Thermo Scientific

Dionex IonPac AS22 and Dionex IonPac AS22-Fast

Column Product Manual

P/N: 065119-08 March 2013

Thermo SCIENTIFIC

Product Manual

for

Dionex IonPacAS22-Fast Analytical Column
(4x150 mm, P/N 079936)
(2x150 mm, P/N 079937)

Dionex IonPacAG22-Fast Guard Column
(4x30 mm, P/N 072784)
(2x30 mm, P/N 072785)

Dionex IonPacAS22 Analytical Column
(4 × 250 mm, P/N 064141)
(2 × 250 mm, P/N 064137)

Dionex IonPac AS22 Capillary Column
(0.4 × 250 mm, P/N 079057)

Dionex IonPacAS22 Guard Column
(4 × 50 mm, P/N 064139)
(2 × 50 mm, P/N 064135)

Dionex IonPac AS22 Capillary Guard Column
(0.4 × 50 mm, P/N 079058)

For Research Use Only. Not for use in diagnostic procedures.

Revision History:

Revision 08, March, 2013; Reformatted for Thermo Scientific. Added new column sizes.

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™ AS22 and Dionex IonPac AS22-Fast columns are designed for compliance monitoring of inorganic anions in accordance with U.S. EPA Method 300.0 (A) and 300.1. The common inorganic anions and low molecular weight organic acids including fluoride, acetate, formate, chloride, nitrite, bromide, nitrate, phosphate and sulfate can be easily separated in a variety of sample matrices including drinking water, wastewater, process streams and scrubber solutions. The selectivity of the Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns has been designed to retain fluoride well out of the water dip (system dip) and to isocratically separate common anions including carbonate. The Dionex IonPac AS22 column is available in standard bore (4 mm i.d.), microbore (2 mm i.d.) and capillary (0.4 mm i.d.) formats by 250 mm long for samples with higher ionic strength matrices. The Dionex IonPac AS22-Fast column is available in standard bore (4 mm i.d.) and microbore (2 mm i.d.) formats by 150 mm long for fast analysis of relatively clean samples. The Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns are compatible with pH 0-14 eluents and eluents containing organic solvents from 0–100% in concentration. The Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns can be used with any suppressible ionic eluent that does not exceed the capacity of the Thermo Scientific Dionex Anion Self-Regenerating Suppressor 300 (Dionex ASRS 300) or the Thermo Scientific Dionex Anion Capillary Electrolytic Suppressor 300 (Dionex ACES 300). The Dionex IonPac AS22 columns have nominal efficiency for sulfate of at least 9,000 plates/column and Dionex IonPac AS22-Fast columns have nominal efficiency for sulfate of at least 5400 plates/column using standard operating conditions.

The Dionex IonPac AS22 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 one-hundredth (1/100) the eluent flow rate. The capillary format has 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 AS22 4 × 250 mm	6	55	210	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG22 4 × 50 mm	11	55	6	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS22 2 × 250 mm	6	55	52.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG22 2 × 50 mm	11	55	1.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS22 0.4 × 250 mm	6	55	2.1	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG22 0.4 × 50 mm	11	55	0.06	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS22-Fast 4 × 150 mm	6	55	126	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG22-Fast 4 × 30 mm	11	55	4	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS22-Fast 2 × 150 mm	6	55	31.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG22-Fast 2 × 30 mm	11	55	1	Alkanol quaternary ammonium	Ultralow

Analytical/Capillary 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)	Standard Flow Rate, mL/min	Maximum Flow Rate, mL/min[c]
Dionex IonPac AS22 4 mm Analytical	≤ 1600 (11.03)	1.2	2.5
Dionex IonPac AG22 4 mm Guard	≤ 300 (2.07)	1.2	2.5
Dionex IonPac AS22 + Dionex IonPac AG22 4 mm columns	**≤ 1900 (13.10)**	**1.2**	**2.5**
Dionex IonPac AS22 2 mm Analytical	≤ 1600 (11.03)	0.3	0.63
Dionex IonPac AG22 2 mm Guard	≤ 300 (2.07)	0.3	0.63
Dionex IonPac AS22 + Dionex IonPac AG22 2 mm columns	**≤ 1900 (13.10)**	**0.3**	**0.63**
Dionex IonPac AS22 0.4 mm Capilllary	≤ 1600 (11.03)	0.012	0.025
Dionex IonPac AG22 0.4 mm Capillary Guard	≤ 300 (2.07)	0.012	0.025
Dionex IonPac AS22 + Dionex IonPac AG22 0.4 mm columns	**≤ 1900 (13.10)**	**0.012**	**0.025**
Dionex IonPac AS22-Fast 4 mm Analytical	≤ 1600 (11.03)	1.2	2.5
Dionex IonPac AG22-Fast 4 mm Guard	≤ 300 (2.07)	1.2	2.5
Dionex IonPac AS22-Fast + Dionex IonPac AG22-Fast 4 mm columns	**≤ 1900 (13.10)**	**1.2**	**2.5**
Dionex IonPac AS22-Fast 2 mm Analytical	≤ 1600 (11.03)	0.3	0.63
Dionex IonPac AG22-Fast 2 mm Guard	≤ 300 (2.07)	0.3	0.63
Dionex IonPac AS22-Fast + Dionex IonPac AG22-Fast 2 mm columns	**≤ 1900 (13.10)**	**0.3**	**0.63**

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.

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.

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 Self-Regenerating Suppressor (SRS), 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 Borate 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 3.12 "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 AS22 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 Thermo Scientific Dionex ICS-4000 capillary system for best performance.

3.3.2 System Requirements for 2 mm Operation

The Dionex IonPac AS22 and Dionex IonPac AS22-Fast 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 IonPacAS22 and Dionex IonPac AS22-Fast 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 AG22 Guard Column or Dionex IonPac AS22-Fast Guard Column can be used for trace anion concentration work with the 2 mm and 4 mm Dionex IonPac AS22 column or the Dionex IonPac AS22-Fast column. For trace anion concentration work with the 0.4 mm Dionex IonPac AS22 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) 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

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 AS22 and Dionex IonPac AS22-Fast 2 mm columns require 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 (Section 2, “Comparison of Ion Chromatography Systems”).

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.

3.6 The Dionex IonPacAG22 and Dionex IonPac AG22-Fast Guard Columns

The Dionex IonPac AG22 Guard/Capillary Guard Column is normally used with the Dionex IonPac AS22 Analytical/Capillary Column. The Dionex IonPac AG22-Fast Guard column is normally used with the Dionex IonPac AS22-Fast Analytical Column. Retention times will increase by approximately 1.5% when a guard/capillary guard column is placed in-line prior to the analytical/capillary column. A guard/capillary guard column is placed prior to the analytical/capillary column to prevent sample contaminants from eluting onto the analytical/capillary column. It is easier to clean or replace a guard/capillary guard column than it is an analytical/capillary column. Replacing the Dionex IonPac AG22 Guard Column or Dionex IonPac AG22-Fast Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the Dionex IonPac AS22 Analytical/Capillary Column or the Dionex IonPac AS22-Fast Column.

3.7 Eluent Storage

Dionex IonPacAS22 and Dionex IonPac AS22-Fast columns are designed to be used with bicarbonate/carbonate eluent systems. Storage under a nitrogen or 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.

3.8 Dionex Anion Self-Regenerating Suppressor (Dionex ASRS 300) Requirements

A Dionex Anion Self-Regenerating 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 ASRS 300 Anion Self-Regenerating Suppressor modes of operation.

NOTE

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

For Dionex IonPac AS22 or Dionex IonPac AS22-Fast 4 mm Analytical Column, use a Dionex ASRS™ 300 (4 mm, P/N 061561).

For Dionex IonPac AS22 or Dionex IonPac AS22-Fsat 2 mm Analytical Column, use a Dionex ASRS 300 (2 mm, P/N 061562).

For Dionex IonPac AS22 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 Self-Regenerating Suppressor, see Document No. 031956, the "Product Manual for the Dionex Anion Self-Regenerating Suppressor 300".

For detailed information on the operation of the Dionex Anion Capillary Electrolytic Suppressor 300, see Document No. 065386, the "Product Manual for the Dionex Anion Capillary Electrolytic Suppressor 300, the Dionex ACES 300".

3.9 Thermo Scientific Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300) Requirements

A Dionex AMMS 300 may be used instead of a Dionex ASRS 300 Anion Self-Regenerating Suppressor (4 mm) for applications that require suppressed conductivity detection. Use a Dionex AMMS 300 (4 mm) (P/N 064558) with the Dionex IonPac AS22 4 mm Analytical column and the Dionex IonPac AS22-Fast 4 mm 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.

3.10 Using Dionex AutoRegen with the Dionex ASRS 300 or 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).

3.11 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.

3.12 Using the Dionex EGC-Carbonate Eluent Generator

The Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns are recommended for use with the Dionex IC 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 P/N 065018.

