

# **Errata**

**Product Manual for Dionex IonPac™ AS9-HC and AG9-HC Columns** 031267-10

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



# PRODUCT MANUAL

# for the

# IONPAC® AG9-HC GUARD COLUMN

(4 x 50 mm, P/N 051791) (2 x 50 mm, P/N 052248)

# **IONPAC® AS9-HC ANALYTICAL COLUMN**

(4 x 250 mm, P/N 051786) (2 x 250 mm, P/N 052244)

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# SECTION 1 - INTRODUCTION TO IONPAC AS9-HC/AG9-HC CHROMATOGRAPHY

The IonPac® AS9-HC Analytical Column in combination with the AG9-HC Guard Column is designed for the analysis of inorganic anions and oxyhalides including bromate, chlorite, and chlorate. The selectivity of the IonPac AS9-HC 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 AS9-HC is compatible with pH 0-12 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS9-HC 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 AS9-HC has nominal efficiency for sulfate using standard operating conditions of at least 6,000 plates/column.

Table 1
IonPac AS9-HC/AG9-HC Packing Specifications

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
AS9-HC 4 x 250 mm	9.0	55	190	Alkyl/Alkanol quaternary ammonium	Medium-low
AG9-HC 4 x 50 mm	9.0	55	6	Alkyl/Alkanol quaternary ammonium	Medium-low
AS9-HC 2 x 250 mm	9.0	55	47.5	Alkyl/Alkanol quaternary ammonium	Medium-low
AG9-HC 2 x 50 mm	9.0	55	1.5	Alkyl/Alkanol quaternary ammonium	Medium-low

<sup>&</sup>lt;sup>a</sup> macroporous (2000 Å) divinylbenzene/ethylvinylbenzene polymer

Table 2
AS9-HC/AG9-HC Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS9-HC 4-mm Analytical	2000 (13.78)	1.0	3.0
AG9-HC 4-mm Guard	250 (1.72)	1.0	3.0
AS9-HC + AG9-HC 4-mm columns	2250 (15.50)	1.0	3.0
AS9-HC 2-mm Analytical	2000 (13.78)	0.25	0.75
AG9-HC 2-mm Guard	250 (1.72)	0.25	0.75
AS9-HC + AG9-HC 2-mm columns	2250 (15.50)	0.25	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 the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the Dionex Offices listed in, "Dionex Worldwide Offices."

# SECTION 2-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 3 - INSTALLATION**

# 3.1 System Requirements

# 3.1.1 System Requirements for 2-mm Operation

The IonPac AS9-HC 2-mm Guard and Analytical Columns are designed to be run on the following Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a GS50/GP50/GP40/IP25, Gradient Pump Module (GPM-2) or an Advanced Gradient Pump (AGP) with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a Microbore GS50/GP50/GP40 or Advanced Gradient Pump (1/16" pistons) must be employed.

# 3.1.2 System Requirements for 4-mm Operation

The IonPac AS9-HC 4-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a GS50/GP50/GP40 or a Gradient Pump Module (GPM-2) or an Advanced Gradient Pump (AGP) with standard pump heads.

### 3.1.3 System Void Volume

When using 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) ID PEEK tubing. 0.010" ID PEEK tubing (P/N 042260) or 0.012" Tefzel tubing (see, "Dionex Product Selection Guide") may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers. If you need assistance in properly configuring your system contact the Dionex Office nearest you (see, "Dionex Worldwide Offices").

### 3.2 The Sample Concentrator

For 4-mm concentrator work, use the IonPac AG9-HC Guard Column, the Anion MicroConcentrator Column (AMC-1, P/N 051760), or the Trace Anion Concentrator Column (TAC-2, P/N 043101) when a single piston pump such as the DQP or DXP pump is used for sample delivery. Use the Low Pressure Trace Anion Concentrator Column (TAC-LP1, P/N 046026) when the sample is delivered with a syringe or with an autosampler. For 2-mm concentrator work, use the IonPac AG9-HC Guard Column or the AMC-1 when a single piston pump such as the DQP or DXP pump is used for sample delivery. The function of the TAC-LP1, the TAC-2, the AMC-1, or the AG9-HC Guard Column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" all anionic analyte species onto the TAC-LP1, TAC-2, AMC-1 or the AG9-HC leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage to the analytical chemist of the TAC-LP1, the TAC-2, the AMC-1, or the AG9-HC in these applications is the capability of performing routine trace analyses of sample matrix ions at  $\mu$ g/L levels without extensive and laborious sample pretreatment.

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

# 3.3.1 The 2-mm System Injection Loop, 2 - 15 $\mu$ L

For most applications on a 2-mm analytical system, a  $2 - 15 \mu L$  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 AS9-HC 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15  $\mu$ L) of the loop volume used with a 4-mm analytical system (Section 2, "Comparison of Ion Chromatography Systems").

# 3.3.2 The 4-mm System Injection Loop, $10 - 50 \mu L$

For most applications on a 4-mm analytical system, a  $10 - 50 \,\mu$ 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. For typical drinking water samples, you can inject up to  $500 \,\mu$ L.

### 3.4 THE IONPAC AG9-HC GUARD COLUMN

An IonPac AG9-HC Guard Column is normally used with the IonPac AS9-HC Analytical Column. Retention times will increase by approximately 3% 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 AG9-HC Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS9-HC 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<sub>2</sub>B<sub>4</sub>O<sub>7</sub> 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  $7\mu S$  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  $10\,\text{mS}$  during a borate gradient eluent concentration ramp from 0 to 70 mM  $Na_2B_4O_7$ . If the baseline shifts are greater than  $10\,\mu S$ , 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<sub>2</sub>B<sub>4</sub>O<sub>7</sub>
- 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 AS9-HC 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 (ASRS ULTRA) 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 AS9-HC4-mm Analytical Column, use an ASRS ULTRA (4-mm, P/N 053946). If you are installing an IonPac AS9-HC2-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."

