Errata

Product Manual for Dionex IonPac™ AS14 and AG14 Columns
031199-07

For new orders of the following parts discussed in this manual, please use the updated part numbers listed below.

Part	Old Part Number in this manual	Updated Part Number to use for new orders
PROD,COL,IP,ATC-3,4X35MM	*059661*	*079932*

Thermo Scientific

Dionex IonPac AS14 Columns

Product Manual

P/N: 031199-07 July 2012

Thermo SCIENTIFIC

PRODUCT MANUAL

for the

IONPAC® AG14 GUARD COLUMN

(4 x 50 mm, P/N 046134)

(2 x 50 mm, P/N 046138)

IONPAC® AS14 ANALYTICAL COLUMN

(4 x 250 mm, P/N 046124)

(2 x 250 mm, P/N 046129)

Document No. 031199
Revision 07
July 2012

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SECTION 1 - INTRODUCTION

The IonPac® AS14 Analytical Column in combination with the AG14 Guard Column is designed for the analysis of fluoride and other inorganic anions. The selectivity of the IonPac AS14 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 AS14 is compatible with pH 0-12 eluents and eluents containing organic solvents from 0–100% in concentration. The AS14 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 AS14 has nominal efficiency for sulfate using standard operating conditions of at least 16,000 plates/meter.

Table 1
IonPac AS14/AG14 Packing Specifications

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

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS14 4-mm Analytical	≤ 1,300 (8.96)	1.2	3.0
AG14 4-mm Guard	≤ 325 (2.24)	1.2	3.0
AS14 + AG14 4-mm Columns	**≤ 1,625 (11.20)**	**1.2**	**3.0**
AS14 2-mm Analytical	≤ 1,300 (8.96)	0.3	0.75
AG14 2-mm Guard	≤ 325 (2.24)	0.3	0.75
AS14 + AG14 2-mm Columns	**≤1,625 (11.20)**	**0.3**	**0.75**

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of Dionex instrumentation and columns through any of the Dionex Offices listed in, "Dionex Worldwide Offices."

SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

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

CONDITION	2-mm	4-mm
Eluent Flow Rate	0.30 mL/min	1.2 mL/min
SRS	ASRS-ULTRA (2-mm) (P/N 053947)	ASRS-ULTRA (4-mm) (P/N 053946)
MMS	AMMS III (2-mm) (P/N 056751)	AMMS III (4-mm) (P/N 056750)
AES	AAES (P/N 056116)	AAES (P/N 056116)
Injection Loop	2 - 15 µL Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.	10 - 50 µL
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 in Microbore 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	2-mm	4-mm
Detectors	AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL, P/N 043120) 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/ED50A or the CD20/CD25/CD25A. Ensure 30-40 psi back pressure after the cell.	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/ED50A or the CD20/CD25/CD25A. Ensure 30-40 psi back pressure after the cell.

Table 3
Tubing Back Pressures

Color	Dionex P/N	ID inches	ID 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

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 2-mm Operation

The 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 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 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 Ultra Low Pressure Trace Anion Concentrator (TAC-ULP1, P/N 061400), the Trace Anion Concentrator Column (TAC-2, P/N 043101), the AMC-1 (P/N 051760), or the IonPac AG14 Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of the TAC-LP1, the TAC-ULP1, the TAC-2 or the AG14 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-ULP1, TAC-2, AMC-1, or the AG14 leading to a lowering of 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. The unique advantage to the analytical chemist of the TAC-LP1, the TAC-ULP1, the AMC-1, the TAC-2 or the AG14 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 and TAC-ULP1) Column Product Manual (Document No. 034972). These techniques can also be applied to either the TAC-2 or the AG14. For information on the AMC-1, refer to the Product Manual for the AMC-1 (Document No. 031262).

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 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. Dionex recommends that a 2.5 µL injection loop be used to avoid overloading the AS14 2-mm Analytical Column. 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 AS14 2-mm 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, “Comparison of 2-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 AS14 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 AG14 Guard Column

An IonPac AG14 Guard Column is normally used with the 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 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 Anion Trap Column, ATC-3

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. 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 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 ATC-3 (2-mm) or ATC-3 (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 ATC. Connect a waste line to the ATC outlet and direct the line to a waste container.

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

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

E. After flushing the ATC with eluent, connect the ATC 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 ASRS in-line and properly functioning. The baseline shift should be no greater than 10 mS 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 ATC should be cleaned using steps A - E above.

The ATC 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 ATC with 30 mL of 70 mM $Na_2B_4O_7$.

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

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

3.6 Eluent Storage

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).

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.

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

If you are installing an IonPac AS14 4-mm Analytical Column, use an ASRS ULTRA (4-mm, P/N 053946).
If you are installing an IonPac AS14 2-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. 031367, 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 2-mm and 4-mm IonPac AS14 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 (4-mm) for applications that require suppressed conductivity detection. Use an AMMS III (P/N 056750) with the 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 AMMS III (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 AutoRegen

To save regenerant preparation time and reduce regenerant consumption and waste, Dionex recommends using an AutoRegen® Accessory (P/N 039594) with the ASRS ULTRA in the Chemical Suppression Mode or with the AMMS III. 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).

3.11 Detector Requirements

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

SECTION 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: AS14 2-mm Analytical Column + AG14 2-mm Guard Column 4-mm: AS14 4-mm Analytical Column + 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
SRS Suppressor:	Anion Self-Regenerating Suppressor ULTRA (2-mm or 4-mm) AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS
Storage Solution:	Eluent

4.2 IonPac AS14 Operation Precautions

CAUTIONS
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. 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 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 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.

B. 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.

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.

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

The AS14 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 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 Self-Regenerating Anion Suppressor (ASRS ULTRA) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

4.6 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."

