PRODUCT MANUAL

for

IonPac® AG9-SC
IonPac® AS9-SC

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PRODUCT MANUAL

for the

IONPAC® AG9-SC GUARD COLUMN

(4 x 50 mm, P/N 043186)

IONPAC® AS9-SC ANALYTICAL COLUMN

(4 x 250 mm, P/N 043185)

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SECTION 1 - INTRODUCTION

The IonPac® AS9-SC Analytical Column (P/N 043185) is designed for the analysis of inorganic anions including oxyhalides, such as chlorate, chlorite and bromate.

The 4 x 250 mm IonPac AS9-SC Analytical Column has an ion exchange capacity of approximately 30 µeq/column. This resin is composed of a highly cross-linked (55%) 13 micron polyethylvinylbenzene/divinylbenzene substrate agglomerated with anion exchange latex that has been completely aminated. The latex has a polyacrylate backbone and carries the actual ion exchange sites. The IonPac AS9-SC has nominal efficiency for sulfate using standard operating conditions of at least 14,000 plates/meter.

The IonPac AS9-SC can be operated at flow rates up to 3.0 mL/min with eluents that have a pH between 2 and 11. Eluents may contain organic solvents from 0 - 100% in concentration. Optimally, the IonPac AS9-SC should operate at a backpressure less than 1,100 psi at 1.0 mL/min. However, the column is capable of operating at backpressures up to 4,000 psi.

CAUTION
Eluent pH must be maintained between 2-11 or irreversible damage to the column will result.

Table 1
AS9-SC/AG9-SC Packing Specifications

Column	Particle Diameter µm	Substrate X-Linking %	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
AS9-SC (4 x 250 mm)	13	55	110	20	30-35	Alkyl quaternary ammonium	Medium-low
AG9-SC (4 x 50 mm)	13	55	110	20	6-7	Alkyl quaternary ammonium	Medium-low

Table 2
AS9-SC/AG9-SC Operating Parameters

Column	Typical Back Pressure psi (MPa) at 30°C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS9-SC (4 x 250 mm) Analytical	≤ 950 (6.55)	1.0	3.0
AG9-SC (4 x 50 mm) Guard	≤ 225 (1.55)	1.0	3.0
AS9-SC Analytical Column + Guard	**≤ 1175 (8.10)**	**1.0**	**3.0**

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 - THE ION CHROMATOGRAPHY SYSTEM

CONDITION	4-mm
Eluent Flow Rate	3 mL/min Maximum Flow Rate
SRS Suppressor	ASRS-ULTRA (4-mm) (P/N 053946)
MMS Suppressor	AMMS III (4-mm) (P/N 056750)
AES Suppressor	AAES (P/N 056116)
Injection Loop	10 - 50 µL
System Void Volume	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2 , GM-3 or recommended gradient mixers.
Pumps	Use the GP40/GP50/IP20/IP25 in Standard-Bore Configuration. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP40/GP50/IP20/IP25 and the DX-300 HPLC Pump.
Detectors	AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) VDM-2 Cell (6-mm, 10 µL) P/N 043113 CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Either the TS-1 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 50-75 psi back pressure.

Table 3
Tubing Back Pressures

Tubing ID in	H_20 Back Pressure Psi/ft at 1 mL/min
0.005	111.4
0.007	29.0
0.010	7.0
0.012	3.4

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 4-mm Operation

The IonPac AS9-SC 4-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 Void Volume

It is important to minimize system void volume. 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). 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 nearest DIONEX Worldwide Office (see, DIONEX Worldwide Offices).

3.2 The Sample Concentrator

The Low Pressure Trace Anion Concentrator Column (TAC-LP1, P/N 046026), the Trace Anion Concentrator Column (TAC-2, P/N 043101), the Anion MicroConcentrator, AMC-1, (P/N 051760) or the IonPac AG9-SC 4-mm Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of the TAC-LP1, the TAC-2, the AMC-1, or the AG14A Guard Column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process “concentrates” all anionic analyte species onto the TAC-LP1, TAC-2, AMC-1 or the AG14A leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage to the analytical chemist of the TAC-LP1, the TAC-2, the AMC-1, or the AG9-SC 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.

