



PRODUCT MANUAL

IONPAC® AG14A GUARD COLUMN

(4 x 50 mm, P/N 056897)

(3 x 30 mm, P/N 056899)

IONPAC® AS14A ANALYTICAL COLUMN

(4 x 250 mm, P/N 056904)

(3 x 150 mm, P/N 056901)

QUICKSTART STEPS AND LINKS

Click blue text below to get started.

1. See [Section 4, "Operation"](#). Note operation precautions and chemical purity requirements.
2. See [Section 5.1, "Preparation of Eluent Stock Solution Concentrates"](#). Make the required stock and working solutions for eluents.
3. See ["Quality Assurance Reports"](#). Run the Production Test Chromatogram as a system check.
4. See [Section 5, "Example Applications"](#) for example applications.
5. See ["Column Care"](#) for column cleanup and long-term storage recommendations.

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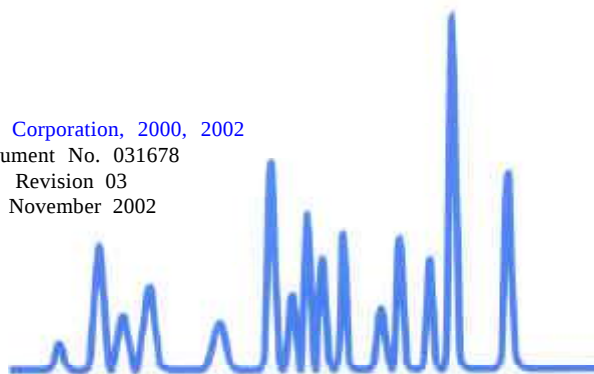


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SECTION 1 - INTRODUCTION TO IONPAC AS14A/AG14A CHROMATOGRAPHY

The IonPac® AS14A Analytical Column in combination with the AG14A Guard Column is designed for the analysis of fluoride and other inorganic anions. The selectivity of the IonPac AS14A Guard plus Analytical Column set has been designed to retain fluoride well out of the water dip (system dip) and to isocratically separate common anions. The AS14A is compatible with pH

0 - 14 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS14A can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor-ULTRA (ASRS-ULTRA). The IonPac AS14A has nominal efficiency for sulfate using standard operating conditions of at least 28,000 plates/meter for the 4 x 250 mm column and 40,000 plates/meter for the 3 x 150 mm column.

Table 1
IonPac AS14A/AG14A Packing Specifications

Column	Particle Diameter ^a μm	Substrate X-linking %	Column Capacity μeq/column	Functional Group	Hydrophobicity
AS14A 4 x 250 mm	7.0	55	120	Alkyl quaternary ammonium	Moderate
AG14A 4 x 50 mm	7.0	55	24	Alkyl quaternary ammonium	Moderate
AS14A 3 x 150 mm	5.0	55	40	Alkyl quaternary ammonium	Moderate
AG14A 3 x 30 mm	5.0	55	8	Alkyl quaternary ammonium	Moderate

^a macroporous (100 Å) divinylbenzene/ethylvinylbenzene polymer

Table 2
AS14A/AG14A Operating Parameters

Column	Typical Back Pressure psi (MPa) at 30°C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS14A 4-mm Analytical	< 1,500 (8.96)	1.0	3.0
AG14A 4-mm Guard	< 300 (1.68)	1.0	3.0
AS14A + AG14A 4-mm columns	< 1,800 (10.61)	1.0	3.0
AS14A 3-mm Analytical	< 1,500 (8.96)	0.5	1.50
AG14A 3-mm Guard	< 300 (1.68)	0.5	1.50
AS14A + AG14A 3-mm columns	<1,800 (10.61)	0.5	1.50

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of DIONEX instrumentation and columns through the DIONEX North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the DIONEX Offices listed in, "DIONEX Worldwide Offices."

SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

The proper configuration of 3-mm system injection volume, mass loading, system void volume and flow rate is based on the ratio of the 3-mm to 4-mm column cross-sectional area which is a factor of 1:2.

CONDITION	3-mm	4-mm
Eluent Flow Rate	Typically 0.50 mL/min	Typically 1.0 mL/min
Injection Loop	5-25 µL Use the Rheodyne Microinjection Valve, Model No. 9126 (DIONEX P/N 044697) for full loop injections <15 µL.	10-50 µL
SRS Suppressor	ASRS-ULTRA (2-mm) (P/N 053947)	ASRS-ULTRA (4-mm) (P/N 053946)
MMS Suppressors	AMMS III (2-mm) (P/N 056751)	AMMS III (4-mm) (P/N 056750)
MMS Regenerant Flow Rate	25 – 100% of 4-mm System	Typically 10 – 15 mL/min. See Suppressor Manual
AES Suppressors	AAES (P/N 056116)	AAES (P/N 056116)
NOTE Do not run a suppressor over 40°C; if applications require higher temperatures, place suppressor outside oven.		
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (2-mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2, GM-3 or recommended gradient mixers.
Pumps	Use the GS50/GP50/GP40/IP20/IP25 in Microbore or Standard Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. No External Gradient Mixer is required for GS50/GP50/GP40 Pump when performing gradient analysis The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography	Use the GP40/GP50/IP20/IP25 in Standard-Bore Configuration. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP40/GP50/IP20/IP25 and the DX-300 HPLC Pump.

CONDITION	3-mm	4-mm
Detectors	AD20/AD25 Cell (6-mm, 7.5 μL, P/N 046423) or an AD20/AD25 cell (10-mm, 9 μL) P/N 049393 VDM-2 Cell (3-mm, 2.0 μL, P/N 043120) or VDM-2 cell (6-mm, 10 μL) P/N 043113 CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 50-75 psi back pressure.	AD20/AD25 Cell (10-mm, 9 μL, P/N 049393) VDM-2 Cell (6-mm, 10 μL) P/N 043113 CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Either the TS-1 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 50-75 psi back pressure.

Table 3
Tubing Back Pressures

Tubing ID in	H ₂ O Back Pressure Psi/ft at 1 mL/min
0.005	111.4
0.007	29.0
0.010	7.0
0.012	3.4

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 3-mm Operation

The IonPac AS14A 3-mm Guard and Analytical Columns are designed to be run on the following 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 GS50/GP50/GP40/IP25, Gradient Pump Module (GPM-2) or an Advanced Gradient Pump (AGP) with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a Microbore GS50/GP50/GP40 or Advanced Gradient Pump (1/16" pistons) must be employed.

3.1.2 System Requirements for 4-mm Operation

The IonPac AS14A 4-mm Guard and Analytical Columns are designed to be 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 GS50/GP50/GP40 or a Gradient Pump Module (GPM-2) or an Advanced Gradient Pump (AGP) with standard pump heads.

3.1.3 System Void Volume

When using 3-mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/2 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) ID PEEK tubing. 0.010" ID PEEK tubing (P/N 042260) or 0.012" Tefzel tubing (see, "DIONEX Product Selection Guide") 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. If you need assistance in properly configuring your system contact the DIONEX Office nearest you (see, "DIONEX Worldwide Offices").

3.2 The Sample Concentrator

The Low Pressure Trace Anion Concentrator Column (TAC-LP1, P/N 046026), the Trace Anion Concentrator Column (TAC-2, P/N 043101), the Anion MicroConcentrator, AMC-1, (P/N 051760) or the IonPac AG14A Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of the TAC-LP1, the TAC-2, the AMC-1, or the AG14A Guard Column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" all anionic analyte species onto the TAC-LP1, TAC-2, AMC-1 or the AG14A leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage to the analytical chemist of the TAC-LP1, the TAC-2, the AMC-1, or the AG14A in these applications is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

For a detailed discussion of anion concentration techniques, refer to Section 3, "Operation," of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column Product Manual (Document No. 034972).

3.3 The Injection Loop

Table 4
Smallest Injectable Volumes (μL)

Valve Type	Using 0.012" ID Tefzel Tubing	Using 0.007" ID Tefzel Tubing	Using 0.010" ID PEEK Tubing	Using 0.005" ID PEEK Tubing
DIONEX BF2 Valve (8 μL Internal Volume) (10 cm Loop)	15.2	10.5	13.1	9.2
DIONEX MicroInject Valve (10.5 μL Internal Volume) (14 cm Loop)	20.5	14.0	17.6	12.2
Rheodyne Microinjection Valve Model 9126 (0.8 μL Internal Volume) (10 cm Loop)	8.0	3.3	5.9	2.0

3.3.1 The 3-mm System Injection Loop, 5 - 25 μL

For most applications on a 3-mm analytical system, a 5 - 25 μL injection loop is sufficient. DIONEX recommends that a 5.0 μL injection loop be used to avoid overloading the AS14A 3-mm Analytical Column. Generally, you should not inject more than 5.0 nanomoles (100 - 200 ppm) of any one analyte onto a 3-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. The AS14A 3-mm requires a microbore HPLC system configuration. Install an injection loop one-half or less ($< 25 \mu\text{L}$) of the loop volume used with a 4-mm analytical system (Section 2, "Comparison of 3-mm and 4-mm 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 will be sufficient. DIONEX recommends that a 10 μL injection loop be used to avoid overloading the AS14A 4-mm Analytical Column. Generally, do 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 IonPac AG14A Guard Column

An IonPac AG14A Guard Column is normally used with the IonPac AS14A 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 AG14A Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS14A Analytical Column.

3.5 Installing the ATC-3 for Eluent Step Change or Gradient Operation

An IonPac Anion Trap Column (ATC-3 (2-mm), P/N 059661 or ATC-3 (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 ATC-3 is filled with high capacity anion exchange resin which helps to minimize the baseline shift caused by increasing carbonate contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis.

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

- A. Remove the Gradient Mixer** installed between the gradient pump pressure transducer and the injection valve.
- B. Connect the gradient pump directly to the ATC-3.** Connect a waste line to the ATC-3 outlet and direct the line to a waste container.
- C. Flush the 4-mm ATC-3 with 100 mL of 2.0 M NaOH or 50 mL for the 2-mm ATC-3 Column.**
- D. Pump 20 mL of eluent through the 4-mm ATC-3 or 10 mL for the 2-mm ATC-3 Column.**
- E. Reconnect the ATC-3 after flushing it with eluent.** Connect the ATC-3 to the eluent line that is connected to the injection valve.

The background conductivity of your system should be between 1.5 μ S and 2.5 μ S when 0.75 mM NaOH is being pumped through the chromatographic system. The baseline shift should be no greater than 5 μ S during a gradient eluent concentration ramp from 0 to 80 mM NaOH. If the baseline shifts are greater than 5 μ S, the ATC-3 should be cleaned using steps B - E above.

At the end of each operating day, the ATC-3 should be flushed to remove any impurities that may have accumulated on it.

Under normal operating conditions, the ATC-3 column should be regenerated at the end of each operational day to remove any contaminants that may have collected on it, including carbonate. The daily regeneration of the ATC-3 column ensures that the IC system is systematically equilibrated for the most reproducible determinations of those anions being eluted by the weak eluents.

