS9-HC column to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Thermo Fisher Scientific, we have obtained consistent results using Optima™ Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the Dionex IonPac AS9-HC column is 4,000 psi (27.57 MPa).

The Dionex IonPac AS9-HC column can withstand common HPLC solvents in a concentration range of 0 - 100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 4 HPLC Solvents for Use with Dionex IonPac AS9-HC Column

Solvent	Maximum Operating Concentration
Acetonitrile	100%
Methanol	100%
2-Propanol	100%
Tetrahydrofuran	20% *

* Higher concentration may only be used for limited duration applications such as column clean up at pressures < 2000 psi

CAUTION

The Dionex Anion Electrically Regenerated Anion Suppressor (Dionex AERS 500) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

4.4 Making Eluents that Contain Solvents

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. For example, if a procedure requires an eluent of 90% acetonitrile, prepare the eluent by adding 900 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.

NOTE

When purging or degassing eluents containing solvents, do not purge or degas the eluent excessively since it is possible that a volatile solvent can be "boiled" off from the solution.

NOTE

Always degas and store all eluents in glass or plastic eluent bottles pressurized with helium. Only helium can be used to purge and degas ionic eluents containing solvents, since nitrogen is soluble in solvent containing eluents.

NOTE

Acetonitrile (ACN) hydrolyzes to ammonia and acetate when left exposed to basic solutions. To prevent eluent contamination from acetonitrile hydrolysis, always add acetonitrile to basic aqueous eluents by proportioning the acetonitrile into the basic eluent with the gradient pump. Keep the acetonitrile in a separate eluent bottle containing only acetonitrile and water.

SAFETY

Never add the acetonitrile directly to the basic carbonate or hydroxide eluent solutions.

4.5 Regenerant Preparation for the Dionex AMMS 300 Suppressor

The Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300) requires the use of a regenerant solution. If you are using the Dionex AMMS 300 suppressor see Document No. 031727, the "Product Manual for the Dionex Anion MicroMembrane Suppressor, Dionex AMMS 300."

5. Example Applications

The chromatograms in this section were obtained using columns that reproduced the Quality Assurance Report on an optimized Ion Chromatograph. Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components and laboratory temperatures.

Ensure that your system is properly configured and that all of the eluents have been made from high purity reagents and deionized water. All water used in the preparation of eluents should be degassed, deionized water with a specific resistance of 18.2 megohm-cm. For chemical purity requirements, see Section 4.3, "Chemical Purity Requirements." After running synthetic standards to calibrate your system, you may find that real sample matrices foul your columns. For this reason it is always advisable to use a guard column to protect the analytical column. If column performance deteriorates and it is determined that the guard or the analytical/capillary column has been fouled, refer to the column cleanup protocols in Appendix A, "Column Care." If your sample matrices are relatively low in ionic concentration, you may be able to increase the sensitivity of your system by using sample concentration techniques (see Section 3.4, "The Sample Concentrator").

5.1 Preparation of Eluent Stock Solution Concentrates

A. **Sodium Carbonate Eluent Concentrate**: The Dionex IonPac AS9-HC Sodium Carbonate Eluent Concentrate (0.5 M Na_2CO_3), P/N 037162, can be used to prepare eluents for the Dionex IonPac AS9-HC column.

To make the eluent concentrate from reagents, thoroughly dissolve 53 g of sodium carbonate (MW 106.00 g/mole) 700 mL of deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

B. Sodium Bicarbonate ($NaHCO_3$) Concentrate: The Dionex Sodium Bicarbonate Concentrate, 0.5 M, 500 mL, is available by ordering P/N 037163.

To make this concentrate from reagents, thoroughly dissolve 21.00 g of $NaHCO_3$ in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

5.2 Eluent Preparation

5.2.1 Eluent: 9 mM Sodium Carbonate

A. Using Dionex IonPac AS9-HC Eluent Concentrate:
By Weight: Weigh 982.0 g of deionized water and add 18.9 g of the Dionex IonPac AS9-HC Eluent Concentrate.

By Volume: To make 1 liter of eluent, pipet 18 mL of the Dionex IonPac AS9-HC Eluent Concentrate into a 1 L volumetric flask and dilute to a final volume of 1 L using deionized water.

NOTE

It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of CO_2 will affect retention times and selectivity. This is particularly important for Capillary IC as a single batch of eluent can last up to 3 months.

5.3 Isocratic Elution Using the Dionex IonPac AS9-HC Column With and Without a Guard Column

Isocratic elution of inorganic anions and oxyhalides using the Dionex IonPac AS9-HC Analytical/Capillary Column has been optimized utilizing a carbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. The Dionex IonPac AS9-HC Analytical/Capillary Column should always be used with the Dionex IonPac AG9-HC Guard/Capillary Guard Column. Note that the Dionex IonPac AG9-HC guard column is packed with a microporous resin of proportionally lower capacity and retention times will increase by approximately 2-3% when a guard/capillary guard column is placed in-line prior to the analytical/capillary column.

Sample Volume:	4 mm: 25 µL Loop + 0.8 µL Injection valve dead volume 2 mm: 5µL Loop + 0.8 µL Injection valve dead volume
Column:	See Chromatograms
Eluent:	9 mM Na_2CO_3
Temperature:	23 °C
Eluent Flow Rate:	1.0 mL/min (4 mm), 0.25 mL/min (2 mm)
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor, Dionex AERS 500 (4 mm or 2 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm or 2 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	24-30 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

Analyte	mg/L (ppm)
1. Fluoride	3.0
2. Chlorite	10.0
3. Bromate	20.0
4. Chloride	6.0
5. Nitrite	15.0
6. Chlorate	25.0
7. Bromide	25.0
8. Nitrate	25.0
9. Phosphate	40.0
10. Sulfate	30.0

Figure 7 Isocratic Elution of Inorganic Anions and Oxyhalides Using the Dionex IonPac AS9-HC Column

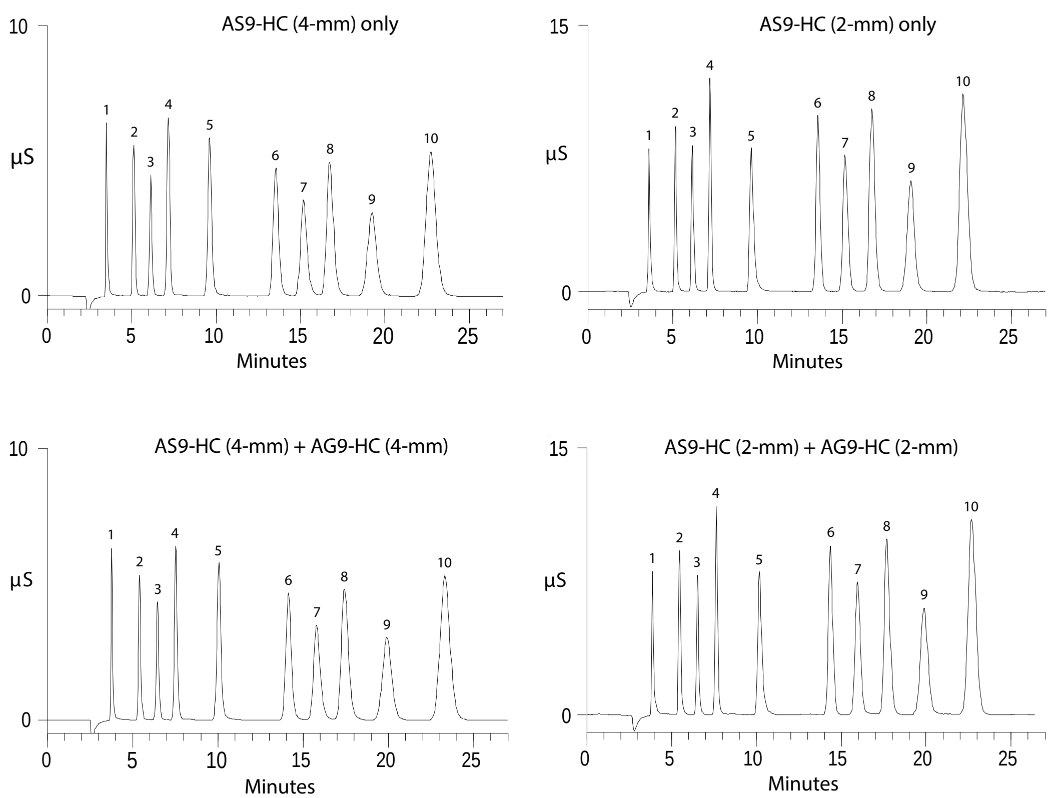
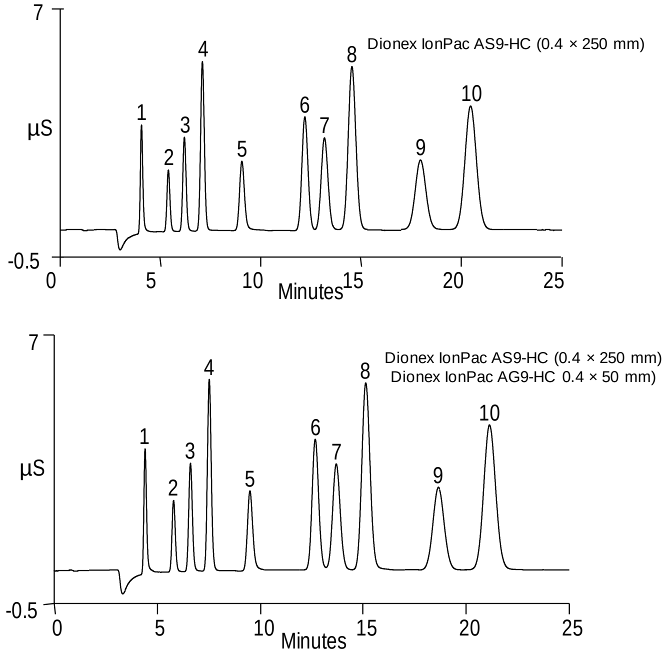