3.13 Detector Requirements

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

3.14 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

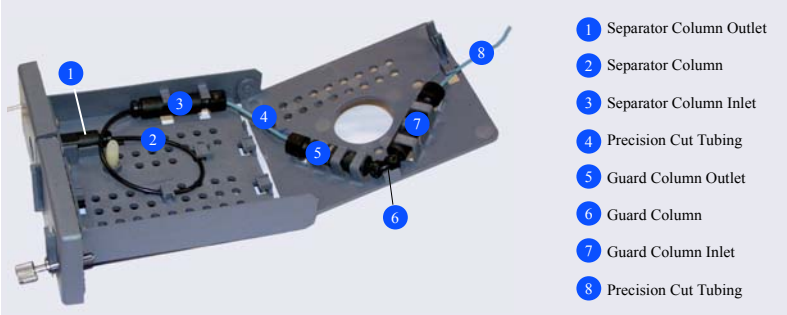
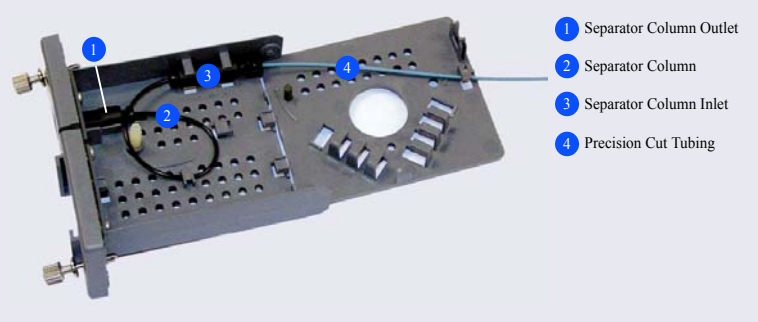


Figure 2 Separator Column Only Installed in Column Cartridge



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

4. 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

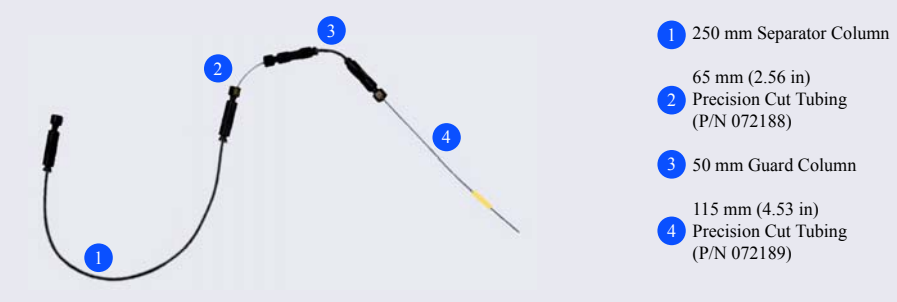
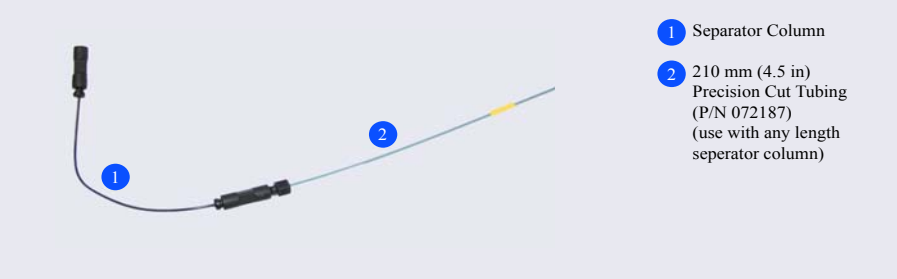
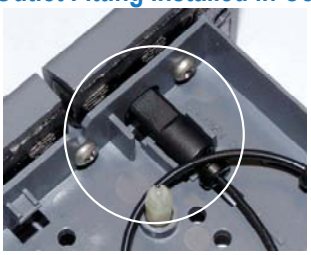


Figure 4 Tubing Connections for Separator Column Only



5. Lift up the lid of the column cartridge to open it.
6. 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



7. 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.
8. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
9. 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.
10. 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

1. Separator Column Outlet
2. Column Inlet Tubing

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 AS22 0.4 mm Capillary Column + Dionex IonPac AG22 0.4 mm Capillary Guard column 2 mm: Dionex IonPac AS22 2 mm Analytical Column + Dionex IonPac AG22 2 mm Guard Column or Dionex IonPac AS22-Fast 2 mm Analytical Column + Dionex IonPac AG22-Fast 2 mm Guard Column 4 mm: Dionex IonPac AS22 4 mm Analytical Column + Dionex IonPac AG22 4 mm Guard Column or Dionex IonPac AS22-Fast 4 mm Analytical Column + Dionex IonPac AG22-Fast 4 mm Guard Column
Eluent:	4.5 mM Na_2CO_3/1.4 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	0.4 mm: 12 µL/min 2 mm: 0.30 mL/min 4 mm: 1.2 mL/min
SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2 or 4 mm) Dionex Anion Capillary Electrolytic Suppression, 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:	20-23 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

4.2 Dionex IonPac AS22 and Dionex IonPac AS22-Fast Operation Precautions

CAUTION

Filter and Degas Eluents
Filter Samples
Eluent pH between 0 and 14
Sample pH between 0 and 14
0.63 mL/min Maximum Flow Rate for 2 mm Columns
2.5 mL/min Maximum Flow Rate for 4 mm Columns
25 µL/min Maximum Flow Rate for 0.4 mm Columns
Maximum Operating Pressure = 3,000 psi (20.68 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.

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 ionic 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 AS22 and Dionex IonPac AS22-Fast columns 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 IonPacAS22 and Dionex IonPac AS22-Fast columns is 3,000 psi (20.68 MPa).

The Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns 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 AS22 and Dionex IonPac AS22-Fast Columns

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

*Higher concentrations may only be used for limited duration applications such as column clean-up at pressures < 4000 psi.

CAUTION

The Dionex Anion Self-Regenerating Anion Suppressor (Dionex ASRS 300) 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

The Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300) requires the use of a regenerant solution. If you are using the Dionex AMMS 300 instead of the Dionex ASRS 300 Anion Self-Regenerating 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 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 3.2, "Sample Concentration").

5.1 Preparation of Eluent Stock Solution Concentrates

A. Sodium Carbonate/Bicarbonate Eluent Concentrate: The Dionex IonPac AS22 Sodium Carbonate/Bicarbonate Eluent Concentrate (0.45 M Na_2CO_3/0.14 M $NaHCO_3$), P/N 063965, can be used to prepare eluents for the Dionex IonPac AS22 column and Dionex IonPac AS22-Fast column.

   To make the eluent concentrate from reagents, thoroughly dissolve 47.7 g of sodium carbonate (MW 106.00 g/mole) plus 11.76 g sodium bicarbonate (MW 84.00 g/mole) in 700 L 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. 0.5 M Sodium Carbonate (Na_2CO_3) Concentrate: The Dionex Carbonate Concentrate, 0.5M, 500 mL is available by ordering P/N 037162.

   To make this eluent concentrate from reagents, thoroughly dissolve 26.49 g of Na_2CO_3 in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

   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 0.5 molar Sodium Carbonate Concentrate 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. We recommend EMD Chemicals sodium carbonate (P/N SX0395) for this purpose. Do not dry sodium carbonate at excessive temperatures (> 110 °C) as this will increase the pH of the salt.

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

   To make this eluent 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: 4.5 mM Sodium Carbonate/1.4 mM Sodium Bicarbonate

A. Using Dionex IonPac AS22 Eluent Concentrate:
By Weight: Weigh 988.0 g of deionized water and add 10.5 g of the Dionex IonPac AS22 Eluent Concentrate.
By Volume: To make 1 liter of eluent, pipette 10 mL of the Dionex IonPac AS22 Eluent Concentrate into a 1 L volumetric flask and dilute to a final volume of 1 L using deionized water.

B. Using 0.5 M Na_2CO_3 and 0.5 M $NaHCO_3$ Concentrates:
By Weight: Weigh 986.2 g of deionized water and add 9.45 g of 0.5 M Na_2CO_3 plus 2.94 g of 0.5 M $NaHCO_3$.
By Volume: Prepare the eluent by pipetting 9.0 mL of 0.5 M Na_2CO_3 plus 2.8 mL of 0.5 M $NaHCO_3$ into a 1 L volumetric flask. Use degassed, deionized water with a specific resistance of 18.2 megohm-cm to dilute the concentrate to a final volume of 1,000 mL.

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.

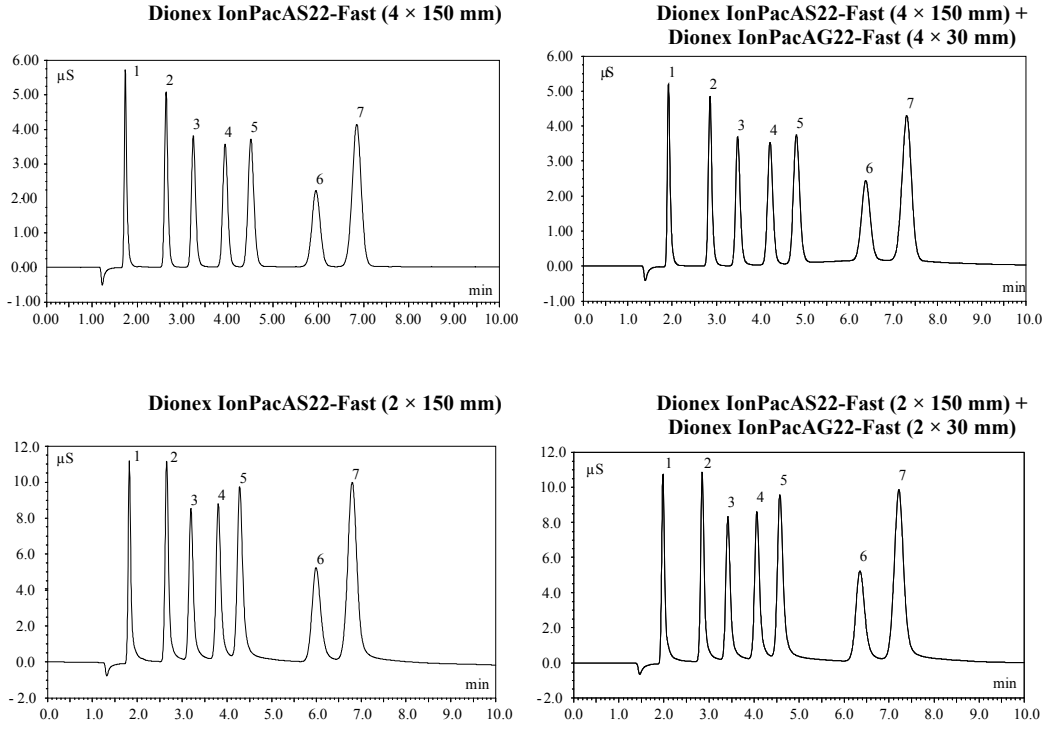
5.3 Isocratic Elution Using the Dionex IonPac AS22-Fast Column With and Without a Guard Column

Isocratic elution of anions on the Dionex IonPac AS22-Fast Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. Fluoride is resolved from acetate and formate. The Dionex IonPac AS22-Fast Analytical Column should always be used with the Dionex IonPac AG22 Guard Column. Note that the Dionex IonPac AS22 guard column is packed with a microporous resin of proportionally lower capacity and retention times will increase by approximately 1.5% when a guard column is placed in-line prior to the analytical column. ***Note that the run time for the Dionex IonPac AS22-Fast column under these conditions would be 40% lower than the run time for the Dionex IonPac AS22 column.***