SECTION 5 - EXAMPLE APPLICATIONS

The chromatograms in this section were obtained using columns that reproduced the Production Test Chromatogram (see Section 5.2, "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 Production Test Chromatogram

Isocratic elution of anions on the 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 IonPac AS14 Analytical Column should always be used with the IonPac AG14 Guard Column. To guarantee that all IonPac AS14 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

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)
SRS Suppressor:	Anion Self-Regenerating Suppressor (2-mm or 4-mm) AutoSuppression® Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
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

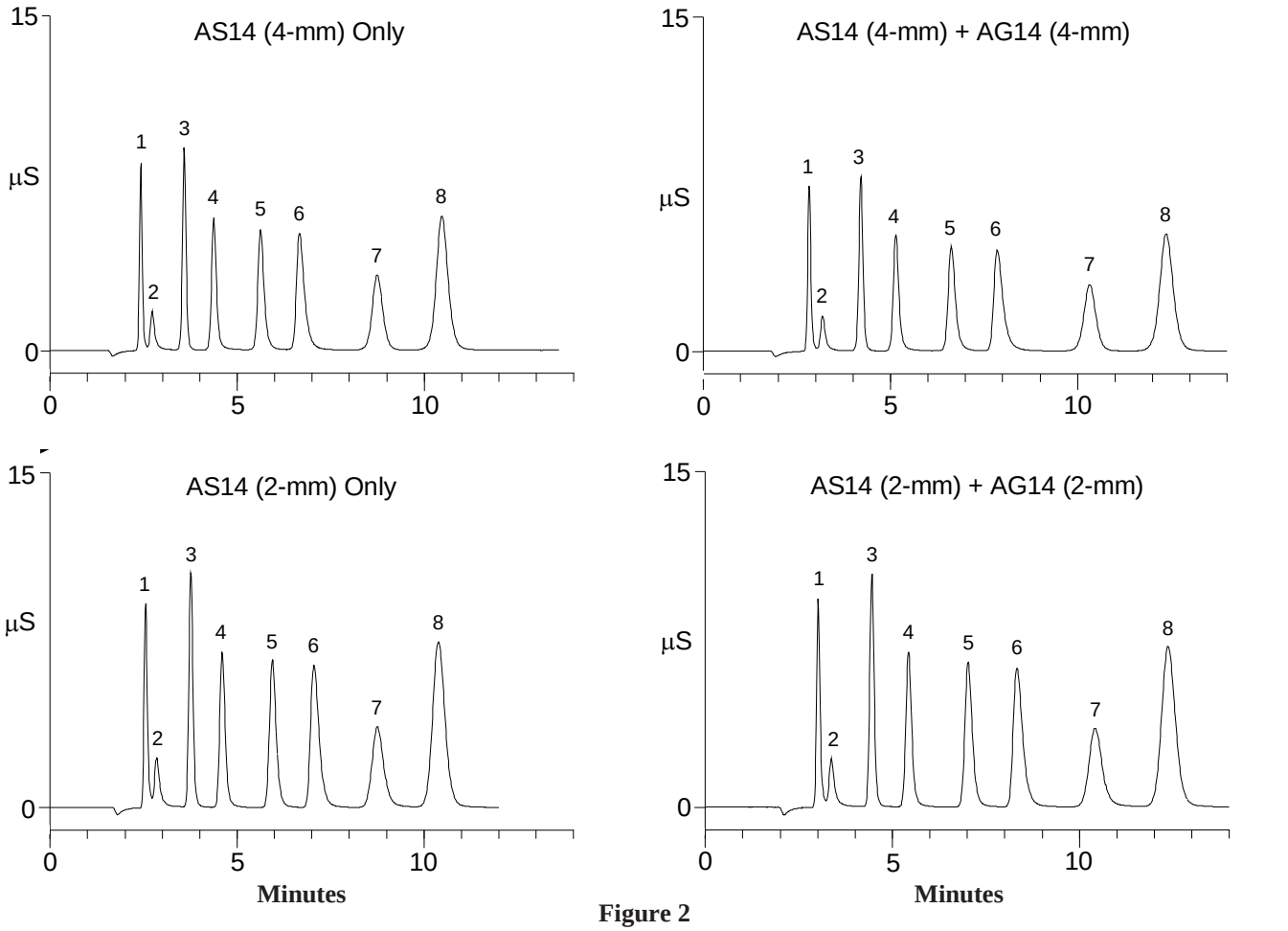


Figure 2
IonPac AS14 Production Test Chromatograms

5.2 Fast Run Analysis without Changes in Selectivity

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

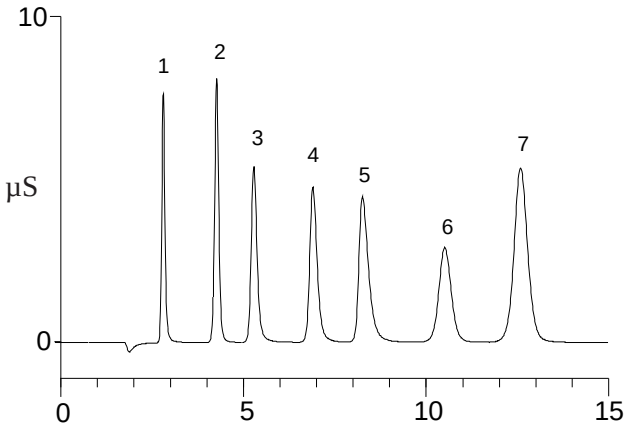
Sample Loop Volume:	10 µL
Column:	IonPac AS14 + IonPac AG14
Eluent:	See Chromatogram
Eluent Flow Rate:	See Chromatogram
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, (if eluent suppression required is less than 25 µeq/min)
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
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