See the conditioning procedure above for regeneration of ATC-3 columns. For detailed information refer to the ATC-3 Product Manual (Document No. 032697).

3.6 Eluent Storage

IonPac AS14A 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).

3.7 Anion Self-Regenerating Suppressor Requirements

An 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 ASRS-ULTRA modes of operation.

CAUTION

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

If you are installing an IonPac AS14A 4-mm Analytical Column, use an ASRS-ULTRA (4-mm, P/N 053946).

If you are installing an IonPac AS14A 3-mm Analytical Column, use an ASRS-ULTRA (2-mm, P/N 053947).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see Document No. 034650, the “Product Manual for the Anion Self-Regenerating Suppressor-ULTRA, the ASRS-ULTRA (4-mm) and the ASRS-ULTRA (2-mm).”

3.8 Atlas Anion Electrolytic Suppressor Requirements

An Atlas Anion Electrolytic Suppressor (AAES) may be used instead of an ASRS-ULTRA for applications that require suppressed conductivity detection. The AAES (P/N 056116) can be used for 3-mm and 4-mm IonPac AS14A applications using eluents up to 25 µeq/min.

For detailed information on the operation of the Atlas Anion Electrolytic Suppressor, see Document No. 031770, the “Product Manual for the Atlas Anion Electrolytic Suppressor.”

3.9 Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS III) may be used instead of an ASRS-ULTRA for applications that require suppressed conductivity detection. Use an AMMS III (4-mm) (P/N 056750) with the IonPac AS14A 4-mm Analytical Column. It is compatible with all solvents and concentrations with which the systems and columns are compatible. For 3-mm operation, use the AMMS III (2-mm) (P/N 056751).

For detailed information on the operation of the Anion MicroMembrane Suppressor, see Document No. 031727, the “Product Manual for the Anion MicroMembrane Suppressor III, the AMMS III.”

3.10 Using Displacement Chemical Regeneration (DCR) with the Chemical Suppression Mode

DIONEX recommends using the Displacement Chemical Regeneration (DCR) Mode for chemical suppression using sulfuric acid and the Anion MicroMembrane Suppressor (AMMS III). See the DCR kit manual, Document P/N 031664, for details.

WARNING

Use proper safety precautions in handling acids and bases.

3.11 Using AutoRegen with the ASRS-ULTRA or the AMMS in the Chemical Suppression Mode

To save regenerant preparation time and reduce regenerant consumption and waste, DIONEX recommends using an AutoRegen® Accessory (P/N 039594). 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 AutoRegen Regenerant Cartridges, see the “Product Manual for the AutoRegen Regenerant Cartridge Refills” (Document No. 032852).

3.12 Detector Requirements

See Section 2, “Comparison of 3-mm and 4-mm Ion Chromatography Systems,” for 3-mm and 4-mm system detector, cell and thermal stabilizer requirements.

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	3-mm: 5.0 μ L Loop + 0.8 μ L Injection valve dead volume 4-mm: 10 μ L Loop + 0.8 μ L Injection valve dead volume
Column:	3-mm: AS14A 3-mm Analytical Column + AG14A 3-mm Guard Column 4-mm: AS14A 4-mm Analytical Column + AG14A 4-mm Guard Column
Eluent:	8.0 mM Na_2CO_3 /1.0 mM NaHCO_3
Eluent Flow Rate:	3-mm: 0.5 mL/min 4-mm: 1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm or 4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	24 - 28 μ S
Storage Solution:	Eluent

4.2 IonPac AS14A Operation Precautions

CAUTIONS
Filter and Degas Eluents
Filter Samples
Eluent pH between 0 and 14
Sample pH between 0 and 14
1.50 mL/min Maximum Flow Rate for 3-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. DIONEX 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 ionized impurities, organics, microorganisms and particulate matter larger than 0.2 μ m. 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 IonPac AS14A 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 DIONEX, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and 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 IonPac AS14A columns is 4,000 psi (27.57 MPa).

The AS14A 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 IonPac AS14A 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 Self-Regenerating Anion Suppressor (ASRS-ULTRA) 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. 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.

CAUTION

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.

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.

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.

CAUTION

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

4.5 Regenerant Preparation for the AMMS III

The Anion MicroMembrane Suppressor III (AMMS III) requires the use of a regenerant solution. If you are using the AMMS III instead of the Anion Self-Regenerating Suppressor-ULTRA (ASRS-ULTRA) see Document No. 031727, the "Product Manual for the Anion MicroMembrane Suppressor III, the AMMS III."

4.6 Sample Concentration

The Low Pressure Trace Anion Concentrator (TAC-LP1), the Trace Anion Concentrator (TAC-2), the Anion MicroConcentrator (AMC-1), or the IonPac AG14A Guard Column can be used for trace anion concentration work required in high purity water analysis. 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. 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 refer to Section 3, "Operation," of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column Product Manual (Document No. 034972). These techniques can also be applied to either the TAC-2, the AMC-1, or the AG14A.

SECTION 5 - EXAMPLE APPLICATIONS

The chromatograms in this section were obtained using columns that reproduced the Production Test Chromatogram (see Section 5.3, "Production Test Chromatogram") on optimized Ion Chromatographs (see Section 3, "Installation"). 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.

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. 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 4.6, "Sample Concentration").

5.1 Preparation of Eluent Stock Solution Concentrates

- A. AS14A Sodium Carbonate/Bicarbonate Eluent Concentrate
(0.8 M Na_2CO_3 /0.1 M NaHCO_3)

Order DIONEX P/N 056937

or

Thoroughly dissolve 84.8 g of sodium carbonate (MW 106.00 g/mole) plus 8.4 g sodium bicarbonate (MW 84.00 g/mole) in 700 mL degassed, 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

Order DIONEX 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.

- C. 0.5 M Sodium Bicarbonate (NaHCO_3) Concentrate

Order DIONEX 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.

5.2 Sodium Carbonate/Sodium Bicarbonate Eluent Preparation

- A. Using AS14A Sodium Carbonate/Bicarbonate Eluent Concentrate
(0.8 M Na_2CO_3 /0.1 M NaHCO_3)

Pipette 10.0 mL of the AS14A Combined Carbonate/Bicarbonate Eluent Concentrate, (100X concentrate = 0.8 M Na_2CO_3 /0.1 M NaHCO_3 , P/N 056937) into a 1 L volumetric flask. Use degassed, deionized water with a specific resistance of 18.2 megohm-cm to dilute to a final volume of 1,000 mL.

or

B. Using 0.5 M Na₂CO₃ and 0.5 M NaHCO₃ Concentrates

Prepare the eluent by pipetting 16.0 mL of 0.5 M Na₂CO₃ plus 2.0 mL of 0.5 M NaHCO₃ 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.

5.3 IonPac AS14A Production Test Chromatograms

Isocratic elution of anions on the IonPac AS14A 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 IonPac AS14A Analytical Column should always be used with the IonPac AG14A Guard Column. To guarantee that all IonPac AS14A Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Volume:	3-mm: 5.0 μ L Loop + 0.8 μ L Injection valve dead volume 4-mm: 10 μ L Loop + 0.8 μ L Injection valve dead volume		
Column:	See Chromatogram		
Eluent:	8.0 mM Na_2CO_3 /1.0 mM NaHCO_3	Analyte	mg/L
Temperature:	30°C	1. Fluoride	5.0
Eluent Flow Rate:	0.5 mL/min (3-mm) 1.0 mL/min (4-mm)	2. Acetate	20.0
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm or 4-mm) AutoSuppression® Recycle Mode	3. Chloride	10.0
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm)	4. Nitrite	15.0
MMS Regenerant:	50 mN H_2SO_4	5. Bromide	25.0
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES	6. Nitrate	25.0
Expected Background Conductivity:	24 - 28 μ S	7. Phosphate	40.0
Storage Solution:	Eluent	8. Sulfate	30.0
		where 1 ppm = 1mg/L	

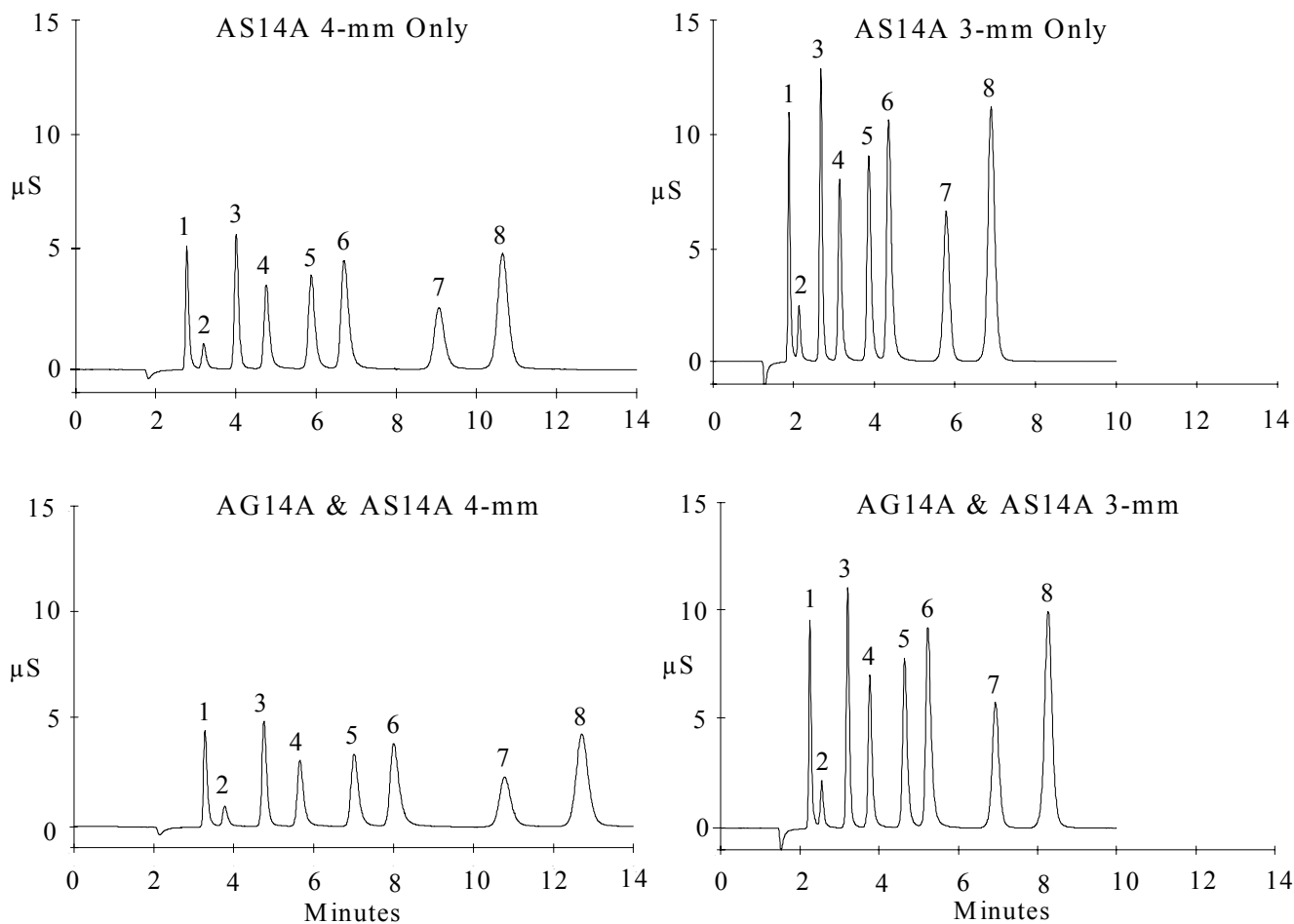


Figure 1
IonPac AS14A Production Test Chromatograms

5.4 Fast Anion Separation of Common Anions Using a Higher Flow Rate on the AS14A 3-mm Column

The following chromatogram shows the fast separation of 8 common anions using a standard carbonate/bicarbonate eluent at 0.8 mL/min and 30°C . Notice that the common anion analysis can be done in less than 6 minutes while still providing separation of fluoride from the system dip and from acetate.