Column:	See Chromatogram
Eluent:	4.5 mM Na_2CO_3/1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min (4 mm) 0.3 mL/min (2 mm)
Temperature:	30 °C
Injection Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume 2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2 mm or 4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Chloride	10.0
3.	Nitrite	15.0
4.	Bromide	25.0
5.	Nitrate	25.0
6.	Phosphate	40.0
7.	Sulfate	30.0

Figure 7 Dionex IonPac AS22-Fast Analytical Column With and Without a Guard Column



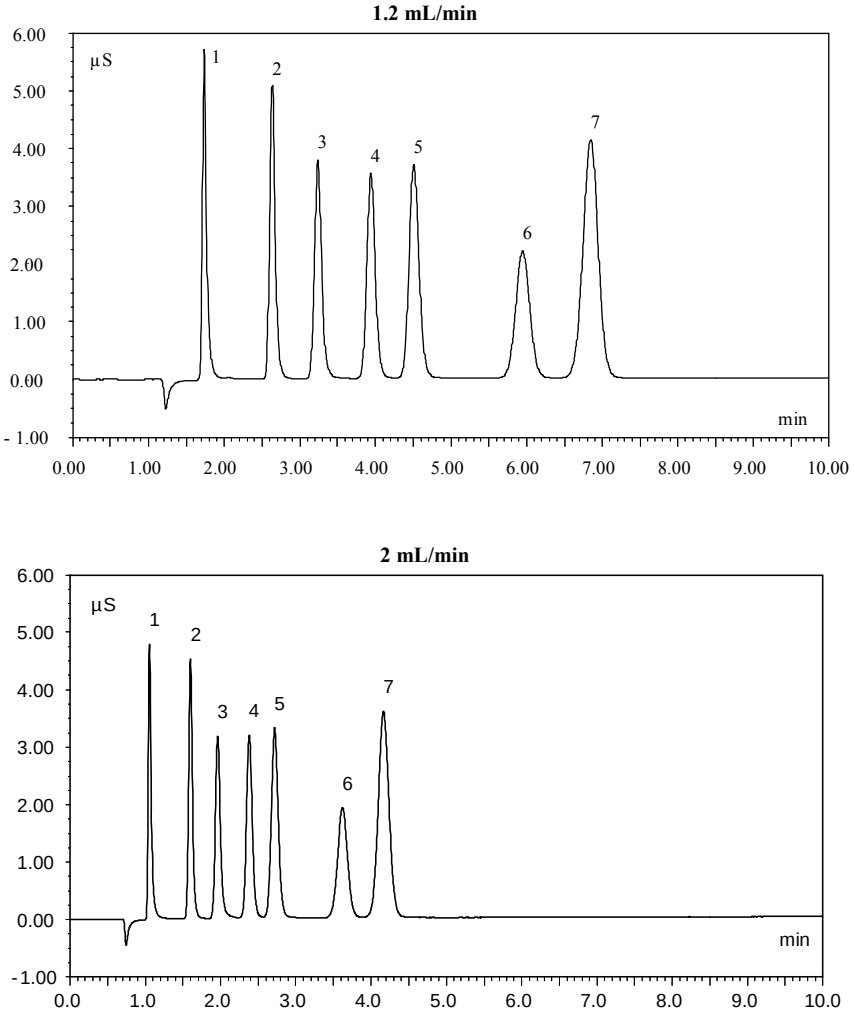
5.4 Fast Analysis of the Common Inorganic Anions using the Dionex IonPac AS22-Fast 4 mm Column with an Optimized Flow Rate

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions. ***Note the maximum flow rate for the various formats for the Dionex IonPac AS22-Fast is listed in Table 2.***

Column:	Dionex IonPac AS22-Fast (4 × 150 mm) Analytical column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	See Chromatogram
Temperature:	30 °C
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Chloride	10.0
3.	Nitrite	15.0
4.	Bromide	25.0
5.	Nitrate	25.0
6.	Phosphate	40.0
7.	Sulfate	30.0

Figure 8 Fast Analysis Without Changes in Selectivity using the Dionex IonPac AS22-Fast Column



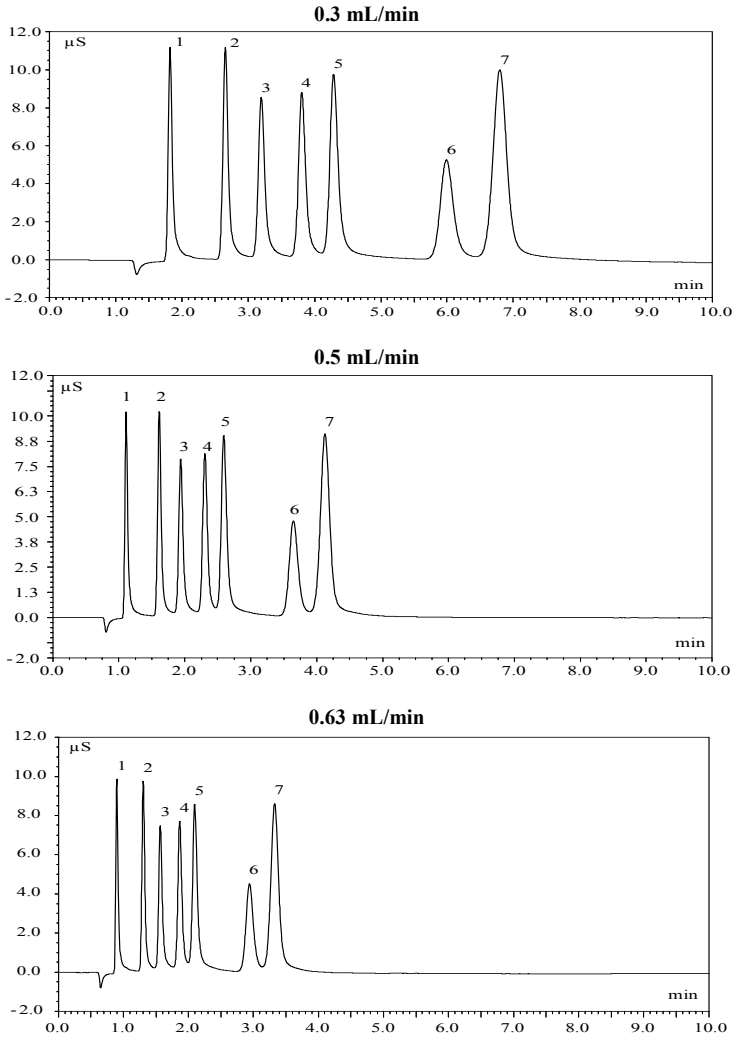
5.5 Fast Analysis of the Common Inorganic Anions using the Dionex IonPac AS22-Fast 2 mm Column with an Optimized Flow Rate

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions. ***Note the maximum flow rate for the various formats for the Dionex IonPac AS22-Fast is listed in Table 2.***

Column:	Dionex IonPac AS22-Fast (2 × 150 mm) Analytical column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	See Chromatogram
Temperature:	30 °C
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (2 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Chloride	10.0
3.	Nitrite	15.0
4.	Bromide	25.0
5.	Nitrate	25.0
6.	Phosphate	40.0
7.	Sulfate	30.0

Figure 9 Fast Analysis Without Changes in Selectivity using the Dionex IonPac AS22-Fast Column



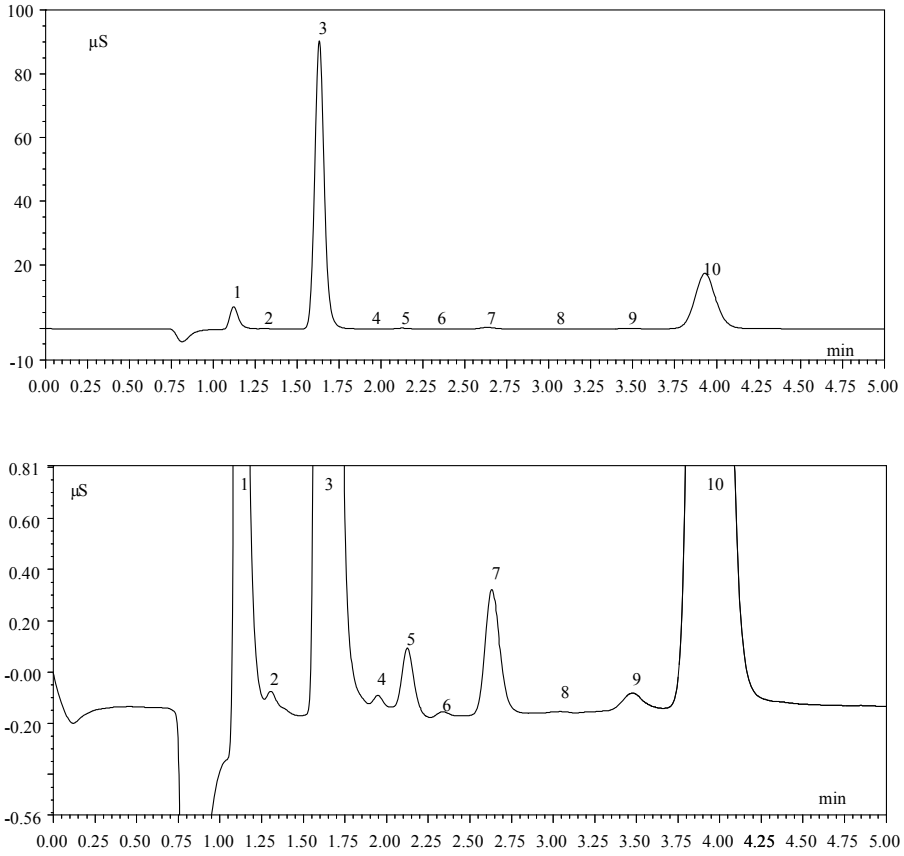
5.6 Analysis of Municipal Drinking Water using the Dionex IonPac AS22-Fast 4 mm Column

The fast analysis of municipal drinking water is demonstrated in the figure below. The eluent flow rate is increased to 2.0 mL/min reducing the run time to less than 5 minutes.