Figure 8 Isocratic Elution of Inorganic Anions and Oxyhalides Using the Dionex IonPac AS9-HC Capillary Column



Column:	see chromatogram
Eluent:	9 mM Sodium Carbonate
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	Ambient
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode

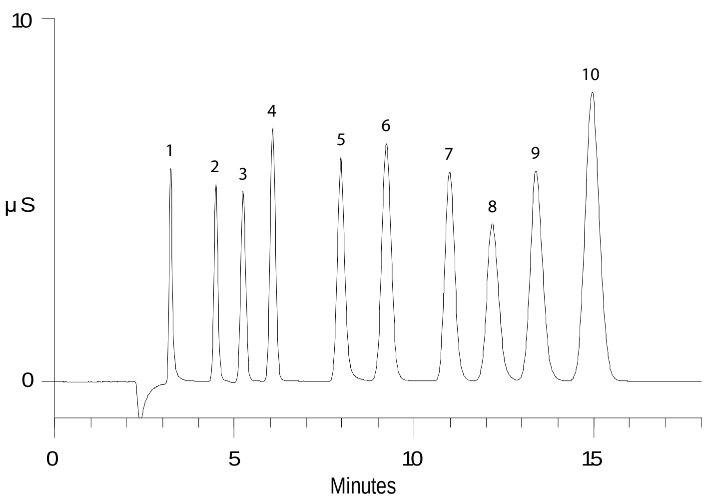
Peaks:	mg/L
1. Fluoride	1.9
2. Chlorite	6.3
3. Bromate	13.5
4. Chloride	3.8
5. Nitrite	9.4
6. Chlorate	15.6
7. Bromide	15.6
8. Nitrate	15.6
9. Phosphate	25
10. Sulfate	18.8

5.4 Fast Analysis of Inorganic Anions and Oxyhalides using the Dionex IonPac AS9-HC Column

The following chromatograms demonstrate a fast separation of inorganic acids and oxyhalides using the Dionex IonPac AS9-HC column. This run shows the selectivity change particularly for phosphate due to the addition of bicarbonate to eluent.

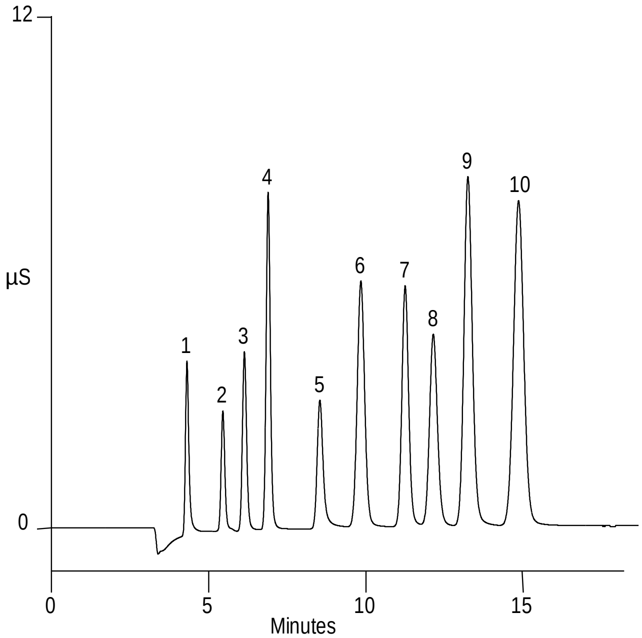
Figure 9 Fast Analysis of Inorganic Anions and Oxyhalides Using the Dionex IonPac AS9-HC Column

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	12.0 mM Carbonate/5 mM Bicarbonate
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	26-30 µS



Peaks:		mg/L
1.	Fluoride	3.0
2.	Chlorite	10.0
3.	Bromate	20.0
4.	Chloride	6.0
5.	Nitrite	15.0
6.	Phosphate	40.0
7.	Bromide	25.0
8.	Chlorate	25.0
9.	Nitrate	25.0
10.	Sulfate	30.0

Figure 10 Fast Analysis of Inorganic Anions Using the Dionex IonPac AS9-HC Capillary Column



Column:	Dionex IonPac AG9-HC/AS9-HC (0.4 × 250 mm)	
Eluent:	12 mM Sodium Carbonate / 5 mM Sodium bicarbonate	
Flow Rate:	10 µL/min	
Inj. Volume:	0.4 µL	
Temperature:	Ambient	
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode	
Peaks:		mg/L
	1. Fluoride	1.9
	2. Chlorite	6.3
	3. Bromate	13.5
	4. Chloride	3.8
	5. Nitrite	9.4
	6. Chlorate	15.6
	7. Bromide	15.6
	8. Nitrate	15.6
	9. Phosphate	25
	10. Sulfate	18.8

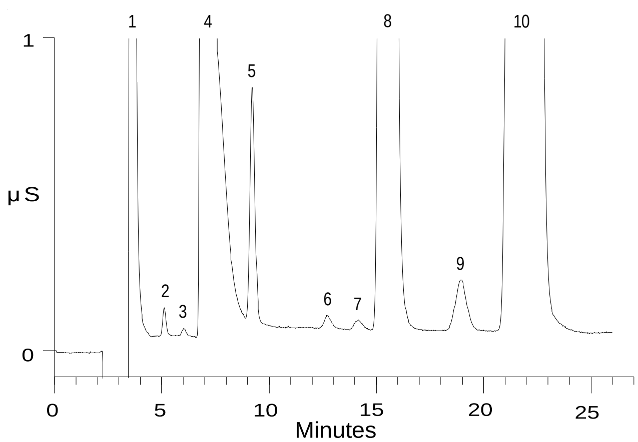
5.5 Analysis of Municipal Drinking Water

The following chromatograms demonstrate the separation of inorganic anions and oxyhalides in a simulated drinking water sample as well as in a tap water sample.

Figure 11 Analysis of Drinking Water Spiked with 5 ppb Bromate Using the Dionex IonPac AS9-HC Column (4mm)

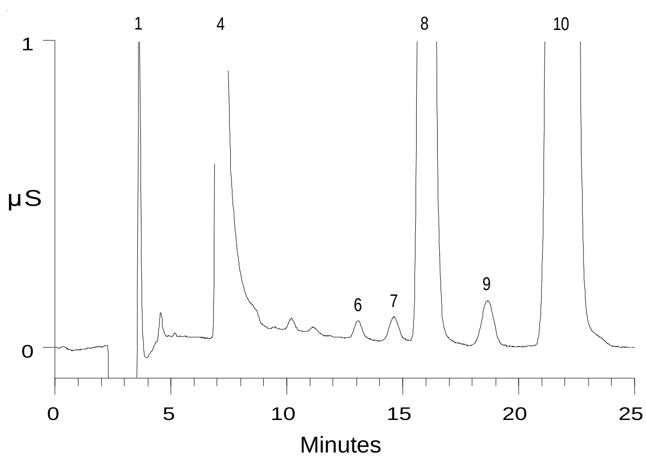
Sample Loop Volume:	200 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9.0 mM Carbonate
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 -30 µS

Simulated Drinking Water



Peaks:	mg/L
1. Fluoride	1.00
2. Chlorite	0.01
3. Bromate	0.005
4. Chloride	50.00
5. Nitrite	0.10
6. Bromide	0.01
7. Chlorate	0.01
8. Nitrate	10.00
9. Phosphate	0.10
10. Sulfate	50.00

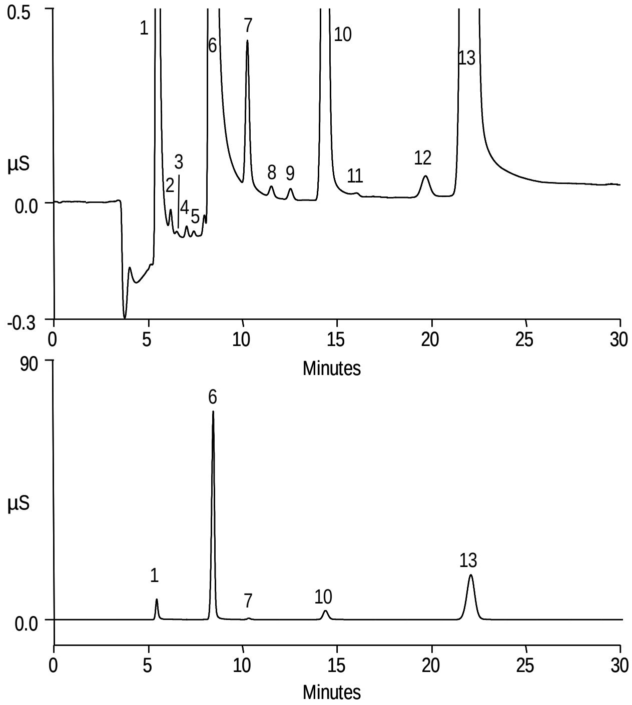
Sunnyvale Drinking Water



Peaks:	mg/L
1. Fluoride	0.04
2. Chlorite	ND
3. Bromate	ND
4. Chloride	16.20
5. Nitrite	ND
6. Bromide	0.03
7. Chlorate	0.04
8. Nitrate	3.90
9. Phosphate	0.15
10. Sulfate	18.30