3.3 The Injection Loop

Table 4
Smallest Injectable Volumes (µL)

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

3.3.1 The 4-mm System Injection Loop, 10 - 50 µL

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop will be sufficient. Generally, do not inject more than 10 nanomoles (100 - 200 ppm) of any one analyte onto the 4-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect peak efficiency and resolution.

3.4 The IonPac AG9-SC Guard Column

An IonPac AG9-SC Guard Column is normally used with the IonPac AS9-SC Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column. Replacing the AG9-SC Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS9-SC 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 (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 (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-3. Connect a waste line to the ATC-3 outlet and direct the line to a waste container.

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

D. Rinse the ATC-3 with the strongest eluent that will be used during the gradient analysis.

E. After flushing the ATC-3 with eluent, connect the ATC-3 to the eluent line that is connected to the injection valve.

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

The ATC-3 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-3 with 30 mL of 70 mM $Na_2B_4O_7$.

B. Prior to next day use of the chromatographic system, flush the ATC-3 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-SC columns are designed to be used with borate or 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 The Anion Self-Regenerating Suppressor, ASRS® -ULTRA

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-SC 4-mm Analytical Column, use an ASRS-ULTRA (4-mm, P/N 053946).

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 The Anion Atlas® Electrolytic Suppressor, AAES

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-SC 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 Atlas Anion Electrolytic Suppressor."

3.9 The Anion MicroMembrane Suppressor, AMMS® III

An Anion MicroMembrane Suppressor, the AMMS III (P/N 056750) can also be used for applications that require suppressed conductivity detection. It is compatible with all solvents and concentrations with which the systems and columns are compatible.

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."

NOTE
Do not run the AMMS III Suppressor over 40°C. If you are using an application where temperatures in excess of 40°C are required, place the suppressor outside of the oven.

To minimize the baseline shift when performing an analysis that requires a borate gradient, a high regenerant flow rate (10 - 15 mL/min) is required. To save regenerant preparation time and reduce regenerant consumption and waste, DIONEX recommends using an AutoRegen® Accessory (P/N 039594).

3.10 Using AutoRegen® with the ASRS-ULTRA orthe 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).

When using an AutoRegen System, specific contaminants are continuously removed from the regenerant solution to restore it to the correct ionic state. It is necessary however to replace the regenerant on a regular basis. If solvents are used in the eluent, ionic

contaminants from the solvent component of the eluent which are not removed by the Anion AutoRegen Regenerant Cartridge may slowly accumulate in the regenerant. This results in slowly increasing background conductivity. The rate at which the background conductivity increases versus the required analysis sensitivity will determine how often the regenerant must be changed.

It is not necessary to change the Anion AutoRegen Regenerant Cartridge until it is completely expended and a sudden jump to very high background conductivity is observed.

3.11 Using Displacement Chemical Regernation (DCR) with the Chemical Suppression Mode

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

SAFETY
Use proper safety precautions in handling acids and bases.

3.12 Detector Requirements

See Section 2, “The Ion Chromatography System,” for 4-mm system detector, cell and thermal stabilizer requirements.

SECTION 4 - OPERATIONS

4.1 General Operating Conditions

Sample Volume:	10 µL Loop + 0.8 µL Injection valve dead volume
Column:	AS9-SC 4-mm Analytical Column + AG9-SC 4-mm Guard Column
Eluent:	1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$
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_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	15 - 20 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

The selectivity of the IonPac AS9-SC 4-mm Analytical Column has been designed to separate F^-, ClO_2^-, BrO_3^-, Cl^-, NO_2^-, Br^-, ClO_3, NO_3^-, HPO_4^{2-} and SO_4^{2-} isocratically in less than 10 minutes. The AS9-SC packing is a highly cross-linked (55%), microporous resin that has been agglomerated with totally permeable latex particles that are completely aminated. The latex particles carry the actual ion exchange function - an alkanol quaternary ammonium group. The polyacrylic structure of the latex MicroBeads make the AS9-SC compatible with pH 2-11 eluents. The highly cross-linked substrate core renders the AS9-SC compatible with eluents containing 0-100% HPLC solvents organic solvents. The AS9-SC can be used with any suppressible ionic eluent that does not exceed the capacity of the suppressor.