Column:	Dionex IonPac AS22-Fast (4 × 150 mm) Analytical column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	2 mL/min
Temperature:	30 °C
Injection Volume:	100 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Analyte
1. Fluoride
2. Formate
3. Chloride
4. Nitrite
5. Unknown
6. Chlorate
7. Bromide
8. Nitrate
9. Carbonate
10. Phosphate

Figure 10 Analysis of Municipal Drinking Water using the Dionex IonPac AS22-Fast Column



5.7 Isocratic Elution Using the Dionex IonPac AS22 Column With and Without a Guard Column

Isocratic elution of anions using the Dionex IonPac AS22 Analytical/Capillary Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. Fluoride is resolved from acetate and formate. The Dionex IonPac AS22 Analytical/Capillary Column should always be used with the Dionex IonPac AG22 Guard/Capillary Guard Column. Note that the Dionex IonPac AG22 guard column is packed with a microporous resin of proportionally lower capacity and retention times will increase by approximately 1.5% when a guard/capillary guard column is placed in-line prior to the analytical/capillary column.

Column:	See Chromatogram
Eluent:	4.5 mM Na_2CO_3/1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min (4 mm) 0.3 mL/min (2 mm)
Temperature:	30 °C
Injection Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume 2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2 mm or 4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Acetate	20.0
3.	Chloride	10.0
4.	Nitrite	15.0
5.	Bromide	25.0
6.	Nitrate	25.0
7.	Phosphate	40.0
8.	Sulfate	30.0

Figure 11A Dionex IonPac AS22 Analytical Columns With and Without a Guard Column

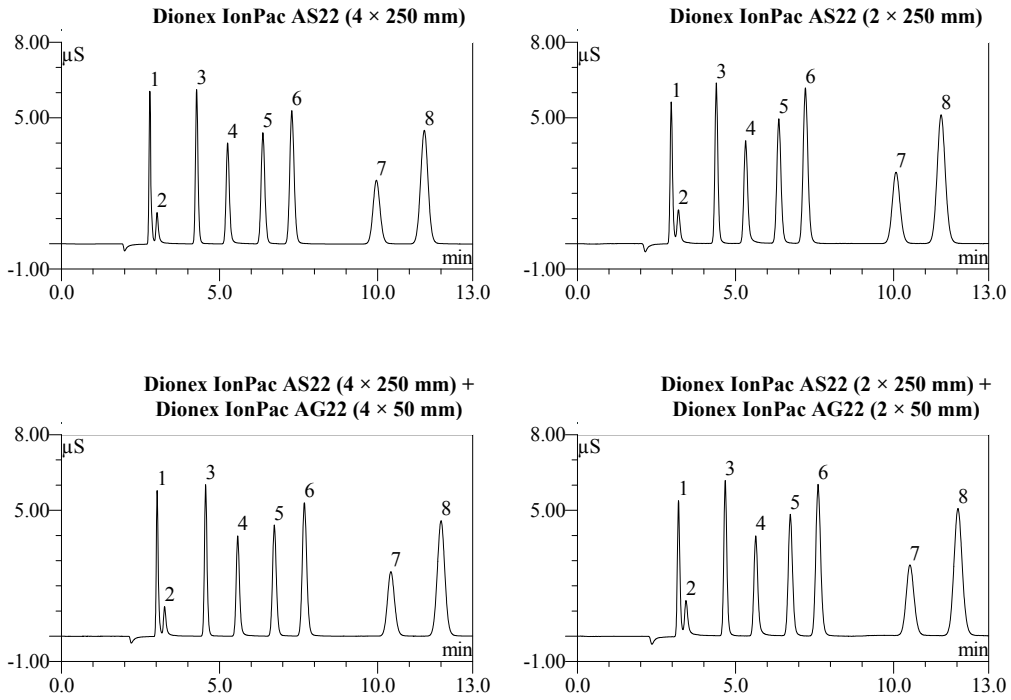
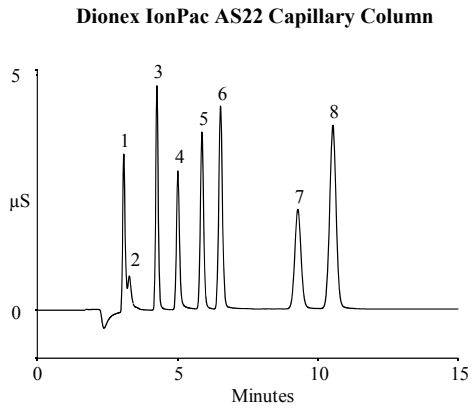
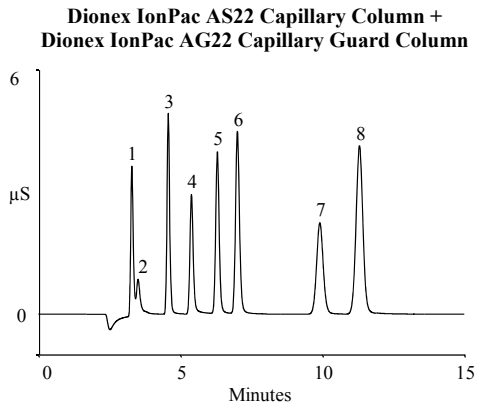


Figure 11B Dionex IonPac AS22 Capillary Column With and Without a Capillary Guard Column

Column:	See chromatograms
Eluent:	4.5 mM Na_2CO_3/1.4 mM $NaHCO_3$
Flow Rate:	12 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode

	Analyte	mg/L (ppm)
1.	Fluoride	1.25
2.	Acetate	5.0
3.	Chloride	2.5
4.	Nitrite	3.75
5.	Bromide	6.25
6.	Nitrate	6.25
7.	Phosphate	10.0
8.	Sulfate	7.5



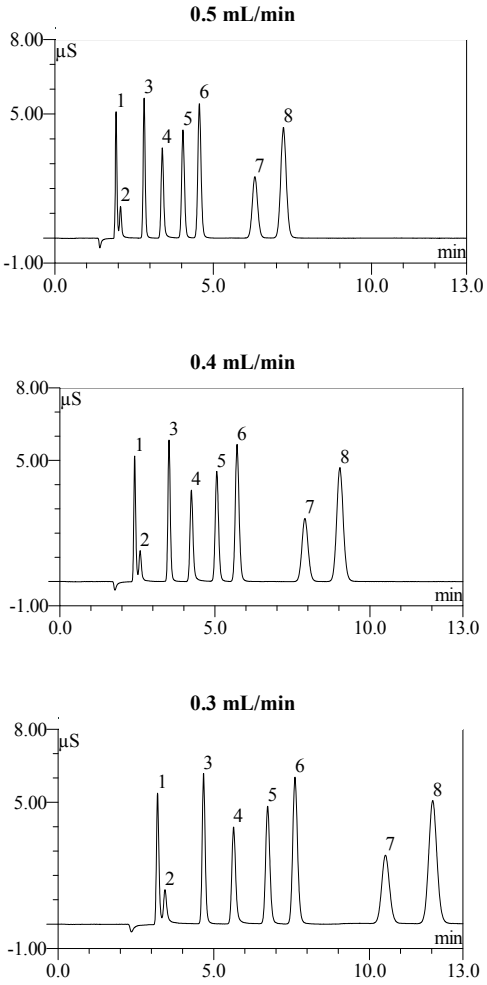


5.8 Fast Analysis of the Common Inorganic Anions using the Dionex IonPac AS22 2 mm Column with an Optimized Flow Rate

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions using the Dionex IonPac AS22 column. For fastest run times use the Dionex IonPac AS22-Fast column.

Column:	Dionex IonPacAS22 (2 × 250 mm) Analytical column Dionex IonPacAG22 (2 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	See Chromatogram
Temperature:	30 °C
Injection Volume:	2.5 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (2 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 12 Fast Analysis Without Changes in Selectivity using the Dionex IonPac AS22 Column



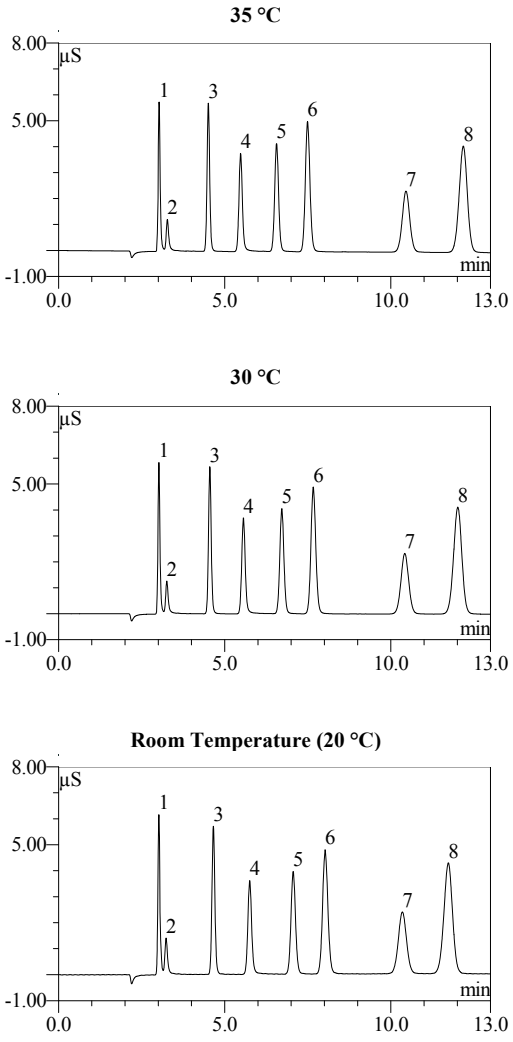
	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Acetate	20.0
3.	Chloride	10.0
4.	Nitrite	15.0
5.	Bromide	25.0
6.	Nitrate	25.0
7.	Phosphate	40.0
8.	Sulfate	30.0

5.9 Effect of Temperature on the Dionex IonPac AS22 Column Selectivity

The following chromatograms demonstrate the effect of temperature on the Dionex IonPac AS22 column selectivity. Notice monovalent inorganic anions have slightly shorter retention time and divalent inorganic anions have slightly longer retention time as temperature changes from room temperature (20 °C) to 35 °C.

