Dionex IonPac AS14 Columns

031199 Revision 08 • November 2015

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Product Manual

for

Dionex IonPac AG14 Guard Column
(4 x 50 mm, P/N 046134)
(2 x 50 mm, P/N 046138)

Dionex IonPac AS14 Analytical Column
(4 x 250 mm, P/N 046124)
(2 x 250 mm, P/N 046129)

Revision History:

Revision 08, November, 2014: Typo correction in Section 1. Document rebranded for Thermo Scientific. Added Column Care Section.

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™ AS14 Analytical Column in combination with the Dionex IonPac AG14 Guard Column is designed for the analysis of fluoride and other inorganic anions. The selectivity of the Dionex IonPac AS14 Guard column plus Analytical Column has been designed to retain fluoride well out of the water dip (system dip) and to isocratically separate common anions. The Dionex IonPac AS14 column is compatible with pH 2-12 eluents and eluents containing organic solvents from 0–100% in concentration. The Dionex IonPac AS14 column can be used with any suppressible ionic eluent that does not exceed the capacity of the Dionex Anion Electrolytically Regenerated Suppressor (Dionex AERS 500). The Dionex IonPac AS14 has nominal efficiency for sulfate using standard operating conditions of at least 16,000 plates/meter.

Table 1 Dionex IonPac AS14/AG14 Column Packing Specifications

Column	Particle Diameter µm [a]	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
Dionex IonPac AS14 Analytical 4 x 250 mm	9.0	55	65	Alkyl quaternary ammonium	Medium-High
Dionex IonPac AG14 Guard 4 x 50 mm	9.0	55	13	Alkyl quaternary ammonium	Medium-High
Dionex IonPac AS14 Analytical 2 x 250 mm	9.0	55	16.25	Alkyl quaternary ammonium	Medium-High
Dionex IonPac AG14 Guard 2 x 50 mm	9.0	55	3.25	Alkyl quaternary ammonium	Medium-High

[a] macroporous (100 Å) divinylbenzene/ethylvinylbenzene polymer

Table 2 Dionex IonPac AS14/AG14 Column Operating Parameters

Column	Typical Back Pressure psi (MPa [a]), 30°C [b]	Standard Flow Rate mL/min	Maximum Flow Rate mL/min [c]
Dionex IonPac AS14 4 mm Analytical	< 1,300 (8.96)	1.2	3.0
Dionex IonPac AG14 4 mm Guard	< 325 (2.24)	1.2	3.0
Dionex IonPac **AS14 + AG14 4 mm Columns**	**< 1,625 (11.20)**	**1.2**	**3.0**
Dionex IonPac AS14 2 mm Analytical	< 1,300 (8.96)	0.3	0.75
Dionex IonPac AG14 2 mm Guard	< 325 (2.24)	0.3	0.75
Dionex IonPac **AS14 + AG14 2 mm Columns**	**<1,625 (11.20)**	**0.3**	**0.75**

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

WARNING

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

NOTE

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

2. Ion Chromatography Systems

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

- For an ICS in 2 mm format, a microbore isocratic pump, standard bore isocratic pump, microbore gradient pump, or standard bore gradient pump is recommended.
- For an ICS in 4 mm format, a standard bore isocratic pump or standard bore gradient pump is recommended.

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

3. Installation

3.1 System Requirements

3.1.1 System Requirements for 2 mm Operation

The Dionex IonPac AS14 2 mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a gradient pump configured for narrow bore operation.

3.1.2 System Requirements for 4 mm Operation

The Dionex IonPac AS14 4 mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a system having a gradient pump configured for standard bore operation.

3.1.3 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" (P/N 044221) i.d. PEEK tubing. 0.010" i.d. PEEK tubing (P/N 042690) 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.

3.2 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 AG14 Guard Column can be used for trace anion concentration work with the 2 mm and 4 mm Dionex IonPac AS14 column.

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.)

3.3 The Injection Loop

3.3.1 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 2.5 nanomoles (100–200 ppm) of any one analyte onto a 2 mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. The Dionex IonPac AS14 2 mm column requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4 mm analytical system (Section 2, "Ion Chromatography Systems").

3.3.2 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 10 nanomoles (100–200 ppm) of any one analyte onto the 4 mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. This phenomenon will be more prevalent at higher concentrations of the analytes of interest.

3.4 The Dionex IonPac AG14 Guard Column

A Dionex IonPac AG14 Guard Column is normally used with the Dionex IonPac AS14 Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column. Replacing the Dionex IonPac AG14 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS14 Analytical Column.

3.5 Installing the Dionex IonPac Anion Trap Column, Dionex IonPac ATC-3 Column

When performing a gradient anion exchange application, a borate eluent system should be used instead of a carbonate system because of its low background conductivity. A Dionex IonPac Anion Trap Column (Dionex IonPac ATC-3 column (2 mm), P/N 079932 or Dionex IonPac ATC-3 column (4 mm), P/N 059660) should be installed between the Gradient Pump and the injection valve. Remove the high pressure Gradient Mixer if present. The Dionex IonPac ATC column is filled with high capacity anion exchange resin which helps to minimize the baseline shift caused by increasing anionic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis.

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

A. Remove the Gradient Mixer, if installed between the gradient pump pressure transducer and the injection valve.
B. Connect the gradient pump directly to the Dionex IonPac ATC column. Connect a waste line to the Dionex IonPac ATC column outlet and direct the line to a waste container.
C. Flush the Dionex IonPac ATC column with 200 mL of 70 mM $Na_2B_4O_7$ at a flow rate of 0.5 mL/min when using the Dionex ATC-3 column (2 mm) or 2.0 mL/min when using the Dionex IonPac ATC-3 column (4 mm).
D. Rinse the Dionex IonPac ATC column with the strongest eluent that will be used during the gradient analysis.
E. After flushing the Dionex IonPac ATC column with eluent, connect the Dionex IonPac ATC column to the eluent line that is connected to the injection valve.

The background conductivity of your system should be less than 7 µS when $Na_2B_4O_7$ is being pumped through the chromatographic system with the Dionex AERS 500 in-line and properly functioning. The baseline shift should be no greater than 10 µS during a borate gradient eluent concentration ramp from 0 to 70 mM $Na_2B_4O_7$. If the baseline shifts are greater than 10 µS, the Dionex IonPac ATC column should be cleaned using steps A - E above.

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

A. Flush the Dionex IonPac ATC column with 30 mL of 70 mM $Na_2B_4O_7$.
B. Prior to using the chromatographic system the next day, flush the Dionex IonPac ATC column with 30 mL of the strongest eluent used in the gradient program.

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

3.6 Eluent Storage

Dionex IonPac AS14 columns are designed to be used with bicarbonate/carbonate eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

NOTE

It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of CO2 will affect retention times and selectivity.

3.7 Dionex Anion Electrolytically Regenerated Suppressor for Carbonate Eluents (Dionex AERS 500 Carbonate) Requirements

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

NOTE

The Dionex AERS 500 suppressor does not support the chemical regeneration mode. The Dionex Chemically Regenerated Suppressor is recommended for chemical regeneration mode.