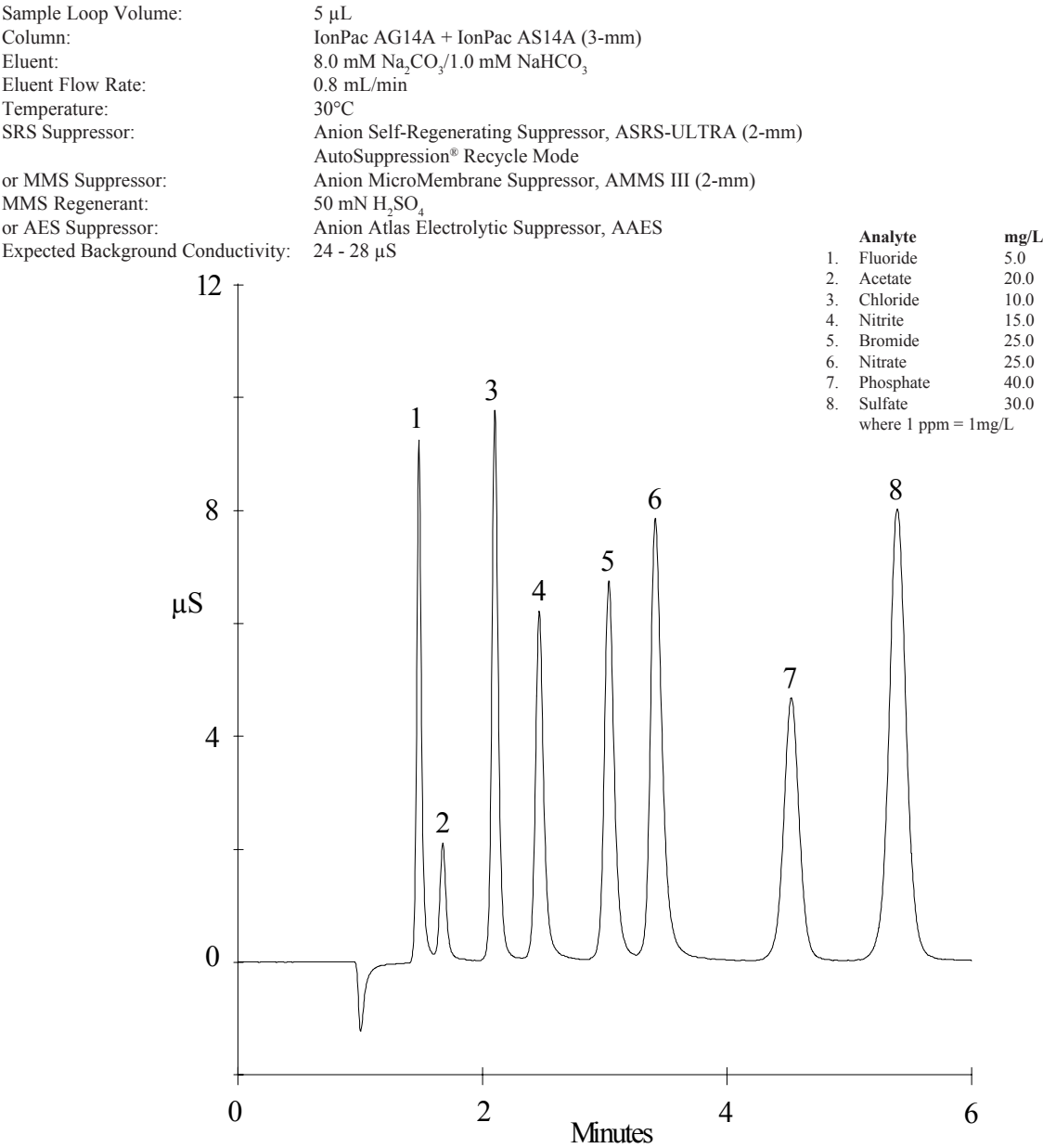


Figure 2
 Fast Anion Separation of Common Anions Using a Higher Flow Rate
 on the AS14A 3-mm Column

5.5 The Effect of Temperature on the Separation of 14 Anions Using the AS14A 4-mm Column

The following chromatograms show the elution order of glycolate, formate, chlorite, bromate, chlorate, and oxalate along with the 8 common anions using a standard carbonate/bicarbonate eluent at room temperature and at 30°C. Notice that chlorate and phosphate are well separated with the standard eluent at 30°C.

Sample Loop Volume: 10 µL
 Column: IonPac AG14A + IonPac AS14A (4-mm)
 Eluent: 8.0 mM Na₂CO₃/1.0 mM NaHCO₃
 Eluent Flow Rate: 1.0 mL/min
 Temperature: See Chromatogram
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS-ULTRA (4-mm)
 AutoSuppression® Recycle Mode
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)
 MMS Regenerant: 50 mN H₂SO₄
 or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES
 Expected Background Conductivity: 24 - 28 µS

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
14. Oxalate	30.0

where 1 mg/L = 1 ppm

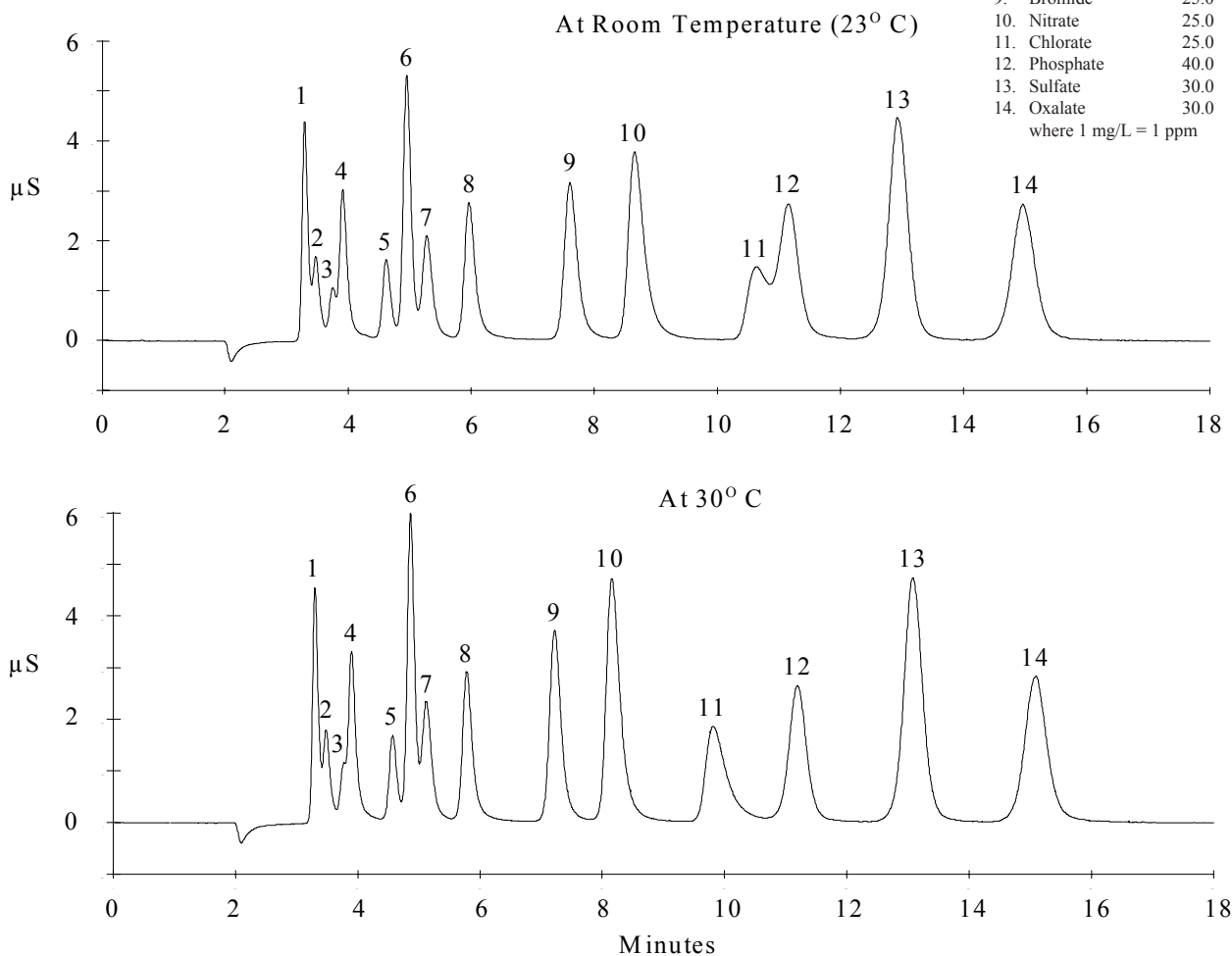


Figure 3
The Effect of Temperature on the Separation of 14 Anions Using the AS14A 4-mm Column

5.6 The Effect of Temperature on the Separation of 14 Anions Using the AS14A 3-mm Column

The following chromatograms show the elution order of glycolate, formate, chlorite, bromate, chlorate, and oxalate along with the 8 common anions using a standard carbonate/bicarbonate eluent at room temperature and at 30°C. Notice that chlorate and phosphate are well separated with the standard eluent at 30°C.