(ND = Not Determined)

Figure 12 Analysis of Drinking Water Spiked with 5 ppb Bromate Using the Dionex IonPac AS9-HC Capillary Column



Column:	Dionex IonPac AG9-HC/AS9-HC (0.4 × 250 mm)
Eluent:	9 mM Sodium Carbonate
Flow Rate:	10 µL/min
Inj. Volume:	2 µL
Temperature:	30° C
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode

Peaks:	mg/L
1. Fluoride	0.5
2. Acetate	-
3. Formate	-
4. Chlorite	0.01
5. Bromate	0.005
6. Chloride	5.0
7. Nitrite	0.1
8. Chlorate	0.01
9. Bromide	0.01
10. Nitrate	1.0
11. Carbonate	5.0
12. Phosphate	0.1
13. Sulfate	5.0

5.6 Effect of Dionex OnGuard Pretreatment Cartridges on Samples with Excessive Chloride Concentrations

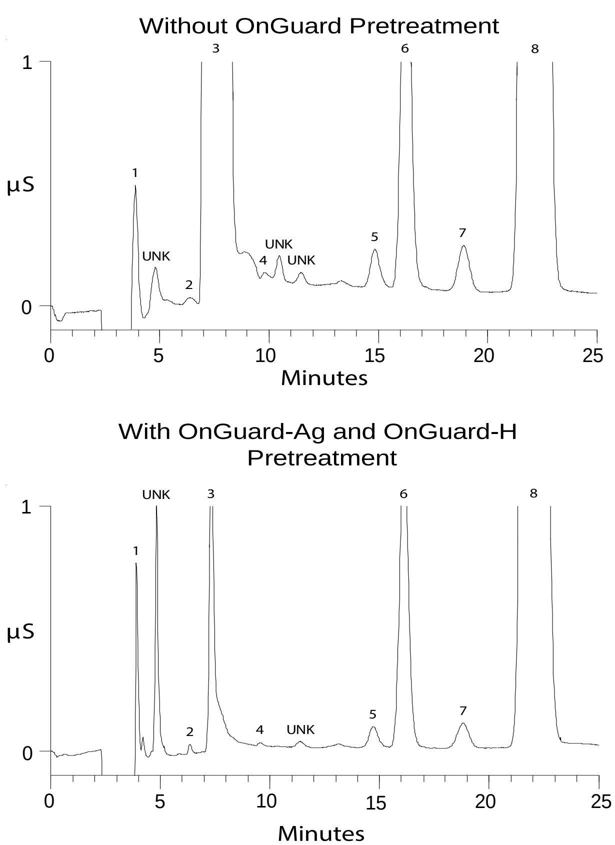
In the presence of very high chloride concentration, the efficiency of early eluting peaks would be compromised because of the overloading effect. A sample pretreatment with Thermo Scientific Dionex OnGuard Ag followed by Dionex OnGuard H, significantly reduces chloride and carbonate; allowing accurate quantification of bromate.

Dionex OnGuard Ag and Dionex OnGuard H sample cleanup procedure:

1. Wash the Dionex OnGuard Ag and Dionex OnGuard H in series with 10 mL of deionized water.
2. Pass 10 mL of sample through the washed cartridge, discarding the first 3 mL of effluent.
3. Sparge the collected pretreated sample with nitrogen or helium for at least 5 minutes in order to remove carbonic acid from the sample.

Sample Loop Volume:	500 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9.0 mM Carbonate
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 13 Effect of OnGuard Pretreatment on Sample



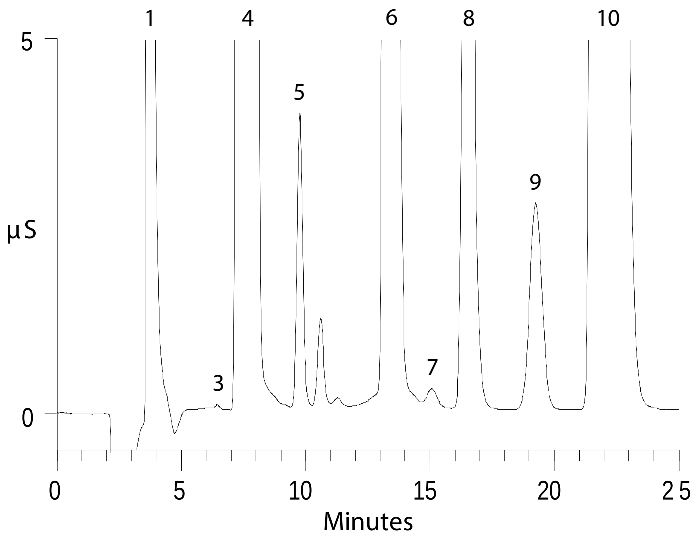
Peaks:		mg/L
1.	Fluoride	0.02
2.	Bromate	0.01
3.	Chloride	240.00
4.	Nitrite	0.002
5.	Chlorate	0.04
6.	Nitrate	0.33
7.	Phosphate	0.10
8.	Sulfate	10.00
	(UNK= Unknown)	

5.7 Analysis of Treated Spa Water Sample

The following chromatogram demonstrates the separation of anions in a spa water sample.

Sample Loop Volume:	500 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9.0 mM Carbonate
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 14 Analysis of Treated Spa Water Sample



Peaks:		mg/L
1.	Fluoride	0.760
2.	Chlorite	ND
3.	Bromate	0.017
4.	Chloride	122.000
5.	Nitrite	0.540
6.	Bromide	46.000
7.	Chlorate	0.076
8.	Nitrate	1.500
9.	Phosphate	1.600
10.	Sulfate	51.000

(ND = Not Determined)

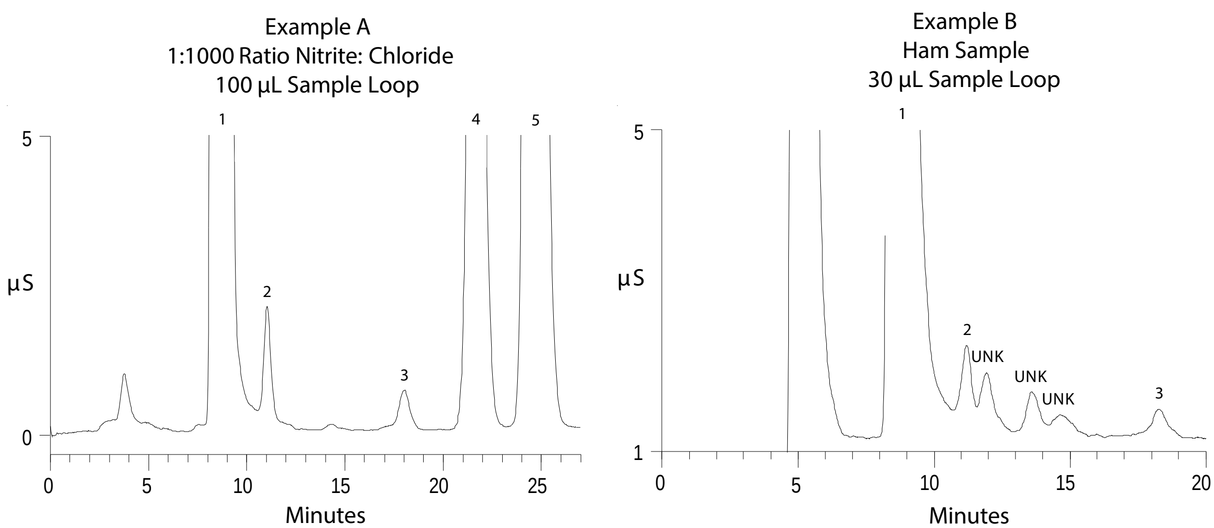
5.8 Determination of Trace Nitrite and Nitrate in High Ionic Strength Matrices

The following chromatograms demonstrate the determination of trace nitrite and nitrate in high ionic strength matrices. Ham sample preparation:

1. Weigh 10.0 g. of the meat sample and transfer to a blender.
2. Add 100.0 mL of deionized water to the meat sample and blend for one minute.
3. Heat the liquefied sample and maintain the temperature of the sample between 70° and 80° C for 15 min.
4. Centrifuge the sample for 10 minutes, and filter sample with a Whatman No. 2 and GF/A filters and 1.2 mm and 0.2mm Acrodisc filters.