Column:	Dionex IonPacAS22 (4 × 250 mm) Analytical column Dionex IonPacAG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Temperature:	See Chromatogram
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 13 Effect of Temperature in Dionex IonPac AS22 Column Selectivity



	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Acetate	20.0
3.	Chloride	10.0
4.	Nitrite	15.0
5.	Bromide	25.0
6.	Nitrate	25.0
7.	Phosphate	40.0
8.	Sulfate	30.0

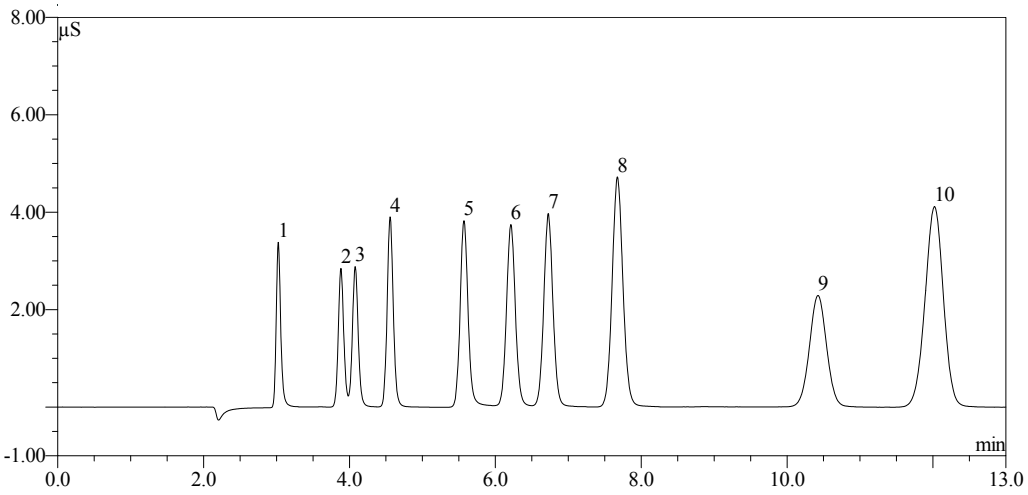
5.10 Separation of Inorganic Anions and Oxyhalides using the Dionex IonPac AS22 4 mm Column

The following chromatograms demonstrate the separation of the common inorganic anions and oxyhalides using the Dionex IonPac AS22 column.

Column:	Dionex IonPacAS22 (4 × 250 mm) Analytical column Dionex IonPacAG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3/1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Temperature:	30 °C
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

	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 14 Separation of Inorganic Anions and Oxyhalides using the Dionex IonPac AS22 Column



5.11 Analysis of Carbonated Water using the Dionex IonPac AS22 Column

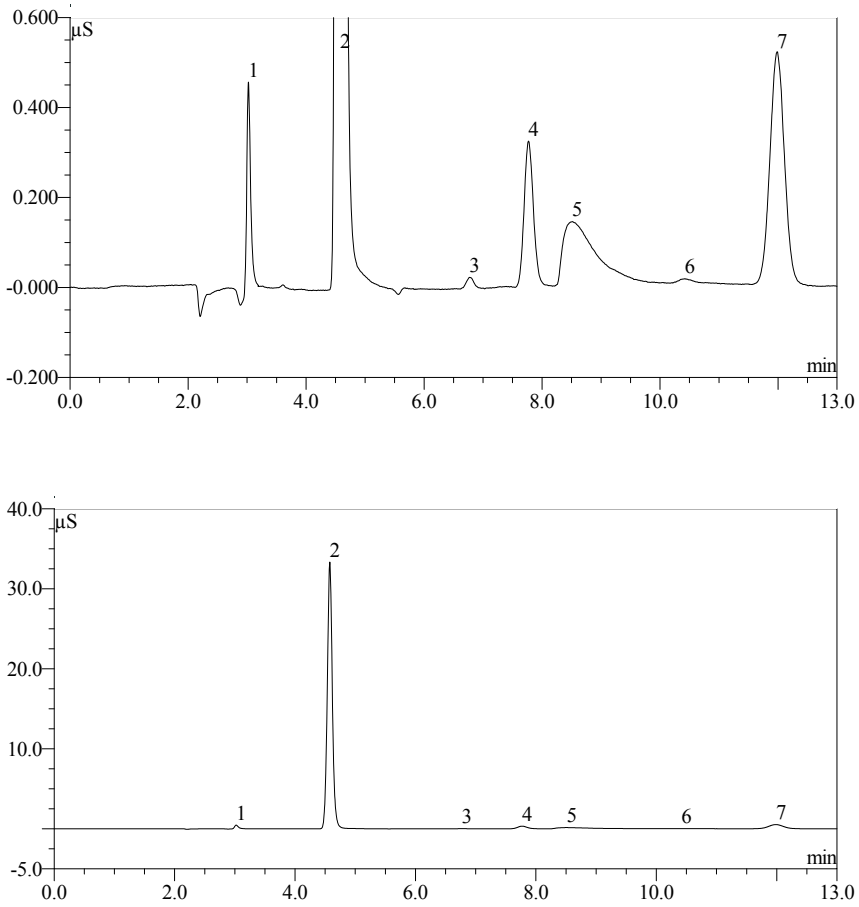
The following chromatograms demonstrate the excellent separation of carbonate from common inorganic anions using the Dionex IonPac AS22 column. The carbonated water sample can be analyzed after a simple 15 to 20 minute sonication. Figure 15A shows the analysis after sonication using the Dionex IonPac AS22 2 mm Column. Figure 15B shows the analysis before and after sonication, as well as a zoomed in view, using the Dionex IonPac AS22 Capillary Column.

Column:	Dionex IonPac AS22 (4 × 250 mm) Analytical column Dionex IonPac AG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Temperature:	30 °C
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4

	Analyte	mg/L (ppm)
1.	Fluoride	0.49
2.	Chloride	61.38
3.	Bromide	0.19
4.	Nitrate	2.53
5.	Carbonate	NQ
6.	Phosphate	0.17
7.	Sulfate	4.4

NQ=Not Quantified

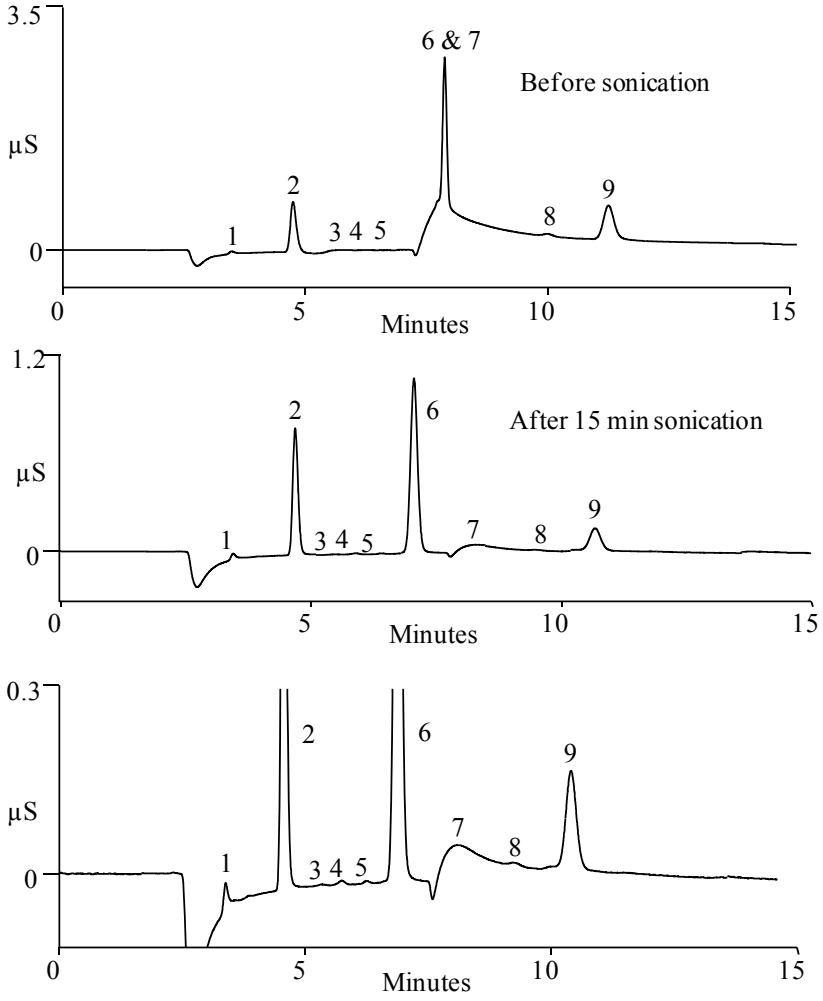
Figure 15A Analysis of Carbonated Water Using the Dionex IonPac AS22 Column



Column:	Dionex IonPac AG22/AS22 (0.4 × 250 mm)
Eluent:	4.5 mM Na_2CO_3 / 1.4 mM $NaHCO_3$
Flow Rate:	12 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode
Sample Prep:	Sonication

	Analyte	mg/L (ppm)
1.	Fluoride	0.022
2.	Chloride	0.802
3.	Nitrite	0.005
4.	Bromide	0.017
5.	Chlorate	NQ
6.	Nitrate	2.440
7.	Carbonate	NQ
8.	Phosphate	0.024
9.	Sulfate	0.374

Figure 15B Analysis of Carbonated Water Using the Dionex IonPac AS22 Capillary Column

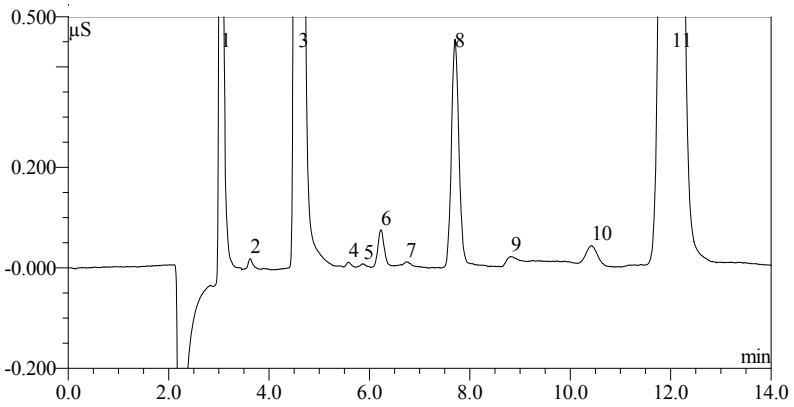


5.12 Analysis of Municipal Drinking Water using the Dionex IonPac AS22 Column

The analysis of two different municipal drinking water samples is demonstrated in the figures below using the Dionex IonPac AS22 4 mm column (Figure 16A) and Dionex IonPac AS22 0.4 mm column (Figure 16B). A run time of less than 13 minutes is achieved using the standard eluent and flow rates.