Sample Loop Volume: 5 µL
 Column: IonPac AG14A + IonPac AS14A (3-mm)
 Eluent: 8.0 mM Na₂CO₃/1.0 mM NaHCO₃
 Eluent Flow Rate: 0.5 mL/min
 Temperature: See Chromatogram
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm)
 AutoSuppression® Recycle Mode
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (2-mm)
 MMS Regenerant: 50 mN H₂SO₄
 or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES
 Expected Background Conductivity: 24 - 28 µS

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
14. Oxalate	30.0

where 1 mg/L = 1 ppm

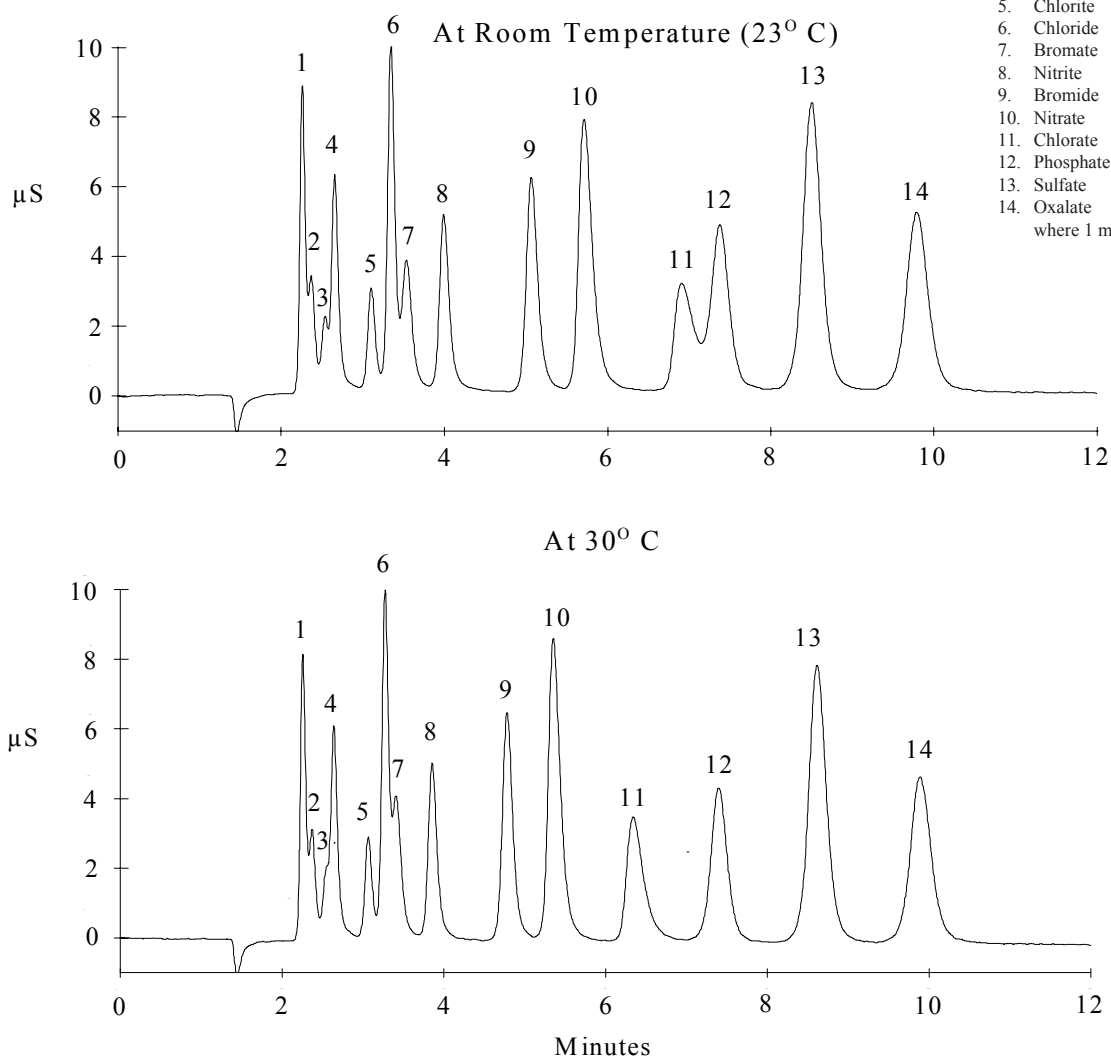


Figure 4
The Effect of Temperature on the Separation of 14 Anions Using the AS14A 3-mm Column

5.7 14 Anion Selectivity Using an Optimized Eluent at Room Temperature on the AS14A 3-mm Column

The following chromatograms show the elution order of glycolate, formate, chlorite, bromate, chlorate, and oxalate along with the 8 common anions using a modified carbonate/bicarbonate eluent at room temperature (23°C). Notice that chlorate and phosphate can be well separated using a 6 mM carbonate / 1 mM bicarbonate eluent.

Sample Loop Volume: 5 µL
 Column: IonPac AG14A + IonPac AS14A (3-mm)
 Eluent: See Chromatogram
 Eluent Flow Rate: 0.5 mL/min
 Temperature: Room Temperature (23°C)
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm)
 AutoSuppression® Recycle Mode
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (2-mm)
 MMS Regenerant: 50 mN H₂SO₄
 or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES
 Expected Background Conductivity: 22 - 28 µS

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
14. Oxalate	30.0

where 1 mg/L = 1 ppm

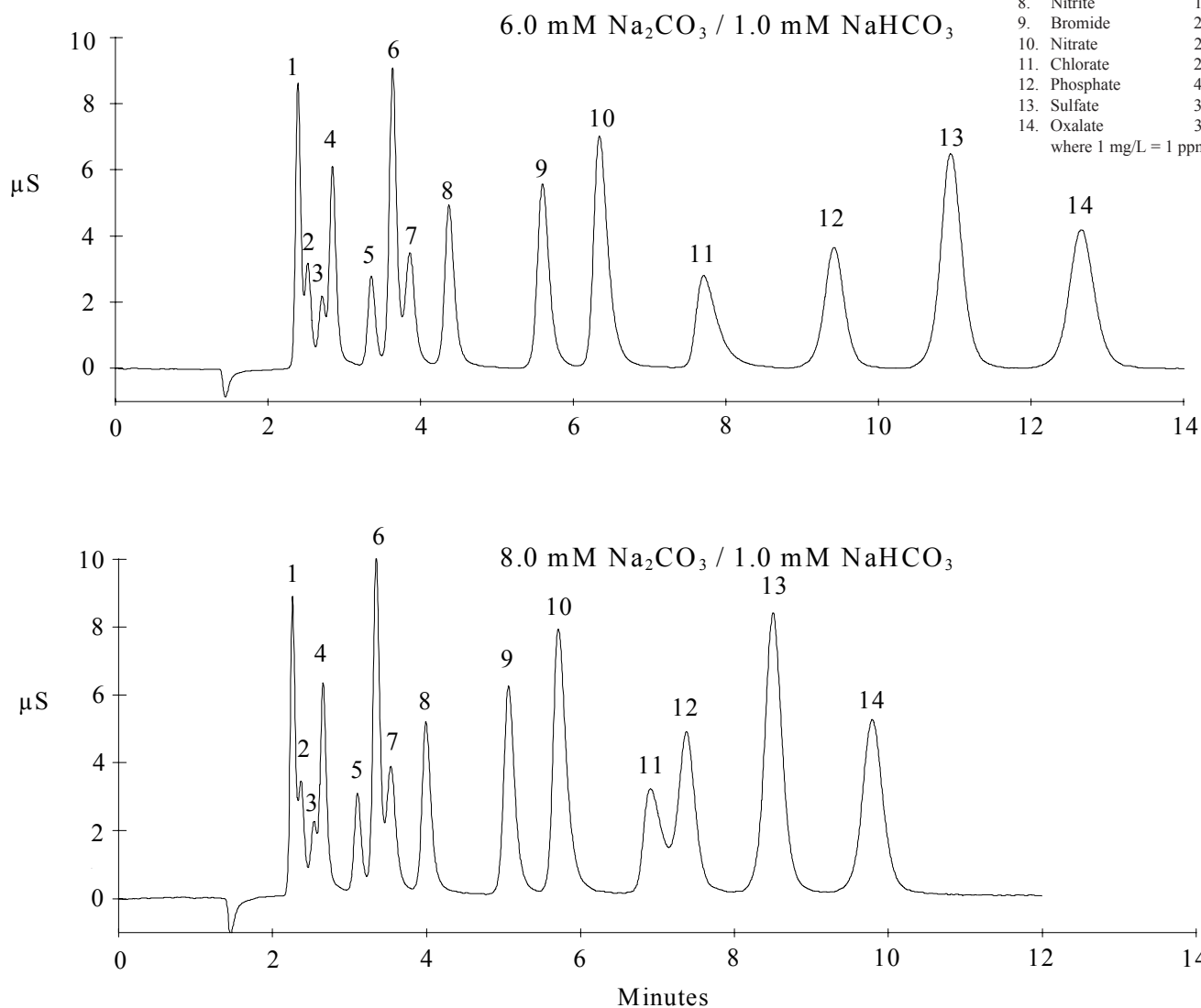


Figure 5
14 Anion Selectivity Using an Optimized Eluent at Room Temperature
on the AS14A 3-mm Column

5.8 Increasing the Sample Loop Size to Increase Sensitivity with the AS14A 4-mm Column

The following chromatograms demonstrate the effect of loop size on the quantification of fluoride and low levels of bromide and phosphate using the AS14A 4-mm column.

Sample Loop	
Volume:	See Chromatograms
Column:	IonPac AG14A + IonPac AS14A (4-mm)
Eluent:	8.0 mM Na ₂ CO ₃ / 1.0 mM NaHCO ₃
Eluent Flow Rate:	1.0 mL/min
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (4-mm) AutoSuppression® Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4-mm)
MMS Regenerant:	50 mN H ₂ SO ₄
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	24 - 28 µS

- Analyte**
1. Fluoride
 2. Acetate
 3. Chloride
 4. Bromide
 5. Nitrate
 6. Chlorate
 7. Phosphate
 8. Sulfate

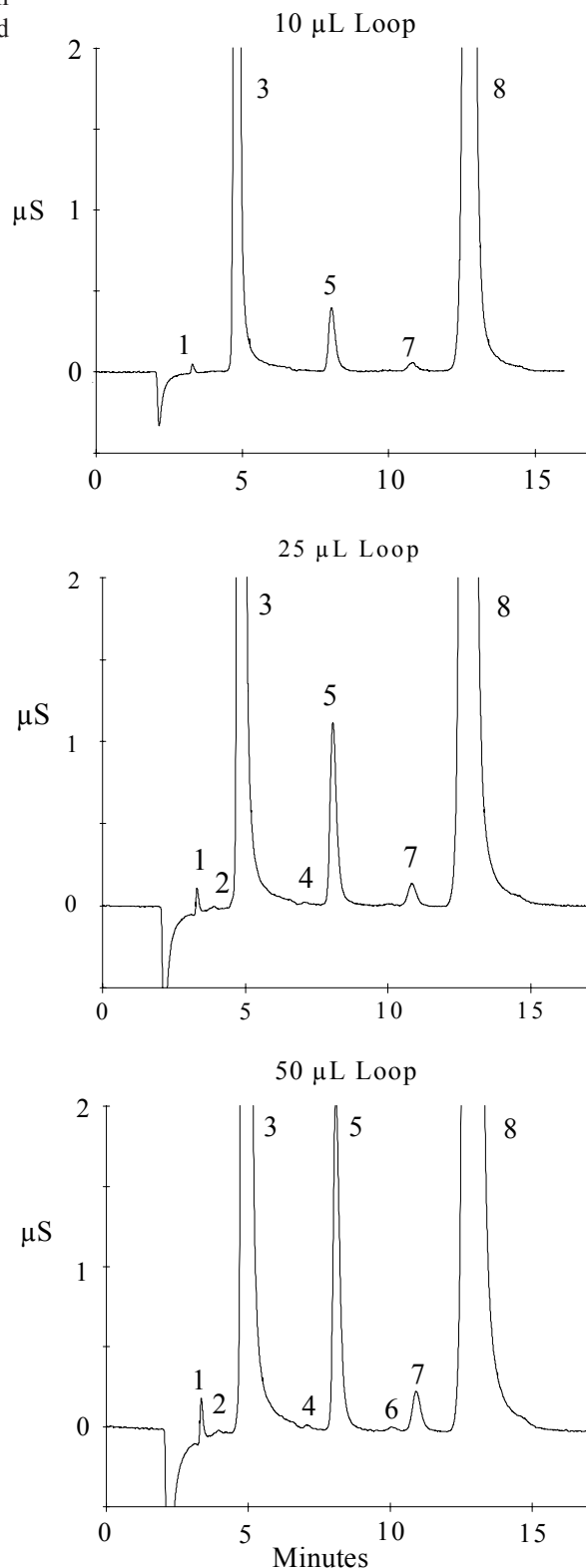


Figure 6
Increasing the Sample Loop Size to Increase Sensitivity
with the AS14A 4-mm Column

5.9 Increasing the Sample Loop Size to Increase Sensitivity with the AS14A 3-mm Column

The following chromatograms demonstrate the effect of loop size on the quantification of fluoride and low levels of bromide and phosphate using the AS14A 3-mm column.

Sample Loop	See Chromatograms
Volume:	IonPac AG14A +
Column:	IonPac AS14A (3-mm)
Eluent:	8.0 mM Na ₂ CO ₃ / 1.0 mM NaHCO ₃
Eluent	
Flow Rate:	0.5 mL/min
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm)
or MMS Suppressor:	AutoSuppression® Recycle Mode Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H ₂ SO ₄
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background	
Conductivity:	24 - 28 µS

Analyte
1. Fluoride
2. Acetate
3. Chloride
4. Bromide
5. Nitrate
6. Chlorate
7. Phosphate
8. Sulfate

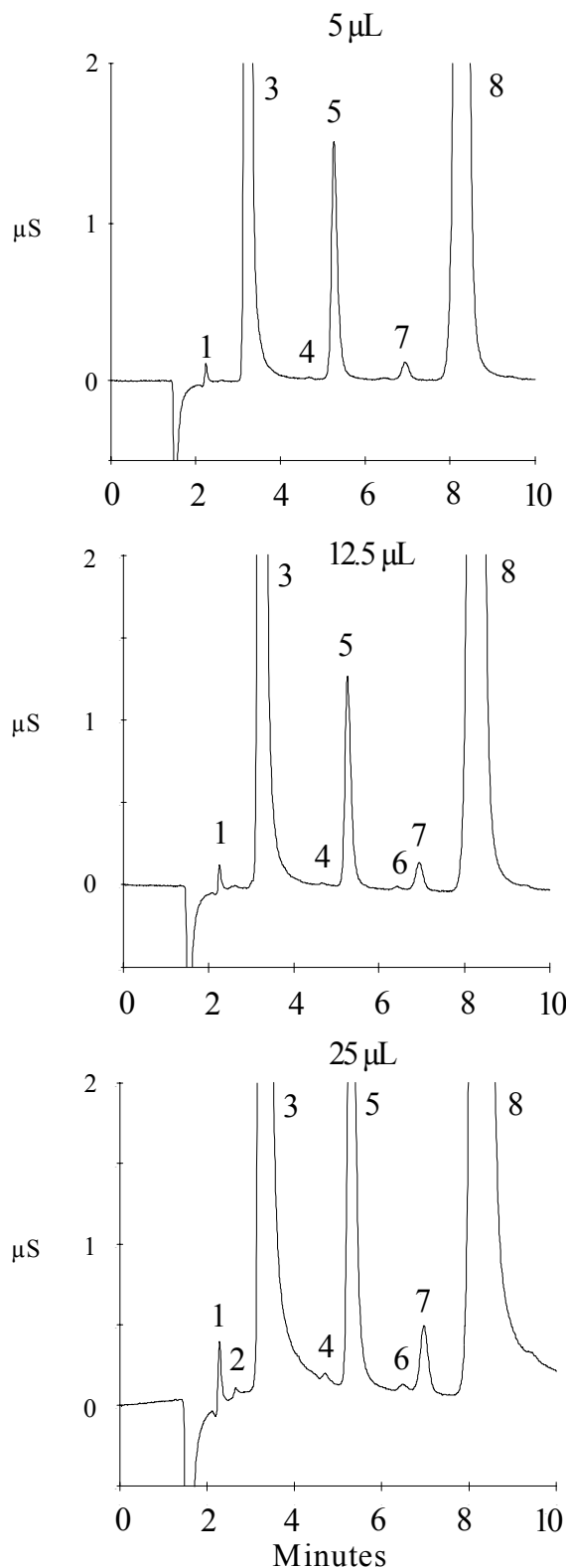


Figure 7
Increasing the Sample Loop Size to Increase Sensitivity
with the AS14A 3-mm Column

5.10 Analysis of Waste Water Using the AS14A 3-mm Column

These chromatograms demonstrate the analyses of waste water collected at various process points from a chemical plant.

Sample Loop: 5 μ L
 Volume: IonPac AG14A +
 Column: IonPac AS14A (3-mm)
 Eluent: 8.0 mM Na_2CO_3 / 1.0 mM NaHCO_3
 Eluent Flow Rate: 0.5 mL/min
 Temperature: 30°C
 SRS Suppressor: Anion Self-Regenerating Suppressor,
 ASRS-ULTRA (2-mm)
 or MMS Suppressor: AutoSuppression® Recycle Mode
 Anion MicroMembrane Suppressor,
 AMMS III (2-mm)
 MMS Regenerant: 50 mN H_2SO_4
 or AES Suppressor: Anion Atlas Electrolytic Suppressor,
 AAES
 Expected Background Conductivity: 24 - 28 μ S

- Analyte**
1. Glycolate
 2. Acetate
 3. Formate
 4. Chloride
 5. Phosphate
 6. Sulfate

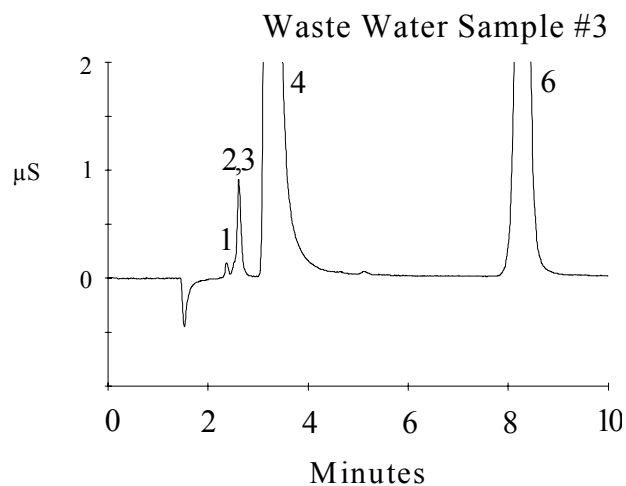
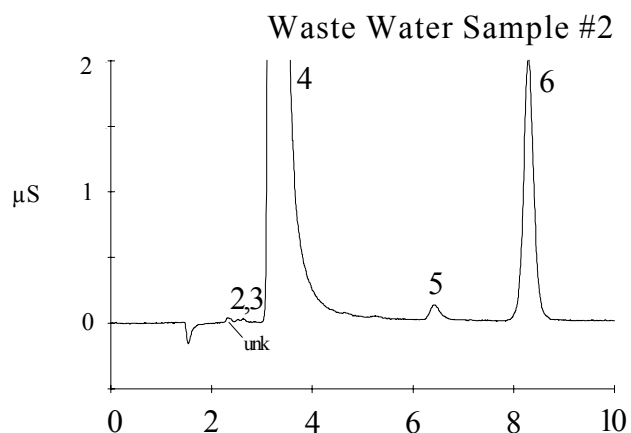
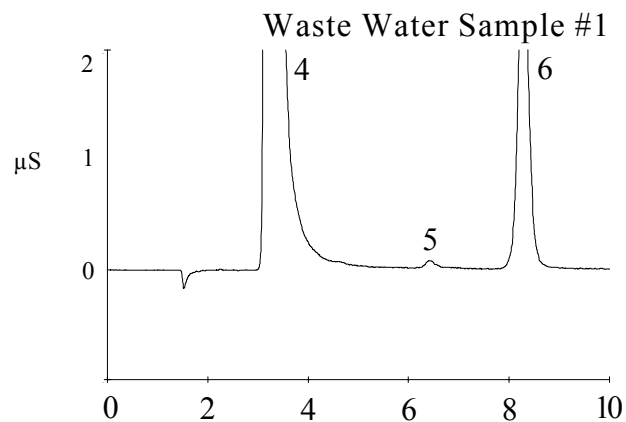


Figure 8
 Analysis of Waste Water Using the AS14A 3-mm Column

5.11 Analysis of Carbonated Water Using the AS14A 4-mm Column

Sonication can be used to remove carbonate from carbonated water samples. 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: IonPac AG14A + IonPac AS14A (4-mm)
Eluent: 8.0 mM Na_2CO_3 / 1.0 mM NaHCO_3
Eluent Flow Rate: 1.0 mL/min
Temperature: 30°C
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS-ULTRA (4-mm)
AutoSuppression® Recycle Mode
or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)
MMS Regenerant: 50 mN H_2SO_4
or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity: 24 - 28 μS