NOTE

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

If you are installing a Dionex IonPac AS14 4 mm Analytical Column, use a Dionex AERS 500 Carbonate Suppressor (4 mm, P/N 085029).

If you are installing a Dionex IonPac AS14 2 mm Analytical Column, use a Dionex AERS 500 Carbonate Suppressor (2 mm, P/N 085028).

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

3.8 Dionex Anion Chemically Regenerated Suppressor (Dionex ACRS 500) Requirements

A Dionex Anion Chemically Regenerated Suppressor (ACRS 500) may be used instead of Dionex AERS 500 suppressor (4 mm) for applications that require suppressed conductivity detection. Use a Dionex ACRS suppressor (P/N 085090) with the Dionex IonPac AS14 4 mm Analytical Column. It is compatible with all solvents and concentrations with which the systems and columns are compatible. For 2 mm operation, use the Dionex ACRS 500 suppressor (P/N 085091).

For detailed information on the operation of the Dionex Anion Chemically Regenerated Suppressor, see Document No. 031727, the "Thermo Scientific Dionex CRS 500 Suppressor Product Manual".

3.9 Using Dionex AutoRegen with the Dionex ACRS 500 Suppressor in the Chemical Suppression Mode

To save regenerant preparation time and reduce regenerant consumption and waste, it is recommended to use a Dionex AutoRegen Accessory (P/N 039564) with the Dionex ACRS 500 suppressor. For more detailed information on the use of the AutoRegen Accessory see the 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).

3.10 Using the Dionex EGC-Carbonate Eluent Generator

The Dionex IonPac AS14 column is recommended for use with the Dionex systems equipped with a Dionex Eluent Generator. The Dionex Eluent Generator is used to automatically produce carbonate and bicarbonate eluents from deionized water. For more information, see the manual for the Dionex Eluent Generator Cartridges Product Manual, Document No. 065018.

4. Operations

4.1 General Operating Conditions

Sample Volume:	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:	2 mm: Dionex IonPac AS14 2 mm Analytical Column + Dionex IonPac AG14 2 mm Guard Column 4 mm: Dionex IonPac AS14 4 mm Analytical Column + Dionex IonPac AG14 4 mm Guard Column
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	2 mm: 0.3 mL/min 4 mm: 1.2 mL/min
ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor for Carbonate Eluents, Dionex AERS 500 Carbonate, (2 mm or 4 mm) AutoSuppression Recycle Mode
or CRS Suppressor:	Anion Chemically Regenerated Suppressor (ACRS 500)
CRS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS
Storage Solution:	Eluent

4.2 Dionex IonPac AS14 Operation Precautions

CAUTION

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

4.3 Chemical Purity Requirements

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

4.3.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label. Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex IonPac AS14 Eluent Concentrate (P/N 053560) is recommended in order to avoid this problem. Otherwise, use of a high purity grade of sodium carbonate to prepare the eluents will generally prevent the problem.

4.3.2 Deionized Water

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

4.4 Preparation of Eluent Stock Solution Concentrates

A. 0.5 M Sodium Carbonate (Na_2CO_3) Concentrate

Order Thermo Scientific Dionex Carbonate Concentrate (P/N 037162)

or

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.

B. 0.5 M Sodium Bicarbonate ($NaHCO_3$) Concentrate

Order Thermo Scientific Dionex Bicarbonate Concentrate (P/N 037163)

or

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.

4.4.1 Eluent Preparation

Eluent: 3.5 mM Sodium Carbonate/1.0 mM Sodium Bicarbonate

Prepare the eluent by pipetting 7.0 mL of 0.5 M Na_2CO_3 plus 2.0 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.

4.5 Making Eluents that Contain Solvents

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. 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 bottle.

The AS14 column can withstand common HPLC solvents in a concentration range of 0–100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 5 HPLC Solvents for Use with Dionex IonPac AS14 Columns

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

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

CAUTION

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

4.6 Regenerant Preparation for the Dionex ACRS 500 Suppressor

The Dionex Anion Chemically Regenerated Suppressor 500 (ACRS 500) requires the use of a regenerant solution. If you are using the Dionex ACRS 500 instead of the Dionex AERS 500 see Document No. 031727, the "Thermo Scientific Dionex CRS 500 Suppressor Product Manual."

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 vary slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components, and laboratory temperatures.

Before attempting any of the following example applications, take the time to ensure that your system is properly configured. Ensure 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 "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, "The Sample Concentrator").

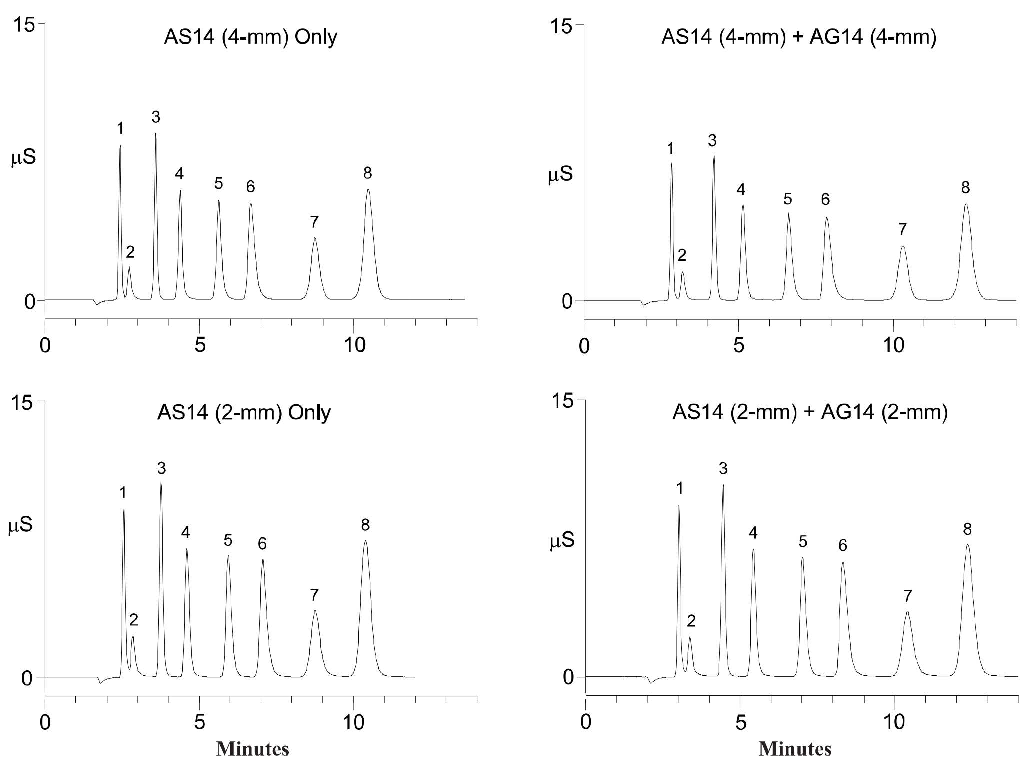
5.1 Isocratic Elution Using the Dionex IonPac AS14 Column With and Without a Guard Column

Isocratic elution of anions on the Dionex IonPac AS14 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 AS14 Analytical Column should always be used with the Dionex IonPac AG14 Guard Column.