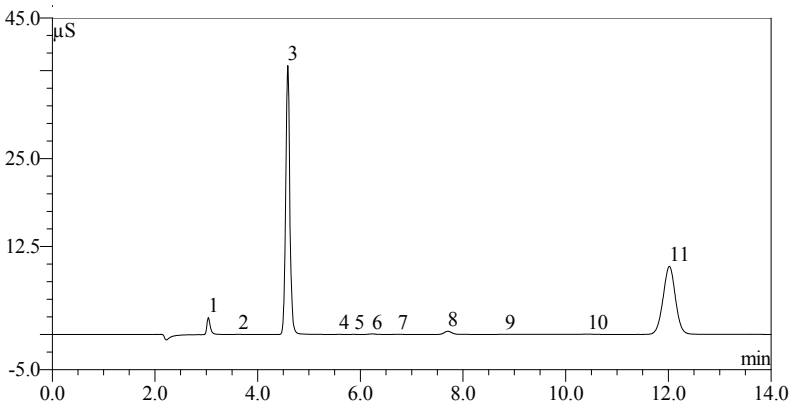
Column:	Dionex IonPac AS22 (4 × 250 mm) Analytical column Dionex IonPac AG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Temperature:	30 °C
Injection Volume:	25 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 16A Analysis of Municipal Drinking Water Using the Dionex IonPac AS22 Column



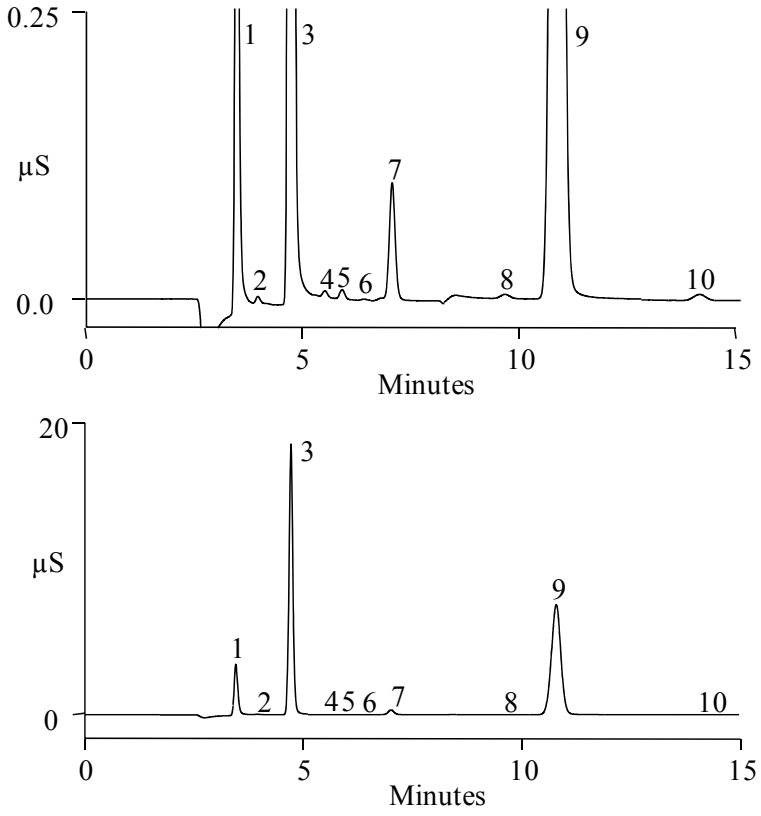
	Analyte	mg/L (ppm)
1.	Fluoride	0.84
2.	Formate	0.03
3.	Chloride	15.59
4.	Nitrite	0.01
5.	Unknown	NQ
6.	Chlorate	0.18
7.	Bromide	0.02
8.	Nitrate	0.89
9.	Carbonate	NQ
10.	Phosphate	0.22
11.	Sulfate	20.45

NQ=Not Quantified



Column:	Dionex IonPac AG22/AS22 (0.4 × 250 mm)
Eluent:	4.5 mM Na_2CO_3, 1.4 mM $NaHCO_3$
Flow Rate:	12 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed conductivity, Dionex ACES 300, recycle mode

Figure 16B Analysis of Municipal Drinking Water Using the Dionex IonPac AS22 Capillary Column



Analyte	mg/L (ppm)
1. Fluoride	2.06
2. Formate	NQ
3. Chloride	16.41
4. Nitrite	0.04
5. Unknown	NQ
6. Bromide	0.01
7. Nitrate	0.86
8. Phosphate	0.10
9. Sulfate	21.6710.
Unknown	NQ

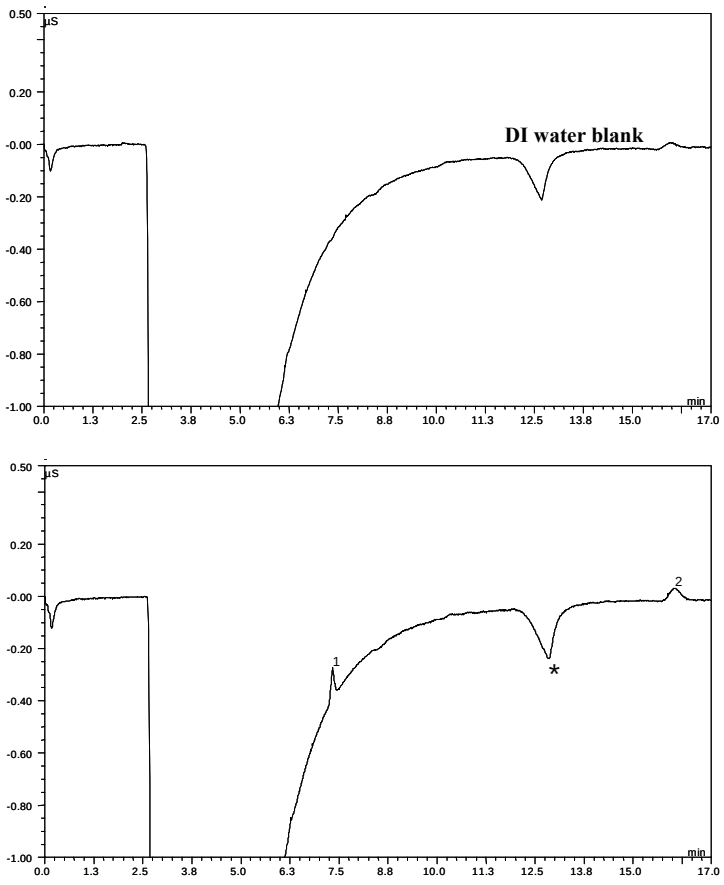
NQ=Not Quantified

5.13 Determination of Trace Chloride and Sulfate in High Purity Water using the Dionex IonPac AS22 Column

The following chromatograms demonstrate the determination of trace chloride and sulfate in high levels of carbonate using the Dionex IonPac AS22 column and a large loop injection of 2 mL. *The carbonate is present as a negative peak because the carbonate concentration in the sample is lower than the carbonate concentration in the eluent.

Column:	Dionex IonPac AS22 (4 × 250 mm) Analytical column Dionex IonPac AG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.0 mL/min
Temperature:	30 °C
Injection Volume:	2 mL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 17 Determination of Trace Chloride and Sulfate in High Purity Water Using the Dionex IonPac AS22 Column



	Analyte	µg/L (ppb)
1.	Chloride	1.0
2.	Sulfate	1.0

*500 ppb Carbonate

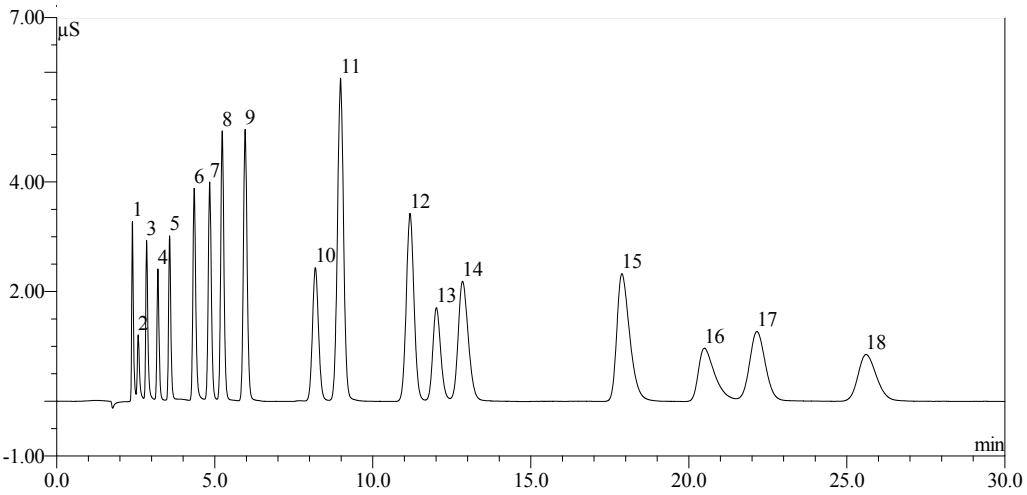
5.14 Separation of 18 Anions using an Isocratic Carbonate Eluent System with the Dionex IonPac AS22

The following chromatogram demonstrates the elution order of 18 anions using an optimized eluent on the Dionex IonPac AS22 column.