Analyte
1. Unknown
2. Chloride
3. Bromide
4. Nitrate
5. Sulfate

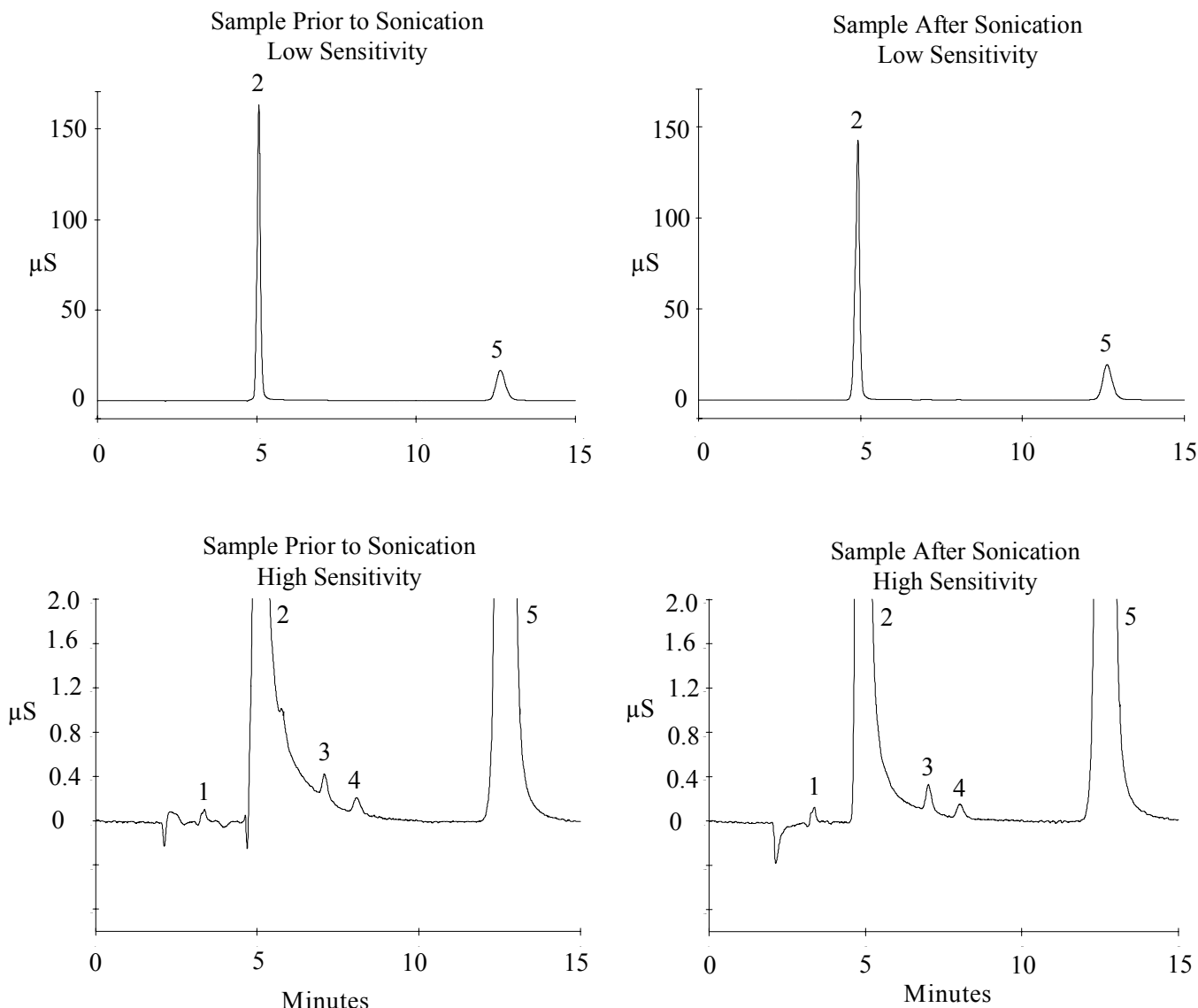


Figure 9
Analysis of Carbonated Water Using the AS14A 4-mm Column

5.12 Analysis of Toothpaste Using the AS14A 3-mm Column

The excellent retention of fluoride on the IonPac AS14A makes it ideal for the determination of fluoride and monofluorophosphate in dental care products. This chromatogram illustrates the separation of monofluorophosphate from phosphate and sulfate which can be present in toothpaste. Notice that this application can be done in less than 10 minutes using the AS14A 3-mm.

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- μ m syringe filter and then through a 0.20- μ m syringe filter before injection.

Sample Loop
Volume: 5 μ L
Column: IonPac AG14A +
IonPac AS14A (3-mm)
Eluent: 8.0 mM Na₂CO₃ / 1.0 mM NaHCO₃
Eluent Flow Rate: 0.5 mL/min
SRS Suppressor: Anion Self-Regenerating Suppressor,
ASRS-ULTRA (2-mm)
or MMS Suppressor: AutoSuppression® Recycle Mode
Anion MicroMembrane Suppressor,
AMMS III (2-mm)
MMS Regenerant: 50 mM H₂SO₄
or AES Suppressor: Anion Atlas Electrolytic Suppressor,
AAES
Expected Background Conductivity: 24 - 28 μ S

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

where 1 ppm = 1 mg/L

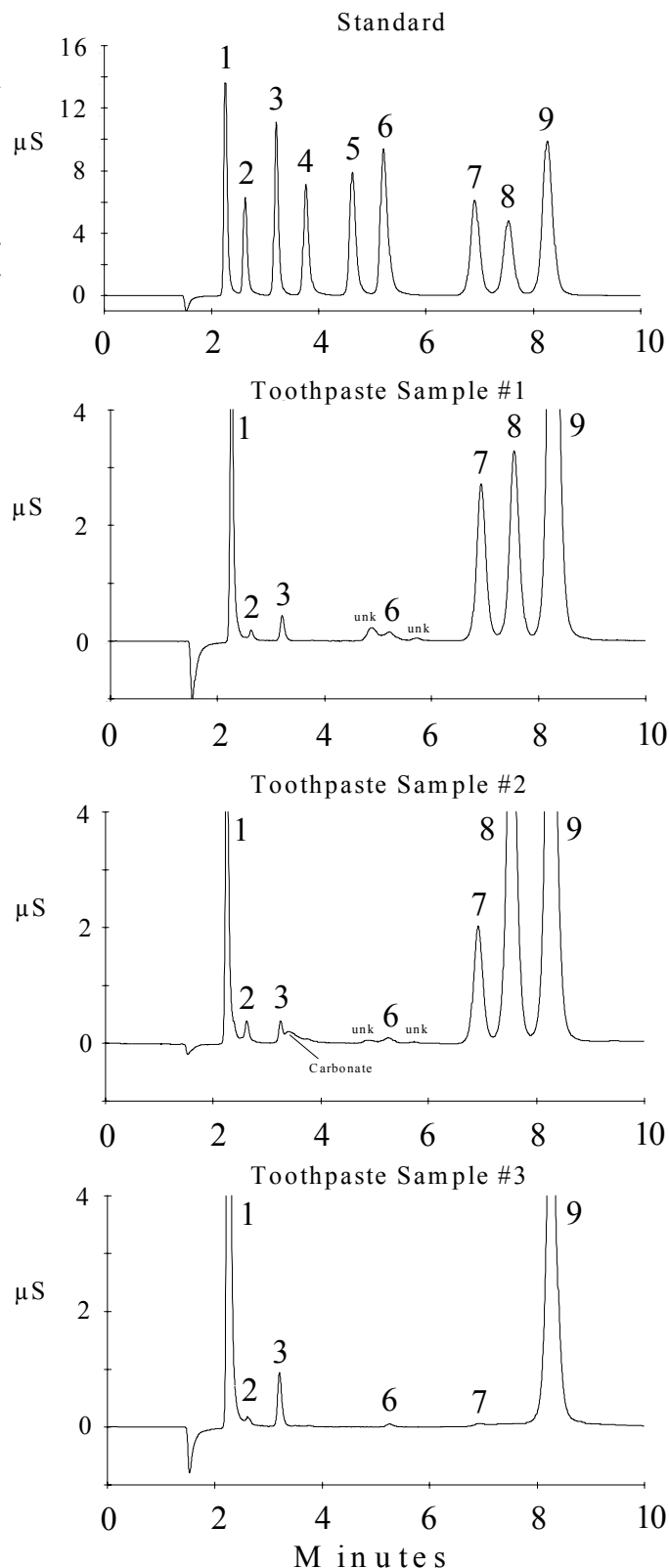


Figure 10
Analysis of Toothpaste Using the AS14A 3-mm Column

5.13 Separation of Weak Organic Acids, Oxyhalides and Common Anions Using a Sodium Hydroxide Gradient on the AS14A 4-mm Column

In the past Dionex has recommended a borate eluent system for analysis of organic acids when gradients were required for carbonate-based columns. With previous stationary phases, the hydroxide eluent system was not sufficiently powerful to elute strongly retained anions. Because the AS14A column has high chemical stability and is moderately hydroxide selective, this column can be used with hydroxide eluent systems. Thus, the AS14A column can be operated with either carbonate or hydroxide eluent systems depending upon the analysis requirements.

With a hydroxide eluent system, a much smaller baseline shift occurs than was previously possible with borate eluent systems. Also, overall chromatographic efficiency with hydroxide eluents is substantially higher than with borate eluents.

Notice that 1.2% boric acid in the standard has no effect on the separation.

Trap Column	ATC-3 (2 required) 1 located after the Pump 1 located at the eluent outlet of the EG40 Degas Module and before the injection valve.	Eluent: Deionized water Offset volume = 0.0 µL	
Sample Loop Volume:	10 µL	Time (min)	Eluent Conc. (mM)
Column:	IonPac AG14A + IonPac AS14A (4-mm)	Equilibrate	
Eluent Source:	EG40	0.0	5
Eluent Flow Rate:	1.2 mL/min	5.0	5
Temperature	30°C	Analysis	
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (4-mm) AutoSuppression® Recycle Mode	5.1	5
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4-mm)	7.0	5
MMS Regenerant:	50 mN H ₂ SO ₄	17.0	5
Expected Background Conductivity:	1 - 2 µS	35.0	80
			Comments
			Start isocratic analysis
			Injection valve to load
			Begin gradient analysis
			End gradient

Analyte	mg/L (ppm)
1. Fluoride	5
2. Glycolate	10
3. Acetate	20
4. Formate	10
5. Chlorite	10
6. Chloride	10
7. Bromate	20
8. Nitrite	15
9. Bromide	25
10. Nitrate	25
11. Chlorate	25
12. Sulfate	30
13. Oxalate	30
14. Phosphate	40

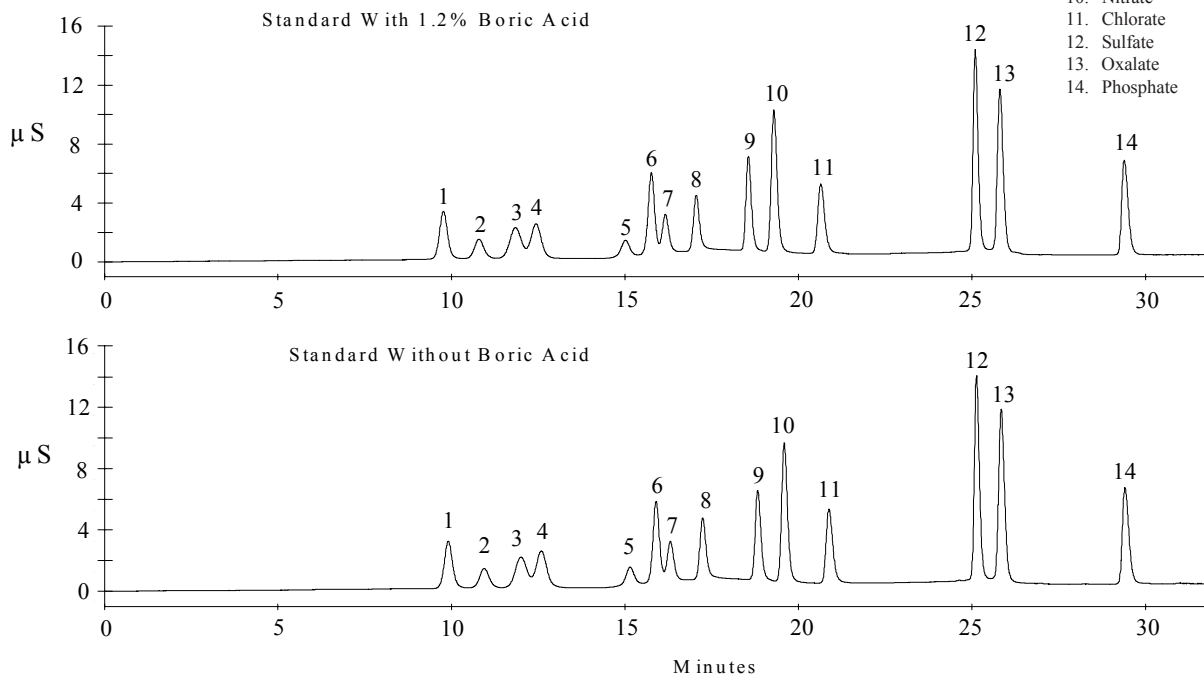


Figure 11
Separation of Weak Organic Acids, Oxyhalides and Common Anions
Using a Sodium Hydroxide Gradient on the AS14A 4-mm Column

5.14 Separation of Weak Organic Acids, Oxyhalides and Common Anions Using a Sodium Hydroxide Gradient on the AS14A 3-mm Column

In the past Dionex has recommended a borate eluent system for analysis of organic acids when gradients were required for carbonate-based columns. With previous stationary phases, the hydroxide eluent system was not sufficiently powerful to elute strongly retained anions. Because the AS14A column has high chemical stability and is moderately hydroxide selective, this column can be used with hydroxide eluent systems. Thus, the AS14A column can be operated with either carbonate or hydroxide eluent systems depending upon the analysis requirements.