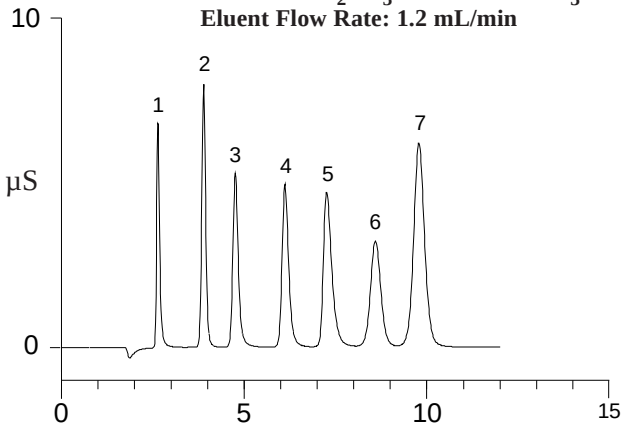
where 1 mg/L = 1 ppm

Reducing Run Time by Changing the Eluent

Eluent: 3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate: 1.2 mL/min



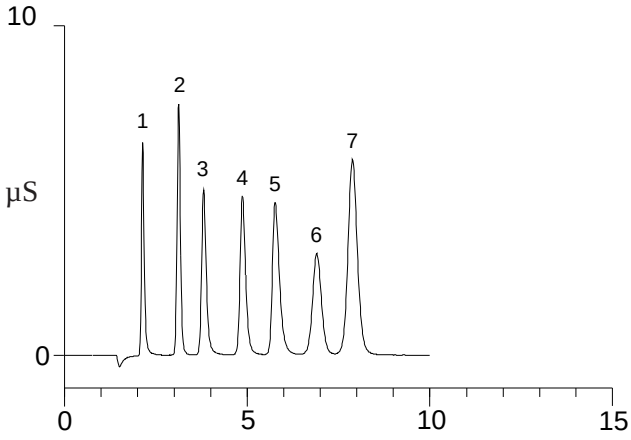
Eluent: 4.8 mM Na_2CO_3/0.6 mM $NaHCO_3$
Eluent Flow Rate: 1.2 mL/min



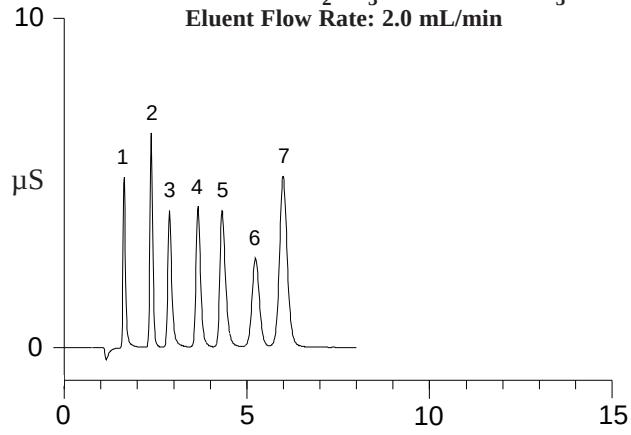
Minutes

Reducing Run Time by Changing the Flow Rate

Eluent: 4.8 mM Na_2CO_3/0.6 mM $NaHCO_3$
Eluent Flow Rate: 1.5 mL/min



Eluent: 4.8 mM Na_2CO_3/0.6 mM $NaHCO_3$
Eluent Flow Rate: 2.0 mL/min



Minutes

Figure 3
Fast Run Analysis Without Changes in Selectivity

5.3 Increasing the Loop Size to Increase Sensitivity

Sample Loop Volume:	See Chromatogram
Column:	IonPac AS14 + IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression External Water Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