Sample Volume:	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:	See Chromatogram
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	0.3 mL/min (2 mm), 1.2 mL/min (4 mm)
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor (2 mm or 4 mm), AutoSuppression Recycle Mode
or recommended CRS Suppressor:	Dionex Anion Chemically Regenerated Suppressor (Dionex ACRS 500)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS
Storage Solution:	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 2 Dionex IonPac AS14 Production Test Chromatograms



5.2 Fast Run Analysis without Changes in Selectivity

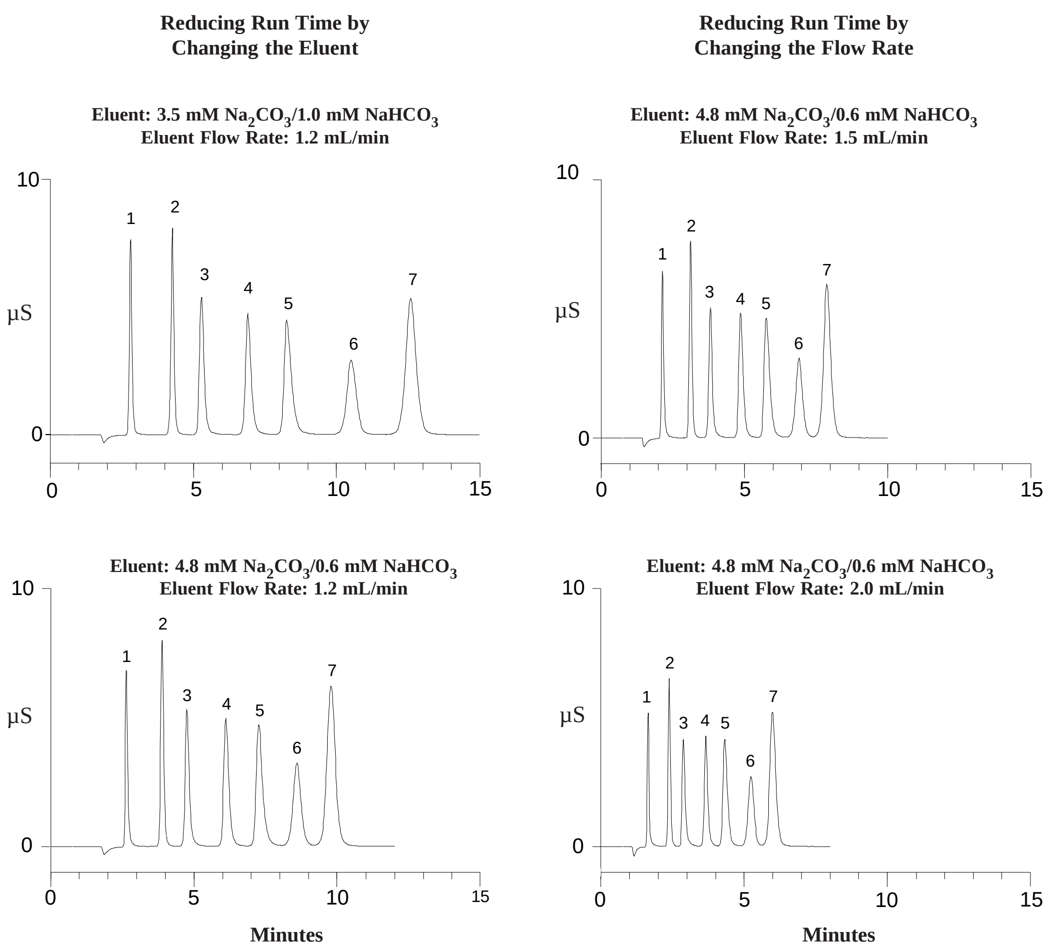
The following chromatograms demonstrate the eluent and flow rate changes for a fast run time.

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	See Chromatogram
Eluent Flow Rate:	See Chromatogram
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

	Analyte	mg/L
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

where 1 mg/L = 1 ppm

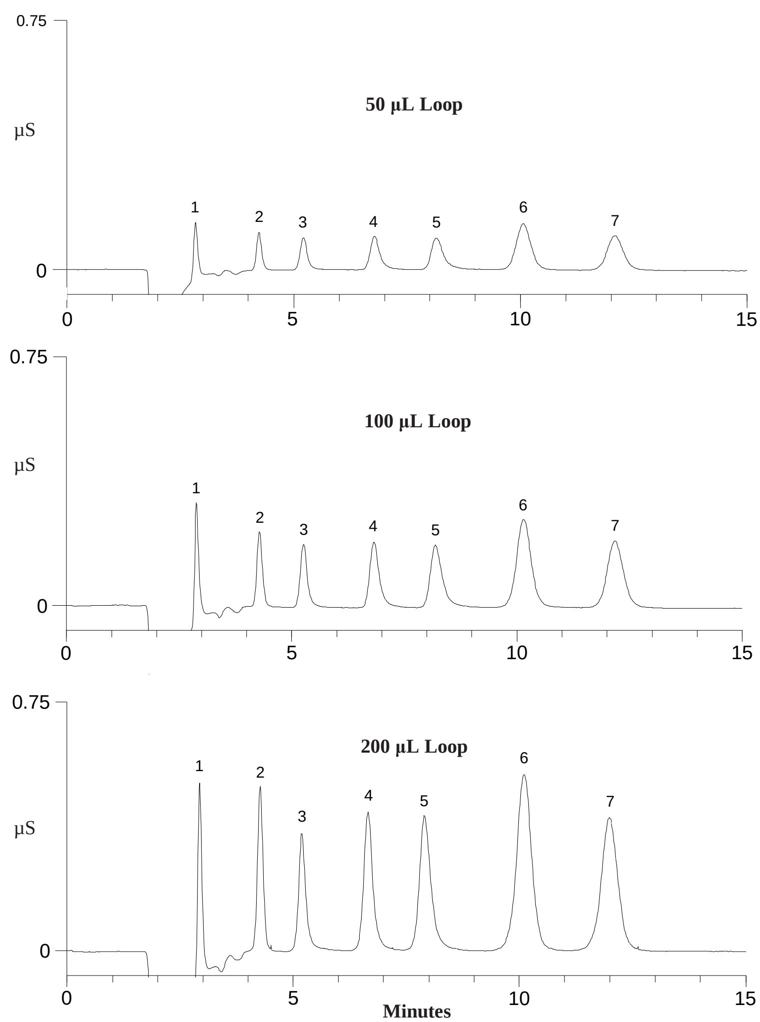
Figure 3 Fast Analysis Without Changes in Selectivity



5.3 Increasing the Loop Size to Increase Sensitivity

Sample Loop Volume:	See Chromatogram
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression External Water Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

Figure 4 Increasing Sensitivity by Increasing Loop Size



	Analyte	mg/L
1.	Fluoride	0.05
2.	Chloride	0.10
3.	Nitrite	0.12
4.	Bromide	0.25
5.	Nitrate	0.25
6.	Phosphate	0.80
7.	Sulfate	0.25