Sample Loop Volume:	See chromatogram
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9.0 mM Carbonate
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 15 Determination of Trace Nitrite and Nitrate in High Ionic Strength Matrices



	Analyte	A mg/L (ppm)	B mg/L (ppm)
1.	Chloride	1000.0	ND
2.	Nitrite	1.0	ND
3.	Nitrate	1.0	ND
4.	Phosphate	100.0	–
5.	Sulfate	100.0	–

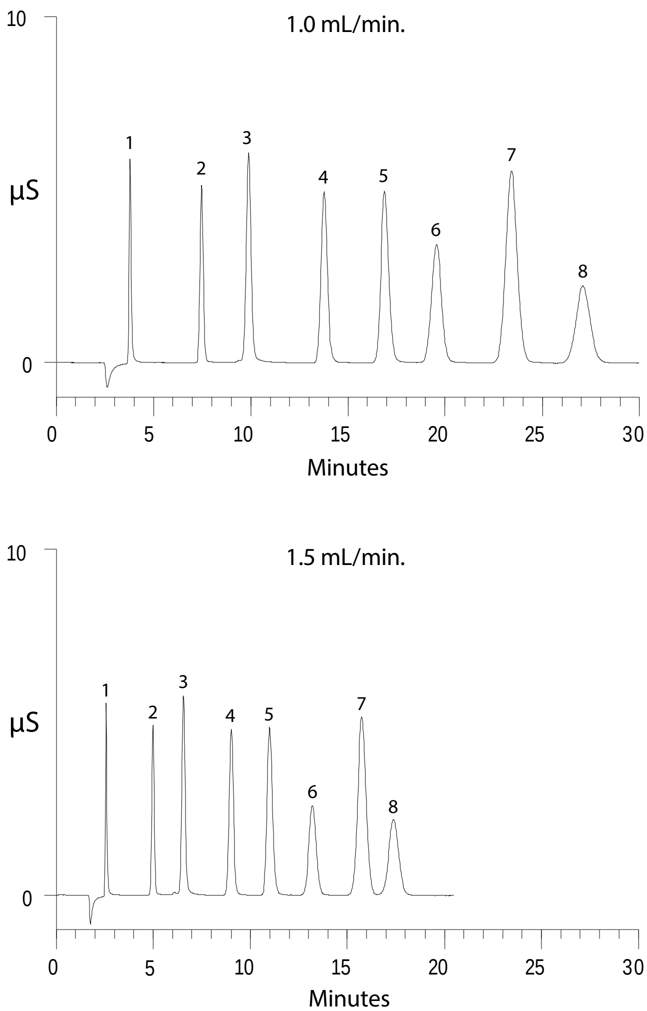
(ND = Not Determined)
(UNK = Unkown)

5.9 Determination of Sulfite in the Presence of Common Anions

The following chromatograms demonstrate the determination of sulfite in the presence of common anions at 1.0 mL/min and 1.5 mL/min.

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	8.0 mM Carbonate/1.5 mM NaOH
Eluent Flow Rate:	See chromatogram
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 16 Determination of Sulfite in the Presence of Common Anions



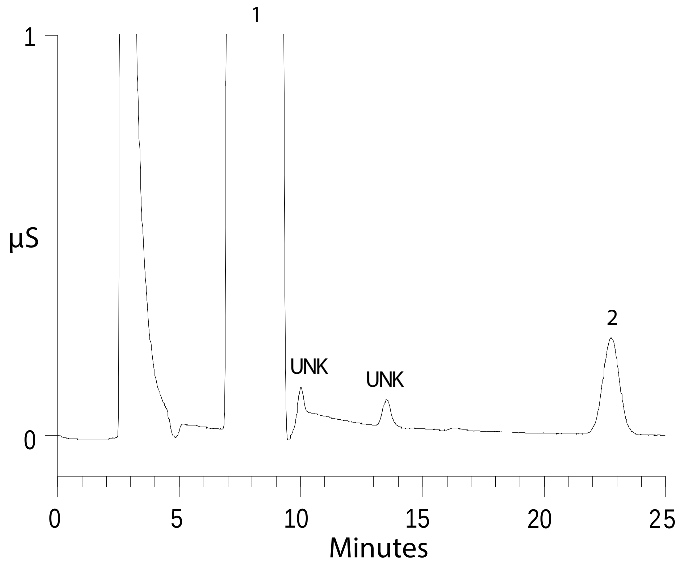
Peaks:		mg/L
1.	Fluoride	3.0
2.	Chloride	6.0
3.	Nitrite	15.0
4.	Bromide	25.0
5.	Nitrate	25.0
6.	Sulfite	30.0
7.	Sulfate	30.0
8.	Phosphate	40.0

5.10 Determination of Trace Sulfate in Brine Sample

The following chromatogram demonstrates the determination of low sulfate in the presence of high chloride (1:1000 ratio).

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9 mM Carbonate
Eluent Flow Rate:	1 mL/min
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 17 Determination of Trace Sulfate in Brine Sample



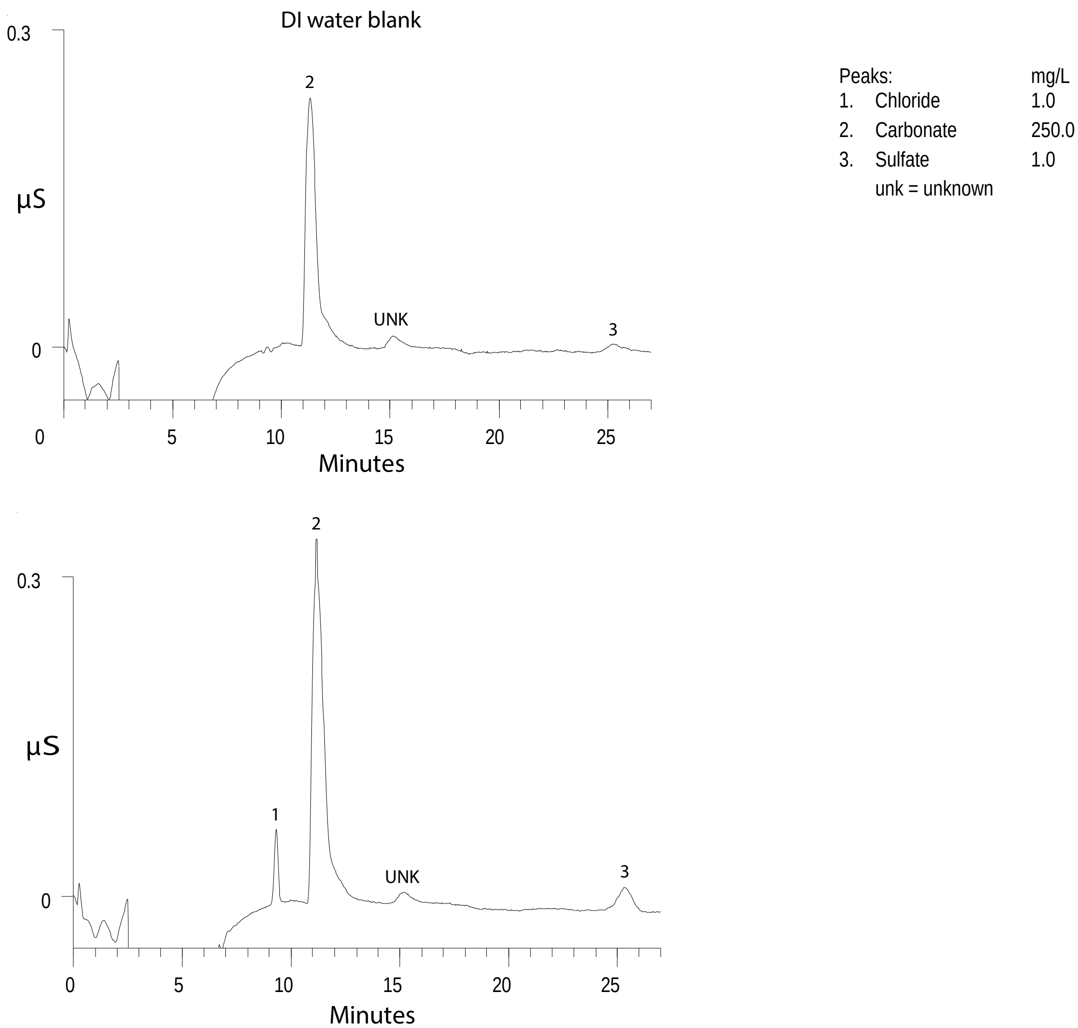
Peaks:	mg/L
1. Chloride	1000.0
2. Sulfate	1.0
unk = unknown	

5.11 Determination of Trace Chloride and Sulfate in High Purity Water

The following chromatograms demonstrate the determination of trace chloride and sulfate in high levels of carbonate using an optimized eluent system.

Sample Loop Volume:	2 mL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	8 mM Carbonate/1.5 mM Bicarbonate
Eluent Flow Rate:	1 mL/min
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 18 Determination of Trace Chloride and Sulfate in High Purity Water

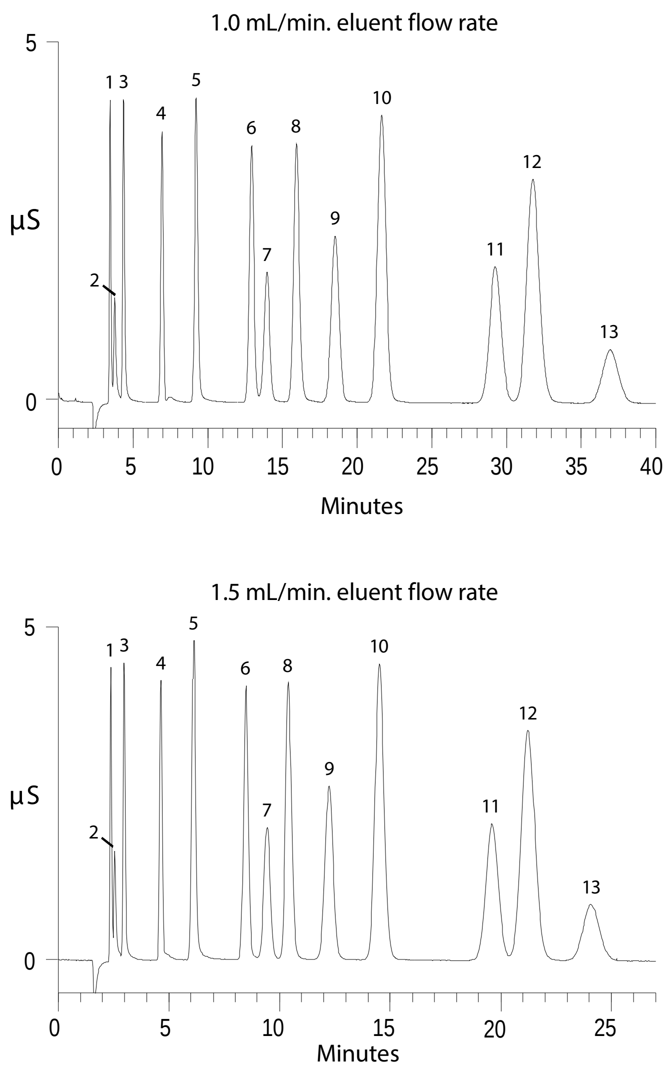


5.12 Analysis of Oxyanions

The following chromatograms demonstrate the analysis of oxyanions in the presence of common inorganic anions. Note that the analysis time can be reduced by increasing the flow rate without changing the selectivity.