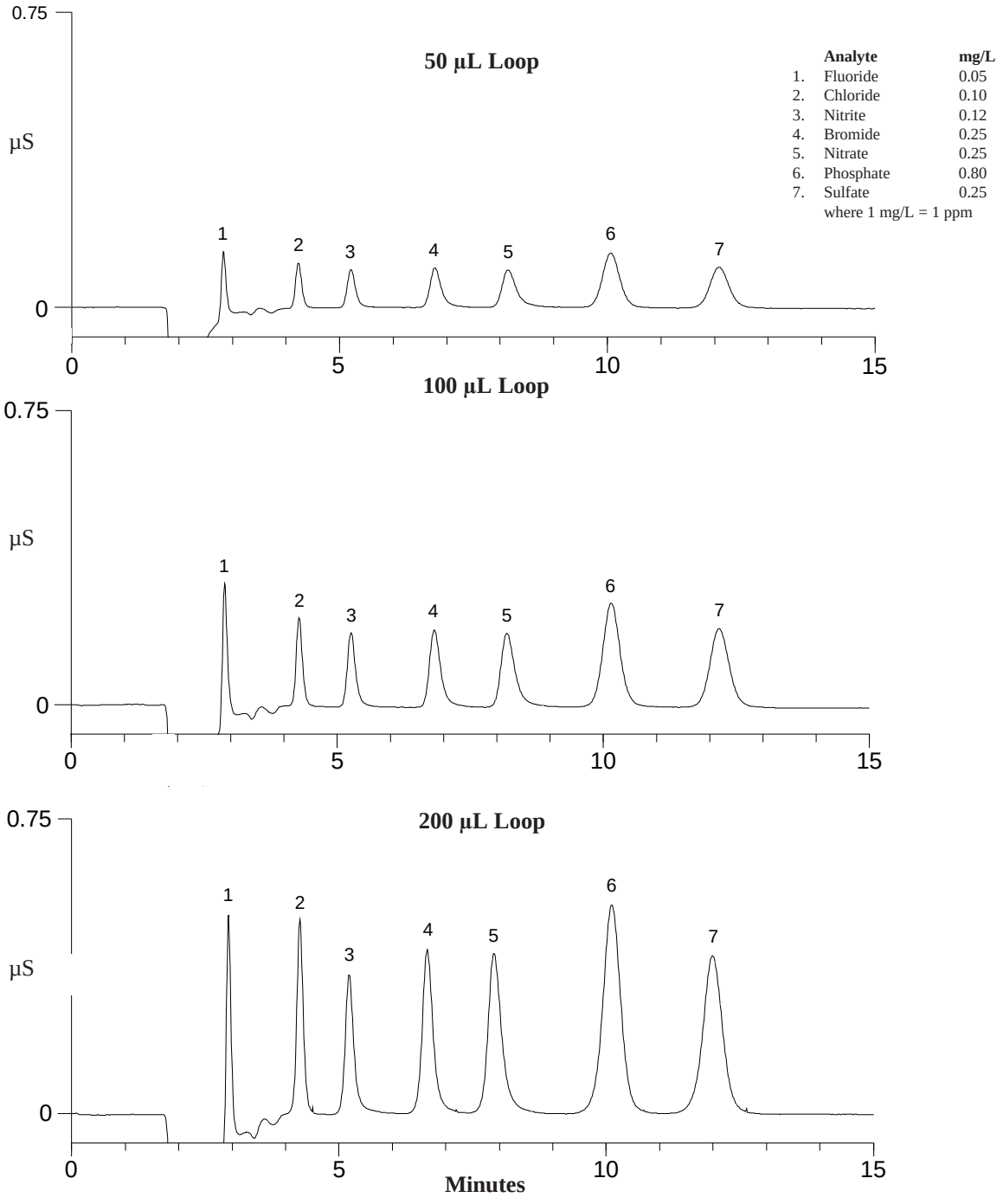


Figure 4
Increasing Sensitivity by Increasing Loop Size

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:	IonPac AS14 + IonPac AG14
Eluent:	2.7 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	15-17 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

where 1 mg/L = 1 ppm

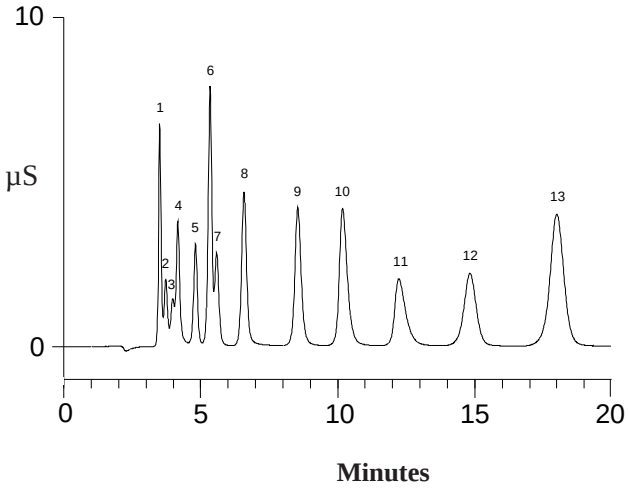


Figure 5
13 Anions Selectivity

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:	IonPac AS14 + IonPac AG14
Eluent:	2.7 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	15-17 µS

	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

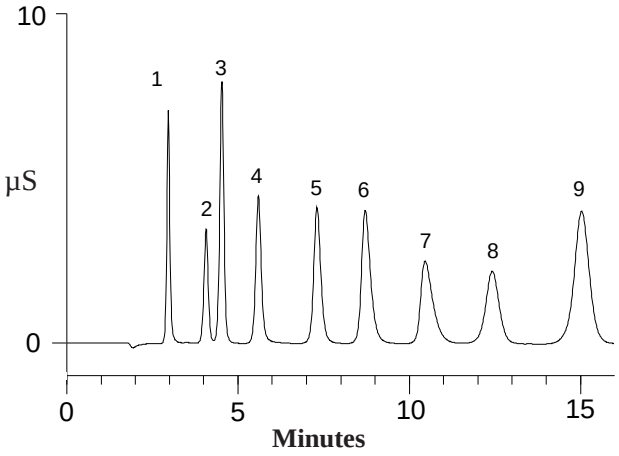


Figure 6
Analysis of Selected Oxyanions

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:	IonPac AS14 + IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
orMMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

Analyte
1. Fluoride
2. Carbonate
3. Chloride
4. Nitrate
5. Phosphate
6. Sulfate

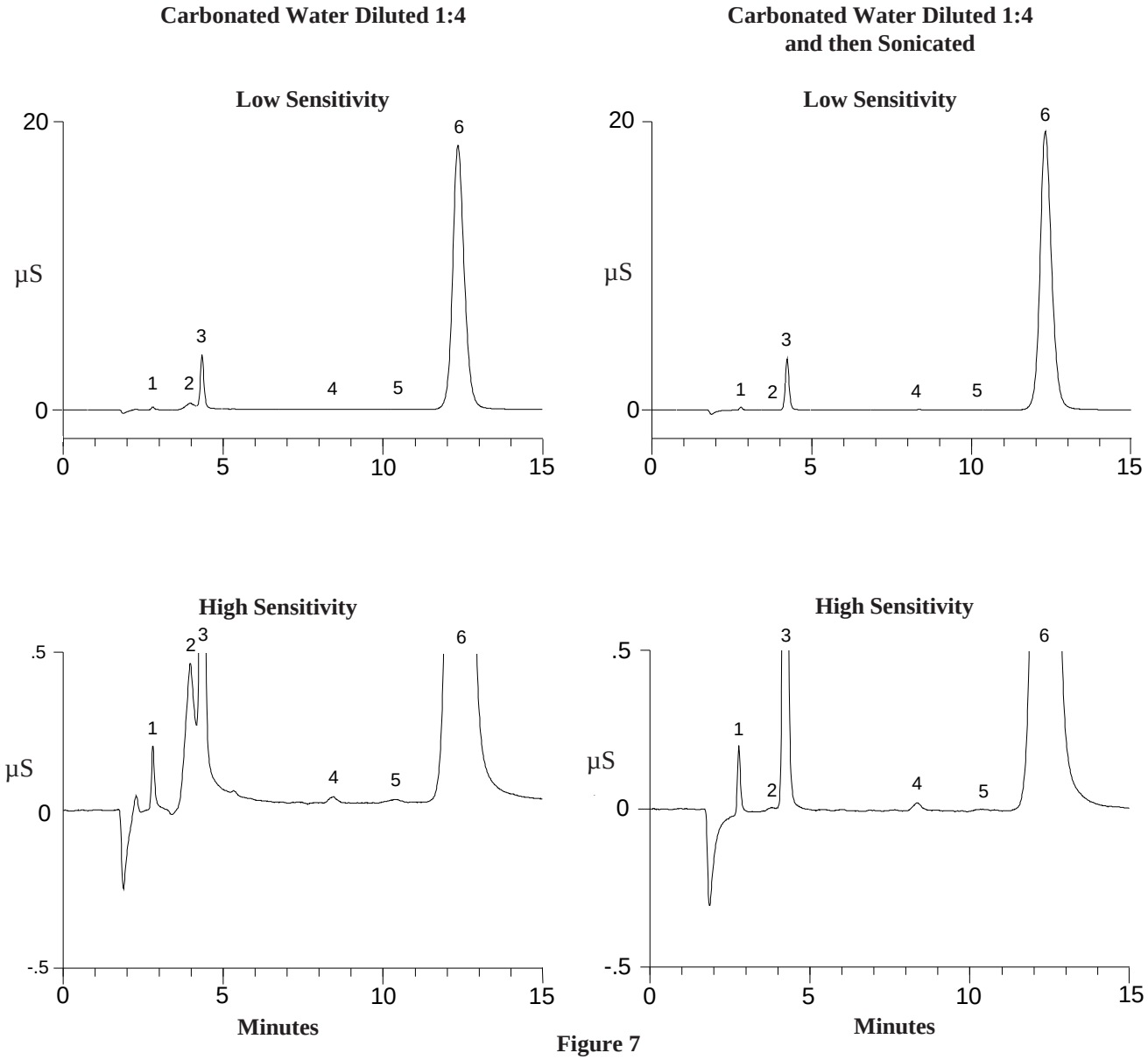


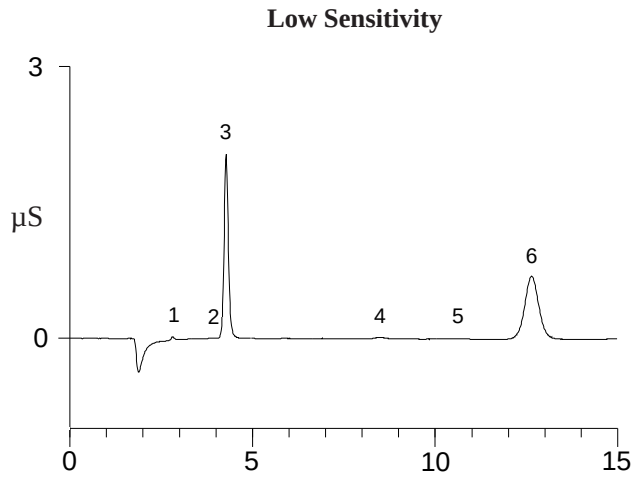
Figure 7
Analysis of Carbonated Water

5.7 Analysis of Municipal Tap Water

Sample Loop Volume:	10 µL
Column:	IonPac AS14 + IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