4.2 IonPac AS9-SC Operation Precautions

CAUTION
Filter and Degas Eluents
Filter Samples
Eluent pH between 2 and 11 and contains no hydroxide
Sample pH between 2 and 13
3 mL/min Maximum Flow Rate

4.3 Chemical Purity Requirements

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

4.3.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label.

4.3.2 Solvents

Since solvents used with the IonPac AS9-SC columns are added to ionic eluents 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.

4.3.3 Deionized Water

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

4.4 Eluent Preparation

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.

The above precautions, if taken when making eluents, ensure smooth, reproducible ramps, with minimum total change in background conductivity when using sodium carbonate/bicarbonate (isocratic) or borate (isocratic and gradient) eluents with the AS9-SC columns.

The following table details the use of the above eluent types:

Table 5
Eluent Type Selection

Eluent	Application
Bicarbonate/Carbonate	Isocratic Analysis
Borate	Isocratic or Gradient Analysis
Hydroxide	**DO NOT USE!**

4.4.1 Preparation of Carbonate Eluent Concentrates

A. **0.5 M Sodium Carbonate (Na_2CO_3) Concentrate**

1. **Order DIONEX P/N 037162** or

2. Thoroughly dissolve 26.49 g of Na_2CO_3 in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

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

1. **Order DIONEX P/N 037163** or

2. Thoroughly dissolve 21.00 g of $NaHCO_3$ in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

C. **200 mM Na_2CO_3/75 mM $NaHCO_3$ (100X)**

The standard test eluent can be readily prepared from this 100X concentrate. This eluent concentrate can be prepared by thoroughly dissolving 21.978 g of sodium carbonate (MW 106.00 g/mole) plus 6.301 g sodium bicarbonate (MW 84.00 g/mole) in 700 mL of degassed, deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL. This solution can be diluted to make

eluents or can be used as a column regenerant.

D. **AS4A Sodium Carbonate/Bicarbonate Concentrate (100X)**
0.18 M Na_2CO_3/0.17 M $NaHCO_3$

1. Order DIONEX P/N 039513 or

2. Thoroughly dissolve 19.078 g of Na_2CO_3 and 14.282 g of $NaHCO_3$ in 700 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 1,000 mL.

CAUTION

Do not use hydroxide eluents or hydroxide to adjust the pH of any eluent higher than pH 11 to effect selectivity changes. Using eluents with pHs greater than 11 may cause irreversible damage to the IonPac AS9-SC/AG9-SC Columns.

4.4.2 Preparation of Carbonate Eluents

A. Eluent: 2.0 mM Carbonate/0.75 mM Bicarbonate

Make the eluent by pipetting 10.0 mL of the eluent concentrate into a 1 L volumetric flask. (100X concentrate = 200 mM Na_2CO_3/75 mM $NaHCO_3$.). See Section 4.1, Preparation of Eluent Concentrates for the preparation of the 100 x eluent concentrate. 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.

4.4.3 Preparation of Borate Eluents

A. Eluent : 22 mM H_3BO_3/22 mM $Na_2B_4O_7$

Thoroughly dissolve 8.391 g $Na_2B_4O_7$/10 H_2O (MW 381.42 g/mole) plus 1.360 g H_3BO_3 (MW 61.84 g/mole) in 700 mL degassed, deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

B. Eluent Preparation for Gradient Program

1. Eluent : 10 mM H_3BO_3/10 mM $Na_2B_4O_7$

Thoroughly dissolve 3.814 g $Na_2B_4O_7$?10 H_2O (MW 381.42 g/mole) plus 0.618 g H_3BO_3 (MW 61.84 g/mole) in 700 mL degassed, deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

2. Purge: 50 mM H_3BO_3/50 mM $Na_2B_4O_7$

Thoroughly dissolve 19.071 g $Na_2B_4O_7$?10 H_2O (MW 381.42 g/mole) plus 3.092 g H_3BO_3 (MW 61.84 g/mole) in 700 mL degassed, deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

4.5 The Borate Eluent System

The borate eluent system gives the same elution order on the AS9-SC column as the carbonate eluent system (see Figure 4, Anion Separation using 22 mM Borate Eluent). However, the borate anion is a weaker “pusher” ion than carbonate. Therefore a higher concentration borate eluent is required to provide the same elution times observed with a carbonate eluent system. The major advantage of the borate system is that early eluting ions (i.e., fluoride, nitrate) can be easily spread out thus improving resolution simply by using a more dilute eluent (see Figure 5, Anion Separation using 10 mM Borate Eluent with Column Purge after Nitrate). Carbonate eluent systems can also be diluted to produce a similar effect but not as reliably due to carbon dioxide intrusion from the air. This improvement in resolution is especially beneficial when doing sub-ppm determinations of early eluting ions such as chlorite and bromate in the presence of high amounts of common anions such as fluoride and chloride.

4.6 Eluents that Contain Solvents

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. For example, 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 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 degassing eluents containing solvents, do not degas the eluent excessively since it is possible that a volatile solvent can be "boiled" off from the solution.

The AS9-SC can withstand common HPLC solvents in a concentration range of 0 - 100%. Solvents and water should be premixed in concentrations to 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 so that salts do not precipitate out in either the pump or the column.

Table 6
HPLC Solvents for Use with IonPac AS9-SC Columns

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

When using a solvent in an ionic eluent, column generated backpressures 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 backpressure limit for the IonPac AS9-SC columns is 4,000 psi. Therefore, any combination of the above, contributing to operating backpressure that totals up to 4,000 psi, can be used.

4.7 Regenerant Preparation for the AMMS III

The regenerant is 50 mN sulfuric acid. Dilute 100 mL (about 100 g) of 0.50 N sulfuric acid (P/N 037164 or P/N 039601) to 1 L using deionized water. If you are not using the AutoRegen Accessory (P/N 039594), prepare several liters of the regenerant.

For a guide to properly adjusting the regenerant flow rate, see Document No. 031727, the Product Manual for the Anion MicroMembrane Suppressor III, the AMMS III.

4.8 The Sample Concentrator

The Low Pressure Trace Anion Concentrator Column (TAC-LP1, P/N 046026), the Trace Anion Concentrator Column (TAC-2, P/N 043101), the Anion MicroConcentrator, AMC-1, (P/N 051760) or the IonPac AG9-SC 4-mm Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of the TAC-LP1, the TAC-2, the AMC-1, or the AG9-SC 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-SC leading to a lowering of detection limits by 2-5 orders of magnitude.

The concentrator column is used in lieu of the sample loop. Pump the sample onto the concentrator column in the **OPPOSITE** direction of the eluent flow. When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column.

For a detailed discussion of anion concentration techniques, refer to Section 3, Operation, of the Trace Anion Concentrator (TAC-2) Column Product Manual (Document No. 034467). For further information on the AMC-1 (P/N 051760) see, Product Manual for AMC-1 Anion MicroConcentrator Column, Document No. 031262.

SECTION 5 - EXAMPLE APPLICATIONS

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 Concentrator).

5.1 Production Test Chromatogram

Isocratic elution of anions on the IonPac AS9-SC Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, mono- and divalent anions, including oxyhalides can be isocratically separated and quantitated in a single injection. To guarantee that all IonPac AS9-SC (4-mm) Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Loop Volume:	25 µL
Analytical Column:	IonPac AS9-SC Analytical Column
Eluent:	2.0 mM Na_2CO_3/0.75 mM $NaHCO_3$
Eluent Flow Rate:	2.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA AutoSuppression Recycle Mode
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III
MMS Regenerant:	25-50 mN H_2SO_4
Expected Background Conductivity:	30 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

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

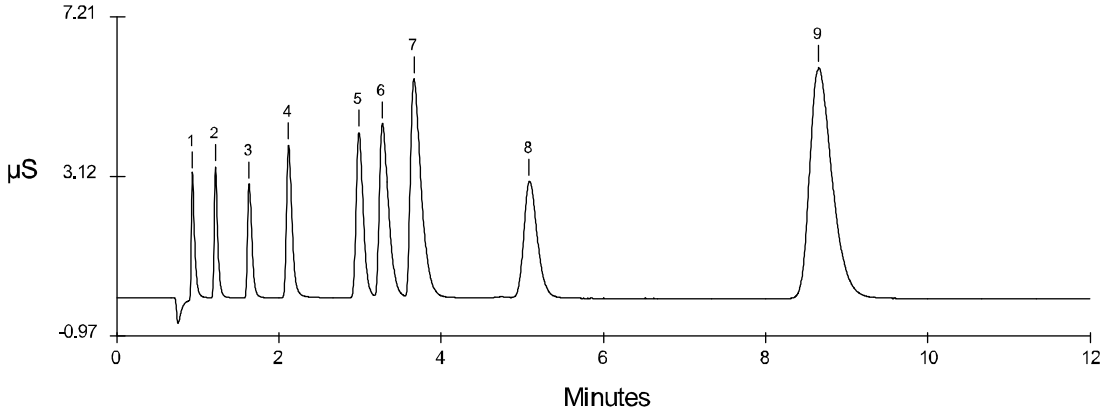


Figure 1
Production Test Chromatogram AS90-SC

5.2 Inorganic Anions Including Chlorate and Chlorite

Separation and elution of anions on the IonPac AS9-SC Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, monovalent and divalent anions can be isocratically separated and quantitated in a single injection. The carbonate/bicarbonate mixture allows the strength of the eluent, and thus the selectivity of the system to be changed by varying the HCO_3^-/CO_3^{2-} ratio. Furthermore, the suppressor reaction product is carbonic acid (H_2CO_3) and results in a low background conductivity (14-18 µS) suitable for isocratic analysis. For samples with similar concentrations of ions, the run time can be decreased to 1/2 the standard run time by doubling the flow rate. For samples with highly variable concentrations, additional resolution can be obtained at lower flow rates (see Section 4.4, Resolution of Low-Concentration Analytes).

Sample Loop Volume:	25 µL
Analytical Column:	IonPac AS9-SC Analytical Column
Eluent:	1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$
Eluent Flow Rate:	See Chromatogram
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA AutoSuppression Recycle Mode
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III
MMS Regenerant:	25-50 mN H_2SO_4
Expected Background Conductivity:	12-16 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent

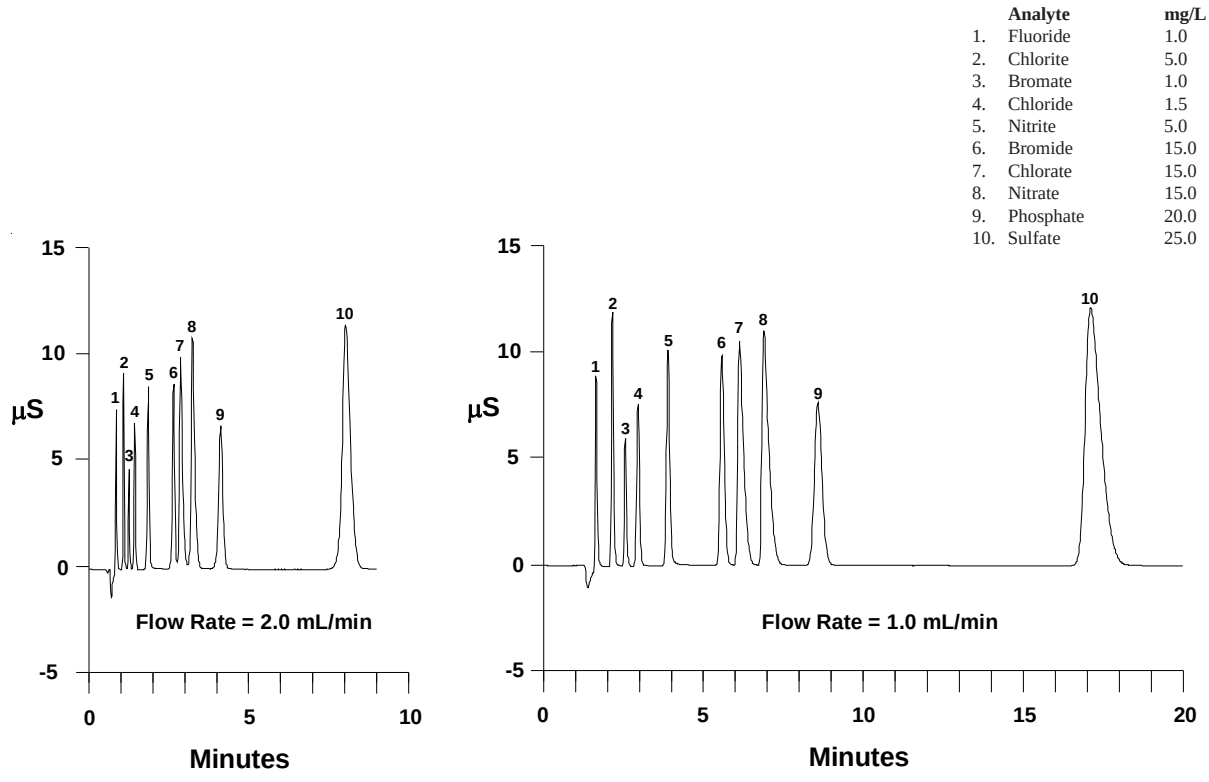
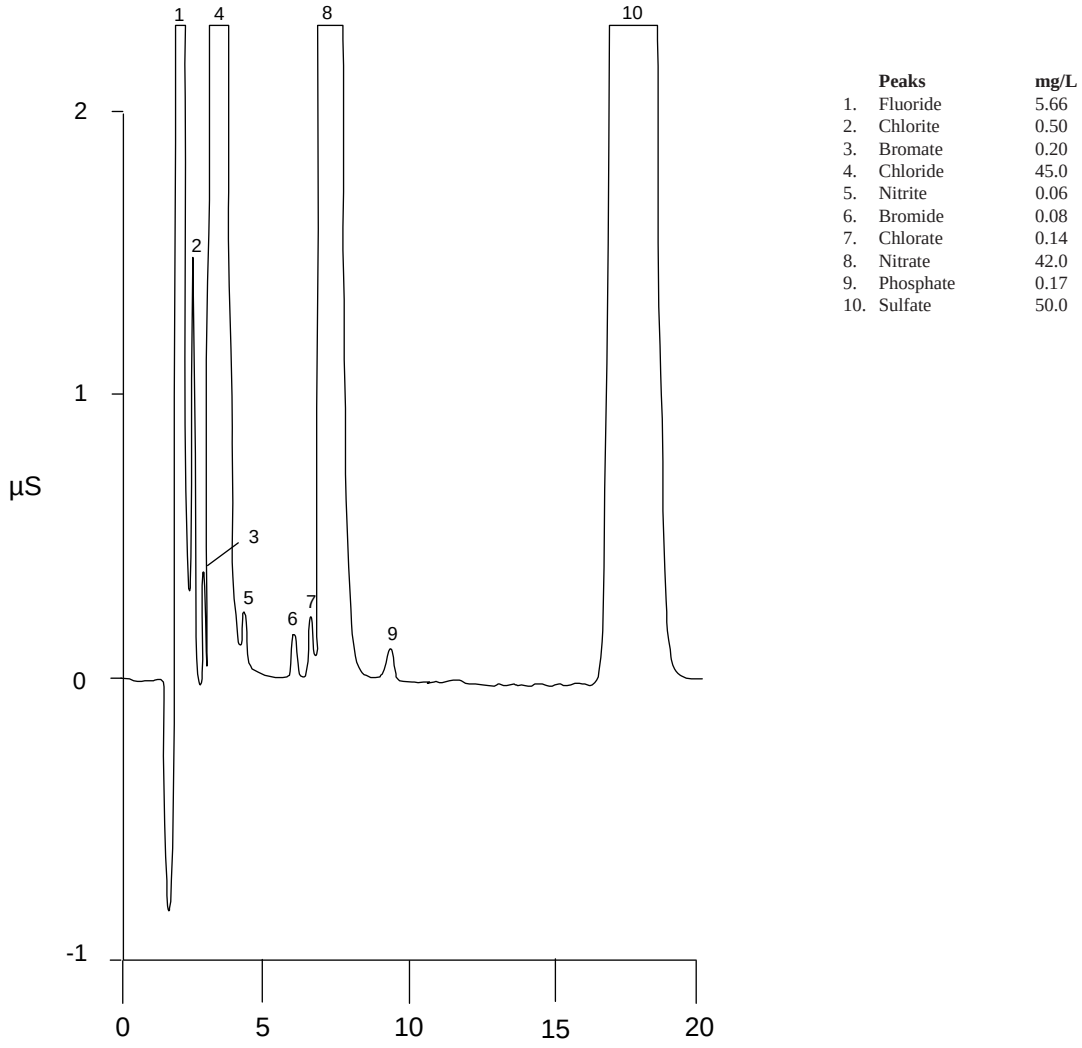


Figure 2
Inorganic Anions including Chlorate, Chlorite and Bromate

5.3 Resolution of Low-Concentration Analytes - EPA Water Matrix

The following example demonstrates the separation of sample analytes having highly variable concentrations. Setting the flow rate at 1.0 mL/min allows for increased resolution of very low concentration analytes such as bromate, chlorite, and chlorate in the following EPA water matrix sample.