Column:	Dionex IonPac AS22 (4 × 250 mm) Analytical column Dionex IonPac AG22 (4 × 50 mm) Guard column
Eluent:	4.8 mM Na_2CO_3 1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
Temperature:	30 °C
Injection Volume:	10 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 18 Analysis of 18 Anions Using the Dionex IonPac AS22 Column

Analyte	mg/L (ppm)	Analyte	mg/L (ppm)
1. Fluoride	3.0	10. Phosphate	40.0
2. Acetate	20.0	11. Sulfate	30.0
3. Formate	10.0	12. Selenate	30.0
4. Bromate	20.0	13. Iodide	40.0
5. Chloride	5.0	14. Arsenate	30.0
6. Nitrite	15.0	15. Thiocyanate	40.0
7. Chlorate	25.0	16. Perchlorate	40.0
8. Bromide	25.0	17. Thiosulfate	40.0
9. Nitrate	25.0	18. Chromate	40.0

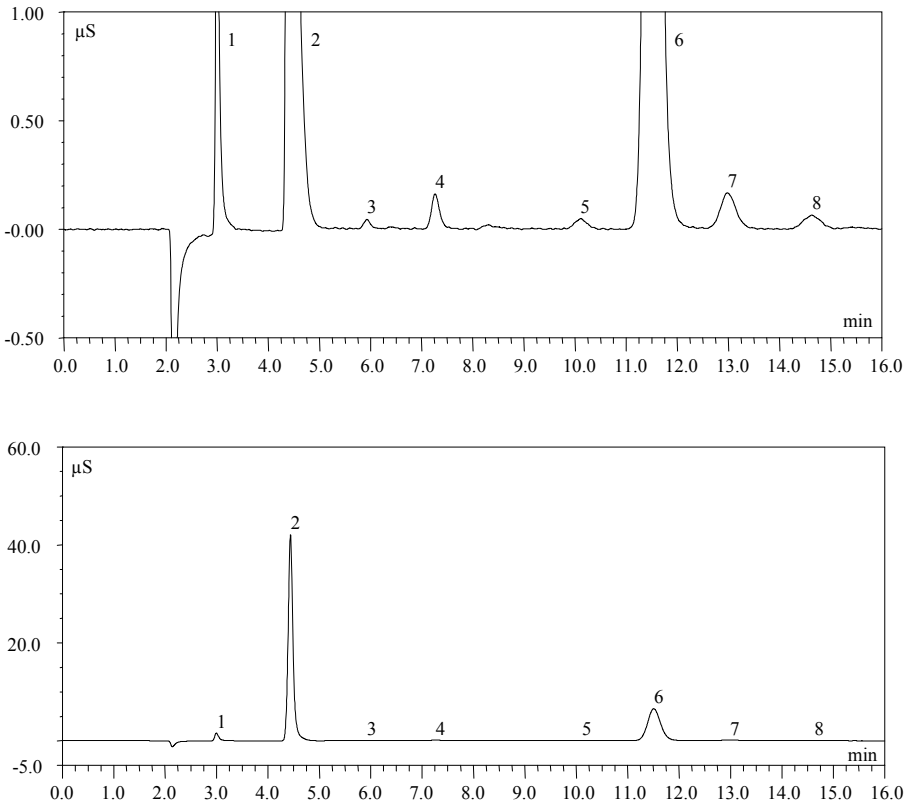


5.15 Separation of Anions in Municipal Drinking Water Spiked with Surrogate Anions using the Dionex IonPac AS22 Column

The following chromatogram shows the analysis of a drinking water sample spiked with 2 ppm of Malonate and Succinate using the Dionex IonPac AS22 column and a 25 µL injection loop. Notice the excellent separation of surrogate anions from sulfate.

Column:	Dionex IonPac AS22 (4 × 250 mm) Analytical column Dionex IonPac AG22 (4 × 50 mm) Guard column
Eluent:	4.5 mM Na_2CO_3 1.4 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Detection:	Conductivity
Temperature:	30 °C
Injection Volume:	25 µL
SRS Suppressor:	Anion Self-Regenerating Suppressor, Dionex ASRS 300 (4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, Dionex AMMS 300 (4 mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

Figure 19 Analysis of Municipal Drinking Water Using the Dionex IonPac AS22 Column



Peaks:	mg/mL
1. Fluoride	0.7
2. Chloride	25.7
3. Chlorate	0.1
4. Nitrate	0.4
5. Phosphate	0.4
6. Sulfate	17.8
7. Malonate	2.0
8. Succinate	2.0

6. Troubleshooting Guide

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac AS22 and Dionex IonPac AS22-Fast columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, 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 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 AG22 Guard/Capillary Guard Column plus the AS22 Analytical/Capillary Column or the Dionex IonPac AG22-Fast Guard Column plus the AS22-Fast Analytical Column when using the test chromatogram conditions should be equal or less than 1,900 psi. If the system pressure is higher than 1,900 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a Thermo Scientific Dionex 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, “Typical Operating Back Pressures”). 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 AS22 4 mm Columns (P/N)	Dionex IonPac AS22 2 mm Columns (P/N)	Dionex IonPac AS22 0.4 mm Columns (P/N)
Analytical Column	064141	064137	079057
Guard Column	064139	064135	079058
Bed Support Assembly	042955	044689	N/A
End Fitting	052809	043278	N/A

Product	Dionex IonPac AS22-Fast 4 mm Columns (P/N)	Dionex IonPac AS22-Fast 2 mm Columns (P/N)
Analytical Column	072782	072783
Guard Column	072784	072785
Bed Support Assembly	042955	044689
End Fitting	052809	043278

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 finger-tight, then an additional 1/4 turn (25 in × 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
4.5 mM Na_2CO_3 /1.4 mM $NaHCO_3$	20 - 23 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 Self-Regenerating Suppressor operation, refer to the Dionex Anion Self-Regenerating Suppressor 300 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

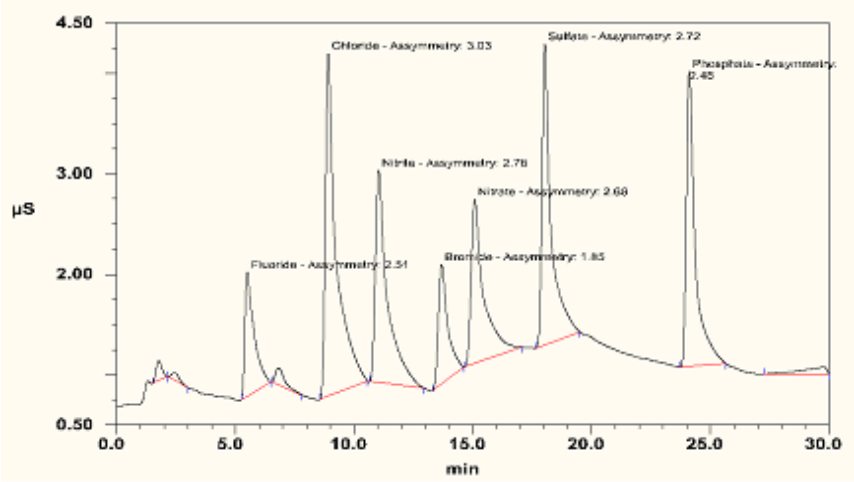
For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

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 re-torqued (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

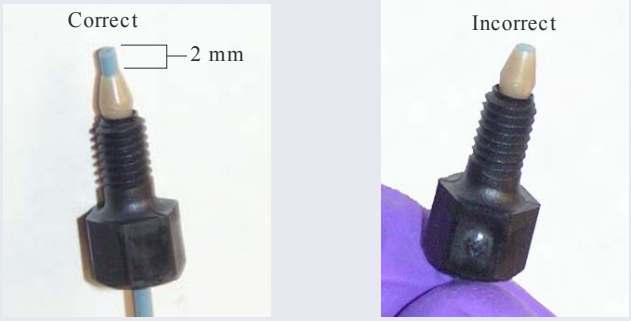
Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks.

Figure 20 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 20 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 21 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 Dionex IonPac AS22 Eluent Concentrate (P/N 063965).
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 IonPacAS22 and Dionex IonPac AS22-Fast columns is 3,000 psi (20.68 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, 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 ≤ 5% levels and the ionic strength of the eluent to ≤ 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

Contamination	Solution
Hydrophilic Contamination of Low Valence	Concentrated carbonate solutions such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.
High Valence Hydrophilic Ions Contamination	Concentrated acid solutions such as 1 to 3 M HCl will remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.
Metal Contamination	Metal contamination often results in asymmetric peak shapes and/or variable analyte recoveries. For example, iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.
	Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.
Nonionic and Hydrophobic Contamination	Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents.
Ionic and Hydrophobic Contamination	Concentrated acid solutions such as 1 to 3 M HCl can be used with compatible organic solvents to remove contamination that is ionic and hydrophobic. The acid suppresses ionization and ion exchange interactions of the contamination with the resin.
	A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

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 – Configuration

Table B1 Configuration

CONFIGURATION	2 mm	4 mm	0.4 mm
Eluent Flow Rate	0.30 mL/min	1.2 mL/min	12 µL/min
SRS Suppressor	Dionex ASRS 300 (2 mm) (P/N 061562)	Dionex ASRS 300 (4 mm) (P/N 061561)	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.	
NOTE: ***Use of a Dionex EGC-KOH cartridge (P/N 074532 or 072076 in conjunction with a Dionex CR-ATC P/N 060477 or 072078) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***			
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

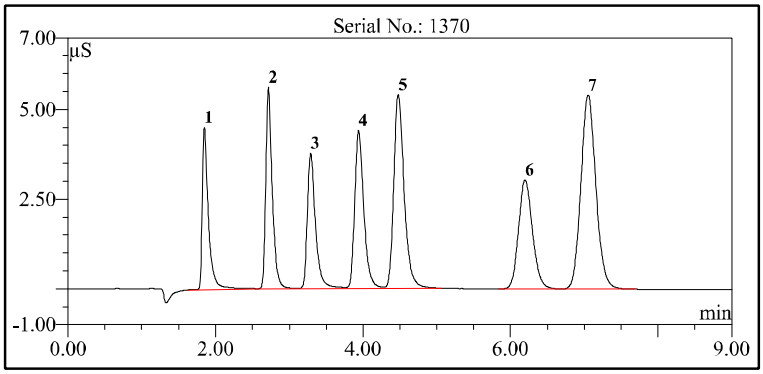
Color	Part Number	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure, Psi/ft. at 1mL/min	Back Pressure, Psi/ft. at 0.25mL/min	Back Pressure, Psi/cm. at 1mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
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 – QAR's