	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



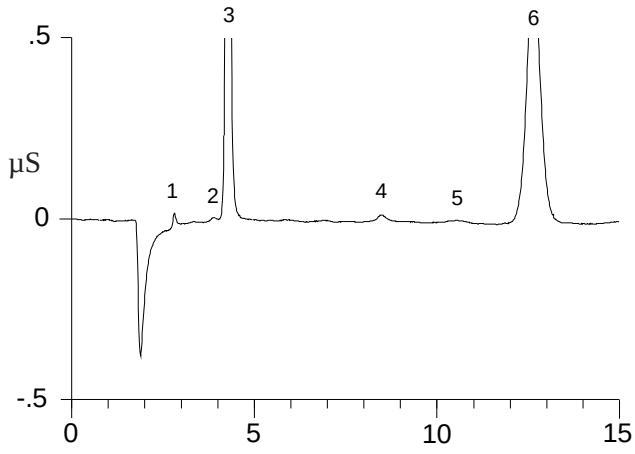
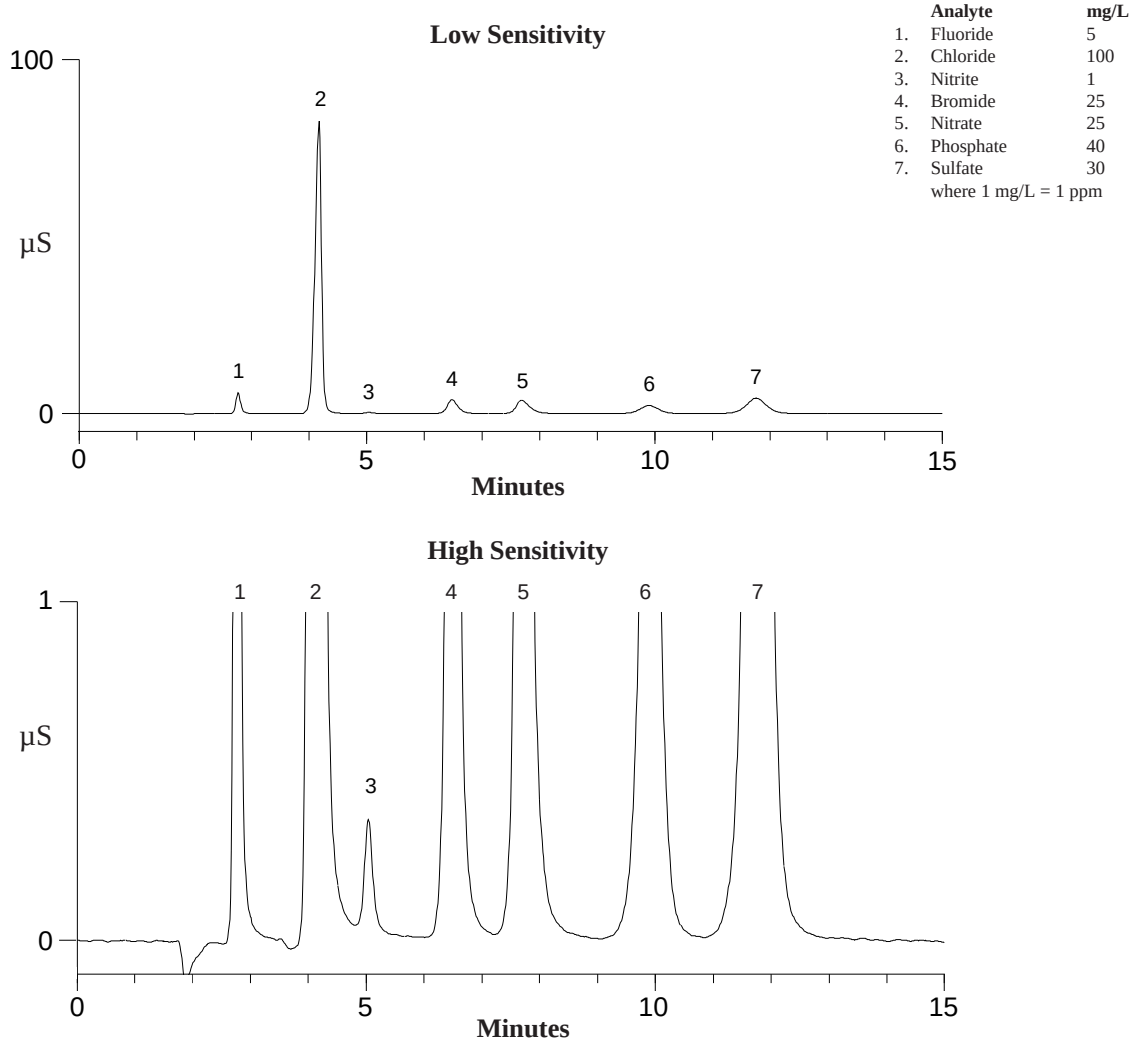


Figure 8
Analysis of Municipal Tap Water

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 AS4A. The 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:	IonPac AS14 + IonPac AG14
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS



	Analyte	mg/L
1.	Fluoride	5
2.	Chloride	100
3.	Nitrite	1
4.	Bromide	25
5.	Nitrate	25
6.	Phosphate	40
7.	Sulfate	30

where 1 mg/L = 1 ppm

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

5.9 Analysis of Tooth Paste

The excellent retention of fluoride on the 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:	IonPac AS14 + IonPac AG14
Eluent:	2.0 mM Na_2CO_3/2.5 mM $NaHCO_3$
Eluent Flow Rate:	1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, if eluent suppression required is less than 25 µeq/min.
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	16-18 µS

	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	

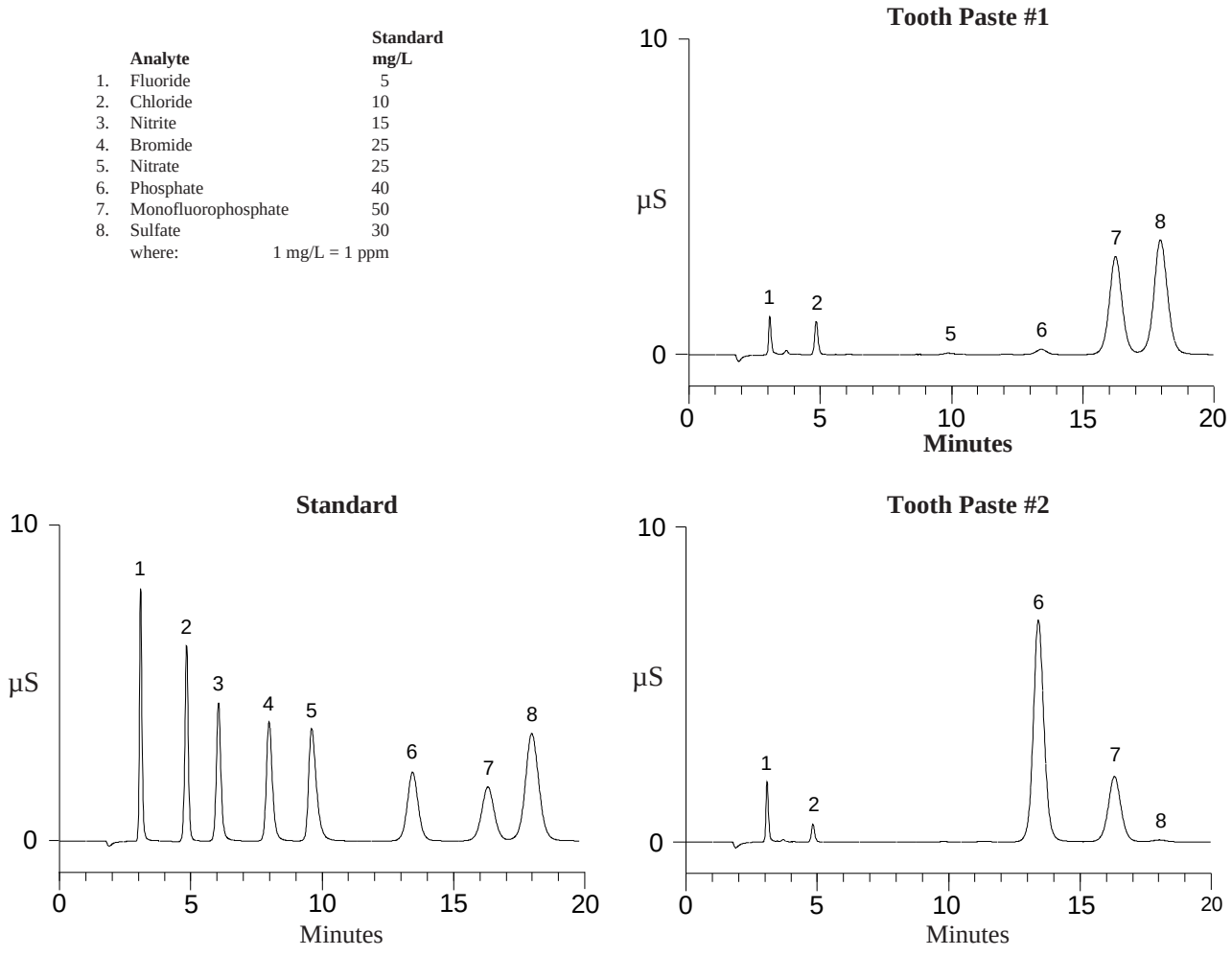


Figure 10
Analysis of Tooth Paste

5.10 Isocratic Analysis of Seven Anions Using a Borate Eluent

Sample Loop Volume:	25 µL
Column:	IonPac AS14 + 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

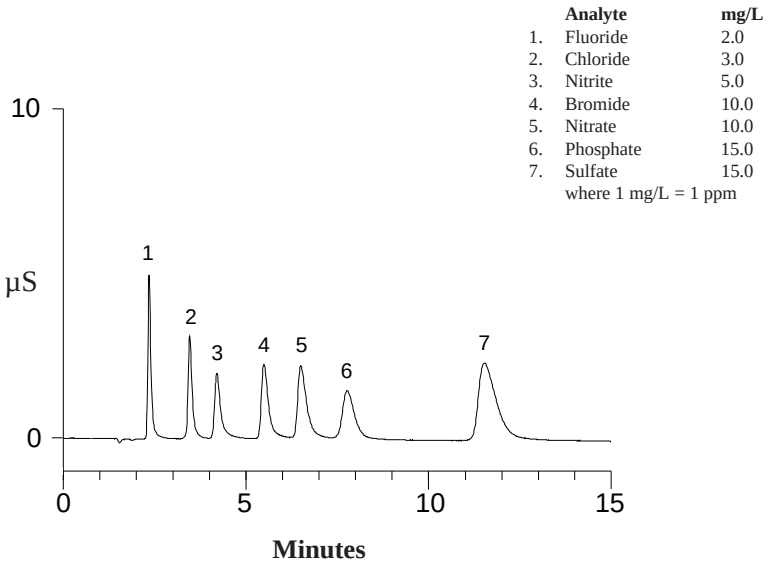


Figure 11
Isocratic Analysis of Seven Anions Using a Borate Eluent

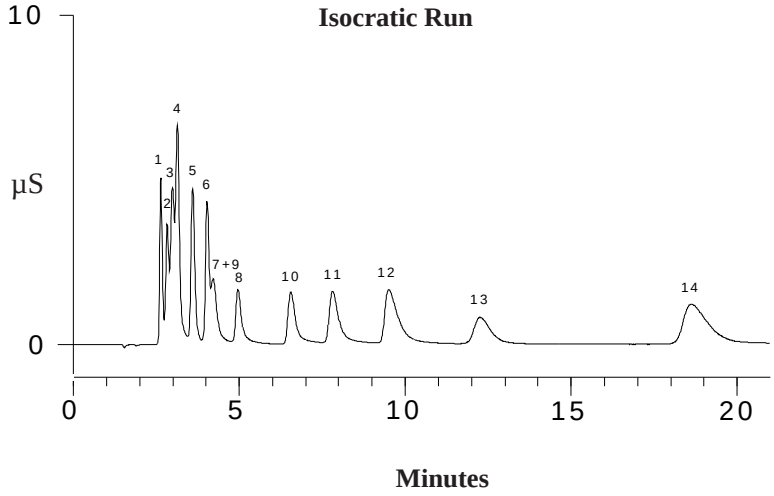
5.11 Borate Gradient Analysis

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:	IonPac AS14 + 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

	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

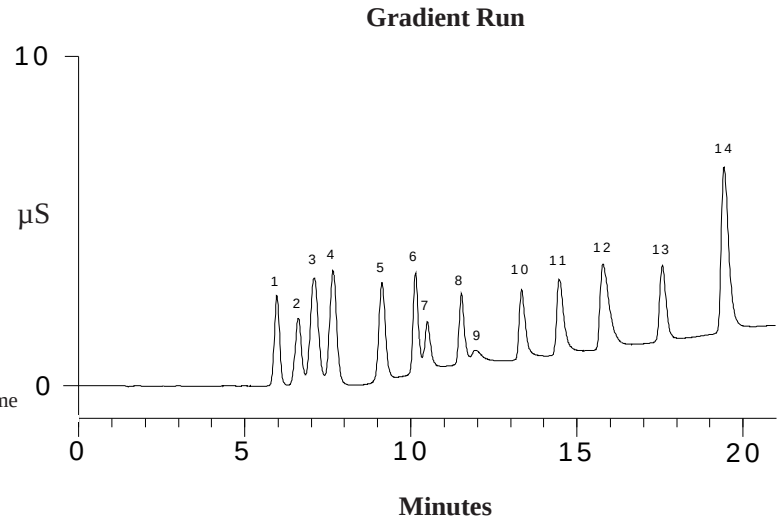


Figure 12
Borate Gradient Analysis

5.12 Clean-up After Humic Acid Samples

Solvent compatibility of the IonPac AS14 permits the use of organic solvents to effectively remove organic contaminates 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.