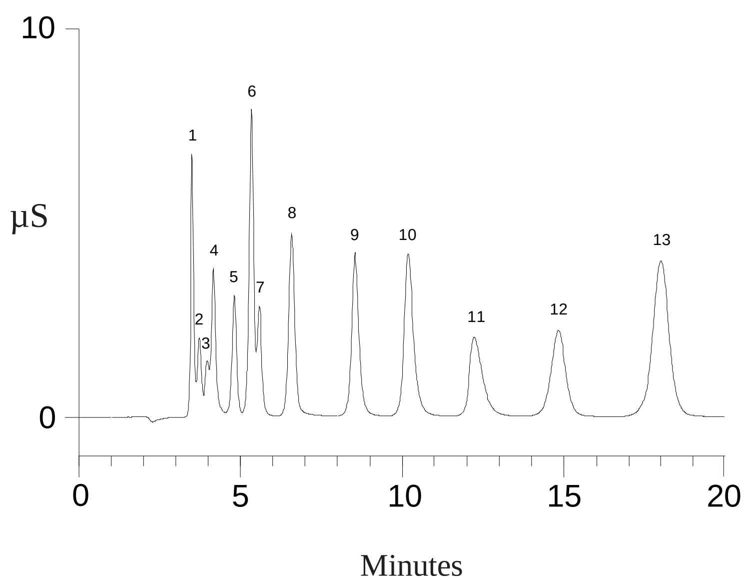
where 1 mg/L = 1 ppm

5.4 13 Anions Selectivity Using Carbonate/Bicarbonate Eluent

The following chromatogram shows the elution order of glycolate, formate, chlorite, bromate and chlorate along with the 8 common anions on the test chromatogram using a modified carbonate/bicarbonate eluent.

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	2.7 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.0 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	15-17 µS

Figure 5 13 Anions Selectivity



	Analyte	mg/L
1.	Fluoride	5.0
2.	Glycolate	10.0
3.	Acetate	20.0
4.	Formate	10.0
5.	Chlorite	10.0
6.	Chloride	10.0
7.	Bromate	20.0
8.	Nitrite	15.0
9.	Bromide	25.0
10.	Nitrate	25.0
11.	Chlorate	25.0
12.	Phosphate	40.0
13.	Sulfate	30.0

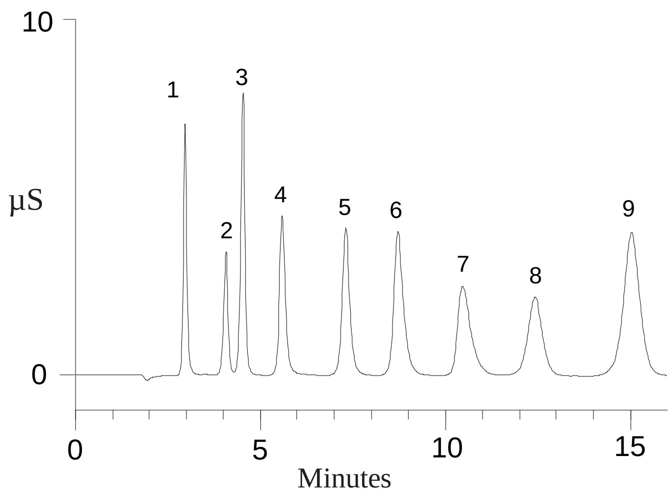
where 1 mg/L = 1 ppm

5.5 Isocratic Analysis of Selected Oxyanions

This slide illustrates optimum conditions for the separation of chlorite and chlorate from the common anions using an isocratic carbonate/bicarbonate eluent.

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	2.7 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	15-17 µS

Figure 6 Analysis of Selected Oxyanions



	Analyte	mg/L
1.	Fluoride	5
2.	Chlorite	10
3.	Chloride	10
4.	Nitrite	15
5.	Bromide	25
6.	Nitrate	25
7.	Chlorate	30
8.	Phosphate	40
9.	Sulfate	30