Dionex IonPac™ AS22-Fast
Analytical (4 x 150 mm)
Product No. 079936

Date: 18-Mar-13 16:20
Serial No. : 001370
Lot No. : 012-26-144

Eluent: 4.5 mM Na2CO3/ 1.4 mM NaHCO3
Flow Rate: 1.2 mL/min
Temperature: 30 ºC
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Self-Regenerating Suppressor (Dionex ASRS™ 300 4mm)
AutoSuppression™ Recycle Mode
Applied Current: 31 mA
Injection Volume: 10 µL
Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	1.85	2.5	6.14	3105	5.0
2	Chloride	2.72	1.7	3.39	5123	10.0
3	Nitrite	3.29	1.7	3.24	4941	15.0
4	Bromide	3.94	1.5	2.32	5346	25.0
5	Nitrate	4.48	1.5	5.84	5232	25.0
6	Phosphate	6.20	1.2	2.41	5231	40.0
7	Sulfate	7.05	1.2	n.a.	5920	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=4860	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	6.2-7.6	Passed
	Pressure	<=1760	744

Production Reference:
Datasource: Column
Directory: CPB\CPB_2
Sequence: 1483468_AS22-Fast_4MM_ZN
Sample No.: 16

6.80 SR12 Build 3578 (207169)

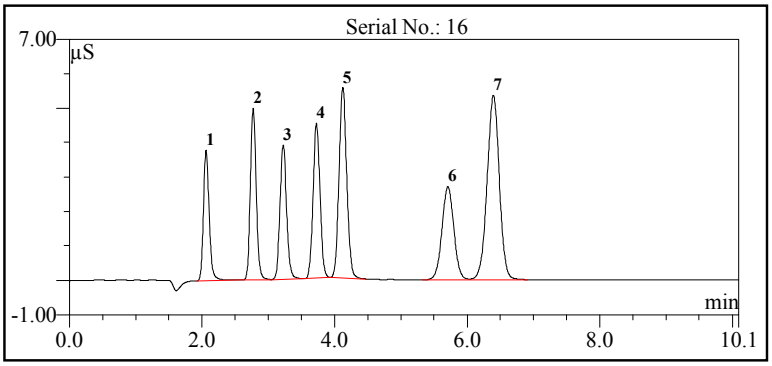
Chromeleon™ Thermo Fisher Scientific

072895-05 (QAR)

Dionex IonPac™ AS22-Fast
Analytical (2 x 150 mm)
Product No. 079937

Date: 26-Oct-09 15:23
Serial No. : 000016
Lot No. : 008-20-158

Eluent: 4.5 mM Na2CO3/ 1.4 mM NaHCO3
Flow Rate: 0.3 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Self-Regenerating Suppressor (Dionex ASRS™ 300 2mm) AutoSuppression™ Recycle Mode
Applied Current: 8 mA
Injection Volume: 2.5 µL
Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.06	1.3	4.58	2881	5.0
2	Chloride	2.77	1.1	2.67	4874	10.0
3	Nitrite	3.22	1.2	2.72	5155	15.0
4	Bromide	3.73	1.1	1.99	6097	25.0
5	Nitrate	4.12	1.1	5.97	6209	25.0
6	Phosphate	5.71	1.0	2.10	4969	40.0
7	Sulfate	6.40	1.0	n.a.	5901	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=4860	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	6.24-7.56	Passed
	Pressure	<=1760	808

Production Reference:
Datasource: QAR
Directory: Anion\AS22-Fast
Sequence: AS22-Fast_2X150MM
Sample No.: 1

6.80 SR12 Build 3578 (207169)

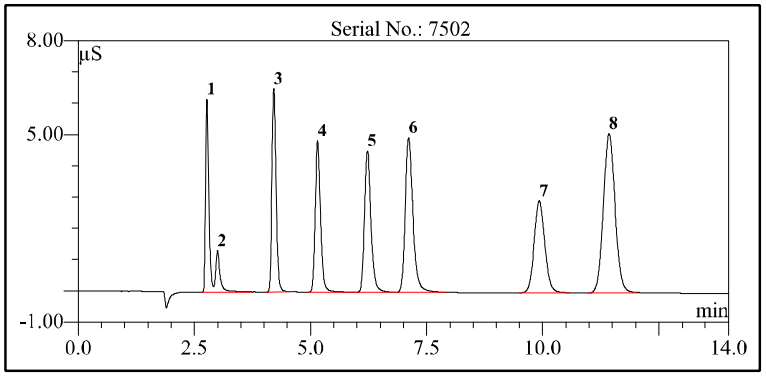
Chromeleon™ Dionex 1994-2013

072896-03 (QAR)

Dionex IonPac™ AS22
Analytical (4 x 250 mm)
Product No. 064141

Date: 04-Mar-13 11:04
Serial No. : 007502
Lot No. : 012-26-144

Eluent: 4.5 mM Na2CO3/ 1.4 mM NaHCO3
Flow Rate: 1.2 mL/min
Temperature: 30 ºC
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Self-Regenerating Suppressor (Dionex ASRS™ 300 4mm)
AutoSuppression™ Recycle Mode
Applied Current: 31 mA
Injection Volume: 10 µL
Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.77	1.6	1.68	8573	5.0
2	Acetate	3.00	n.a.	7.89	6295	20.0
3	Chloride	4.21	1.3	5.28	11494	10.0
4	Nitrite	5.16	1.3	4.81	10550	15.0
5	Bromide	6.23	1.4	3.32	10311	25.0
6	Nitrate	7.11	1.5	7.98	9568	25.0
7	Phosphate	9.93	1.2	3.46	9119	40.0
8	Sulfate	11.43	1.1	n.a.	10090	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=8100	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	10.4-12.6	Passed
	Pressure	<=1760	1473

Production Reference:
Datasource: Column
Directory: CPA\CPA_5
Sequence: 1482422_AS22_4MM _ZN
Sample No.: 135

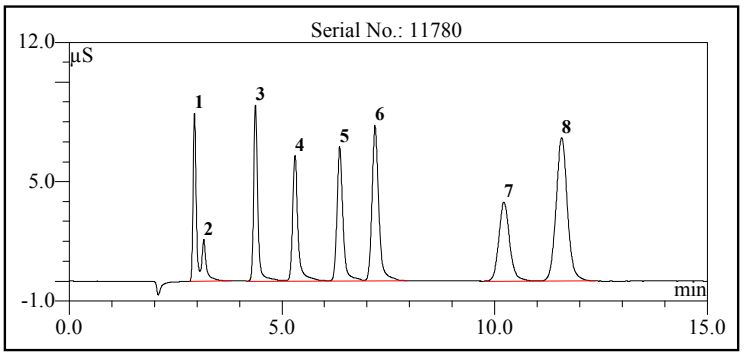
6.80 SR12 Build 3578 (207169) (Demo-Installation)
Chromeleon ™ Thermo Fisher Scientific

066544-04 (QAR)

Dionex IonPac™ AS22 2-mm Analytical (2 x 250 mm) Product No. 064137

Date: 16-Nov-12 15:42
Serial No. : 011780
Lot No. : 011-33-096

Eluent: 4.5 mM Na2CO3/ 1.4 mM NaHCO3
Flow Rate: 0.3 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Self-Regenerating Suppressor (Dionex ASRS™ 300 2mm) AutoSuppression™ Recycle Mode
Applied Current: 8 mA
Injection Volume: 2.5 µL
Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.95	1.4	1.54	9367	5.0
2	Acetate	3.16	n.a.	7.59	6230	20.0
3	Chloride	4.38	1.2	5.07	11819	10.0
4	Nitrite	5.31	1.4	4.75	10582	15.0
5	Bromide	6.36	1.3	3.28	11571	25.0
6	Nitrate	7.19	1.3	8.45	11282	25.0
7	Phosphate	10.22	1.2	2.96	8303	40.0
8	Sulfate	11.58	1.1	n.a.	9636	30.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=8100	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	10.40-12.60	Passed
	Pressure	<=1760	1205

Production Reference:
Datasource: Column
Directory: CPA\CPA_6
Sequence: 1477807 _AS22_2 MM_ AK
Sample No.: 40

6.80 SR11 Build 3160 (183147) (Demo-Installation)

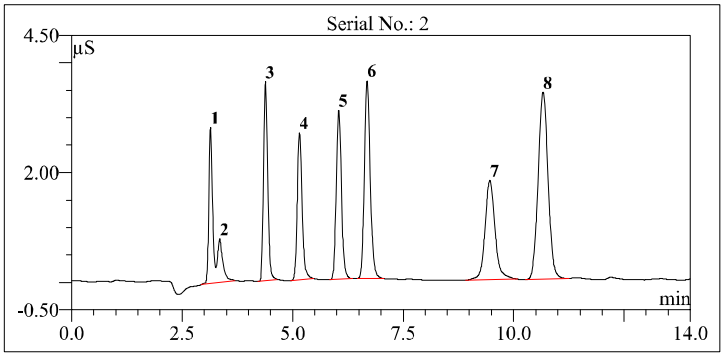
Chromeleon ™ Thermo Fisher Scientific

066547-04 (QAR)

Dionex IonPac™ AS22
Capillary (0.4 x 250 mm)
Product No. 079057

Date: 03-Apr-13 15:15
Serial No. : 000002
Lot No. : 2012-08-003

Eluent: 4.5 mM Na2CO3/ 1.4 mM NaHCO3
Flow Rate: 12 µL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Anion Capillary Electrolytic Suppressor (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	3.14	1.5	1.15	6989	1.25
2	Acetate	3.35	n.a.	5.54	4033	5.00
3	Chloride	4.39	1.2	4.25	11448	2.50
4	Nitrite	5.15	1.3	4.31	10871	3.75
5	Nitrate	6.04	1.2	2.85	12697	6.25
6	Bromide	6.69	1.2	8.74	12430	6.25
7	Phosphate	9.46	1.2	3.00	9106	10.00
8	Sulfate	10.67	1.1	n.a.	10883	7.50

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Efficiency	>=8100	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	10.4-12.6	Passed
	Pressure	<=1760	1533

Production Reference:
Datasource: QAR
Directory: Cap\AS22
Sequence: AS22_0p4x250
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific