

## Errata

### ***Product Manual for Dionex IonPac™ AS22 and AG22 Columns***

065119-07

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

<b>Part</b>	<b>Old Part Number in this manual</b>	<b>Updated Part Number to use for new orders</b>
<i>PROD,COL,IP,UTAC-ULP2,5X23MM</i>	<i>072780</i>	<i>079918</i>
<i>PROD,COL,IP,AS22-FAST,4X150MM</i>	<i>072782</i>	<i>079936</i>
<i>PROD,COL,IP,AS22-FAST,2X150MM</i>	<i>072783</i>	<i>079937</i>



**Thermo Scientific**

**Dionex IonPac AS22 Fast**

**Product Manual**

**P/N: 065119-07      July 2012**

## **Product Manual**

**for**

### **IonPac AS22 Analytical Columns**

(4 x 250 mm, P/N 064141)  
(2 x 250 mm, P/N 064137)

### **IonPac AS22 Guard Columns**

(4 x 50 mm, P/N 064139)  
(2 x 50 mm, P/N 064135)

### **IonPac AS22-Fast Analytical Columns**

(4x150 mm, P/N 072782)  
(2x150 mm, P/N 072783)

### **IonPac AG22-Fast Guard Columns**

(4x30 mm, P/N 072784)  
(2x30 mm, P/N 072785)

© 2012 Thermo Fisher Scientific

Document No. 065119  
Revision 07  
July 2012

## TABLE OF CONTENTS

<b>SECTION 1 – INTRODUCTION TO IONPAC AS22/AG22/ AS22-Fast/AG22-Fast CHROMATOGRAPHY .....</b>	<b>4</b>
<b>SECTION 2 – INSTALLATION.....</b>	<b>7</b>
<b>2.1. System Requirements.....</b>	<b>7</b>
2.1.1. System Requirements for 2 mm Operation .....	7
2.1.2. System Requirements for 4 mm Operation .....	7
2.1.3. System Void Volume .....	7
<b>2.2. The Sample Concentrator.....</b>	<b>7</b>
<b>2.3. The Injection Loop .....</b>	<b>8</b>
2.3.1. The 2 mm System Injection Loop, 2 - 15 µL .....	8
2.3.2. The 4 mm System Injection Loop, 10 - 50 µL .....	8
<b>2.4. The IonPac AG22/AG22-Fast Guard Column.....</b>	<b>8</b>
<b>2.5. Eluent Storage.....</b>	<b>8</b>
<b>2.6. Anion Self-Regenerating Suppressor (ASRS 300) Requirements .....</b>	<b>8</b>
<b>2.7. Anion Atlas Electrolytic Suppressor (AAES) Requirements.....</b>	<b>9</b>
<b>2.8. Anion MicroMembrane Suppressor (AMMS 300) Requirements .....</b>	<b>9</b>
<b>2.9. Using AutoRegen with the ASRS 300 or the AMMS 300 in the Chemical Suppression Mode .....</b>	<b>9</b>
<b>2.10. Using Displacement Chemical Regeneration (DCR) with the Chemical Suppression Mode.....</b>	<b>9</b>
<b>2.11. Detector Requirements .....</b>	<b>9</b>
<b>SECTION 3 – OPERATION .....</b>	<b>10</b>
<b>3.1. General Operating Conditions .....</b>	<b>10</b>
<b>3.2. IonPac AS22/AS22-Fast Operation Precautions.....</b>	<b>10</b>
<b>3.3. Chemical Purity Requirements .....</b>	<b>10</b>
3.3.1. Inorganic Chemicals.....	10
3.3.2. Deionized Water.....	11
3.3.3. Solvents.....	11
<b>3.4. Making Eluents that Contain Solvents .....</b>	<b>12</b>
<b>3.5. Regenerant Preparation for the AMMS 300.....</b>	<b>12</b>

<b>SECTION 4 – EXAMPLE APPLICATIONS .....</b>	<b>13</b>
4.1. Preparation of Eluent Stock Solution Concentrates.....	13
4.2. Eluent Preparation .....	14
4.3. Production Test Chromatogram (AS22) .....	15
4.4. Production Test Chromatogram (AS22-Fast).....	16
4.5. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22-Fast).....	17
4.6. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22-Fast).....	18
4.7. Analysis of Municipal Drinking Water (AS22-Fast) .....	19
4.8. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22) .....	20
4.9. Effect of Temperature on the AS22 Selectivity .....	21
4.10. Separation of Inorganic Anions and Oxyhalides on the AS22.....	22
4.11. Analysis of Carbonated Water (AS22) .....	23
4.12. Analysis of Municipal Drinking Water (AS22).....	24
4.13. Determination of Trace Chloride and Sulfate in High Purity Water (AS22).....	25
4.14. Analysis of 18 Anions with Isocratic Elution Using a Carbonate Eluent System (AS22).....	26
4.15. Separation of Anions in Municipal Drinking Water Spiked with Surrogate Anions (AS22) .....	27
<b>SECTION 5 – TROUBLESHOOTING GUIDE .....</b>	<b>28</b>
5.1. High Back Pressure .....	29
5.1.1. Finding the Source of High System Pressure .....	29
5.1.2. Replacing Column Bed Support Assemblies .....	30
5.2. High Background or Noise .....	31
5.2.1. Preparation of Eluents .....	31
5.2.2. A Contaminated Guard or Analytical Column.....	31
5.2.3. Contaminated Hardware.....	31
5.2.4. A Contaminated Anion Self-Regenerating Suppressor, ASRS 300 .....	31
5.2.5. A Contaminated Anion MicroMembrane Suppressor, AMMS 300.....	32
5.2.6. A Contaminated Anion Atlas Electrolytic Suppressor, AAES.....	33
5.3. Poor Peak Resolution .....	34
5.3.1. Loss of Column Efficiency .....	34
5.3.2. Poor Resolution Due to Shortened Retention Times .....	34
5.3.3. Loss of Front End Resolution.....	35
5.3.4. Spurious Peaks .....	35
5.3.5. Poor Resolution of Only Phosphate and Sulfate .....	36
5.3.6. Poor Resolution of Only Fluoride and Acetate .....	36
<b>APPENDIX A - COLUMN CARE .....</b>	<b>37</b>
A.1 Recommended Operating Pressure .....	37
A.2 Column Start-Up .....	37
A.3 Column Storage .....	37
A.4 Column Cleanup.....	37
A.4.1 Choosing the Appropriate Cleanup Solution.....	38
A.4.2 Column Cleanup Procedure .....	38

## SECTION 1 – INTRODUCTION TO IONPAC AS22/AG22/ AS22-FAST/AG22-FAST CHROMATOGRAPHY

The IonPac AS22/AS22-Fast Analytical Column in combination with the AG22/AG22-Fast Guard Column is designed for the analysis of the common inorganic anions including Fluoride, Chloride, Nitrite, Bromide, Nitrate, Phosphate, and Sulfate. The selectivity of the IonPac AG22/AG22-Fast 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 including carbonate. The AS22/AS22-Fast is compatible with pH 0-14 eluents and eluents containing organic solvents from 0–100% in concentration. The AS22/AS22-Fast can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor 300 (ASRS<sup>®</sup> 300). The IonPac AS22/AS22-Fast has nominal efficiency for sulfate using standard operating conditions of at least 9,000 plates/column.

**Table 1**  
**IonPac AS22/AG22/AS22Fast/ASG22Fast Packing Specifications**

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
AS22 4 x 250 mm	65	55	210	Alkanol quaternary ammonium	Ultralow
AG22 4 x 50 mm	110	55	6	Alkanol quaternary ammonium	Ultralow
AS22 2 x 250 mm	65	55	525	Alkanol quaternary ammonium	Ultralow
AG22 2 x 50 mm	110	55	15	Alkanol quaternary ammonium	Ultralow
AS22-Fast 4 x 150 mm	6.5	55	126	Alkanol quaternary ammonium	Ultralow
AG22-Fast 4 x 30 mm	11	55	4	Alkanol quaternary ammonium	Ultralow
AS22-Fast 2 x 150 mm	6.5	55	31.5	Alkanol quaternary ammonium	Ultralow
AG22-Fast 2 x 30 mm	11	55	1	Alkanol quaternary ammonium	Ultralow

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

**Table 2**  
**AS22/AG22/AS22-Fast/AG22-Fast Operating Parameters**

<b>Column</b>	<b>Typical Back Pressure psi (MPa)</b>	<b>Standard Flow Rate mL/min</b>	<b>Maximum Flow Rate mL/min</b>
AS22 4 mm Analytical	≤ 1600 (11.03)	1.2	2.5
AG22 4 mm Guard	≤ 300 (2.07)	1.2	2.5
<b>AS22 + AG22 4 mm columns</b>	<b>≤ 1900 (13.10)</b>	<b>1.2</b>	<b>2.5</b>
AS22-Fast 4 mm Analytical	≤ 1600	1.2	2.5
AG22-Fast 4 mm Guard	≤ 300	1.2	2.5
<b>AS22-Fast + AG22-Fast 4 mm</b>	<b>≤ 1900</b>	<b>1.2</b>	<b>2.5</b>
AS22 2 mm Analytical	≤ 1600 (11.03)	0.3	0.63
AG22 2 mm Guard	≤ 300 (2.07)	0.3	0.63
<b>AS22 + AG22 2 mm columns</b>	<b>≤ 1900 (13.10)</b>	<b>0.3</b>	<b>0.63</b>
AS22-Fast 2 mm Analytical	≤ 1600	0.3	0.63
AG22-Fast 2 mm Guard	≤ 300	0.3	0.63
<b>AS22-Fast + AG22-Fast 2 mm</b>	<b>≤ 1900</b>	<b>0.3</b>	<b>0.63</b>

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."



**WARNING**

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

## ION CHROMATOGRAPHY SYSTEMS

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

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

See Appendix B, Comparison of Ion Chromatography Systems for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor (SRS), MicroMembrane Suppressor (MMS), injection loop, system void volume, detectors, and tubing back pressure.



## SECTION 2 – INSTALLATION

### 2.1. System Requirements

#### 2.1.1. System Requirements for 2 mm Operation

The IonPac AS22/AS22-Fast 2 mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a pump with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a microbore pump (1/16" pistons) is recommended.

#### 2.1.2. System Requirements for 4 mm Operation

The IonPac AS22/AS22-Fast 4 mm Guard and Analytical Columns are designed to run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a pump with a standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons).

#### 2.1.3. 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" ID PEEK tubing (P/N 044221). 0.010" ID PEEK tubing (P/N 042260) may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

### 2.2. The Sample Concentrator

For 2 mm or 4 mm concentrator work, use the IonPac AG22/AG22-Fast Guard Column when a single piston pump is used for sample delivery. Use the Trace Anion Concentrator Low Pressure Column (TAC-LP1, P/N 046026) or Trace Anion Concentrator Ultra Low Pressure Column (TAC-ULP1, P/N 061400) when the sample is delivered with a syringe or with an autosampler. Alternatively, use the Ultra Trace Anion Concentrator Low Pressure Column (UTAC-LP1, P/N 063079), Ultra Trace Anion Concentrator Ultra Low Pressure Column (UTAC-ULP1, P/N 063475), Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459), UTAC-LP2 (P/N 072799), UTAC-ULP2 (P/N 072780), UTAC-XLP2 (P/N 072781). The TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, UTAC-LP2, UTAC-ULP2, UTAC-XLP2 or the IonPac AG22/AG22-Fast Guard Column can be used for trace anion concentration work. 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 as this 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 function of the TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, UTAC-LP2, UTAC-ULP2, UTAC-XLP2 or the AG22/AG22-Fast 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, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, or the AG22/AG22-Fast leading to a lowering of detection limits by 2–5 orders of magnitude. The unique advantage to the analytical chemist of the TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, UTAC-LP2, UTAC-ULP2, UTAC-XLP2 or the AG22/AG22-Fast 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 Trace Anion Concentrator (TAC-LP1 and TAC-ULP1) Column Product Manual (Document No. 034972) or Section 3, "Operation," of the Ultra Trace Anion Concentrator (UTAC-LP1, UTAC-ULP1, and UTAC-XLP1) Column Product Manual (Document No. 065091).

## 2.3. The Injection Loop

### 2.3.1. The 2 mm System Injection Loop, 2 - 15 $\mu\text{L}$

For most applications on a 2 mm analytical system, a 2 - 15  $\mu\text{L}$  injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. The AS22/AS22-Fast 2 mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15  $\mu\text{L}$ ) of the loop volume used with a 4 mm analytical system (Section 2, "Comparison of Ion Chromatography Systems").

### 2.3.2. The 4 mm System Injection Loop, 10 - 50 $\mu\text{L}$

For most applications on a 4 mm analytical system, a 10 - 50  $\mu\text{L}$  injection loop is sufficient. Generally, you should not inject more than 50 nanomoles of any one analyte onto the 4 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity.

## 2.4. The IonPac AG22/AG22-Fast Guard Column

An IonPac AG22/AG22-Fast Guard Column is normally used with the IonPac AS22/AS22-Fast Analytical Column. Retention times will increase by approximately 4% 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 AG22/AG22-Fast Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS22/AS22-Fast Analytical Column.

## 2.5. Eluent Storage

IonPac AS22/AS22-Fast 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).

## 2.6. Anion Self-Regenerating Suppressor (ASRS 300) 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 300 modes of operation.



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

### NOTE

If you are installing an IonPac AS22/AS22-Fast 4 mm Analytical Column, use an ASRS 300 (4 mm, P/N 064554).  
If you are installing an IonPac AS22/AS22-Fast 2 mm Analytical Column, use an ASRS 300 (2 mm, P/N 064555).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see Document No. 031956, the Product Manual for the Anion Self-Regenerating Suppressor 300 (ASRS 300.)"

## 2.7. Anion Atlas Electrolytic Suppressor (AAES) Requirements

An Atlas Anion Electrolytic Suppressor (AAES) may be used instead of an ASRS 300 for applications that require suppressed conductivity detection. The AAES (P/N 056116) can be used for AS22/AS22-Fast 2 mm and 4 mm 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 Anion Atlas Electrolytic Suppressor.”

## 2.8. Anion MicroMembrane Suppressor (AMMS 300) Requirements

An Anion MicroMembrane Suppressor (AMMS 300) may be used instead of an ASRS 300 (4 mm) for applications that require suppressed conductivity detection. Use an AMMS 300 (P/N 064558) with the IonPac AS22/AS22-Fast 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 300 (P/N 064559).

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

## 2.9. Using AutoRegen with the ASRS 300 or the AMMS 300 in the Chemical Suppression Mode

To save regenerant preparation time and reduce regenerant consumption and waste, Dionex recommends using an AutoRegen<sup>®</sup> 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).

## 2.10. 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 (H<sub>2</sub>SO<sub>4</sub>) and the Anion MicroMembrane Suppressor (AMMS 300). See the DCR kit manual, Document P/N 031664, for details.



*Use proper safety precautions in handling acids and bases.*

### SAFETY

## 2.11. Detector Requirements

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

## SECTION 3 – OPERATION

### 3.1. 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: AS22 2 mm Analytical Column + AG22 2 mm Guard Column 4 mm: AS22 4 mm Analytical Column + AG22 4 mm Guard Column 2 mm: AS22-Fast 2 mm Analytical Column + AG22-Fast 2 mm Guard Column 4 mm: AS22-Fast 4 mm Analytical Column + AG22-Fast 4 mm Guard Column
Eluent:	4.5 mM Na <sub>2</sub> CO <sub>3</sub> /1.4 mM NaHCO <sub>3</sub>
Temperature:	30 °C
Eluent Flow Rate:	2 mm: 0.30 mL/min 4 mm: 1.2 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 (2 mm or 4 mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300 (2 mm or 4 mm)
MMS Regenerant:	50 mN H <sub>2</sub> SO <sub>4</sub>
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	20-23 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

### 3.2. IonPac AS22/AS22-Fast Operation Precautions



**CAUTION**

*Filter and Degas Eluents*

*Filter Samples*

*Eluent pH between 0 and 14*

*Sample pH between 0 and 14*

*0.63 mL/min Maximum Flow Rate for 2 mm Columns*

*2.5 mL/min Maximum Flow Rate for 4 mm Columns*

*Maximum Operating Pressure = 3,000 psi (20.68 MPa)*

### 3.3. 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.

#### 3.3.1. Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label. Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex AS22/AS22-Fast Eluent Concentrate (P/N 063965) is recommended in order to avoid this problem. Otherwise, use of a high purity grade of sodium carbonate to prepare eluents will generally prevent the problem. We recommend EMD Chemicals sodium carbonate (P/N SX0395) for this purpose. Do not dry sodium carbonate at excessive temperatures (> 110°C) as this will increase the pH of the salt.

### 3.3.2. 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  $\mu\text{m}$ . 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.

### 3.3.3. Solvents

Solvents can be added to the ionic eluents used with IonPac AS22/AS22-Fast columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima<sup>®</sup> 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 AS22/AS22-Fast columns is 3,000 psi (20.68 MPa).

The AS22/AS22-Fast 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 3**  
**HPLC Solvents for Use with IonPac AS22/AS22-Fast 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 300) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.*

### 3.4. 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.



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

**NOTE**



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

**NOTE**



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

**NOTE**



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

**SAFETY**

### 3.5. Regenerant Preparation for the AMMS 300

The Anion MicroMembrane Suppressor (AMMS 300) requires the use of a regenerant solution. If you are using the AMMS 300 instead of the Anion Self-Regenerating Suppressor 300 (ASRS 300) see Document No. 031727, the “Product Manual for the Anion MicroMembrane Suppressor, the AMMS 300.”

## SECTION 4 – 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 3.2, “Sample Concentration”).

### 4.1. Preparation of Eluent Stock Solution Concentrates

- A. AS22/AS22-Fast Sodium Carbonate/Bicarbonate Eluent Concentrate (0.45 M Na<sub>2</sub>CO<sub>3</sub>/0.14 M NaHCO<sub>3</sub>)

**Order DIONEX P/N 063965**

or

Thoroughly dissolve 47.7 g of sodium carbonate (MW 106.00 g/mole) plus 11.76 g sodium bicarbonate (MW 84.00 g/mole) in 700 L of deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

- B. 0.5 M Sodium Carbonate (Na<sub>2</sub>CO<sub>3</sub>) Concentrate

**Order Dionex P/N 037162**

or

Thoroughly dissolve 26.49 g of Na<sub>2</sub>CO<sub>3</sub> in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex 0.5 molar Sodium Carbonate Concentrate is recommended in order to avoid this problem. Otherwise, use of a high purity grade of sodium carbonate to prepare eluents will generally prevent the problem. We recommend EMD Chemicals sodium carbonate (P/N SX0395) for this purpose. Do not dry sodium carbonate at excessive temperatures (> 110°C) as this will increase the pH of the salt.

- C. 0.5 M Sodium Bicarbonate (NaHCO<sub>3</sub>) Concentrate

**Order Dionex P/N 037163**

or

Thoroughly dissolve 21.00 g of NaHCO<sub>3</sub> in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

## 4.2. Eluent Preparation

### Eluent: 4.5 mM Sodium Carbonate/1.4 mM Sodium bicarbonate

A. Using AS22/AS22-Fast Eluent Concentrate

By Weight: Weigh 988.0 g of deionized water and add 10.5 g of the AS22/AS22-Fast Eluent Concentrate.

By Volume: To make 1 liter of eluent, pipette 10 mL of the AS22/AS22-Fast Eluent Concentrate into a 1 L volumetric flask and dilute to a final volume of 1 L using deionized water.

B. Using 0.5 M Na<sub>2</sub>CO<sub>3</sub> and 0.5 M NaHCO<sub>3</sub> Concentrates

By Weight: Weigh 986.2 g of deionized water and add 9.45 g of 0.5 M Na<sub>2</sub>CO<sub>3</sub> plus 2.94 g of 0.5 M NaHCO<sub>3</sub>.

By Volume: Prepare the eluent by pipetting 9.0 mL of 0.5 M Na<sub>2</sub>CO<sub>3</sub> plus 2.8 mL of 0.5 M NaHCO<sub>3</sub> 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.



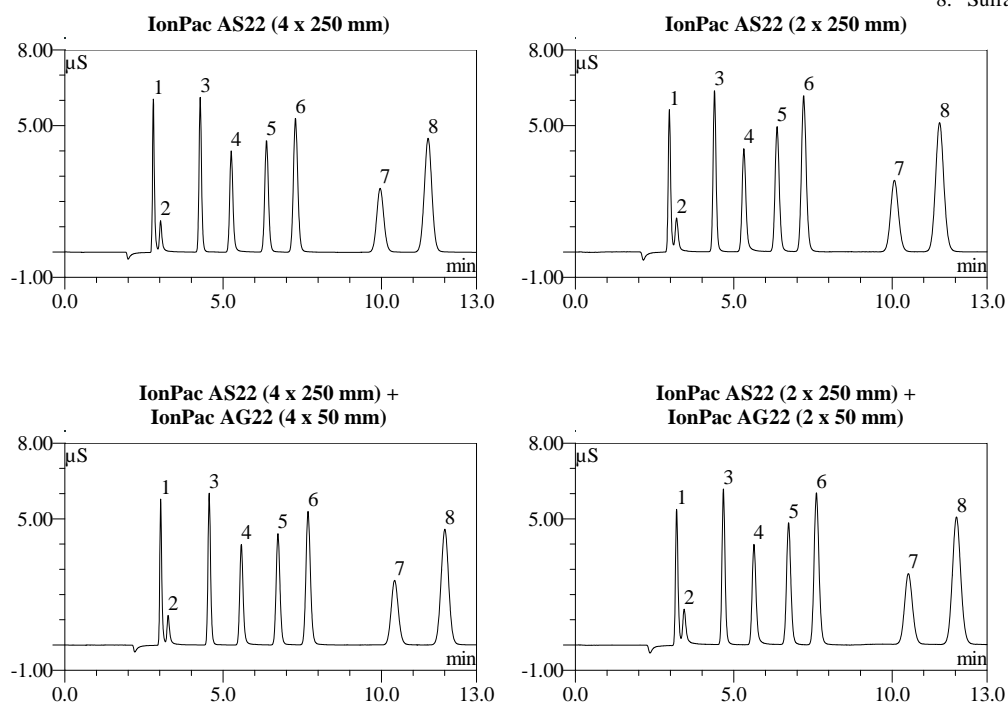
### 4.3. Production Test Chromatogram (AS22)

Isocratic elution of anions on the IonPac AS22 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 AS22 Analytical Column should always be used with the IonPac AG22 Guard Column. To guarantee that all IonPac AS22 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Column:	See Chromatogram	
Eluent:	4.5 mM Na <sub>2</sub> CO <sub>3</sub> /1.4 mM NaHCO <sub>3</sub>	
Eluent Flow Rate:	1.2 mL/min (4 mm) 0.3 mL/min (2 mm)	
Temperature:	30 °C	
Injection Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume 2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume	
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 (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 300)	
MMS Regenerant:	50 mN H <sub>2</sub> SO <sub>4</sub>	
Expected Background Conductivity:	20-23µS	
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate	
Short-term Storage Solution (< 1 week):	Eluent	

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



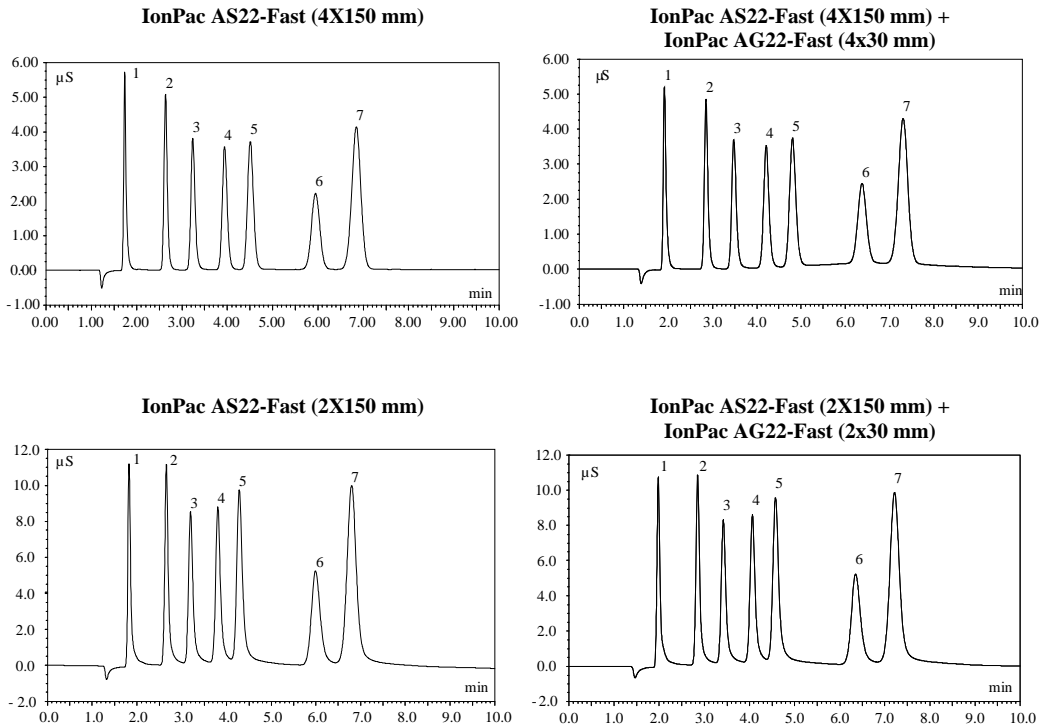
**Figure 1**  
**IonPac AS22 Production Test Chromatograms**

### 4.4. Production Test Chromatogram (AS22-Fast)

Isocratic elution of anions on the IonPac AS22-Fast Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. Fluoride is resolved from acetate and formate. The IonPac AS22-Fast Analytical Column should always be used with the IonPac AG22 Guard Column. To guarantee that all IonPac AS22-Fast Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. **Note that the run time for the IonPac AS22-Fast column under these conditions would be 40% lower than the run time for the IonPac AS22 column.**

Column: See Chromatogram  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>/1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: 1.2 mL/min (4 mm)  
 0.3 mL/min (2 mm)  
 Temperature: 30 °C  
 Injection Volume: 4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume  
 2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23µS  
 Long-term Storage Solution (> 1 week): 100 mM Sodium Bicarbonate  
 Short-term Storage Solution (< 1 week): Eluent

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



**Figure 2**  
**IonPac AS22-Fast Production Test Chromatograms**

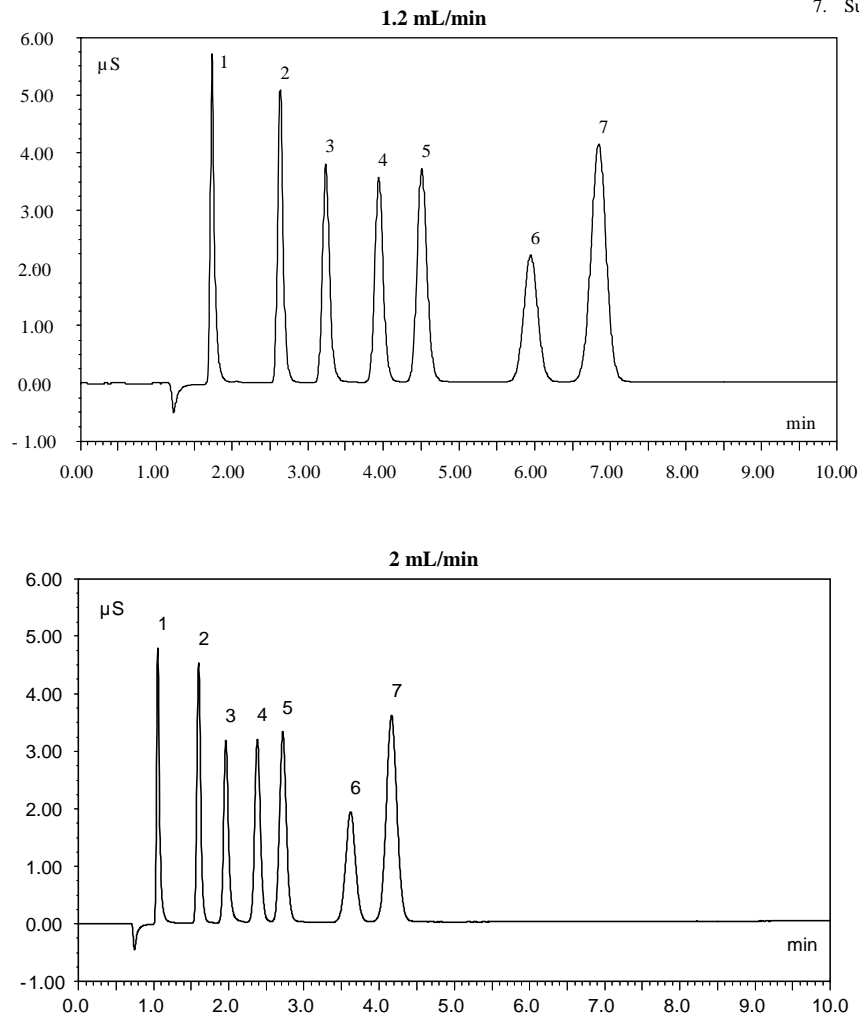
#### 4.5. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22-Fast)

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions.

*Note, the maximum flow rate for the various formats for the IonPac AS22-Fast is listed in Table 2.*

Column: IonPac AS22-Fast (4 x 150 mm) Analytical column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: See Chromatogram  
 Temperature: 30 °C  
 Injection Volume: 10 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (2 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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23 µS

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



**Figure 3**  
**Fast Run Analysis Without Changes in Selectivity (AS22-Fast)**

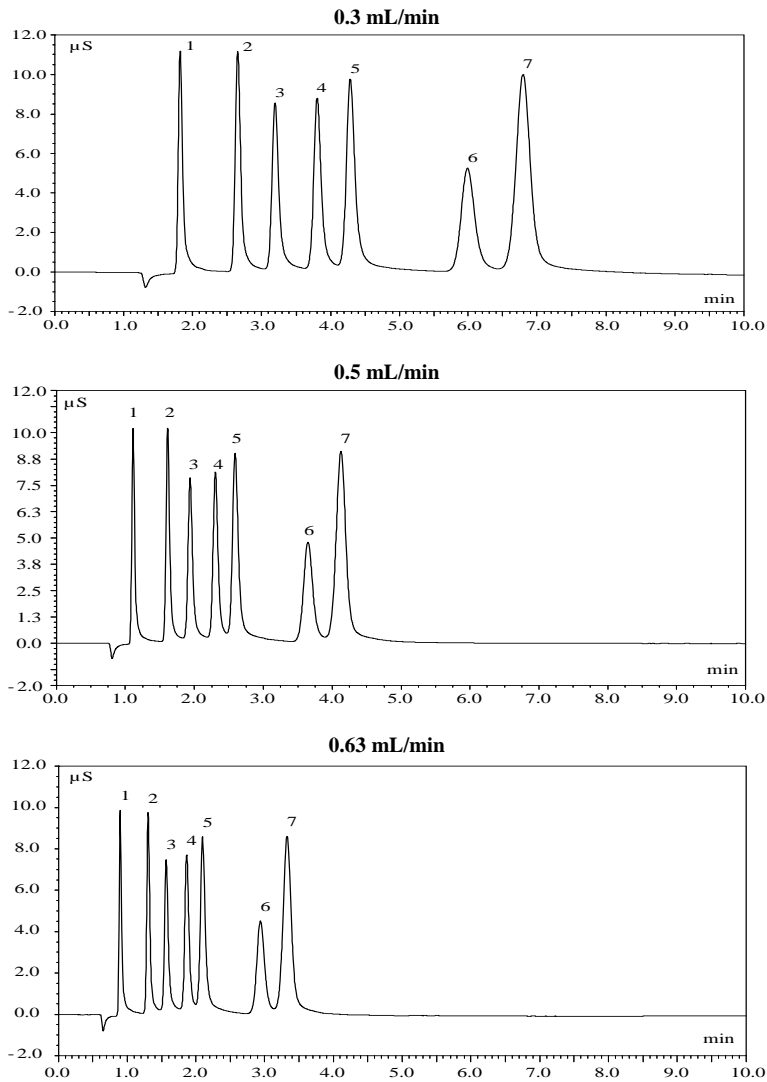
### 4.6. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22-Fast)

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions.

**Note, the maximum flow rate for the various formats for the IonPac AS22-Fast is listed in Table 2.**

Column: IonPac AS22-Fast (2 x 150 mm) Analytical column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: See Chromatogram  
 Temperature: 30 °C  
 Injection Volume: 10 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (2 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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23 µS

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

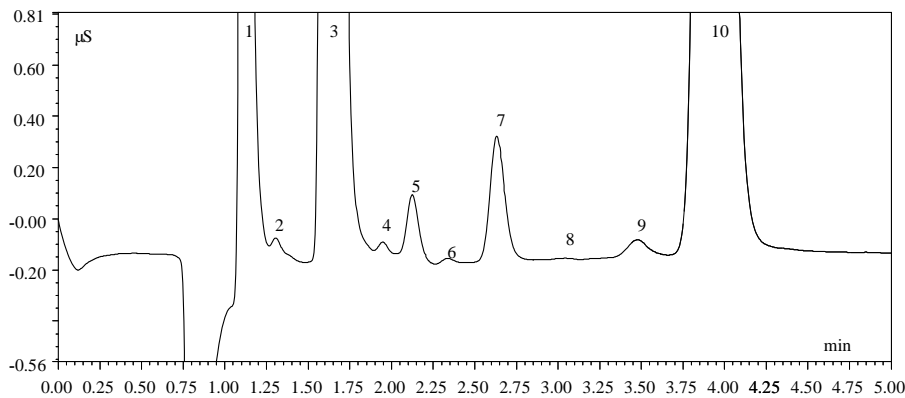
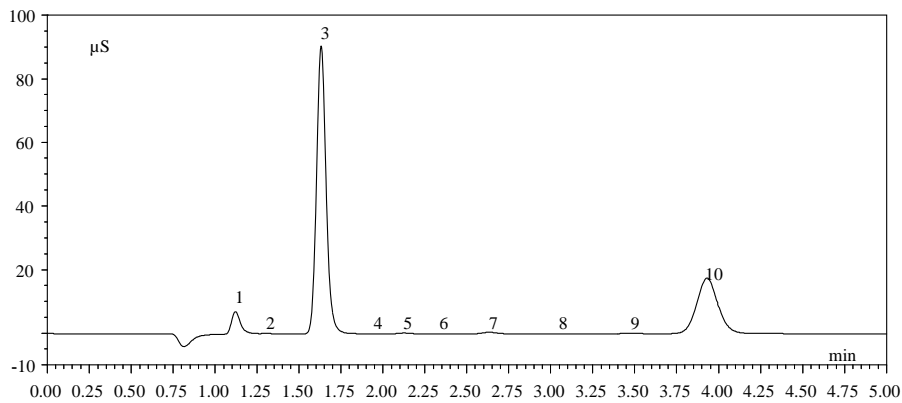


**Figure 4**  
**Fast Run Analysis Without Changes in Selectivity (AS22-Fast)**

### 4.7. Analysis of Municipal Drinking Water (AS22-Fast)

Column: IonPac AS22-Fast (4 x 150 mm) Analytical column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: 2 mL/min  
 Temperature: 30 °C  
 Injection Volume: 100 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23 µS

- | Analyte       | mg/L (ppm) |
|---------------|------------|
| 1. Fluoride   |            |
| 2. Formate    |            |
| 3. Chloride   |            |
| 4. Nitrite    |            |
| 5. Unknown    |            |
| 6. Chlorate   |            |
| 7. Bromide    |            |
| 8. Nitrate    |            |
| 9. Carbonate  |            |
| 10. Phosphate |            |



**Figure 5**  
**Analysis of Municipal Drinking Water (AS22-Fast)**

#### 4.8. Fast Analysis of the Common Inorganic Anions using an Optimized Flow Rate (AS22)

The following chromatograms demonstrate the use of a high flow rate for the fast analysis of the common inorganic anions.

Column: IonPac AS22 (2 x 250 mm) Analytical column  
IonPac AG22 (2 x 50 mm) Guard column

Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
1.4 mM NaHCO<sub>3</sub>

Eluent Flow Rate: See Chromatogram

Temperature: 30 °C

Injection Volume: 2.5 µL

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (2 mm)  
AutoSuppression Recycle Mode

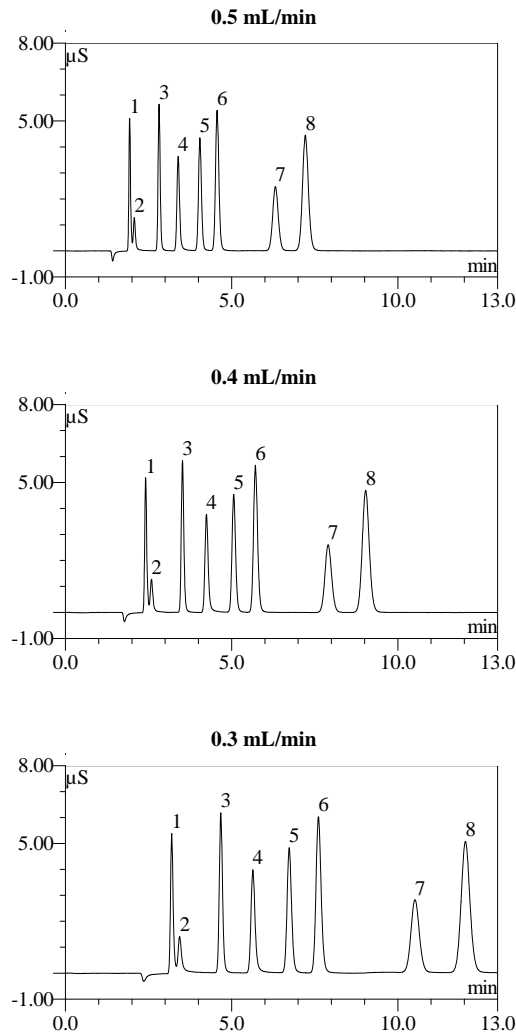
or AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES,  
(if eluent suppression required is less than 25 µeq/min)

or MMS Suppressor: Anion MicroMembrane Suppressor (AMMS 300)

MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

Expected Background Conductivity: 20-23 µS

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



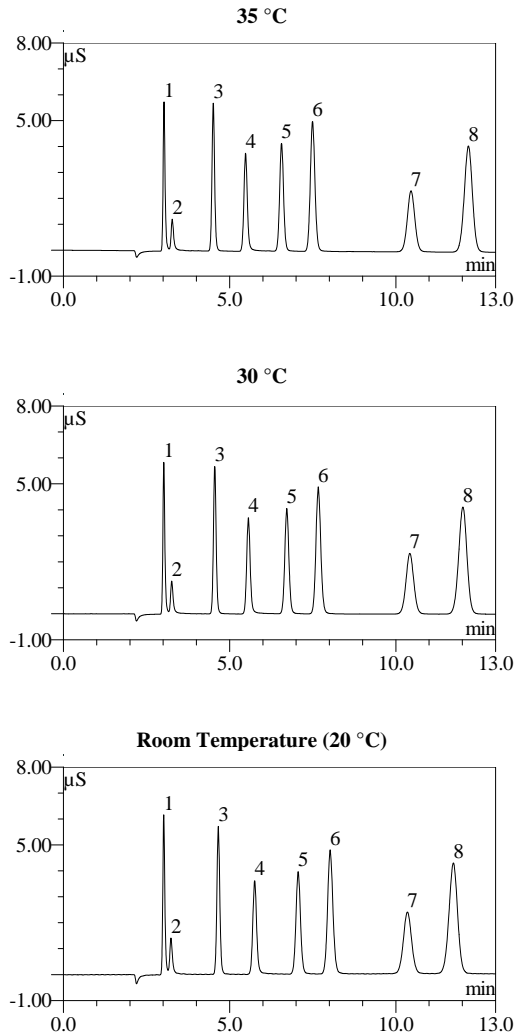
**Figure 6**  
Fast Run Analysis Without Changes in Selectivity (AS22)

### 4.9. Effect of Temperature on the AS22 Selectivity

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

Column: IonPac AS22 (4 x 250 mm) Analytical column  
 IonPac AG22 (4 x 50 mm) Guard column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: 1.2 mL/min  
 Temperature: See Chromatogram  
 Injection Volume: 10 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23 µS

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



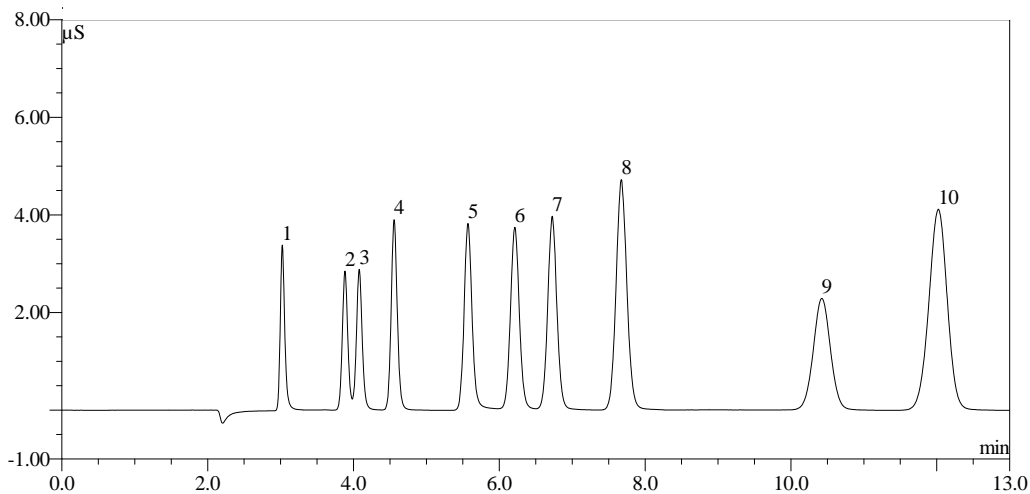
**Figure 7**  
**Effect of Temperature in AS22 Selectivity**

## 4.10. Separation of Inorganic Anions and Oxyhalides on the AS22

The following chromatograms demonstrate the separation of the common inorganic anions and oxyhalides on the AS22 column.

Column: IonPac AS22 (4 x 250 mm) Analytical column  
IonPac AG22 (4 x 50 mm) Guard column  
Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>/1.4 mM NaHCO<sub>3</sub>  
Eluent Flow Rate: 1.2 mL/min  
Temperature: 30 °C  
Injection Volume: 10 µL  
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
Expected Background Conductivity: 20-23 µS

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



**Figure 8**  
**Separation of Inorganic Anion and Oxyhalides on AS22**



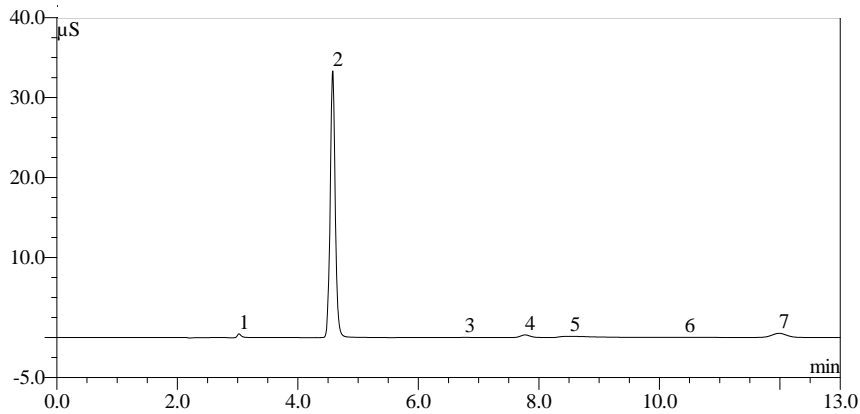
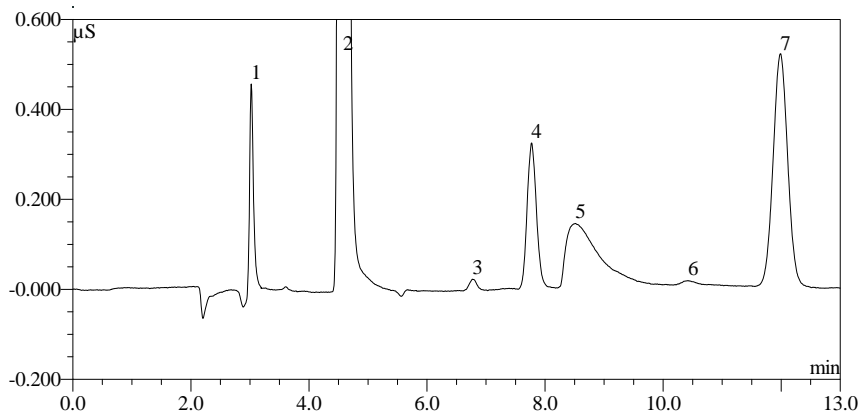
### 4.11. Analysis of Carbonated Water (AS22)

The following chromatograms demonstrate the excellent separation of carbonate from common inorganic anions. The carbonated water sample can be analyzed after a simple 15 to 20 minute sonication.

Column: IonPac AS22 (4 x 250 mm) Analytical column  
 IonPac AG22 (4 x 50 mm) Guard column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: 1.2 mL/min  
 Temperature: 30 °C  
 Injection Volume: 10 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

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

NQ=NotQuantified



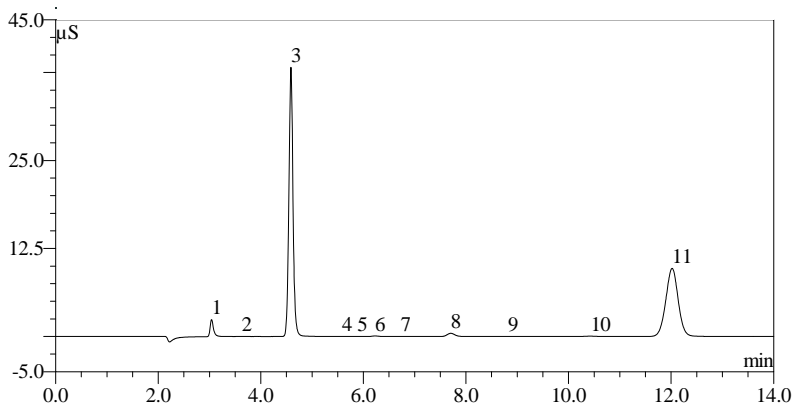
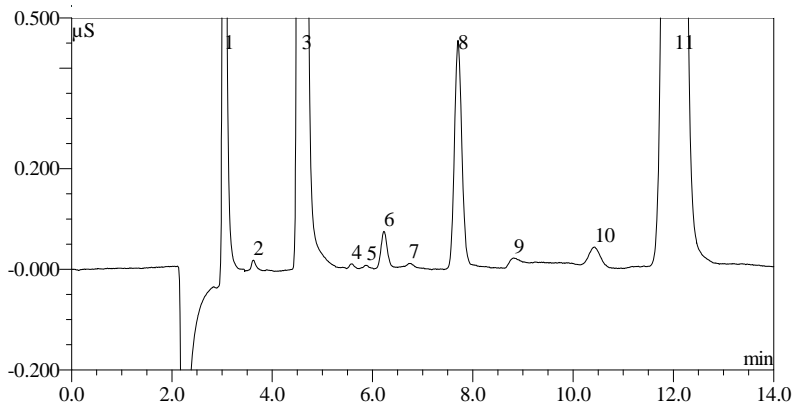
**Figure 9**  
**Analysis of Carbonated Water (AS22)**

### 4.12. Analysis of Municipal Drinking Water (AS22)

Column: IonPac AS22 (4 x 250 mm) Analytical column  
 IonPac AG22 (4 x 50 mm) Guard column  
 Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
 1.4 mM NaHCO<sub>3</sub>  
 Eluent Flow Rate: 1.2 mL/min  
 Temperature: 30 °C  
 Injection Volume: 25 µL  
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (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 300)  
 MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>  
 Expected Background Conductivity: 20-23 µS

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

NQ=NotQuantified



**Figure 10**  
**Analysis of Municipal Drinking Water (AS22)**

### 4.13. Determination of Trace Chloride and Sulfate in High Purity Water (AS22)

The following chromatograms demonstrate the determination of trace chloride and sulfate in high levels of carbonate using an AS22 column. The carbonate is present as a negative peak because the carbonate concentration in the sample is lower than the carbonate concentration in the eluent.

Column: IonPac AS22 (4 x 250 mm) Analytical column  
IonPac AG22 (4 x 50 mm) Guard column

Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>  
1.4 mM NaHCO<sub>3</sub>

Eluent Flow Rate: 1.0 mL/min

Temperature: 30 °C

Injection Volume: 2 mL

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4 mm)  
AutoSuppression Recycle Mode

or AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES,  
(if eluent suppression required is less than 25 µeq/min)

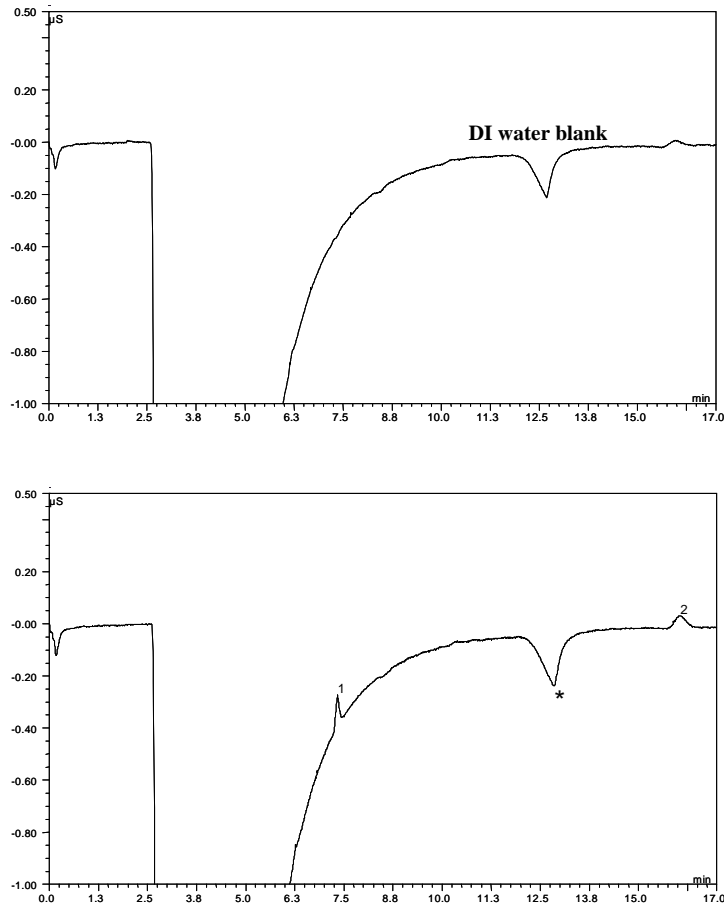
or MMS Suppressor: Anion MicroMembrane Suppressor (AMMS 300)

MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

Expected Background Conductivity: 20-23 µS

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

\* 500 ppb Carbonate



**Figure 11**  
**Determination of trace Chloride and Sulfate in High Purity Water (AS22)**

#### 4.14. Analysis of 18 Anions with Isocratic Elution Using a Carbonate Eluent System (AS22)

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

Column: IonPac AS22 (4 x 250 mm) Analytical column  
IonPac AG22 (4 x 50 mm) Guard column

Eluent: 4.8 mM Na<sub>2</sub>CO<sub>3</sub>  
1.0 mM NaHCO<sub>3</sub>

Eluent Flow Rate: 1.5 mL/min

Temperature: 30 °C

Injection Volume: 10 µL

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4 mm)  
AutoSuppression Recycle Mode

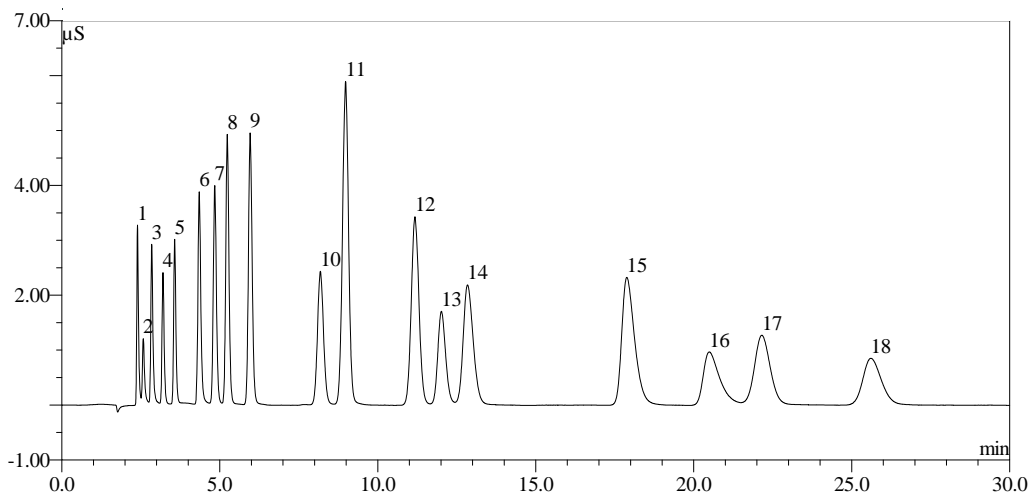
or AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES,  
(if eluent suppression required is less than 25 µeq/min)

or MMS Suppressor: Anion MicroMembrane Suppressor (AMMS 300)

MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

Expected Background Conductivity: 20-23 µS

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

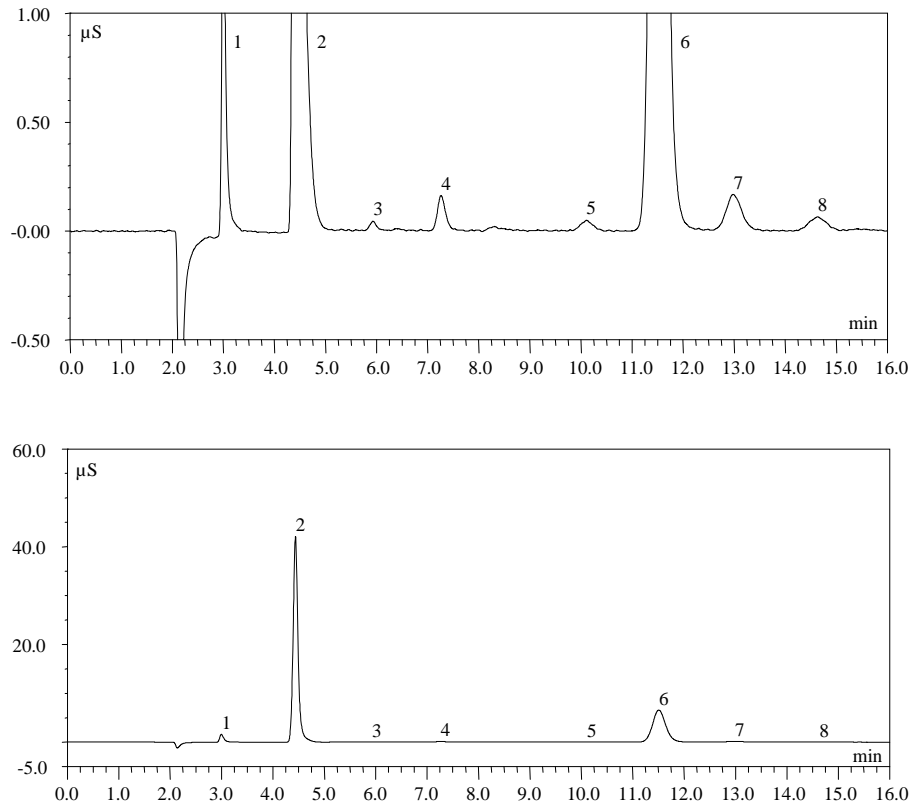


**Figure 12**  
Analysis of 18 Anions (AS22)

### 4.15. Separation of Anions in Municipal Drinking Water Spiked with Surrogate Anions (AS22)

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

Column:	IonPac AS22 (4 x 250 mm) Analytical column IonPac AG22 (4 x 50 mm) Guard column	
Eluent:	4.5 mM Na <sub>2</sub> CO <sub>3</sub> 1.4 mM NaHCO <sub>3</sub>	
Eluent Flow Rate:	1.2 mL/min	
Detection:	Conductivity	
Temperature:	30 °C	
Injection Volume:	25 µL	
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 (4 mm) AutoSuppression Recycle Mode	<b>Peaks:</b> <b>mg/mL</b>
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, (if eluent suppression required is less than 25 µeq/min)	1. Fluoride 0.7
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS 300)	2. Chloride 25.7
MMS Regenerant:	50 mN H <sub>2</sub> SO <sub>4</sub>	3. Chlorate 0.1
Expected Background Conductivity:	20-23 µS	4. Nitrate 0.4
		5. Phosphate 0.4
		6. Sulfate 17.8
		7. Malonate 2.0
		8. Succinate 2.0



**Figure 13**  
**Analysis of Municipal Drinking Water (AS22)**

## SECTION 5 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS22/AS22-Fast columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the Dionex Office nearest you (see, "Dionex Worldwide Offices").

**Table 4**  
**AS22/AG22/AS22-Fast/AG22-Fast Troubleshooting Summary**

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed	Replace Bed Supports	6.1.2
	Supports		
High Background Conductivity	Other System Components	Unplug, Replace	Component Manual
	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.2, 7.4
	Contaminated ASRS 300, AAES or AMMS 300	Clean Suppressor	6.2.4, Component Manual
Poor Resolution	Contaminated Hardware	Clean Component	Component Manual
	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.1.A, Component Manual
Poor Resolution of Only Phosphate and Sulfate	Column Headspace	Replace Column	6.3.1B
	Sodium Carbonate Contaminated with Sodium Hydroxide, Inadequate Equilibration after Use of an Alkaline Buffer, Sodium Carbonate Dried at Temperatures >110°C	Use Dionex 0.5 M Sodium Carbonate (P/N 037162), Dry Sodium Carbonate at Lower Temperature	6.3.5
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, 7.4
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, 7.4
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual

## 5.1. High Back Pressure

### 5.1.1. Finding the Source of High System Pressure

Total system pressure for the IonPac AG22/AG22-Fast (4 mm) Guard Column plus the AS22/AS22-Fast (4 mm) Analytical Column when using the test chromatogram conditions should be equal or less than 1900 psi. If the system pressure is higher than 1900 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 5, "Typical AS22/AG22/AS22-Fast/AG22-Fast Operating Back Pressures").  
The Anion Self-Regenerating Suppressor 300 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 5**  
**Typical AS22/AG22/AS22-Fast/AG22-Fast Operating Back Pressures**

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS22 4 mm Analytical	≤ 1600 (11.03)	1.2
AG22 4 mm Guard	≤ 300 (2.07)	1.2
<b>AS22 + AG22 4 mm columns</b>	<b>1900 (13.10)</b>	<b>1.2</b>
AS22 2 mm Analytical	≤ 1600 (11.03)	0.30
AG22 2 mm Guard	≤ 300 (2.07)	0.30
<b>AS22 + AG22 2 mm columns</b>	<b>1900 (13.10)</b>	<b>0.30</b>
AS22-Fast 4 mm Analytical	≤ 1600	1.2
AG22-Fast 4 mm Guard	≤ 300	1.2
<b>AS22-Fast + AG22-Fast 4 mm</b>	<b>≤ 1900</b>	<b>1.2</b>
AS22-Fast 2 mm Analytical	≤ 1600	0.3
AG22-Fast 2 mm Guard	≤ 300	0.3
<b>AS22-Fast + AG22-Fast 2 mm</b>	<b>≤ 1900</b>	<b>0.3</b>

### 5.1.2. 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 AS22 4 mm Columns (P/N)	IonPac AS22 2 mm Columns (P/N)
Analytical Column	064141	064137
Guard Column	064139	064135
Bed Support Assembly	042955	044689
End Fitting	052809	043278

	IonPac AS22-Fast 4 mm columns (P/N)	IonPac AS22-Fast 2 mm columns (P/N)
Analytical column	072782	072783
Guard Column	072784	072785
Bedsupport assembly	042955	044689
Endfitting	052809	043278



#### CAUTION

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

- E. **Screw the end fitting back onto the column.** Tighten it finger-tight, then an additional 1/4 turn (25 in 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.*



## 5.2. High Background or Noise

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

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
4.5 mM Na <sub>2</sub> CO <sub>3</sub> /1.4 mM NaHCO <sub>3</sub>	20 - 23 µS

### 5.2.1. Preparation of Eluents

- Make sure that the eluents and the regenerant are made correctly.
- Make sure that the eluents are made from chemicals with the recommended purity.
- Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

### 5.2.2. A Contaminated Guard or Analytical Column

Remove the IonPac AG22/AG22-Fast Guard and AS22/AS22-Fast 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 AG22/AG22-Fast 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").

### 5.2.3. Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the columns and the Anion Self-Regenerating 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.

### 5.2.4. A Contaminated Anion Self-Regenerating Suppressor, ASRS 300

This section describes routine cleanup procedures for the Anion Self-Regenerating Suppressors (ASRS 300) 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 300 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



#### NOTE

*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."*

- Turn off the SRS Control unit.
- Disconnect the analytical (and guard) column(s) from the injection valve and the ASRS 300. 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 300 **REGEN IN** port.
- D. Disconnect the liquid line from the ASRS 300 **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 300 (4 mm) at 1-2 mL/min for 30 minutes. For 2 mm systems pump this solution through the ASRS 300 (2 mm) at 0.25-0.50 mL/min for 30 minutes.

**NOTE**

*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 300 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 300 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.

### 5.2.5. A Contaminated Anion MicroMembrane Suppressor, AMMS 300

- 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 1.2 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.

## 5.2.6. 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.



#### 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.*

- 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 H<sub>2</sub>SO<sub>4</sub>/90% acetonitrile. H<sub>2</sub>SO<sub>4</sub>/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<sub>2</sub>SO<sub>4</sub> 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.*

- 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.

### 5.3. Poor Peak Resolution

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

#### 5.3.1. 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.

#### 5.3.2. 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 North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

### 5.3.3. 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.

### 5.3.4. 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 AS22/AS22-Fast columns, contact the North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or 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 re-torqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.  
For DX-300 systems equipped with a Rheodyne Microinjection Valve, Model 9126 (Dionex P/N 044697), consult the accompanying manual for service instructions.

### 5.3.5. Poor Resolution of Only Phosphate and Sulfate

#### A. Causes

1. Sodium carbonate is contaminated with sodium hydroxide,
2. Inadequate equilibration after use of an alkaline buffer or hydroxide eluent,
3. Sodium carbonate was dried at temperatures  $> 110^{\circ}\text{C}$ .

#### B. Action

1. Use Dionex AS22/AS22-Fast Eluent Concentrate (P/N 063965).
2. Use a high purity sodium carbonate salt.
3. Dry the sodium carbonate at a lower temperature. See section 4.3.1 and section 5.1.

### 5.3.6. Poor Resolution of Only Fluoride and Acetate

#### A. Causes

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

#### B. Action

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

## APPENDIX A - COLUMN CARE

### A.1 Recommended Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS22/AS22-Fast columns is 3,000 psi (20.68 MPa).

### A.2 Column Start-Up

The column is shipped using the column test 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.

### A.3 Column Storage

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

### A.4 Column Cleanup

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



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

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

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

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

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

### A.4.1 Choosing the Appropriate Cleanup Solution

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

### A.4.2 Column Cleanup Procedure

Use the following cleanup procedures to clean the AG22/AG22-Fast and AS22/AS22-Fast.

- Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in, "Choosing the Appropriate Cleanup Solution".
- Disconnect the ASRS 300, AAES, or AMMS 300 from the IonPac AS22/AS22-Fast 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. If not, the contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.*

- Set the pump flow rate to 1.0 mL/min for an AS22/AS22-Fast 4 mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS22/AS22-Fast 2 mm Analytical or Guard Column.
- Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.
- Pump the cleanup solution through the column for at least 60 minutes.
- Rinse the column for 10 minutes with deionized water before pumping eluent over the column.
- Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.
- Reconnect the ASRS 300, AAES, or AMMS 300 to the AS22/AS22-Fast Analytical Column
- Place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.



**Table A1  
Configuration**

CONFIGURATION	2 mm	4 mm
Eluent Flow Rate	0.30 mL/min	1.20 mL/min
SRS Suppressor	ASRS 300 (2 mm) (P/N 064555)	ASRS 300 (4 mm) (P/N 064554)
MMS Suppressor	AMMS 300 (2 mm) (P/N 064559)	AMMS 300 (4 mm) (P/N 064558)
AAE Suppressor	AAES (P/N 056116)	AAES (P/N 056116)
Injection Loop	2 - 15 $\mu$ L Rheodyne Microinjection Valve (P/N 044697) for full loop injections <15 $\mu$ L.	10-50 $\mu$ L
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the 2 mm GM-4 Mixer (P/N 049135).	Minimize dead volume. Switching valves, couplers can be used. Use the GM-2 , GM-3 or recommended gradient mixers.
Pumps	Use the DP/SP/GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2 mm) Gradient Mixer.  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 DP/SP/GP40/GP50/IP20/IP25 in Standard-Bore Configuration.  The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP50. Note: The GP40 has an active mixer.
Detectors	AD20 Cell (6 mm, 7.5 $\mu$ L, P/N 046423)  VDM-2 Cell (3 mm, 2.0 $\mu$ L) (P/N 043120)  DC/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)  Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-2 (P/N 043117) is optimized for 2 mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi	AD25 Cell (10 mm, 9 $\mu$ L, P/N 049393)  VDM-2 Cell (6 mm, 10 $\mu$ L) (P/N 043113)  DC/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)  Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-1 or TS-2 (P/N 043117) is optimized for 4 mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi

**Table A2**  
**Tubing Back Pressures**

Color	Dionex P/N	ID Inches	ID cm	Volume $\mu\text{L}/\text{cm}$	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	4.560	0.086	0.021	0.003
Orange	042855	0.020	0.051	2.027	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.856	2.437	0.609	0.081
Black	042690	0.010	0.025	0.507	6.960	1.740	0.232
Red	044221	0.005	0.013	0.127	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.046	859.259	214.815	28.642