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9 mM Carbonate
Eluent Flow Rate:	See Chrmoatogram
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	24 - 30 µS

Figure 19 Analysis of Oxyanions



Peaks:	mg/L
1. Fluoride	3.0
2. Acetate	20.0
3. Formate	10.0
4. Chloride	6.0
5. Nitrite	15.0
6. Bromide	25.0
7. Selenite	30.0
8. Nitrate	25.0
9. Phosphate	40.0
10. Sulfate	30.0
11. Selenate	30.0
12. Oxalate	50.0
13. Arsenate	50.0

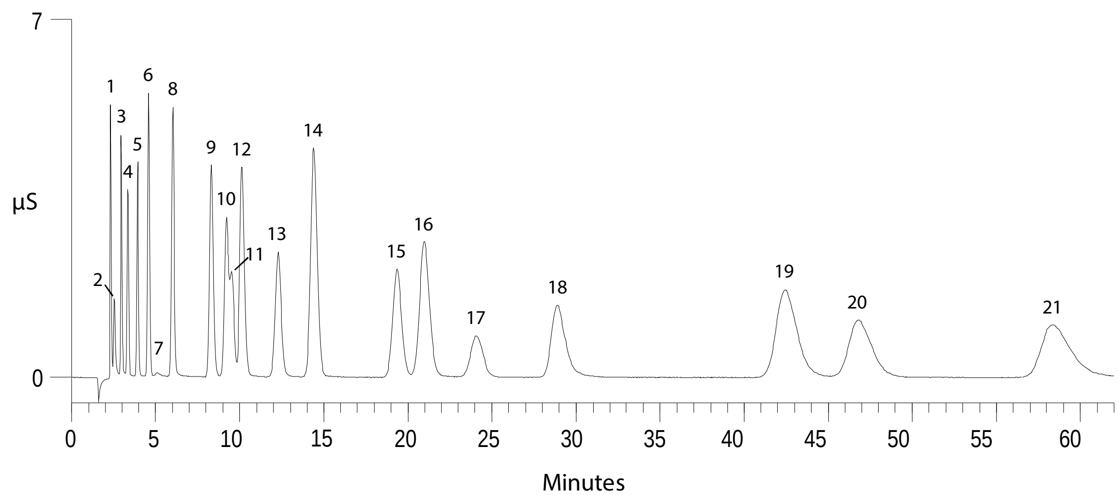
5.13 Analysis of 21 Anions with Isocratic Elution Using a Carbonate Eluent System

The following chromatogram demonstrates the elution order of 21 anions using the standard eluent with the Dionex IonPac AS9-HC column.

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9 mM Carbonate
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS

Figure 20 Analysis of 21 Anions

Peaks:	mg/L	Peaks:	mg/L
1. Fluoride	3.0	12. Nitrate	25.0
2. Acetate	20.0	13. Phosphate	40.0
3. Formate	10.0	14. Sulfate	30.0
4. Chlorite	10.0	15. Selenate	30.0
5. Bromate	20.0	16. Oxalate	40.0
6. Chloride	6.0	17. Arsenate	50.0
7. Bicarbonate	50.0	18. Iodide	70.0
8. Nitrite	15.0	19. Thiosulfate	70.0
9. Bromide	25.0	20. Thiocyanate	70.0
10. Chlorate	25.0	21. Chromate	70.0
11. Selenite	30.0		

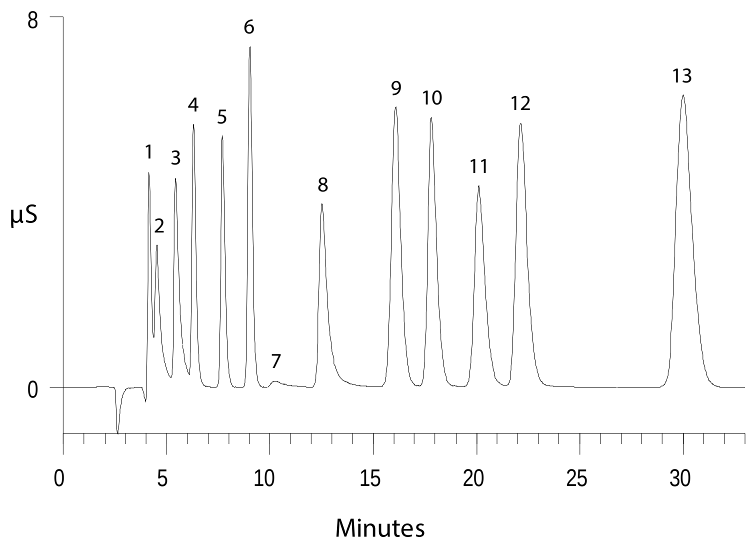


5.14 Isocratic Analysis of 13 Anions Using a Borate Eluent System

The following chromatogram demonstrates the isocratic separation of acetate, formate, and inorganic anions including oxyhalides using a sodium tetraborate eluent system. Using these conditions, glycolate and acetate will coelute.

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	50 mM sodium tetraborate
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (4 mm) External Water Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	7-10 µS

Figure 21 Isocratic Analysis of 13 Anions Using a Borate Eluent System



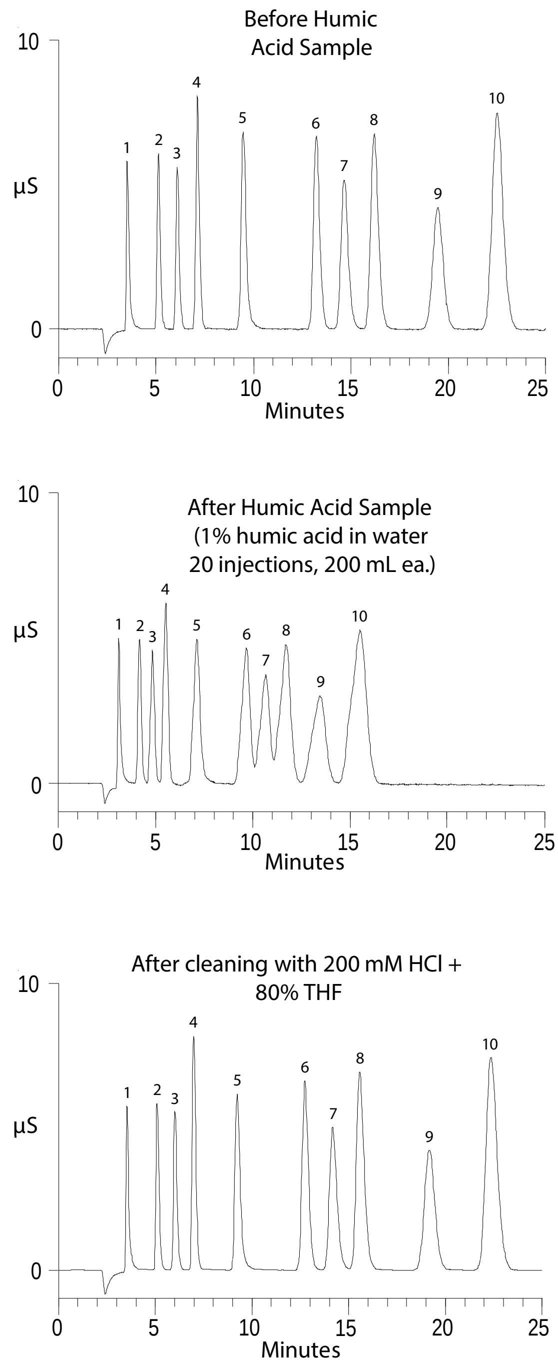
Peaks:	mg/L
1. Fluoride	3.0
2. Acetate	20.0
3. Formate	10.0
4. Chlorite	10.0
5. Bromate	20.0
6. Chloride	6.0
7. Bicarbonate	50.0
8. Nitrite	15.0
9. Phosphate	40.0
10. Bromide	25.0
11. Chlorate	25.0
12. Nitrate	25.0
13. Sulfate	30.0

5.15 Clean-up After Humic Acid Samples

Solvent compatibility of the Dionex IonPac AS9-HC column permits the use of organic solvents to effectively remove organic contaminates from the column. A Dionex IonPac AS9-HC column, after losing over 30% of its original capacity due to fouling with humic acid samples, can easily be restored to original performance by cleaning for 60 minutes with 200 mM HCl + 80% THF.

Figure 22 Clean-up After Humic Acid Sample

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS9-HC Analytical Column (4 mm) + Dionex IonPac AG9-HC Guard Column (4 mm)
Eluent:	9 mM Carbonate
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS ULTRA (4 mm)
External Water Mode or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS III (4 mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Dionex Anion Atlas Electrolytic Suppressor, Dionex AAES
Expected Background Conductivity:	24 - 30 µS



Peaks:		mg/L
1.	Fluoride	3.0
2.	Chlorite	10.0
3.	Bromate	20.0
4.	Chloride	6.0
5.	Nitrite	15.0
6.	Bromide	25.0
7.	Chlorate	25.0
8.	Nitrate	25.0
9.	Phosphate	40.0
10.	Sulfate	30.0

6. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac AS9-HC column. For more information on problems that originate with the Ion Chromatograph (IC) or other consumables such as the suppressor, trap or concentrator columns, refer to the Troubleshooting Guide in the appropriate operator's manual.

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

Table 5 Dionex IonPac AS9-HC/AG9-HC Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2
	Other System Components	Unplug, Replace	Component Manual
High Background Conductivity	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.2,
	Contaminated Suppressor	Clean Suppressor	6.2.4, Component Manual
	Contaminated Hardware	Clean Component	6.2.3, Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.1.B, Component Manual
	Column Headspace	Replace Column	6.3.1A
Poor Resolution of Only Phosphate and Sulfate	Sodium Carbonate Contaminated with Sodium Hydroxide, Inadequate Equilibration after Use of an Alkaline Buffer, Sodium Carbonate Dried at Temperatures >110°C	Use Dionex 0.5 M Sodium Carbonate (P/N 037162), Dry Sodium Carbonate at Lower Temperature	6.3.6
Short Retention Times	Flow Rate Too fast	Recalibrate Pump	6.3.2.A, Component Manual
	Conc. Incorrect Eluents	Remake Eluents	6.3.2.B
	Column Contamination	Clean Column	6.3.2.C, 6.3.2.D,
Poor Front End Resolution	Conc. Incorrect Eluents	Remake Eluents	6.3.3.A
	Column Overloading	Reduce Sample Size	6.3.3.B, 3.3
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual
	Large System Void Volumes	Replumb System	6.3.3.D, Component Manual
Poor Resolution of only Fluoride and Acetate	Poor plumbing, incorrect eluent	Replumb system, check eluent	6.3.7
Spurious Peaks	Column Contaminated	Clean Column	6.3.4.A,
	Sluggish Injection Valve	Service Valve	6.3.3.B, Component Manual
Poor Efficiency using Capillary Columns	Poor Connections	Remake Connections	6.3.5

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the Dionex IonPac AG9-HC Guard/Capillary Guard Column plus the Dionex IonPac AS9-HC Analytical/Capillary Column when using the test chromatogram conditions should be equal or less than 2,250 psi. If the system pressure is higher than 2,250 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated.

A. **Make sure that the pump is set to the correct eluent flow rate.** Higher than recommended eluent flow rates will cause higher pressure. Measure the pump flow rate if necessary with a stop watch and graduated cylinder.

B. **Determine which part of the system is causing the high pressure.** High pressure could be due to a plugged tubing or tubing with collapsed walls, an injection valve with a clogged port, a column with particulates clogging the bed support, a clogged High-Pressure In-Line Filter, the suppressor or the detector cell.

   To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi. Continue adding system components (injection valve, column(s), suppressor and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 2, “Operating Parameters”).

   The suppressor may add up to 100 psi (0.69 MPa). No other components should add more than 100 psi (0.69 MPa) of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

6.1.2 Replacing Column Bed Support Assemblies for 2 mm and 4 mm Columns

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

A. **Disconnect the column from the system.**

B. **Carefully unscrew the inlet (top) column fitting.** Use two open-end wrenches.

C. **Remove the bed support.** Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you **do not scratch the walls of the end fitting.** Discard the old bed support assembly.

D. **Place a new bed support assembly into the end fitting.** Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

Table 6 Ordering Information

Product	Dionex IonPac AS9-HC 4 mm Columns (P/N)	Dionex IonPac AS9-HC 2 mm Columns (P/N)	Dionex IonPac AS9-HC 0.4 mm Columns (P/N)
Analytical Column	051786	052244	082319
Guard Column	051791	052248	088296
Bed Support Assembly	042955	044689	N/A
End Fitting	052809	043278	N/A

CAUTION

If the column tube end is not clean when inserted into the end fitting, particulate matter may obstruct a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.

E. **Screw the end fitting back onto the column.** Tighten it fingertight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.

F. **Reconnect the column to the system and resume operation.**

NOTE

Replace the outlet bed support ONLY if high pressure persists after replacement of the inlet fitting.

6.2 High Background or Noise

In a properly working system, the background conductivity level for the standard eluent system is shown below:

Eluent	Expected Background Conductivity
9.0 mM Na_2CO_3	24-30 µS

6.2.1 Preparation of Eluents

A. Make sure that the eluents and the regenerant are made correctly.

B. Make sure that the eluents are made from chemicals with the recommended purity.

C. Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

6.2.2 A Contaminated Guard or Analytical Column

Remove the guard and analytical columns from the system. If the background conductivity decreases, the column(s) is (are) the cause of the high background conductivity. Clean or replace the columns at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in Appendix A "Column Care".

6.2.3 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the columns and the suppressor. Pump deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

6.2.4 A Contaminated Suppressor

If the above items have been checked and the problem persists, the suppressor is probably causing the problem. For details on Dionex Anion Electrolytically Regenerated Suppressor operation, refer to the Dionex Anion Electrolytically Regenerated Suppressor 500 Product Manual (Document No. 031956). For details on Dionex Anion Membrane Suppressor 300 operation, refer to the Product Manual (Document No. 031727) for assistance. For details on the Dionex Anion Capillary Electrolytic Suppressor 300 (Dionex ACES 300) operation, refer to the product manual (Document No. 065388) for assistance.

6.3 Poor Peak Resolution

Poor peak resolution can also be due to any or all of the following factors:

6.3.1 Loss of Column Efficiency

A. Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 6.1.2, "Replacing Column Bed Support Assemblies"). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

B. **Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient.** Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4 mm systems or no greater than 0.005" for 2 mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks.

6.3.2 Poor Resolution Due to Shortened Retention Times

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

A. **Check the flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

B. **Check to see if the eluent compositions and concentrations are correct.** An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent, components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

C. **Column contamination can lead to a loss of column capacity.** This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to Appendix A "Column Care", for recommended column cleanup procedures.
Possible sources of column contamination are impurities in chemicals and in the deionized water used for eluents or components of the sample matrix. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

D. **Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times.** If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see Appendix A, "Column Care").

NOTE

For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

6.3.3 Loss of Front End Resolution

If poor resolution or efficiency is observed for the peaks eluting near the system void volume compared to the later eluting peaks, check the following:

A. **Improper eluent concentration may be the problem.** Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity.

B. **Column overloading may be the problem.** Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.

C. **Sluggish operation of the injection valve may be the problem.** Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.

D. **Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem.** Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change.

6.3.4 Spurious Peaks

A. **The columns may be contaminated.** If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, the retention times for the analytes will then decrease and be spurious, inefficient (broad) peaks that can show up at unexpected times. Clean the column as indicated in Appendix A, “Column Care”.

NOTE

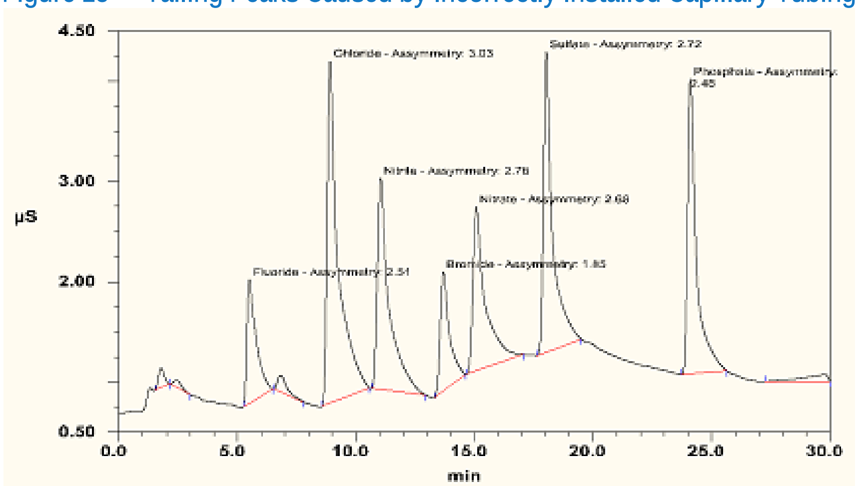
For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

B. **The injection valve may need maintenance.** When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

6.3.5 Poor Efficiency Using Capillary Columns

Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks.

Figure 23 Tailing Peaks Caused by Incorrectly Installed Capillary Tubing Fittings



When connecting a capillary tube fitting, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing. Insert the tubing hard and hold it in place while tightening the fitting. Figure 24 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing. If necessary to hold the ferrule and nut securely, turn the pump off while making capillary connections.