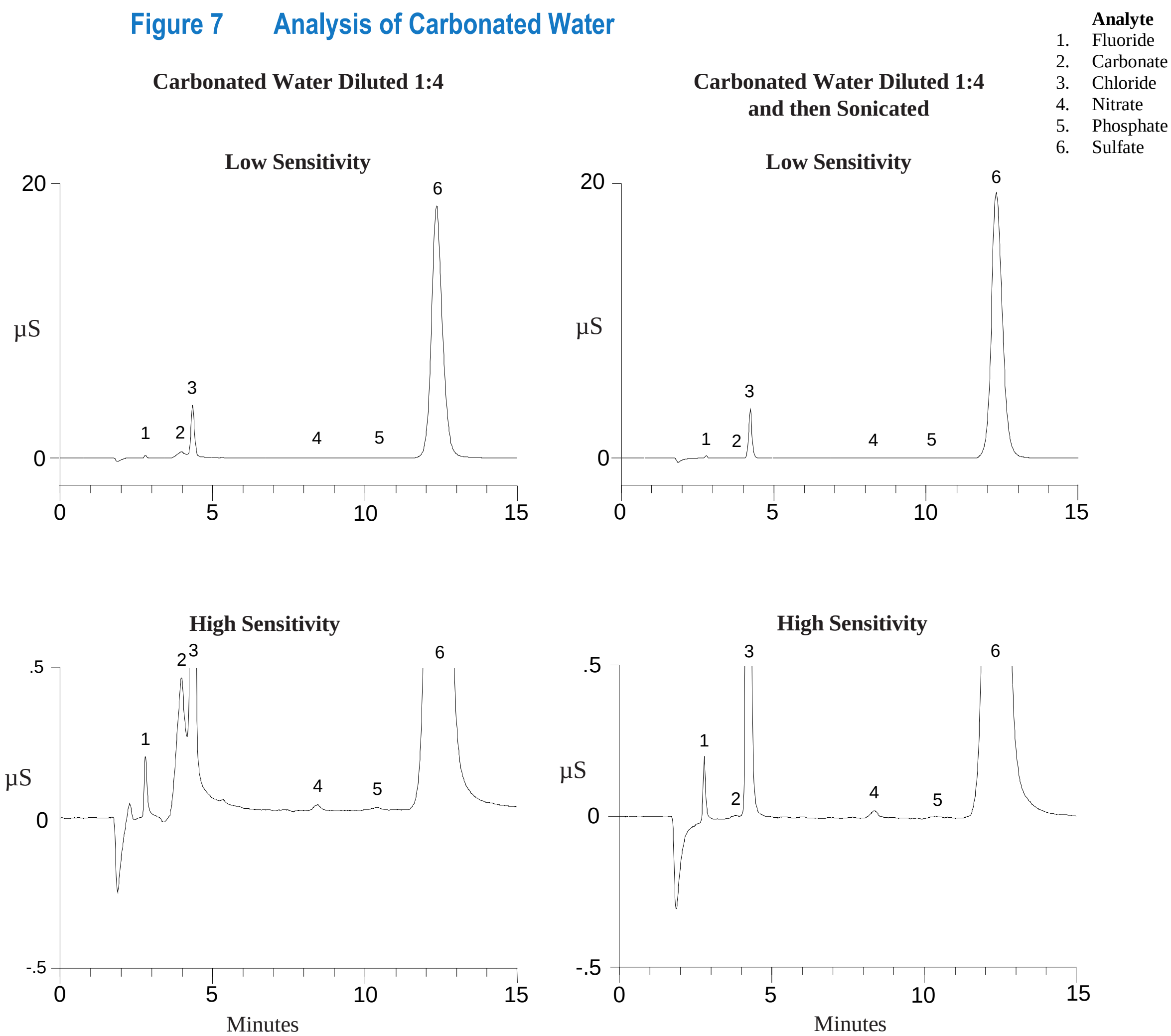
where 1 mg/L = 1 ppm

5.6 Analysis of Carbonated Water

Sonication can be used to remove carbonate from the sample. Carbonate can interfere with the quantification of chloride when large amounts of carbonate are present in the sample. The first chromatogram set (low and high sensitivity) demonstrates the carbonate interference with chloride. The second chromatogram set (low and high sensitivity) demonstrates the removal of the carbonate interference by sonicating the sample at room temperature for 5 minutes.

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

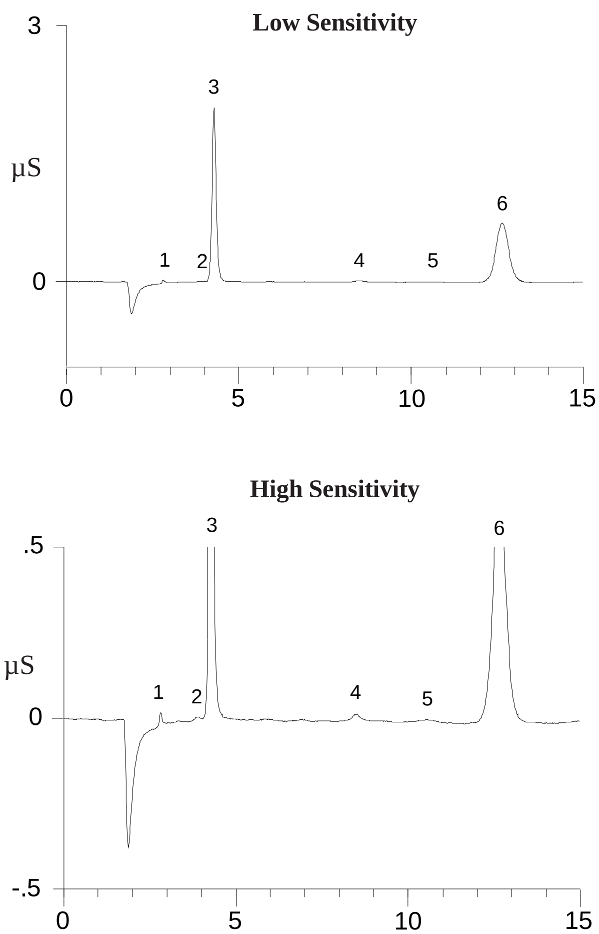
Figure 7 Analysis of Carbonated Water



5.7 Analysis of Municipal Tap Water

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

Figure 8 Analysis of Municipal Tap Water



	Analyte	mg/L
1.	Fluoride	0.03
2.	Bicarbonate	30.42
3.	Chloride	3.12
4.	Nitrate	0.15
5.	Phosphate	0.04
6.	Sulfate	4.45

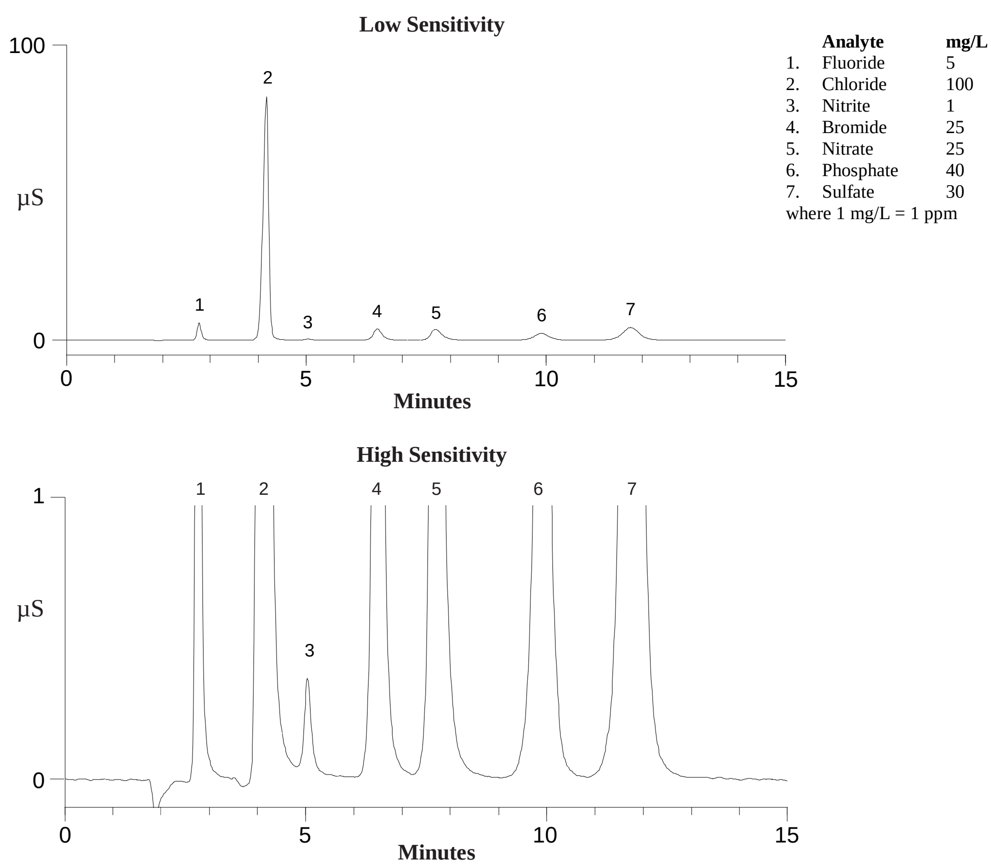
where 1 mg/L = 1 ppm

5.8 Isocratic Analysis of Low Levels of Nitrite in High Levels of Chloride (1:100 Ratio)

The quantification of nitrite in the presence of excess chloride is often difficult on the Dionex IonPac AS4A. The Dionex IonPac AS14 gives increased separation between these two peaks, resulting in easier integration of nitrite. Nitrite is sufficiently resolved from chloride so that it can be quantified at the 0.1 mg/L level even when chloride is at the 100 mg/L level.

Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

Figure 9 Isocratic Analysis of Low Levels of Nitrite in High Levels of Chloride (1:100)



5.9 Analysis of Toothpaste

The excellent retention of fluoride on the Dionex IonPac AS14 makes it ideal for the determination of fluoride and monofluorophosphate in dental care products. This slide illustrates optimized conditions for the separation of monofluorophosphate from phosphate and sulfate which can be present in toothpaste.