With a hydroxide eluent system, a much smaller baseline shift occurs than was previously possible with borate eluent systems. Also, overall chromatographic efficiency with hydroxide eluents is substantially higher than with borate eluents.

Trap Column	ATC-3 (2 required) 1 located after the Pump 1 located at eluent outlet of the EG40 Degas Module and before the injection valve.
Sample Loop Volume:	5 μ L
Column:	IonPac AG14A + IonPac AS14A (3-mm)
Eluent Source:	EG40
Eluent Flow Rate:	0.6 mL/min
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (2-mm) AutoSuppression® Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm)
MMS Regenerant:	50 mN H ₂ SO ₄
Expected Background Conductivity:	1 - 2 μ S

Eluent: Deionized water
Offset volume = 0.0 μ L

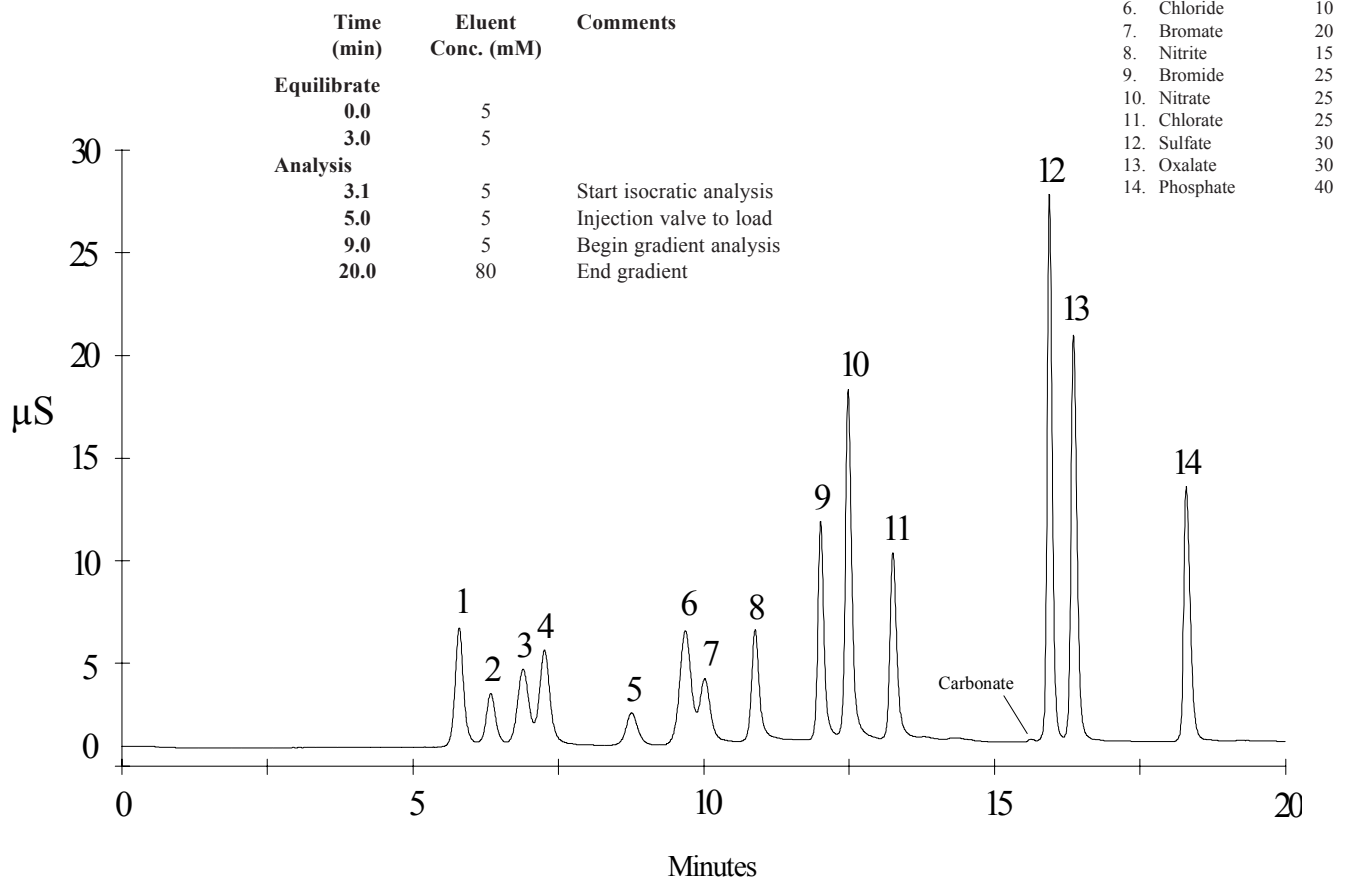


Figure 12
Separation of Weak Organic Acids, Oxyhalides and Common Anions
Using a Sodium Hydroxide Gradient on the AS14A 3-mm Column

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS14A columns. For more information on problems that originate with the Ion Chromatograph (IC) or the Anion Self-Regenerating Suppressor-ULTRA, the ASRS-ULTRA, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the DIONEX Office nearest you (see, "DIONEX Worldwide Offices").

Table 6
AS14A/AG14A 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, 6.2.3
	Contaminated ASRS, AAES, or AMMS	Clean Suppressor	6.2.5, 6.2.6, 6.2.7 Component Manual
	Contaminated Hardware	Clean Component	6.2.4, Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System	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
	Incorrect Eluent Conc.	Remake Eluents	6.3.2.B
	Column Contamination	Clean Column	6.3.2.C, 6.3.2.D
Poor Front End Resolution	Incorrect Eluent Conc.	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 IonPac AG14A (4-mm) Guard Column plus the AS14A (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 AS14A/AG14A Operating Back Pressures”).

The Anion Self-Regenerating Suppressor-ULTRA 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 AS14A/AG14A Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS14A 4-mm Analytical	< 1,500 (8.96)	1.0
AG14A 4-mm Guard	< 300 (1.68)	1.0
AS14A + AG14A 4-mm columns	< 1,800 (11.20)	1.0
AS14A 3-mm Analytical	< 1,500 (8.96)	0.5
AG14A 3-mm Guard	< 300 (1.68)	0.5
AS14A + AG14A 3-mm columns	< 1,800 (11.20)	0.5

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.

	IonPac AS14A 4-mm Columns (P/N)	IonPac AS14A 3-mm Columns (P/N)
Analytical Column	056904	056901
Guard Column	056897	056899
Bed Support Assembly	042955	056823
End Fitting	052809	052809

CAUTION

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

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

CAUTION

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
8.0 mM Na ₂ CO ₃ /1.0 mM NaHCO ₃	24 - 28 μ S

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 Trap Column

When doing gradient analysis, ensure that the Anion Trap Column, the ATC-3 (2-mm) or the ATC-3 (4-mm) has been installed correctly. If it has not, install one as directed in Section 3.5, "Installing the ATC-3 for Eluent Step Change or Gradient Operation," 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).

Determine if the ATC is the source of high background conductivity. Remove the ATC. If the background conductivity remains high, then the ATC is not the problem. 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-3 (4-mm) with 100 mL of 200 mM NaOH or 50 mL of 200 mM NaOH for 2-mm ATC-3. 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. Pump 20 mL of eluent through 4-mm ATC-3 or 10 mL for the 2-mm ATC-3.
- D. If the problem persists, replace the ATC.

6.2.3 A Contaminated Guard or Analytical Column

Remove the IonPac AG14A Guard and AS14A 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 AG14A 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 μ S. 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 A Contaminated ASRS-ULTRA Suppressor

This section describes routine cleanup procedures for the Anion Self-Regenerating Suppressors (ASRS-ULTRA) in the case of contamination. Consult the Troubleshooting Guide (see Section 4, “Troubleshooting Guide”) to first determine that the system is operating properly. If the ASRS-ULTRA is determined to be the source of higher than normal back pressure, higher than anticipated conductivity, decreased suppression capacity or decreased sensitivity, cleaning the membrane may restore the performance of the system. Use the following procedures to clean the membrane.

Metal Contaminants or Precipitates

CAUTION

The suppressor voltage is a good indicator of the resistance across the suppressor. Higher resistance may indicate contamination of the suppressor. For more information regarding monitoring the voltage, see Document No. 031814-02, “Removal of Iron Contamination from Electrolytic Suppressors.”

- A. Turn off the SRS Control unit.
- B. Disconnect the analytical (and guard) column(s) from the injection valve and the ASRS-ULTRA. Refer to the specific analytical column Product Manual for column cleanup procedures.
- C. If you are running in the **AutoSuppression External Water Mode**, turn off the external water and disconnect the external water line from the ASRS-ULTRA **REGEN IN** port.
- D. Disconnect the liquid line from the ASRS-ULTRA **ELUENT OUT** port to the cell at the cell fitting and reconnect it to the **REGEN IN** port.
- E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.2 M oxalic acid. Pump this solution through the ASRS-ULTRA (4-mm) at 1-2 mL/min for 30 minutes. For 2-mm systems pump this solution through the ASRS-ULTRA (2-mm) at 0.25-0.50 mL/min for 30 minutes.

CAUTION

Bypassing internal pump manifolds when temporarily pumping high concentration cleaning solutions significantly reduces the time required to reequilibrate the system to low concentration eluents.