Figure 24 Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

6.3.6 Poor Resolution of Only Phosphate and Sulfate

A. Causes
1. Sodium carbonate is contaminated with sodium hydroxide,
2. Inadequate equilibration after use of an alkaline buffer or hydroxide eluent,
3. Sodium carbonate was dried at temperatures > 110°C.

B. Action
1. Use Dionex IonPac AS9-HC Eluent Concentrate (P/N 064161).
2. Use a high purity sodium carbonate salt.
3. Dry the sodium carbonate at a lower temperature. See section 4.3.1 and section 5.1.

NOTE

It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of CO_2 will affect retention times and selectivity. This is particularly important for Capillary IC as a single batch of eluent can last up to 3 months.

6.3.7 Poor Resolution of Only Fluoride and Acetate

A. Causes
1. Lower fluoride and acetate peak efficiency due to extra column effects.
2. Excessive peak tailing for the early eluting peaks.
3. Short run time for sulfate.

B. Action
1. Check the system plumbing especially connecting tubes between injection valve and column.
2. Remake the eluent and check the pump flow rate (see Section 6.3.2).

Appendix A – Column Care

A.1 Recommended Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for Dionex IonPac AS9-HC column is 4,000 psi (27.57 MPa).

A.2 Column Start-Up

The column is shipped using 100 mM Sodium bicarbonate as the storage solution.

Prepare the eluent shown on the Quality Assurance Report (QAR), install the column in the chromatography module and direct the column effluent to waste for 30 minutes and then connect to the suppressor. Test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

If peak efficiencies or resolution on the capillary column are poorer than the QAR, see Sections 3.13 “Installation of the Capillary Column” and 6.3.5 “Poor Efficiency using Capillary Columns” for information regarding proper connections.

A.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), use 100 mM Sodium Bicarbonate for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

A.4 Column Cleanup

The following column cleanup protocols have been divided into three general isocratic protocols to remove acid-soluble, base-soluble, or organic contaminants. They can be combined into one gradient protocol if desired; however, the following precautions should be observed.

WARNING

Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column.

High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column.

High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent which may create a neutralization pressure band.

The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to $\leq$ 5% levels and the ionic strength of the eluent to $\leq$ 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

A.4.1 Choosing the Appropriate Cleanup Solution

CAUTION

When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately. Do not pass the column effluent through the suppressor.

A. Hydrophilic Ionic Contamination of Low Valency

E1: Test eluent shown on the Quality Assurance Report (QAR)

E2: 10X concentrate of test eluent on the Quality Assurance Report (QAR): 90 mM sodium carbonate

1. Prepare the cleanup solutions above.
2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.
3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
4. Pump the cleanup solution E2 through the column(s) for 60 minutes.
5. Equilibrate the column(s) with eluent (E1) before resuming normal operation for at least 15 minutes.
6. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

NOTE

Be sure to wash bottle, lines and pump with deionized water after using 10X eluent on the test system.

B. Metal Contamination

E1: Test eluent shown on the Quality Assurance Report (QAR)

E2: 0.1 M oxalic acid

NOTE

Iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

1. Prepare the cleanup solutions above.
2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.
3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
4. Pump the cleanup solution E2 through the column(s) for 60 minutes it is aluminum contamination or for overnight (14-18 hours) if it is iron contamination..

5. Equilibrate the column(s) with eluent (E1) before resuming normal operation for at least 30 minutes.
6. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

NOTE

Be sure to wash the bottle, lines and pump with deionized water after using oxalic acid on the test system.

C. High Valency Hydrophobic Ions - Isocratic Cleanup Option

E1: Test eluent shown on the Quality Assurance Report (QAR).

E2: 5% acetonitrile in the test eluent shown on the Quality Assurance Report (QAR).

E3: 150 mM potassium nitrate in 80% acetonitrile

1. Prepare the cleanup solutions above.
2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.
3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
4. Rinse the column for 15 minutes with E2.
5. Pump the cleanup solution E3 through the column(s) for 60 minutes.
6. Rinse the column for 15 minutes with E2.
7. Equilibrate the column(s) with eluent E1 before resuming normal operation for at least 30 minutes.
8. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

NOTE

Be sure to wash the bottles, lines and pump with deionized water after using acetonitrile and potassium nitrate on the system.

D. High Valency Hydrophobic Ions - Gradient Cleanup Option

E1: Test eluent shown on the Quality Assurance Report (QAR).

E2: 100% acetonitrile (Note: Acetonitrile must be stored in a separate reservoir bottle because it slowly breaks down in acidic aqueous solutions and high percentages of acetonitrile tend to be non-miscible in high molarity salt solutions. These limitations are overcome by step gradient operation.

E3: 1.0 mM NaCl using degassed Type I Reagent Grade Water with a specific resistance of 18.2 megaohm-cm. Adjust the pH to 2.0 using HCl

E4: Degassed Type I Reagent Grade Water with a specific resistance of 18.2 megaohm-cm.

1. Prepare the cleanup solutions above.
2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard and an

3. analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.
4. Set the flow rate to 0.5 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
5. Proceed with the following gradient:
6. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

NOTE

Be sure to wash the bottles, lines and pump with deionized water after using acetonitrile and sodium chloride on the test system

E. Humic Acid Clean-up

E1: Test eluent shown on the Quality Assurance Report (QAR).

E2: DI water

E3: 1M HCl

E4: 100% THF

1. Prepare the cleanup solutions as above.
2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.
3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns. Set the upper pressure limit on the pump to be 1000psi to avoid any damage to the column PPK tubing and other PEEK tubing on the system.
4. Rinse the column for 10 minutes with E2.
5. Pump the 20% of E3 and 80% of E4 (200mM HCL and 80% THF) solution through the column(s) for 120 minutes.
6. Rinse the column for 15 minutes with E2.
7. Equilibrate the column(s) with eluent E1 before resuming normal operation for at least 60 minutes.
8. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

NOTE

Be sure to wash the bottles, lines and pump with deionized water after using THF and HCl on the test system.

A.4.2 Column Cleanup Procedure

A. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in Section A.4.1, "Choosing the Appropriate Cleanup Solution".

B. Disconnect the suppressor form the columns and direct the effluent to waste.

C. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path.

D. Double check that the eluent flows in the direction designated on each of the column labels.

CAUTION

When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. If not, the contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

E. Set the pump flow rate to 1.0 mL/min for a 4 mm analytical and/or guard column, 0.25 mL/min for a 2 mm analytical and/or guard Column and 10 µL/min for 0.4 mm capillary and/or capillary guard column.

F. Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.

G. Pump the cleanup solution through the column for at least 60 minutes. If the column is heavily contaminated, then clean the column for four hours to overnight.

H. Rinse the column for 10 minutes with deionized water before pumping eluent over the column.

I. Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.

J. Reinstall the guard/capillary guard column in line between the injection valve and the analytical/capillary column and reconnect the analytical/capillary column to the suppressor.

Appendix B – System Configuration

Table B1 System Configuration

CONFIGURATION	2 mm	4 mm	0.4 mm
Eluent Flow Rate	0.25 mL/min	1.0 mL/min	10 µL/min
ERS Suppressor	Dionex AERS 500 (2 mm) (P/N 082541)	Dionex AERS 500 (4 mm) (P/N 082540)	N/A
MMS Suppressor	Dionex AMMS (2 mm) (P/N 056751)	Dionex AMMS (4 mm) (P/N 056750)	N/A
ACES Suppressor	N/A	N/A	Dionex ACES 300 (P/N 072052)
Injection Loop	2 - 15 µL	10-50 µL	0.4 µL (typical)
	Rheodyne Microinjection Valve (P/N 044697) for full loop injections <15 µL.		
System Void Volume	Eliminate switching valves, couplers and the Dionex GM-3 Gradient Mixer. Use only the 2 mm Dionex GM-4 Mixer (P/N 049135).	Minimize dead volume. Switching valves, couplers can be used. Use the Dionex GM-2, GM-3 or recommended gradient mixers.	Use only in an IC system equipped for capillary analysis.
Pumps	Use the Dionex ICS 2100/5000, or Dionex GS50/GP50/GP40/ IP20/IP25 in Microbore Configuration with a Microbore Dionex GM-4 (2 mm) Gradient Mixer.	Use the Dionex ICS 2100/5000, or Dionex GP40/GP50/IP20/ IP25 in Standard-Bore Configuration.	Use only a pump designed for capillary flow rates such as the Dionex ICS-5000 capillary pump.
	The Dionex GPM-2 can be used for 2 mm isocratic chromatography at flow rates of 0.5 mL/min or greater. Note: The GPM-2 should not be used for 2 mm gradient chromatography.	The Dionex GM-3 Gradient Mixer should be used for gradient analysis on systems other than the Dionex GP50. Note: The Dionex GP40 has an active mixer.	
Chromatographic Module	A thermally controlled column oven such as the Dionex LC25,LC30,ICS-10,11,15,16,20,2100,3000,5000 DC	A thermally controlled column oven such as the Dionex LC25,LC30,ICS-10,11,15,16,20,2100,3000, 5000 DC	A thermally controlled column compartment such as the Dionex ICS-5000 DC or Dionex IC-Cube.

CONFIGURATION	2 mm	4 mm	0.4 mm
Detectors	Dionex Conductivity Detector P/N 061830 Dionex AD20/AD25 Cell (6 mm, 7.5 µL, P/N 046423) Dionex VDM-2 Cell (3 mm, 2.0 µL) (P/N 043120) Dionex CD20, CD25, CD25A, ED40, ED50, or ED50A Dionex Conductivity Cell with Dionex DS3 P/N 044130 or Dionex Conductivity Cell with shield P/N 044132 Dionex CDM-2/CDM-3 Cell P/N 042770 Replace the Dionex TS-1 with the Dionex TS-2 (P/N 043117) on the Dionex CDM-2 or the Dionex CDM-3. The Dionex TS-2 has been optimized for 2 mm operation. Do not use the Dionex TS-2 or the Dionex TS-1 with the Dionex ED40/ED50/ED50A or the Dionex CD20/CD25/CD25A. Ensure 30–40 psi back pressure.	Dionex Conductivity Detector P/N 061830 Dionex AD20/AD25 Cell (10 mm, 9 µL, P/N 049393) Dionex VDM-2 Cell (6 mm, 10 µL) P/N 043113 Dionex CD20, CD25, CD25A, ED40, ED50, or ED50A Dionex Conductivity Cell with Dionex DS3 P/N 044130 or Dionex Conductivity Cell with shield P/N 044132 Dionex CDM-2/CDM-3 Cell P/N 042770 Either the Dionex TS-1 with the Dionex TS-2 can be used with the Dionex CDM-2 or the Dionex CDM-3. Do not use the Dionex TS-2 or the Dionex TS-1 with the Dionex ED40/ED50/ED50A or the Dionex CD20/CD25/CD25A. Ensure 30–40 psi back pressure.	Use only a conductivity detector designed for capillary flow rates such as the Dionex ICS-5000$^+$ Capillary CD.

Table B2 Tubing Back Pressures

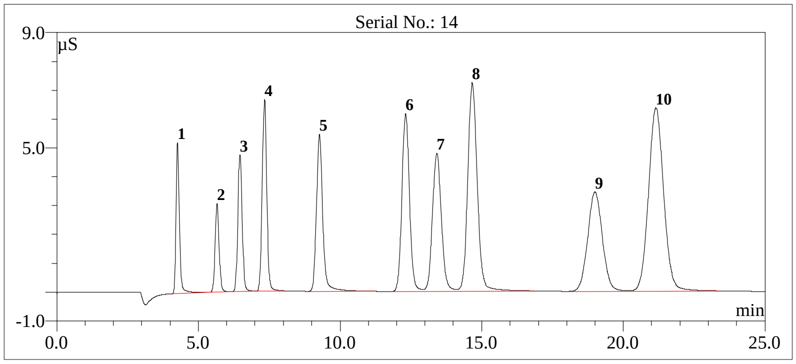
Color	Part Number	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure, Psi/ft. at 1mL/min	Back Pressure, Psi/ft. at 0.25mL/min	Back Pressure, Psi/cm. at 1mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642
Light Blue	071870	0.0025	0.006	0.0009	1766.0	441.0	58.0

Appendix C – Quality Assurance Reports (QAR)

Dionex IonPac™ AS9-HC
Capillary (0.4 x 250 mm)
Product No. 082319

Date: 10-Apr-14 08:32
Serial No. : 000014
Lot No. : 013-29-037

Eluent:	9 mM Na2CO3
Flow Rate:	10 µL/min
Temperature:	Ambient Temperature (23 °C)
Detection:	Suppressed Conductivity
Suppressor:	Dionex Anion Capillary Electrolytic Suppressor (Dionex ACES™ 300) AutoSuppression™ Recycle Mode
Applied Current:	7 mA
Injection Volume:	0.4 µL
Storage Solution:	100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	4.27	1.3	5.91	6507	1.9
2	Chlorite	5.66	1.1	3.00	7573	6.3
3	Bromate	6.47	1.1	3.03	8424	13.5
4	Chloride	7.33	1.1	5.73	10169	3.8
5	Nitrite	9.27	1.2	7.00	9151	9.4
6	Bromide	12.31	1.1	2.11	10390	15.6
7	Chlorate	13.41	1.2	2.15	9043	15.6
8	Nitrate	14.66	1.2	5.66	9673	15.6
9	Phosphate	19.00	1.1	2.26	6514	25.0
10	Sulfate	21.15	1.1	n.a.	7677	18.8

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=5400	Passed
Sulfate	Asymmetry	1.0-2.0	Passed
Sulfate	Retention Time	19.85-23.15	Passed
	Pressure	<=2200	1591

Production Reference:

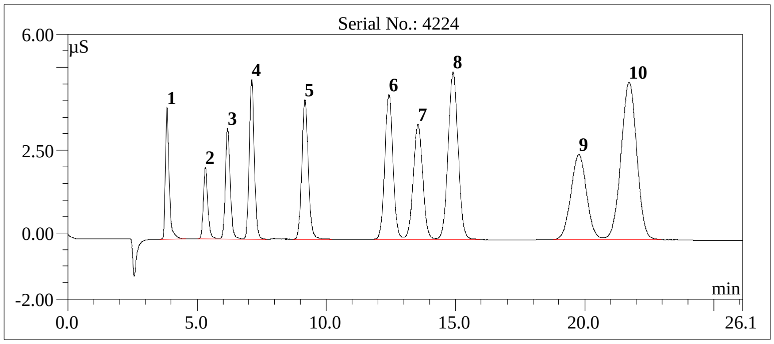
Datasource: QAR
Directory: Cap\AS9HC
Sequence: AS9HC 0p4X250mm
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)

Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ AS9-HC	**Date:**	14-Nov-13 10:35
Analytical (2 x 250 mm)	**Serial No. :**	004224
Product No. 052244	**Lot No. :**	011-28-051F

Eluent: 9 mM Na2CO3
Flow Rate: 0.25 mL/min
Temperature: Ambient Temperature
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Self-Re generatin g Suppressor (Dionex ASRS™ 300 2mm)
AutoSu ppression™ Rec ycle Mode
Applied Current: 12 mA
Injection Volume: 5 µL
Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	3.83	1.6	5.55	4204	3.0
2	Chlorite	5.32	1.3	2.72	4980	10.0
3	Bromate	6.18	1.2	2.72	5477	20.0
4	Chloride	7.11	1.1	5.14	6585	6.0
5	Nitrite	9.17	1.1	6.32	6564	15.0
6	Bromide	12.42	1.0	1.82	7380	25.0
7	Chlorate	13.55	1.0	1.99	6620	25.0
8	Nitrate	14.91	1.0	5.38	7183	25.0
9	Phosphate	19.78	1.0	1.75	5068	40.0
10	Sulfate	21.72	1.0	n.a.	6060	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=5400	Passed
Sulfate	Asymmetry	1.0-1.8	Passed
Sulfate	Retention Time	19.85-23.15	Passed
	Pressure	<=2200	1577

Production Reference:
Datasource: QAR
Directory: Anion\AS9_HC
Sequence: AS9-HC_2X250MM
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)

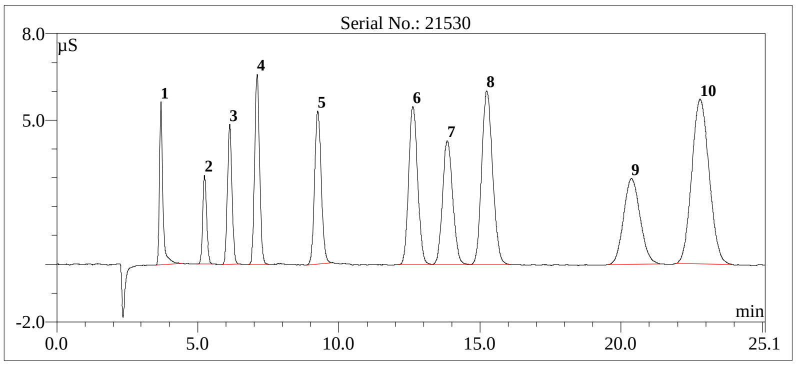
Chromeleon™ Thermo Fisher Scientific

066894-04(QAR)

Dionex IonPac™ AS9-HC
Analytical (4 x 250 mm)
Product No. 051786

Date: 13-Feb-14 10:55
Serial No. : 021530
Lot No. : 013-10-125

Eluent:	9 mM Na2CO3
Flow Rate:	1.0 mL/min
Temperature:	Ambient Temperature
Detection:	Suppressed Conductivity
Suppressor:	Dionex Anion Self-Regenerating Suppressor (Dionex ASRS™ 300 4mm) AutoSuppression™ Recycle Mode
Applied Current:	45 mA
Injection Volume:	25 µL
Storage Solution:	100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	3.69	1.6	6.75	5352	3.0
2	Chlorite	5.24	1.2	3.21	6551	10.0
3	Bromate	6.13	1.1	3.14	6830	20.0
4	Chloride	7.10	1.1	5.67	7665	6.0
5	Nitrite	9.25	1.2	6.63	7202	15.0
6	Bromide	12.62	1.2	1.95	7458	25.0
7	Chlorate	13.84	1.2	2.03	6878	25.0
8	Nitrate	15.24	1.2	5.72	7248	25.0
9	Phosphate	20.37	1.2	2.19	5660	40.0
10	Sulfate	22.80	1.1	n.a.	6393	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=5400	Passed
Sulfate	Asymmetry	1.0-1.8	Passed
Sulfate	Retention Time	19.85-23.15	Passed
	Pressure	<=2200	1739

Production Reference:

Datasource: QAR
Directory: Anion\AS9_HC
Sequence: AS9-HC_4X250MM
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)

Chromeleon™ Thermo Fisher Scientific

066900-04 (QAR)