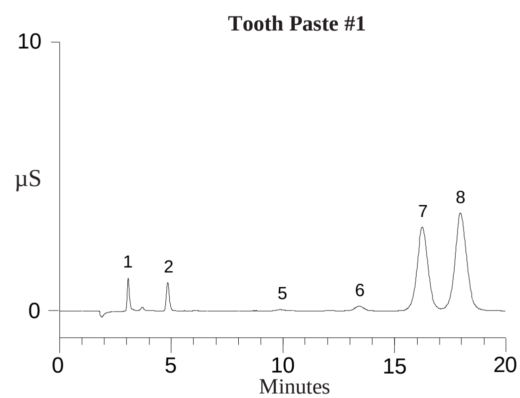
The following analyses demonstrate the quantification of monofluorophosphate in tooth paste. In each case, 1 g of sample was diluted to 100 g total weight with deionized water and then filtered first through a 0.45 µm syringe filter and then through a 0.20 µm syringe filter before injection.

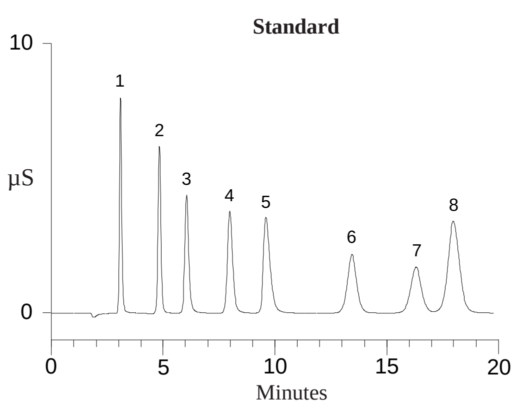
Sample Loop Volume:	10 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Eluent:	2.0 mM Na_2CO_3/2.5 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Recommended ERS Suppressor:	Dionex Anion Electrolytically Regenerated Suppressor AutoSuppression Recycle Mode
or MMS Suppressor:	Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

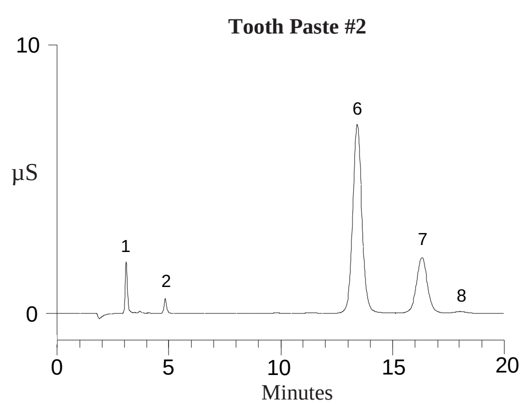
Figure 10 Analysis of Toothpaste

	Analyte	Standard mg/L
1.	Fluoride	5
2.	Chloride	10
3.	Nitrite	15
4.	Bromide	25
5.	Nitrate	25
6.	Phosphate	40
7.	Monofluorophosphate	50
8.	Sulfate	30

where:1 mg/L = 1 ppm



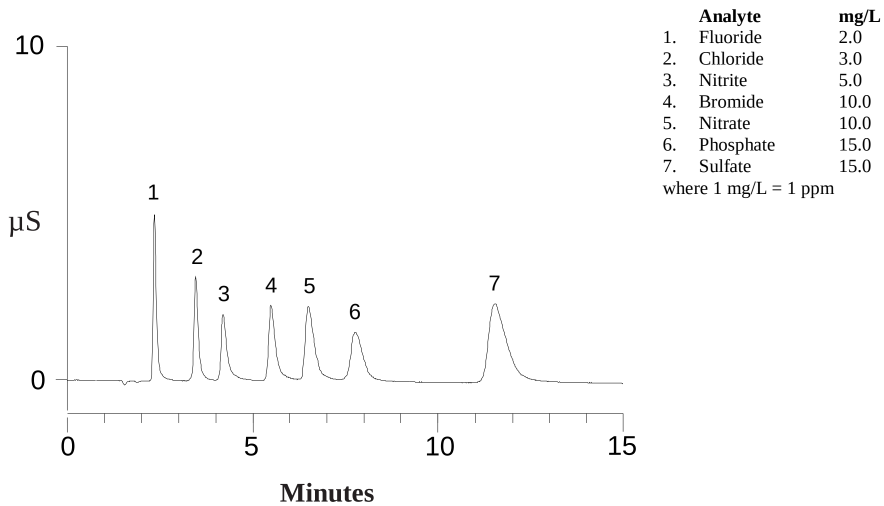




5.10 Isocratic Analysis of Seven Anions Using a Borate Eluent

Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Isocratic Eluent:	9 mM $Na_2B_4O_7$
Eluent Flow Rate:	1.5 mL/min
Suppressor:	Anion Self-Regenerating Suppressor Recycle Suppression Mode
Expected Background Conductivity:	3-5 µS

Figure 11 Isocratic Analysis of Seven Anions Using a Borate Eluent



5.11 Borate Gradient Analysis

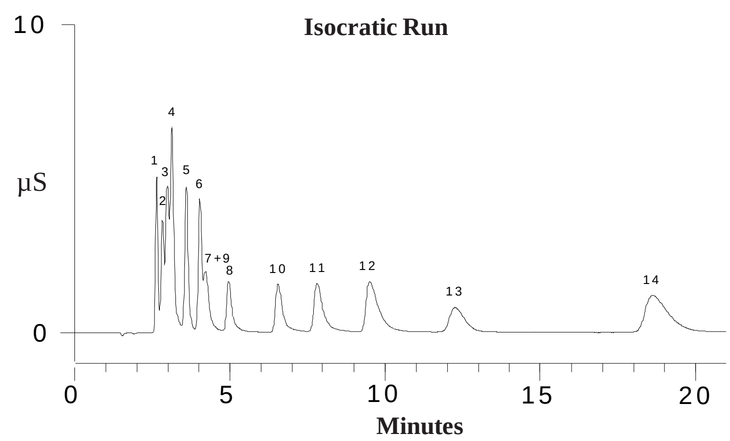
The following analyses demonstrate the advantages of gradient analysis. Borate gradient elution enhances the resolution of the weakly retained analytes, allows better positioning of carbonate, and accelerates the elution of the strongly retained analytes. The ASRS ULTRA should be used in the External Water Suppression mode when using borate eluents.