# 3.8 Anion Atlas Electrolytic Suppressor (AAES) 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 AS9-HC 2-mm and 4-mm applications using eluents up to 25  $\mu$ eq/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."

# 3.9 Anion MicroMembrane Suppressor (AMMS III) 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 AS9-HC 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 with the ASRS ULTRA or the AMMS III in the Chemical Suppression Mode

To save regenerant preparation time and reduce regenerant consumption and waste, Dionex recommends using an AutoRegen® Accessory (P/N 039594). For more detailed information on the use of the AutoRegen Accessory see the AutoRegen Accessory manual (Document No. 032853). For more detailed information on the use of AutoRegen Regenerant Cartridges, see the "Product Manual for the AutoRegen Regenerant Cartridge Refills" (Document No. 032852).

# 3.11 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 III). See the DCR kit manual, Document P/N 031664, for details.

# SAFETY Use proper safety precautions in handling acids and bases.

### 3.12 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 - OPERATION**

# 4.1 General Operating Conditions

Sample Volume: 2-mm:  $5 \mu \text{L Loop} + 0.8 \mu \text{L Injection valve dead volume}$ 

4-mm:  $25 \mu L$  Loop +  $0.8 \mu L$  Injection valve dead volume

Column: 2-mm: AS9-HC 2-mm Analytical Column + AG9-HC 2-mm Guard Column

4-mm: AS9-HC 4-mm Analytical Column + AG9-HC 4-mm Guard Column

Eluent:  $9.0 \text{ mM Na}_2\text{CO}_3$ Eluent Flow Rate: 2-mm: 0.25 mL/min

4-mm: 1.0 mL/min

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm or 4-mm)

AutoSuppression Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm)

Regenerant is 50 mN H<sub>2</sub>SO<sub>4</sub>

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

Expected Background Conductivity: 24-30μS

Long-term Storage Solution (> 1 week): 100 mM NaHCO<sub>3</sub>

Short-term Storage Solution (< 1 week): Eluent

# 4.2 IonPac AS9-HC Operation Precautions

**CAUTIONS** 

Filter and Degas Eluents
Filter Samples
Eluent pH between 0 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. Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely effect the resolution of phosphate and sulfate. Use of Dionex 0.5 molar Sodium Carbonate Concentrate (P/N 037162) 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.

### 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  $0.2\,\mu\text{m}$ . 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.

### 4.3.3 Solvents

Solvents can be added to the ionic eluents used with IonPac AS9-HC columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of watermethanol and water-acetonitrile mixture varies. The practical back pressure limit for the IonPac AS9-HC columns is 4,000 psi (27.57 MPa).

The AS9-HC 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 AS9-HC Columns

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

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

### **CAUTION**

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

# 4.4 Making Eluents that Contain Solvents

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 90% acetonitrile, prepare the eluent by adding 900 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.

### 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.

### **SAFETY**

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

### 4.5 Regenerant Preparation for the AMMS III

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

# 4.6 Sample Concentration

The Low Pressure Trace Anion Concentrator (TAC-LP1), the Anion MicroConcentrator Column (AMC-1), the Trace Anion Concentrator (TAC-2), or the IonPac AG9-HC Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of a concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the concentrator column, lowering detection limits by 2-5 orders of magnitude. The concentrator column is used in lieu of the sample loop. Pump the sample onto the concentrator column in the **OPPOSITE** direction of the eluent flow. When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column. For more detailed information on sample concentration techniques for high sensitivity work refer to Section 3, "Operation," of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column Product Manual (Document No. 034972). These techniques can also be applied to the TAC-2, AMC-1, or the AG9-HC.

### SECTION5-EXAMPLE APPLICATIONS

The chromatograms in this section were obtained using columns that reproduced the Production test Chromatogram (see Section 5.3, "Production Test Chromatogram") on optimized Ion Chromatographs (see Section 3, "Installation"). Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components and laboratory temperatures.

Before attempting any of the following example applications, take the time to ensure that your system is properly configured. Ensure that all of the eluents have been made from high purity reagents and deionized water. All water used in the preparation of eluents should be degassed, deionized water. For chemical purity requirements, see Section 4.3, "Chemical Purity Requirements." After running synthetic standards to calibrate your system, you may find that real sample matrices foul your columns. For this reason it is always advisable to use a guard column to protect the analytical column. If column performance deteriorates and it is determined that the guard or the analytical column has been fouled, refer to the column cleanup protocols in, "Column Care." If your sample matrices are relatively low in ionic concentration, you may be able to increase the sensitivity of your system by using sample concentration techniques (see Section 4.6, "Sample Concentration").

### **5.1** Preparation of Eluent Stock Solution Concentrates

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

#### Order Dionex P/N 037162

OI

Thoroughly dissolve 26.49 g of  $\text{Na}_2\text{CO}_3$  in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely effect 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.

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

### Order Dionex P/N 037163

or

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

# **5.2** Eluent Preparation

# **Eluent: 9.0 mM Sodium Carbonate**

Prepare the eluent by pipetting 18.0 mL of  $0.5 \text{ M Na}_2\text{CO}_3$  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.

# **5.3** Production Test Chromatograms

Isocratic elution of inorganic anions and oxyhalides on the IonPac AS9-HC Analytical Column has been optimized utilizing a carbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. The IonPac AS9-HC Analytical Column should always be used with the IonPac AG9-HC Guard Column. To guarantee that all IonPac AS9-HC Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Volume:	2-mm: 5 $\mu$ L Loop + 0.8 $\mu$ L Injection valve dead volume			
	4-mm: 25 $\mu$ L Loop + 0.8 $\mu$ L Injection valve dead volume			
Column:	See Chromatogram		Analyte	mg/L (ppm)
Eluent:	9.0 mM Carbonate	1.	Fluoride	3.0
Eluent Flow Rate:	0.25 mL/min (2-mm), 1.0 mL/min (4-mm)	2.	Chlorite	10.0
		3.	Bromate	20.0
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (2-mm or 4-mm)	4.	Chloride	6.0
	AutoSuppression® Recycle Mode	5.	Nitrite	15.0
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm)	6.	Bromide	25.0
MMS Regenerant:	50 mN H <sub>2</sub> SO <sub>4</sub>	7.	Chlorate	25.0
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES	8.	Nitrate	25.0
Expected Background Conductivity:	24 - 30 µS	9.	Phosphate	40.0
Long-term Storage Solution (> 1 week):	,	10.	Sulfate	30.0
	3			
Short-term Storage Solution (< 1 week):	Eluelit			

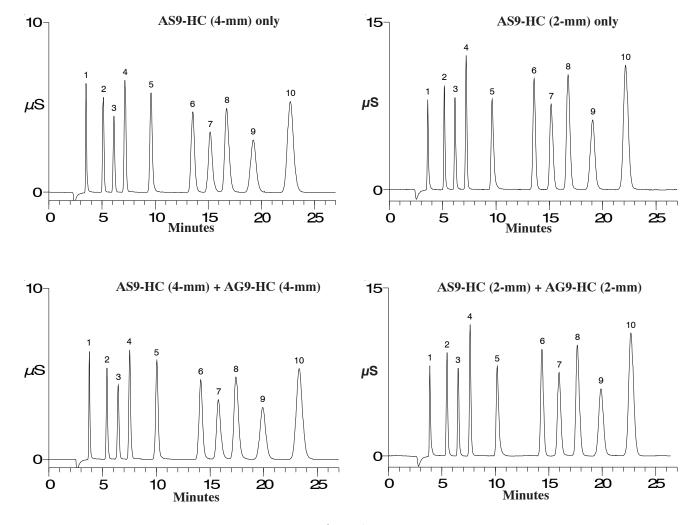


Figure 1
IonPac AS9-HC Production Test Chromatograms

# 5.4 IonPac AS9-HC Fast Run Analysis

The following chromatogram demonstrates a fast run analysis using the AS9-HC analytical column. This run shows the selectivity change particularly for phosphate due to the addition of bicarbonate to eluent.

Sample Loop Volume: 25  $\mu$ L

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 12.0 mM Carbonate/5 mM Bicarbonate

Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	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.	Phosphate	40.0
7.	Bromide	25.0
8.	Chlorate	25.0
9.	Nitrate	25.0
10.	Sulfate	30.0

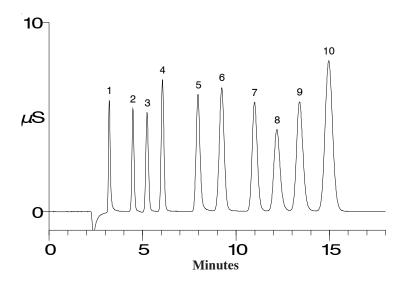


Figure 2
IonPac AS9-HC Fast Run Analysis

# 5.5 Analysis of Municipal Drinking Water

The following chromatogram demonstrates the separation of inorganic anions and oxyhalides in a simulated drinking water sample as well as in a tap water sample.

Sample Loop Volume:  $200 \,\mu\text{L}$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Carbonate
Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

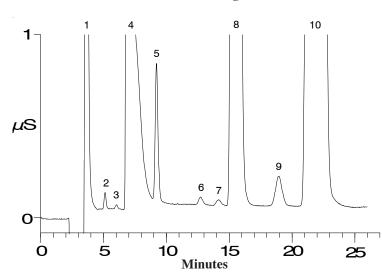
MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

Expected Background Conductivity:  $24 - 30 \mu S$ 

# **Simulated Drinking Water**

	Analyte	mg/L (ppm)
1.	Fluoride	1.00
2.	Chlorite	0.01
3.	Bromate	0.005
4.	Chloride	50.00
5.	Nitrite	0.10
6.	Bromide	0.01
7.	Chlorate	0.01
8.	Nitrate	10.00
9.	Phosphate	0.10
10.	Sulfate	50.00



### **Sunnyvale Drinking Water**

	Analyte	mg/L (ppm)
1.	Fluoride	0.04
2.	Chlorite	ND
3.	Bromate	ND
4.	Chloride	16.20
5.	Nitrite	ND
6.	Bromide	0.03
7.	Chlorate	0.04
8.	Nitrate	3.90
9.	Phosphate	0.15
10.	Sulfate	18.30
	(ND = Not	Determined)

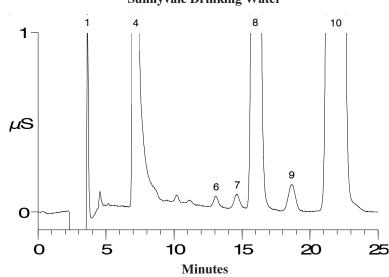


Figure 3
Analysis of 11 Anions in Drinking Water Samples

# 5.6 Effect of OnGuard™ Pretreatment on Samples with Excessive Chloride Concentrations

In the presence of very high chloride concentration, the efficiency of early eluting peaks would be compromised because of the overloading effect. A sample pretreatment with OnGuard-Ag followed by OnGuard-H, significantly reduces chloride and carbonate; allowing accurate quantification of bromate.

OnGuard - Ag and OnGuard - H sample cleanup procedure:

- 1. Wash the OnGuard Ag and OnGuard H in series with 10 mL of deionized water.
- 2. Pass 10 mL of sample through the washed cartridge, discarding the first 3 mL of effluent.
- 3. Sparge the collected pretreated sample with nitrogen or helium for at least 5 minutes in order to remove carbonic acid from the sample.

Sample Loop Volume: 500 µL

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Carbonate
Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

External Water Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

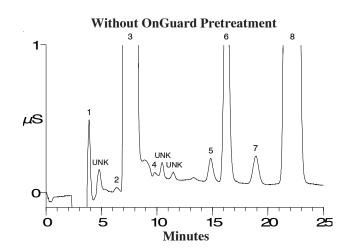
MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>4</sub>

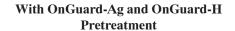
or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

Expected Background Conductivity:  $24 - 30 \mu S$ 

	Analyte	mg/L (ppm)
1.	Fluoride	0.02
2.	Bromate	0.01
3.	Chloride	240.00
4.	Nitrite	0.002
5.	Chlorate	0.04
6.	Nitrate	0.33
7.	Phosphate	0.10
8.	Sulfate	10.00
	(UNK= Unknown)	

(UNK= Unknown)





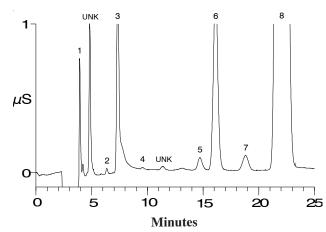


Figure 4
Effect of OnGuard Pretreatment on Sample

# 5.7 Analysis of a Treated Spa Water Sample

The following chromatogram demonstrates the separation of anions in a spa water sample.

Sample Loop Volume: 500 μL

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Carbonate

Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	mg/L (ppm)	
1.	Fluoride	0.760	
2.	Chlorite	ND	
3.	Bromate	0.017	
4.	Chloride	122.000	
5.	Nitrite	0.540	
6.	Bromide	46.000	
7.	Chlorate	0.076	
8.	Nitrate	1.500	
9.	Phosphate	1.600	
10.	Sulfate	51.000	
	(ND = Not Determined)		

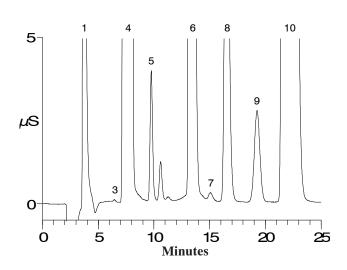


Figure 5 Analysis of 11 Anions in Spa Water

# 5.8 Determination of Trace Nitrite and Nitrate in High Ionic Strength Matrices

The following chromatograms demonstrates the determination of trace nitrite and nitrate in high ionic strength matrices.

### Ham sample preparation:

- 1. Weigh 10.0 g. of the meat sample and transfer to a blender.
- 2. Add 100.0 mL of deionized water to the meat sample and blend for one minute.
- 3. Heat the liquified sample and maintain the temperature of the sample between 70° and 80°C for 15 min.
- 4. Centrifuge the sample for 10 minutes, and filter sample with a Whatman No. 2 and GF/A filters and 1.2 mm and 0.2 mm Acrodisc filters.

Sample Loop Volume: See chromatogram

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Sodium Carbonate

Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

Expected Background Conductivity: 24-30 μS

		A	В
	Analyte	mg/L	mg/L
		(ppm)	(ppm)
1.	Chloride	1000.0	ND
2.	Nitrite	1.0	ND
3.	Nitrate	1.0	ND
4.	Phosphate	100.0	_
5.	Sulfate	100.0	_

(ND = Not Determined) (UNK = Unkown)

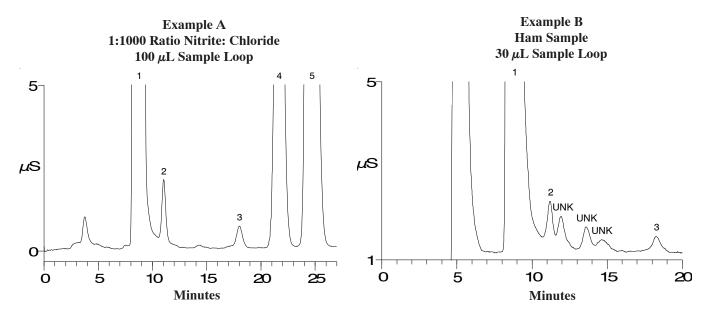


Figure 6
Determination of Trace Nitrite and Nitrate
in High Ionic Strength Matrices

# 5.9 Determination of Sulfite in the Presence of Common Anions

The following chromatograms demonstrate the determination of sulfite in the presence of common anions at  $1.0 \, \text{mL/min}$  and  $1.5 \, \text{mL/min}$ .

Sample Loop Volume:  $25 \mu L$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 8 mM Carbonate/1.5 mM NaOH

Eluent Flow Rate: See Chromatogram

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

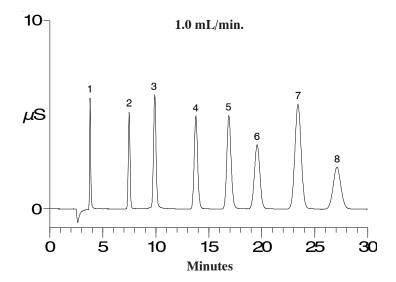
AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	mg/L
		(ppm)
1.	Fluoride	3.0
2.	Chloride	6.0
3.	Nitrite	15.0
4.	Bromide	25.0
5.	Nitrate	25.0
6.	Sulfite	30.0
7.	Sulfate	30.0
8	Phosphate	40.0



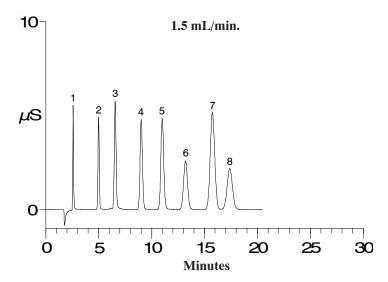


Figure 7
Determination of Sulfite in the Presence of Common Anions

# 5.10 Determination of Trace Sulfate in Brine Sample

The following chromatograms demonstrate the determination of low sulfate in the presence of high chloride (1:1000 ratio).

Sample Loop Volume:  $25 \mu L$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9 mM Carbonate

Eluent Flow Rate: 1 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

External Water Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	mg/L
		(ppm)
1.	Chloride	1000.0
2.	Sulfate	1.0
	unk = unknown	

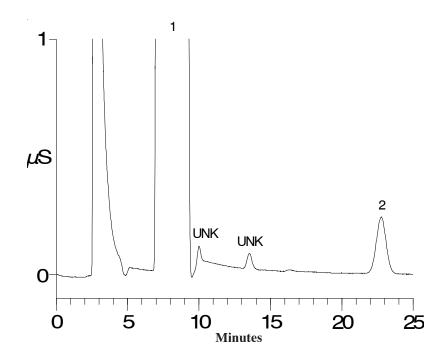


Figure 8
Determination of Trace Sulfate in Brine Sample

# 5.11 Determination of Trace Chloride and Sulfate in High Purity Water

The following chromatograms demonstrate the determination of trace chloride and sulfate in high levels of carbonate using an optimized eluent system.

Sample Loop Volume: 2 mL

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 8 mM Carbonate/1.5 mM Bicarbonate

Eluent Flow Rate: 1 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

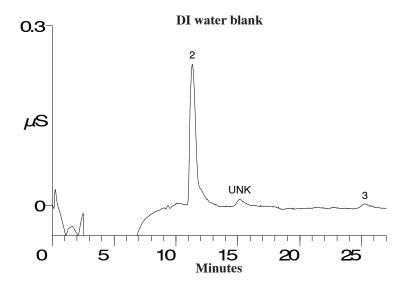
External Water Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

MMS Regenerant:  $50 \text{ mN H}_2\text{SO}_4$ 

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	$\mu g/L$
		(ppb)
1.	Chloride	1.0
2.	Carbonate	250.0
3.	Sulfate	1.0
	unk = unknown	



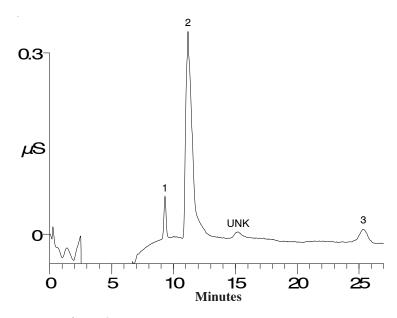


Figure 9
Determination of trace Chloride and Sulfate in High Purity Water

# 5.12 Analysis of Oxyanions

The following chromatogram demonstrates the analysis of oxyanions in the presence of common inorganic anions. Note that analysis time can be reduced by increasing the flow rate without changing selectivity.

Sample Loop Volume:  $25 \mu L$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Carbonate
Eluent Flow Rate: See chromatogram

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

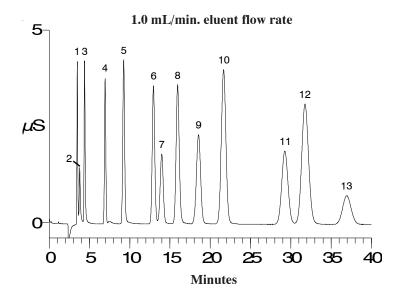
AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	mg/L
		(ppm)
1.	Fluoride	3.0
2.	Acetate	20.0
3.	Formate	10.0
4.	Chloride	6.0
5.	Nitrite	15.0
6.	Bromide	25.0
7.	Selenite	30.0
8.	Nitrate	25.0
9.	Phosphate	40.0
10.	Sulfate	30.0
11.	Selenate	30.0
12.	Oxalate	50.0
13.	Arsenate	50.0



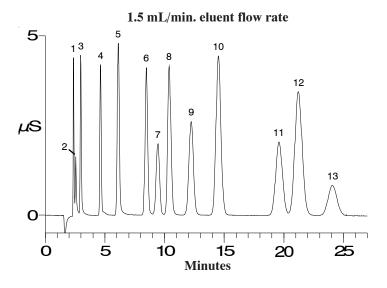


Figure 10 Analysis of Oxyanions

# 5.13 Analysis of 21 Anions with Isocratic Elution Using a Carbonate Eluent System

The following chromatogram demonstrates the elution order of 21 anions using the standard eluent on the IonPac AS9-HC.

Sample Loop Volume:  $25 \mu L$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 9.0 mM Carbonate
Eluent Flow Rate: 1.5 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

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

or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

	Analyte	mg/L (ppm)		Analyte	mg/L (ppm)
1.	Fluoride	3.0	12.	Nitrate	25.0
2.	Acetate	20.0	13.	Phosphate	40.0
3.	Formate	10.0	14.	Sulfate	30.0
4.	Chlorite	10.0	15.	Selenate	30.0
5.	Bromate	20.0	16.	Oxalate	40.0
6.	Chloride	6.0	17.	Arsenate	50.0
7.	Bicarbonate	50.0	18.	Iodide	70.0
8.	Nitrite	15.0	19.	Thiosulfate	70.0
9.	Bromide	25.0	20.	Thiocyanate	70.0
10.	Chlorate	25.0	21.	Chromate	70.0
11.	Selenite	30.0			

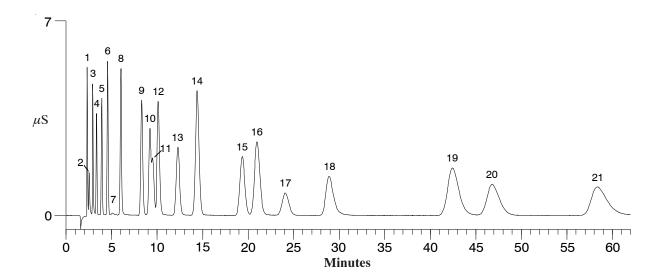


Figure 11 Analysis of 21 Anions

# 5.14 Isocratic Analysis of 13 Anions Using a Borate Eluent System

The following chromatogram demonstrates the isocratic separation of acetate, formate, and inorganic anions including oxyhalides using a sodium tetraborate eluent system. Using these conditions, glycolate and acetate will coelute.

Sample Loop Volume:  $25 \mu L$ 

Column: IonPac AS9-HC Analytical Column (4-mm) + IonPac AG9-HC Guard Column (4-mm)

Eluent: 50 mM sodium tetraborate

Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm)

External Water Mode

or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS III (4-mm)

MMS Regenerant: 50 mN  $H_2SO_4$ Expected Background Conductivity: 7-10  $\mu S$ 

	Analyte	mg/L (ppm)
1.	Fluoride	3.0
2.	Acetate	20.0
3.	Formate	10.0
4.	Chlorite	10.0
5.	Bromate	20.0
6.	Chloride	6.0
7.	Bicarbonate	50.0
8.	Nitrite	15.0
9.	Phosphate	40.0
10.	Bromide	25.0
11.	Chlorate	25.0
12.	Nitrate	25.0
13.	Sulfate	30.0

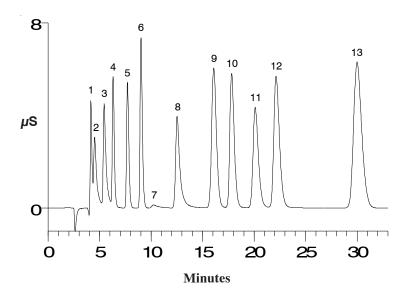


Figure 12
Isocratic Analysis of 13 Anions Using a Borate Eluent System

### **5.15** Clean-up After Humic Acid Samples

Solvent compatibility of the IonPac AS9-HC permits the use of organic solvents to effectively remove organic contaminates from the column. An AS9-HC 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 200 mM HCl + 80% THF.

Sample Loop Volume: 25 µL

Column: IonPac AS9-HC (4-mm) + IonPac AG9-HC

Eluent: 9 mM Carbonate
Eluent Flow Rate: 1.0 mL/min.

SRS Suppressor: Anion Self-Regenerating Suppressor,

ASRS ULTRA (4-mm)

AutoSuppression® Recycle Mode

or MMS Suppressor: Anion MicroMembrane Suppressor,

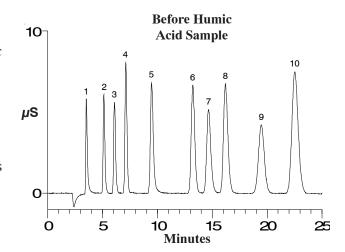
AMMS III (4-mm)

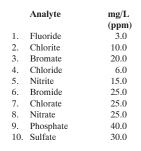
MMS Regenerant: 50 mN H<sub>2</sub>SO<sub>2</sub>

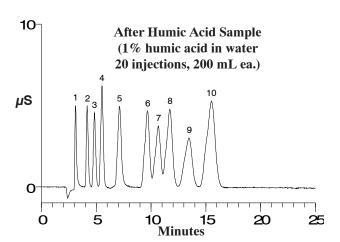
or AES Suppressor: Anion Atlas Electrolytic Suppressor, AAES

Expected Background

Conductivity:  $24-30 \mu S$ 







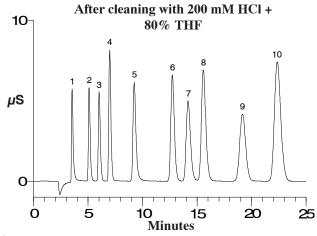


Figure 13 Clean-up After Humic Acid Sample

# **SECTION 6 - TROUBLESHOOTING GUIDE**

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS9-HC 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 6
AS9-HC/AG9-HC 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, 7.4
	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
Poor Resolution of Only Phosphate and Sulfate	Sodium Carbonate Contaminated with Sodium Hydroxide, Inadequate Equilibration after Use of an Alkaline Buffer, Sodium Carbonate Dried at Temperatures >110°C	Use Dionex 0.5 M Sodium Carbonate (P/N 037162), Dry Sodium Carbonate at Lower Temperature	6.3.5
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

### 6.1 High Back Pressure

# **6.1.1** Finding the Source of High System Pressure

Total system pressure for the IonPac AG9-HC (4-mm) Guard Column plus the AS9-HC (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,250 psi. If the system pressure is higher than 2,250 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 AS9-HC/AG9-HC Operating Back Pressures").

The Anion Self-Regenerating Suppressor ULTRA may add up to 100 psi (0.69 MPa). No other components should add more than 100 psi (0.69 MPa) of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

Table 7
Typical AS9-HC/AG9-HC Operating Back Pressures

Column psi (MPa)	Typical Back Pressure mL/min	Flow Rate
AS9-HC 4-mm Analytical	2000 (13.78)	1.0
AG9-HC 4-mm Guard	250 (1.72)	1.0
AS9-HC + AG9-HC 4-mm columns	2250 (15.50)	1.0
AS9-HC 2-mm Analytical	2000 (13.78)	0.25
AG9-HC 2-mm Guard	250 (1.72)	0.25
AS9-HC + AG9-HC 2-mm columns	2250 (15.50)	0.25

# 6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

- A. Disconnect the column from the system.
- **B.** Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.
- C. Remove the bed support. Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you DO NOT SCRATCH THE WALLS OF THE END FITTING. Discard the old bed support assembly.
- **D.** Place a new bed support assembly into the end fitting. Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

	IonPac AS9-HC 4-mm Columns (P/N)	IonPac AS9-HC 2-mm Columns (P/N)
Analytical Column	051786	052244
Guard Column	051791	052248
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

9.0 mM Na<sub>2</sub>CO<sub>3</sub>

 $24 - 30 \mu S$ 

# **6.2.1 Preparation of Eluents**

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

# 6.2.2 A Contaminated Guard or Analytical Column

Remove the IonPac AG9-HC Guard and AS9-HC 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 AG9-HC 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").

### 6.2.3 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.

# 6.2.4 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  $\mu$ S. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

# 6.2.5 A Contaminated Anion Self-Regenerating Suppressor, ASRS ULTRA

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

### Metal Contaminants or Precipitates

### **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."

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

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

### 6.2.6 A Contaminated Anion MicroMembrane Suppressor, AMMS III

A. Check the regenerant flow rate at the REGEN OUT port of the AMMS. For the example isocratic applications, this flow rate should be 3 - 5 mL/min.

- **B.** Check the eluent flow rate. In general, the eluent flow rate for 4-mm applications, it should be 1.0 mL/min. Refer to the Anion MicroMembrane Suppressor Product Manual (Document No. 034449-02) for assistance in determining that the eluent is within suppressible limits.
- C. If you are using an AutoRegen Accessory with the SRS (in the Chemical Suppression Mode) or the MMS, prepare fresh regenerant solution. Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.
  - 1. If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge, you probably need to clean or replace your SRS or MMS.
  - 2. If the background conductivity is low when freshly prepared regenerant is run through the SRS or MMS without an AutoRegen Accessory in-line, test the AutoRegen Regenerant Cartridge to see if it is expended. Connect the freshly prepared regenerant to the AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is high after placing the AutoRegen Accessory in-line, you probably need to replace the AutoRegen Regenerant Cartridge. Refer to the "AutoRegen Regenerant Cartridge Refill Product Manual" (Document No. 032852) for assistance.

# 6.2.7 A Contaminated Anion Atlas Electrolytic Suppressor, AAES

# Metal Contaminants or Precipitates

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

By passing 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 REGENIN 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  ${
  m H_2SO_4/90\%}$  acetonitrile.  ${
  m H_2SO_4/acetonitrile}$  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  ${
  m H_2SO_4}$  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.

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

### 6.3 Poor Peak Resolution

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

# **6.3.1** Loss of Column Efficiency

- A. Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 6.1.2, "Replacing Column Bed Support Assemblies"). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.
- B. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4-mm systems or no greater than 0.005" for 2-mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks.

# 6.3.2 Poor Resolution Due to Shortened Retention Times

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

- **A.** Check the flow rate. See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.
- **B.** Check to see if the eluent compositions and concentrations are correct. An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent, components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

C. Column contamination can lead to a loss of column capacity. This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to "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").

### 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 AS9-HC 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 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.

# 6.3.5 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 °C.

# B. Action

- 1. Use Dionex 0.5 molar Sodium Carbonate Concentrate (P/N 037162).
- 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.

### APPENDIX A - COLUMN CARE

### A.1 Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS9-HC columns is 4,000 psi.

# A.2 Column Start-Up

The test eluent on the Quality Assurance Report (QAR) is 9 mM sodium carbonate.

Prepare the eluent shown on the QAR. Install the column in the chromatography module and test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

### A.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), 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 hydrophilic ionic contamination of low valency, metal contamination, and high valency hydrophobic ions (with isocratic or gradient cleanup options).

Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column. High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column. High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent which may create a neutralization pressure band. The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to  $\leq 5\%$  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

CAUTION: When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately. Do not pass the column effluent through the suppressor.

### A. Hydrophilic Ionic Contamination of Low Valency

- E1: Test eluent shown on the Quality Assurance Report (QAR)
- E2: 10X concentrate of test eluent on the Quality Assurance Report (QAR): 90 mM sodium carbonate
- 1. Prepare the cleanup solutions above.
- 2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard 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.
- 3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
- 4. Pump the cleanup solution E2 through the column(s) for 60 minutes.
- 5. Equilibrate the column(s) with eluent (E1) before resuming normal operation for at least 15 minutes.
- 6. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

Note: Be sure to wash bottle, lines and pump with di water after using 10X eluent on the test system.