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

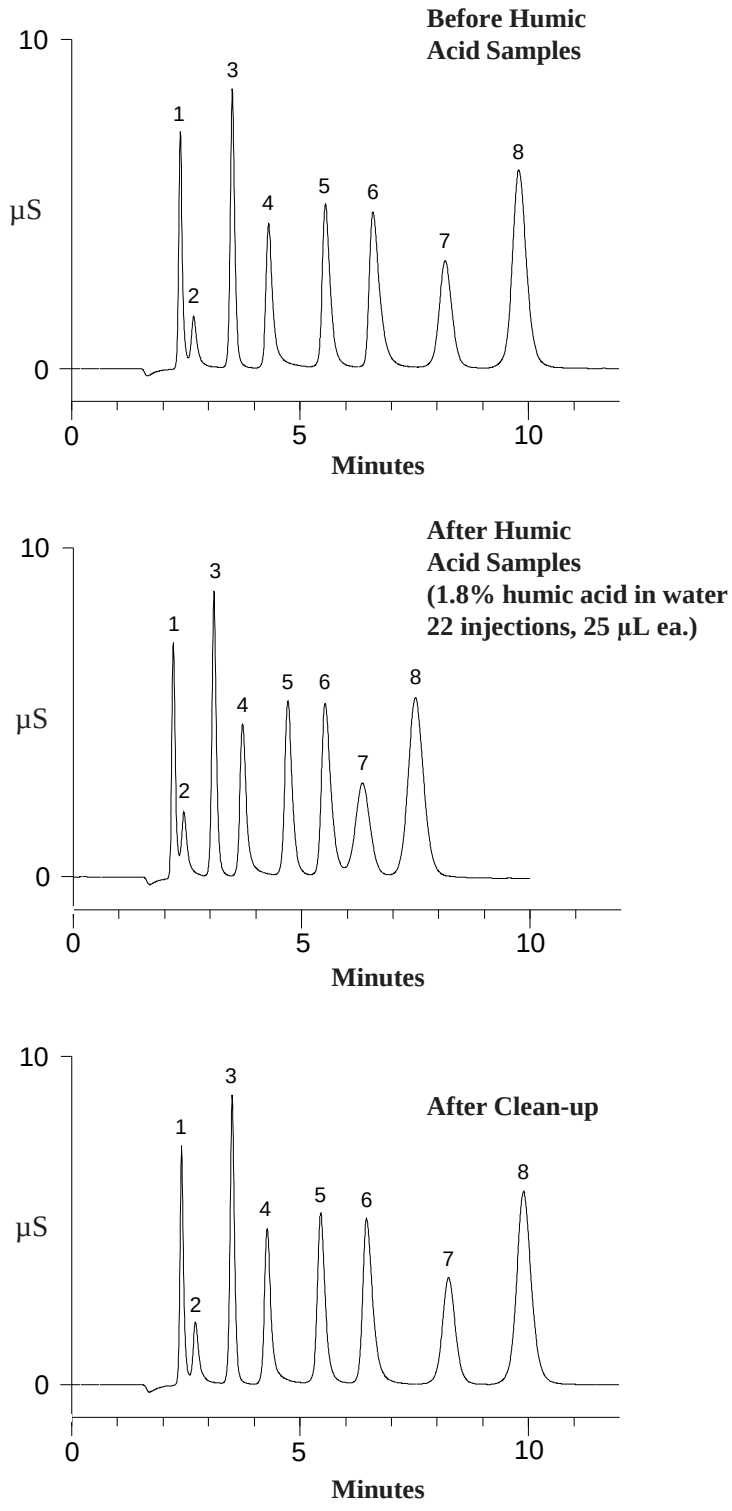


Figure 13
Clean-up after Humic Acid Samples

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using 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. If you can not solve the problem on your own, call the Dionex Office nearest you (see, "Dionex Worldwide Offices").

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	Component Manual
High Background Conductivity	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.2, Column Care
	Contaminated ASRS, AAES or AMMS	Clean Suppressor	6.2.4, Component Manual
	Contaminated Hardware	Clean Component	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
	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 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 psi (MPa)	Flow Rate 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	IonPac AS14 4-mm Columns (P/N)	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.

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

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

NOTE

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

6.2 High Background or Noise

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

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
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 1–3 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 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

A. AMMS and ASRS Suppressors

If the above items have been checked and the problem persists, the Anion MicroMembrane Suppressor is probably causing the problem.

1. **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.

2. **Check the eluent flow rate.** In general, the eluent flow rate for 4-mm applications, it should be 1.5 mL/min. Refer to the Anion MicroMembrane Suppressor Product Manual (Document No. 031727) for assistance in determining that the eluent is within suppressible limits.

3. **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.

a. 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.

b. 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.

B. Contaminated Anion Atlas Electrolytic Suppressor, AAES

Metal Contaminants or Precipitates

1. Turn off the power to the AAES.

2. 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.

3. 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.

4. 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.

5. 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.

NOTE

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.

6. Flush the AAES with deionized water at 2 mL/min for 30 minutes.

7. 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

1. Turn off the power to the AAES.

2. 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.

3. 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.

4. 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.

5. 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 H_2SO_4/90% acetonitrile. H_2SO_4/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 H_2SO_4 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.

NOTE

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.

6. Flush the AAES with deionized water at 2 mL/min for 30 minutes.

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

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 at least 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 AS14 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.

For DX-300 systems equipped with a Rheodyne Microinjection Valve, Model 9126 (Dionex P/N 044697), consult the accompanying manual for service instructions.