Anion Trap Column:	ATC-3 (4 mm)
Sample Loop Volume:	25 µL
Column:	Dionex IonPac AS14 + Dionex IonPac AG14
Isocratic Eluent:	7 mM $Na_2B_4O_7$
Gradient Eluents:	E1: 50 mM $Na_2B_4O_7$
	E2: Deionized water
Eluent Flow Rate:	1.5 mL/min
Suppressor:	Anion Self-Regenerating Suppressor
	AutoSuppression External Water Mode
Expected Background Conductivity:	3-5 µS

Figure 12 Borate Gradient Analysis

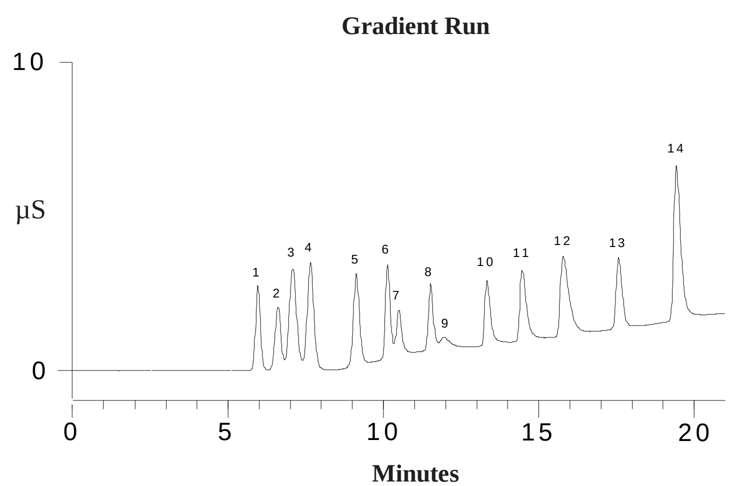
	Analyte	mg/L
1.	Fluoride	2.0
2.	Gylcolate	10.0
3.	Acetate	20.0
4.	Formate	10.0
5.	Chlorite	10.0
6.	Chloride	3.0
7.	Bromate	10.0
8.	Nitrite	5.0
9.	Carbonate	100
10.	Bromide	10.0
11.	Nitrate	10.0
12.	Chlorate	20.0
13.	Phosphate	15.0
14.	Sulfate	15.0

where 1 mg/L = 1 ppm



Gradient Conditions

Time	%E1	%E2	Comments
Init	4	96	Initial Eluent
0.00	4	96	Load Position
0.10	4	96	Inject
0.30	4	96	Load Position
6.00	4	96	Beginning of Gradient
18.00	30	70	End of Gradient
9.00	30	70	Isocratic Strong Eluent
19.10	4	96	Initial Eluent
24.00	4	96	5-Minute Equilibration Time



5.12 Clean-up After Humic Acid Samples

Solvent compatibility of the Dionex IonPac AS14 permits the use of organic solvents to effectively remove organic contaminants from the column. An AS14 column, after losing over 30% of its original capacity due to fouling with humic acid samples, can easily be restored to original performance by cleaning for 60 minutes with 80% tetrahydrofuran/20% 1.0 M NaCl, pH 2. Flow rate for cleanup is 0.6 mL/min.

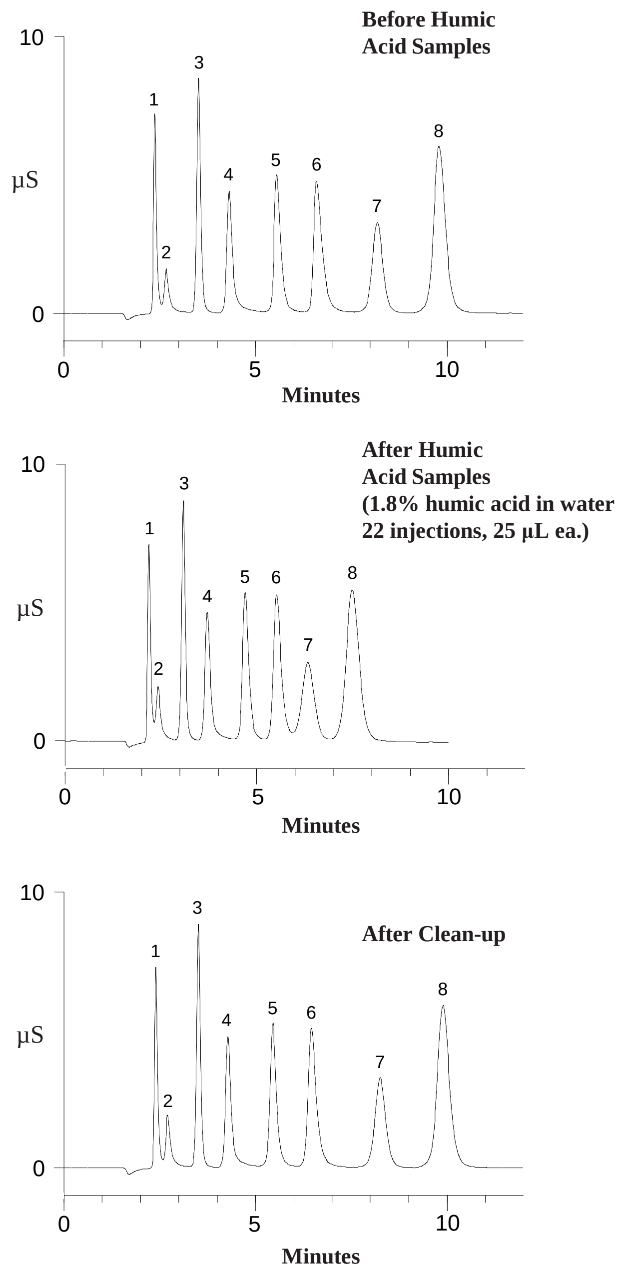
Figure 13 Clean-up after Humic Acid Samples

Column:	IonPac AS14
Eluent:	3.5 mM Sodium Carbonate/ 1.0 mM Sodium Bicarbonate
Flow Rate:	1.2 mL/min
Detection:	Suppressed Conductivity

	Peaks:	mg/L
1.	Fluoride	5
2.	Acetate	20
3.	Chloride	10
4.	Nitrite	15
5.	Bromide	25
6.	Nitrate	25
7.	Phosphate	40
8.	Sulfate	30

Clean-up Method
E1 - Deionized water
E2 - 1 M NaCl, pH 2
E3 - 100% Tetrahydrofuran
Flow Rate: 0.6 mL/min

Time	E1	E2	E3
0.0	75%	20%	5%
10.0	75	20	5
10.1	0	20	80
70.0	0	20	80
70.1	75	20	5
80.0	75	20	5



6. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac AS14 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. 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 6 AS14/AG14 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, Filter Eluents and Samples	Component Manual
High Background Conductivity	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.2, Column Care
	Contaminated Suppressor	Clean Suppressor	6.2.4, Component Manual
	Contaminated Hardware	Clean Component	Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Optimize method	6.3.1.A, Component Manual
	Column Headspace	Replace Column	6.3.1.B
Short Retention Times	Flow Rate Too fast	Recalibrate Pump	6.3.2.A
	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, 3.3.1, 3.3.2
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual
	Large System Void Volumes	Replumb System	6.3.3.D, Component Manual
Spurious Peaks	Sample Contaminated	Pretreat Samples	6.3.4.A, 6.3.4.B
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the Dionex IonPac AG14 (4 mm) Guard Column plus the AS14 (4 mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,000 psi. If the system pressure is higher than 2,000 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated.

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

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

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi. Continue adding system components (injection valve, column(s), suppressor, and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 7, "Typical AS14/AG14 Operating Back Pressures").

The Anion Self-Regenerating 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.