- F. Flush the ASRS-ULTRA with deionized water for 10 minutes.
- G. Perform steps A - D of the procedure in Section 4.1, “Small Analyte Peak Areas.”
- H. Turn on the SRS Control unit for the **AutoSuppression Recycle or External Water Modes** of operation. Ensure that the SRS Control unit is **off** for the **Chemical Suppression Mode** of operation.
- I. Flush the ASRS-ULTRA with eluent for 10 minutes.
- J. Reinstall the analytical (and guard) column(s). Begin pumping eluent through the system at the flow rate required for your analysis and equilibrate the system.

6.2.6 A Contaminated Suppressor AMMS III Suppressor

- A. **Check the regenerant flow rate at the REGEN OUT port of the AMMS.** For the example isocratic applications, this flow rate should be 3 - 5 mL/min.
- B. **Check the eluent flow rate.** In general, the eluent flow rate for 4-mm applications, it should be 1.0 mL/min. Refer to the Anion MicroMembrane Suppressor Product Manual (Document No. 034449-02) for assistance in determining that the eluent is within suppressible limits.

- C. If you are using an AutoRegen Accessory with the SRS (in the Chemical Suppression Mode) or the MMS, prepare fresh regenerant solution. Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.
1. If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge, you probably need to clean or replace your SRS or MMS.
 2. If the background conductivity is low when freshly prepared regenerant is run through the SRS or MMS without an AutoRegen Accessory in-line, test the AutoRegen Regenerant Cartridge to see if it is **expended**. Connect the freshly prepared regenerant to the AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is high after placing the AutoRegen Accessory in-line, you probably need to replace the AutoRegen Regenerant Cartridge. Refer to the “AutoRegen Regenerant Cartridge Refill Product Manual” (Document No. 032852) for assistance.

6.2.7 A Contaminated Anion Atlas Electrolytic Suppressor (AAES)

Metal Contaminants or Precipitates

- A. Turn off the power to the AAES.
- B. Disconnect the analytical (and guard) column(s) from the injection valve and the AAES. Refer to the specific analytical column Product Manual for column cleanup procedures.
- C. If you are running in the **AutoSuppression External Water Mode**, turn off the external water and disconnect the external water line from the AAES **REGEN IN** port.
- D. Disconnect the liquid line from the AAES **ELUENT OUT** port to the cell at the cell fitting and reconnect it to the **REGEN IN** port.
- E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.5 M oxalic acid. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 2.0 mL/min for 30 minutes.

CAUTION

Bypassing internal pump manifolds when temporarily pumping high concentration cleaning solutions significantly reduces the time required to re-equilibrate the system to low concentration eluents.

- F. Flush the AAES with deionized water at 2 mL/min for 30 minutes.
- G. Reinstall the AAES according to procedures in Section 4.2.1, “Eluent and Regenerant Flow Path Connections in the AutoSuppression Recycle Mode Operation” or Section 4.3.1, “Eluent Flow Path Connections in the AutoSuppression External Water Mode Operation” and resume operation.

Organic Contaminants

- A. Turn off the power to the AAES.
- B. Disconnect the analytical (and guard) column(s) from the injection valve and the AAES. Refer to the specific analytical column Product Manual for column cleanup procedures.
- C. If you are running in the **AutoSuppression External Water Mode**, turn off the external water and disconnect the external water line from the AAES **REGEN IN** port. If you are running in the **AutoSuppression Recycle Mode**, proceed to D.
- D. Disconnect the liquid line from the AAES **ELUENT OUT** port to the cell at the cell fitting and reconnect it to the **REGEN IN** port.

- E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of freshly prepared 10% 1.0 M H₂SO₄/90% acetonitrile. H₂SO₄/acetonitrile solutions are not stable during long term storage so this cleanup solution must be made immediately before each column cleanup. Alternatively, it can be proportioned from 1 bottle containing 1.0 M H₂SO₄ and another bottle containing 100% acetonitrile. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 1.0 mL/min for 60 minutes.

CAUTION

Bypassing internal pump manifolds when temporarily pumping high concentration cleaning solutions significantly reduces the time required to re-equilibrate the system to low concentration eluents.

- F. Flush the AAES with deionized water at 2 mL/min for 30 minutes.
- G. Reinstall the AAES according to procedures in Section 4.2.1, "Eluent and Regenerant Flow Path Connections in the AutoSuppression Recycle Mode Operation" or Section 4.3.1, "Eluent Flow Path Connections in the AutoSuppression External Water Mode Operation" and resume operation.

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. **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 3-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.
- B. **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.

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, "Column Cleanup" (see, "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, "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. If you need assistance in solving resolution problems, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices")

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").

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS14A columns, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

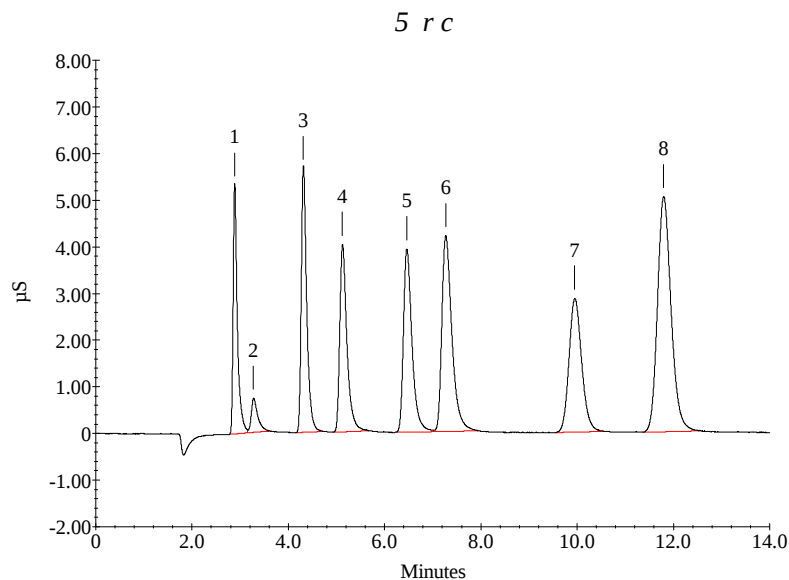
- B. The injection valve may need maintenance.** When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or retorqued (see valve manual). 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.

IonPac® AS14A 4-mm
Analytical (4 x 250 mm)
Product No. 056904

Serial No. : 5 r c

Pressure (PSI) : 1470

Date : 4/26/00 10:39:07 AM



Eluent: 8.0 mM Na₂CO₃/1.0 mM NaHCO₃

Flow Rate: 1.0 mL/min

Temperature: 30° C

Detection: Suppressed Conductivity at 10 µSFS
ASRS® -ULTRA
 AutoSuppression® Recycle Mode

Injection Volume: 10 µL

Storage Solution: Eluent

Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	2.88	Fluoride	5.0	6132	2.5	2.07
2	3.27	Acetate	20.0	3181	2.6	4.81
3	4.31	Chloride	10.0	7555	2.1	3.59
4	5.13	Nitrite	15.0	6326	2.0	4.70
5	6.46	Bromide	25.0	6875	1.9	2.38
6	7.27	Nitrate	25.0	6232	2.0	6.38
7	9.95	Phosphate	40.0	6996	1.3	3.76
8	11.79	Sulfate	30.0	8655	1.4	n/a

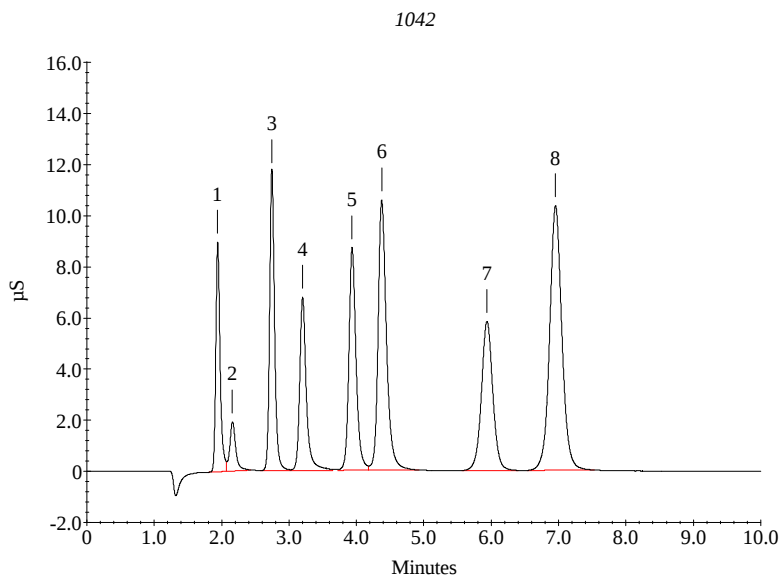
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IonPac® AS14A 3-mm
Analytical (3 x 150 mm)
Product No. 056901

Serial No. : 1042

Pressure (PSI) : 1160

Date : 5/15/00 10:45:15 AM



Eluent: 8.0 mM Na₂CO₃/1.0 mM NaHCO₃

Flow Rate: 0.5 mL/min

Temperature: 30° C

Detection: Suppressed Conductivity at 10 μSFS
ASRS®-ULTRA, 2-mm
 AutoSuppression® Recycle Mode

Injection Volume: 5 μL

Storage Solution: Eluent

Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	1.94	Fluoride	5.0	4943	1.5	1.67
2	2.16	Acetate	20.0	3307	n/a	4.17
3	2.74	Chloride	10.0	7085	1.5	3.12
4	3.20	Nitrite	15.0	6071	1.5	4.22
5	3.93	Bromide	25.0	7303	1.4	2.23
6	4.38	Nitrate	25.0	6787	1.5	6.10
7	5.93	Phosphate	40.0	6249	1.1	3.29
8	6.95	Sulfate	30.0	7544	1.2	n/a

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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 AS14A columns is 4,000 psi (27.57 MPa).

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.

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.

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.

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.

CHOOSING THE APPROPRIATE CLEANUP SOLUTION

A. Hydrophilic ionic contamination of low valency

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

B. High valency hydrophobic ions

1. 200 mM HCl in 80% acetonitrile
The acetonitrile solution is stored in a separate eluent bottle because acetonitrile slowly breaks down in acidic aqueous solutions. Prepare two bottles (E1 and E2) with the following 500-mL solutions:
E1: 100% acetonitrile
E2: 1 M HCl using degassed Type I Reagent Grade Water with a specific resistance of 17.8 megohm-cm.

You may make 200 mM HCl in 80% acetonitrile in a single bottle but this solution must be made immediately before each use and cannot be stored because of acetonitrile degradation

C. Metal contamination

1. 0.1 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. Regardless of the cleanup solution chosen, use the following cleanup procedure, "Column Cleanup Procedure", to clean the AG14A and AS14A.**

COLUMN CLEANUP PROCEDURE

- A. Prepare a 500 mL solution of the appropriate cleanup solution** using the guidelines, "Choosing the Appropriate Cleanup Solution", above.
- B. Disconnect the ASRS-ULTRA or AMMS** from the IonPac AS14A 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 AS14A 4-mm Analytical or Guard Column, or set the pump flow rate to 0.5 mL/min for an AS14A 3-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 at least 60 minutes.**
- 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 ASRS-ULTRA or AMMS** to the AS14A 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.