### B. Metal Contamination

E1: Test eluent shown on the Quality Assurance Report (QAR)

E2: 0.1 Moxalic acid

# Note: Iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

- 1. Prepare the cleanup solutions above.
- 2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard 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.
- 3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
- 4. Pump the cleanup solution E2 through the column(s) for 60 minutes it is aluminum contamination or for overnight (14-18 hours) if it is iron contamination..
- 5. Equilibrate the column(s) with eluent (E1) before resuming normal operation for at least 30 minutes.
- 6. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

Note: Be sure to wash the bottle, lines and pump with di water after using oxalic acid on the test system.

### C. High Valency Hydrophobic Ions - Isocratic Cleanup Option

- E1: Test eluent shown on the Quality Assurance Report (QAR).
- E2: 5% acetonitrile in the test eluent shown on the Quality Assurance Report (QAR).
- E3: 150 mM potassium nitrate in 80% acetonitrile
- 1. Prepare the cleanup solutions above.
- 2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard 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.
- 3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
- 4. Rinse the column for 15 minutes with E2.
- 5. Pump the cleanup solution E3 through the column(s) for 60 minutes.
- 6. Rinse the column for 15 minutes with E2.
- 7. Equilibrate the column(s) with eluent E1 before resuming normal operation for at least 30 minutes.
- 8. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

Note: Be sure to wash the bottles, lines and pump with di water after using acetonitrile and potassium nitrate on the system.

### D. High Valency Hydrophobic Ions - Gradient Cleanup Option

E1: Test eluent shown on the Quality Assurance Report (QAR).

E2: 100% acetonitrile (Note: Acetonitrile must be stored in a separate reservoir bottle because it slowly breaks down in acidic aqueous solutions and high percentages of acetonitrile tend to be non-miscible in high molarity salt solutions. These limitations are overcome by step gradient operation.

E3: 1.0 mM NaCl using degassed Type I Reagent Grade Water with a specific resistance of 18.2 megaohm-cm. Adjust the pH to 2.0 using HCl

E4: Degassed Type I Reagent Grade Water with a specific resistance of 18.2 megaohm-cm.

- 1. Prepare the cleanup solutions above.
- 2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard 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.
- 3. Set the flow rate to 0.5 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns.
- 4. Proceed with the following gradient:

Time (min)	%E1*	%E2	%E3	%E4	Comments**
0.0	0	5	20	75	Low Solvent
15.0	0	5	20	75	
15.1	0	80	20	0	High Solvent
75.0	0	80	20	0	
75.1	0	5	20	75	Low Solvent
90.0	0	5	20	75	

<sup>\*</sup>Equilibrate the column set to E1 at least 30 minutes.

5. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

# Note: Be sure to wash the bottles, lines and pump with di water after using acetonitrile and sodium chloride on the test system

### E Humic Acid Clean-up

E1: Test eluent shown on the Quality Assurance Report (QAR).

E2: DI water E3: 1MHCl E4: 100% THF

- 1. Prepare the cleanup solutions as above.
- 2. Disconnect the suppressor from the analytical column. If your system is configured with both a guard 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.
- 3. Set the flow rate to 0.25 mL/min for 2-mm columns, or 1.0 mL/min for 4-mm columns. Set the upper pressure limit on the pump to be 1000psi to avoid any damage to the column PPK tubing and other PEEK tubing on the system.
- 4. Rinse the column for 10 minutes with E2.
- 5. Pump the 20% of E3 and 80% of E4 (200mM HCL and 80% THF) solution through the column(s) for 120 minutes.
- 6. Rinse the column for 15 minutes with E2.
- 7. Equilibrate the column(s) with eluent E1 before resuming normal operation for at least 60 minutes.
- 8. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system is configured with a guard column.

Note: Be sure to wash the bottles, lines and pump with di water after using THF and HCl on the test system.

<sup>\*\*</sup>Equilibration is complete when consecutive injections of the standard give reproducible retention times.

# APPENDIX B - CONFIGURATION

Table 1 Configuration

CONFIGURATION	2-mm	4-mm
Fluent Flow Rate	0.25 mL/min	1.0 mL/min
SRS Suppressor	ASRS ULTRA II (2-mm) (P/N 061562)	ASRS ULTRA II (4-mm) (P/N 061561)
	( )( )	, , ,
MMS Suppressor	AMMS III (2-mm) (P/N 056751)	AMMS III (4-mm) (P/N 056750)
AAE Suppressor	AAES (P/N 056116)	AAES (P/N 056116)
Injection Loop	2 - 15 μL	10-50 μL
	Rheody ne Microinjection Valve (P/N 044697) for full 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.	Use the DP/SP/GP40/GP50/IP20/IP25 in Standard-Bore Configuration.
	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.	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 μL, P/N 046423)	AD25 Cell (10-mm, 9 μL, P/N 049393)
	VDM-2 Cell (3-mm, 2.0 μL) (P/N 043120)	VDM-2 Cell (6-mm, 10 μL) (P/N 043113)
	DC/CD20, CD25, CD25A, ED40, ED50, or ED50A	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	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	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 2
Tubing Back Pressures

Color	Dionex P/N	ID Inches	ID cm	Volume mL/cm	Back Presure psi/ft at 1 mL/min	Back Presure psi/ft at 0.25 mL/min	Back Presure 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