Table 7 Typical AS14/AG14 Operating Back Pressures

Column	Typical Back Pressure	Flow Rate
		psi (MPa) mL/min
AS14 4 mm Analytical	≤ 1,300 (8.96)	1.2
AG14 4 mm Guard	≤ 325 (2.24)	1.2
AS14 + AG14 4 mm columns	**≤ 1,625 (11.20)**	**1.2**
AS14 2 mm Analytical	≤ 1,300 (8.96)	0.3
AG14 2 mm Guard	≤ 325 (2.24)	0.3
AS14 + AG14 2 mm columns	**≤ 1,625 (11.20)**	**0.3**

6.1.2 Replacing Column Bed Support Assemblies

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.

Part	**Dionex IonPac AS14 4 mm Columns (P/N)**	**Dionex IonPac AS14 2 mm Columns (P/N)**
Analytical Column	046124	046129
Guard Column	046134	046138
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.

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

B. **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 CONDUCTIVITY	EXPECTED BACKGROUND
3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$	16–18 µS
2 mM $Na_2B_4O_7$ - 15 mM $Na_2B_4O_7$	2–5 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 Anion Trap Column, ATC-3

When doing gradient analysis, has the Anion Trap Column, the ATC-3 (2 mm) or the ATC-3 (4 mm) been installed correctly? If it has not, install one as directed in Section 3.5, Installing the Anion Trap Column, and watch the background conductivity. If the background conductivity is now low, this means that the ATC is trapping contaminants from the eluent. The eluents probably have too many impurities (see items A – C above).

If the ATC is already installed, remove it. Is the background conductivity still high? If the background conductivity decreases, the ATC is the source of the high background conductivity.

A. Disconnect either the ATC-3 (2 mm) or the ATC-3 (4 mm) from the injection valve and direct the outlet to waste.

B. Flush the ATC with 200 mL of 70 mM $Na_2B_4O_7$. Use a flow rate of 0.5 mL/min on a 2 mm system or a flow rate of 2.0 mL/min on a 4 mm system.

C. Equilibrate the ATC with the strongest eluent used during the gradient run. Use a flow rate of 0.5 mL/min on a 2 mm system or a flow rate of 2.0 mL/min on a 4 mm system.

D. If the problem persists, replace the ATC.

6.2.3 A Contaminated Guard or Analytical Column

Remove the Dionex IonPac AG14 Guard and AS14 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 AG14 at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in, “Column Cleanup” (see, “Column Care”).

6.2.4 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.

6.2.5 Contaminated Suppressor

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

6.3 Poor Peak Resolution

Poor peak resolution can 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

NOTE

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.

NOTE

If you are using a gradient pump to proportion the eluent 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, "Column Cleanup" (see, "Column Care"), for recommended column cleanup procedures.

NOTE

Possible sources of column contamination are impurities in the chemicals and 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, "Column Cleanup" in "Column Care").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. No water wash is necessary. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column.

NOTE

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

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 “Column Cleanup” (see, “Column Care”).

NOTE

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

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

7. Column Care

7.1 Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS14 columns is 4,000 psi (27.57 MPa).

7.2 Column Start-Up

The column is shipped using the eluent as the storage solution.

Prepare the eluent shown on the test chromatogram, install the column in the chromatography module, and test the column performance under the conditions described in the test chromatogram. 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.

7.3 Column Storage

For both short-term and long-term storage, use eluent for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution (eluent). Cap both ends securely using the plugs supplied with the column.

7.4 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, but 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 column bed packing 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.

7.4.1 Choosing the Appropriate Cleanup Solution

A. Hydrophilic ionic contamination of low valency

1. 10X eluent concentrate
(35 mM Na_2CO_3/10 mM $NaHCO_3$ = 10X concentrate of the production test eluent)

B. High valency hydrophobic ions

1. pH 2.0 200 mM NaCl in 80% acetonitrile

The acetonitrile solution is stored in a separate eluent bottle because acetonitrile slowly breaks down in acidic aqueous solutions. Prepare three bottles (E1, E2, and E3) with the following 500-mL solutions:

El: 100% acetonitrile

E2: 1 M NaCl using degassed Type I Reagent Grade Water with a specific resistance of 17.8 megohm-cm. Adjust the pH to 2.0 using HCl

E3: Degassed Type I Reagent Grade Water with a specific resistance of 17.8 megohm-cm

Wash the column with the following solutions:

For 10 minutes: 5% ACN (El) + 20% 1M NaCl (E2) + 75% deionized water (E3)

For 60 minutes: 80% ACN (El) + 20% 1M NaCl (E2) + 0% deionized water (E3)

C. Metal contamination

1. 0.2 M oxalic acid.
Iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

D. Humic acid clean-up

For clean-up of humic acid see section 5.12, "Clean-up After Humic Acid Samples".

E. Regardless of the cleanup solution chosen, use the following cleanup procedure in Section 7.4.2, "Column Cleanup Procedure", to clean the AG14 and AS14.

7.4.2 Column Cleanup Procedure

A. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in Section 7.4.1, "Choosing the Appropriate Cleanup Solution".

B. Disconnect the suppressor from the IonPac AS14 Analytical Column. 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. 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. 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.

C. Set the pump flow rate to 1.0 mL/min for an AS14 4 mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS14 2 mm Analytical or Guard Column.
D. Rinse the column for 15 minutes with deionized water before pumping the chosen cleanup solution over the column.
E. Pump the cleanup solution through the column for 60 minutes. Note in the case of Section 7.4.1, "Choosing the Appropriate Cleanup Solution", Part B, "High valency hydrophobic ions", a step gradient is used for column cleanup.
F. Rinse the column for 15 minutes with deionized water before pumping eluent over the column.
G. Equilibrate the column(s) with eluent before resuming normal operation for at least 30 minutes.
H. Reconnect the suppressor to the AS14 Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

Appendix B – System Configuration

Table B1 Configuration

CONFIGURATION	2 mm	4 mm
Eluent Flow Rate	0.3 mL/min	1.2 mL/min
AERS Suppressor	Dionex AERS 500 Carbonate (P/N 085028) *recommended* or Dionex AERS 500 (P/N 082541)	Dionex AERS 500 Carbonate (P/N 085029) *recommended* or Dionex AERS 500 (P/N 082540)
ACRS Suppressor	Dionex ACRS 500 (P/N 085091)	Dionex ACRS 500 (P/N 085090)
Injection Loop	2 – 15 µL	10 – 50 µL
System Void Volume	Eliminate switching valves, couplers, and use only the 2 mm Dionex GM-4 Mixer (P/N 049135).	Eliminate switching valves, couplers, and use the Dionex GM-2, GM-3, or recommended gradient mixers.
Pumps	Use the Dionex ICS 1100/1600/2100/5000+.	Use the Dionex ICS 1100/1600/2100/5000+.
Chromatographic Module	Use a thermally controlled column compartment such as the Dionex ICS-5000+ DC or ICS 1100/1600/2100 column heater.	Use a thermally controlled column compartment such as the Dionex ICS-5000+ DC or ICS 1100/1600/2100 column heater.

Table B2 Tubing Back Pressures

Color	Part Number	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure Psi/ft. at 1 mL/min	Back Pressure Psi/ft. at 0.25 mL/min	Back Pressure Psi/cm. at 1 mL/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