Sample Loop Volume:	100 µL
Analytical Column:	IonPac AS9-SC Analytical Column
Eluent:	1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA (4-mm) AutoSuppression Recycle Mode
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4-mm)
MMS Regenerant:	25-50 mN H_2SO_4
Expected Background Conductivity:	12-16 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent



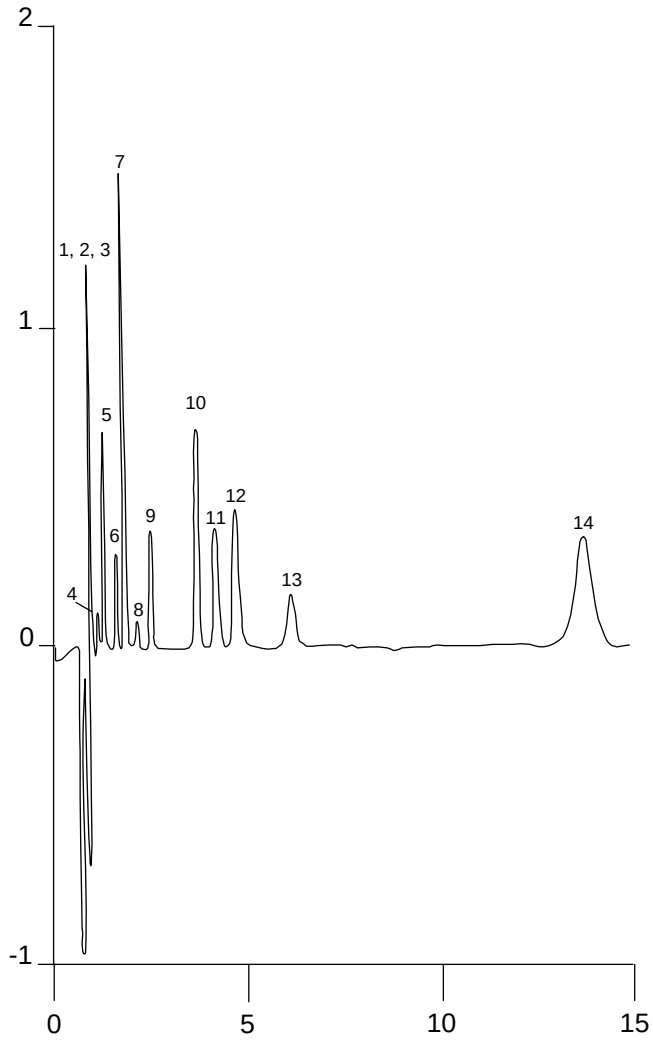
	Peaks	mg/L
1.	Fluoride	5.66
2.	Chlorite	0.50
3.	Bromate	0.20
4.	Chloride	45.0
5.	Nitrite	0.06
6.	Bromide	0.08
7.	Chlorate	0.14
8.	Nitrate	42.0
9.	Phosphate	0.17
10.	Sulfate	50.0

Figure 3
EPA Water Matrix

5.4 Varying the Eluent System - 22 mM Borate

The borate eluent gives the same elution order as the carbonate eluent traditionally used on the IonPac AS9. However, since the borate anion is a "weaker" eluent ion, higher concentrations are required to achieve the same elution time. The advantage of the borate eluent is increased resolution of early eluting ions.

Sample Loop Volume:	50 µL
Analytical Column:	IonPac AS9-SC Analytical Column
Eluent:	22 mM H_3BO_3/22 mM $Na_2B_4O_7$
Eluent Flow Rate:	2.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS-ULTRA AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III
MMS Regenerant:	25-50 mN H_2SO_4
Expected Background Conductivity:	5-6 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent



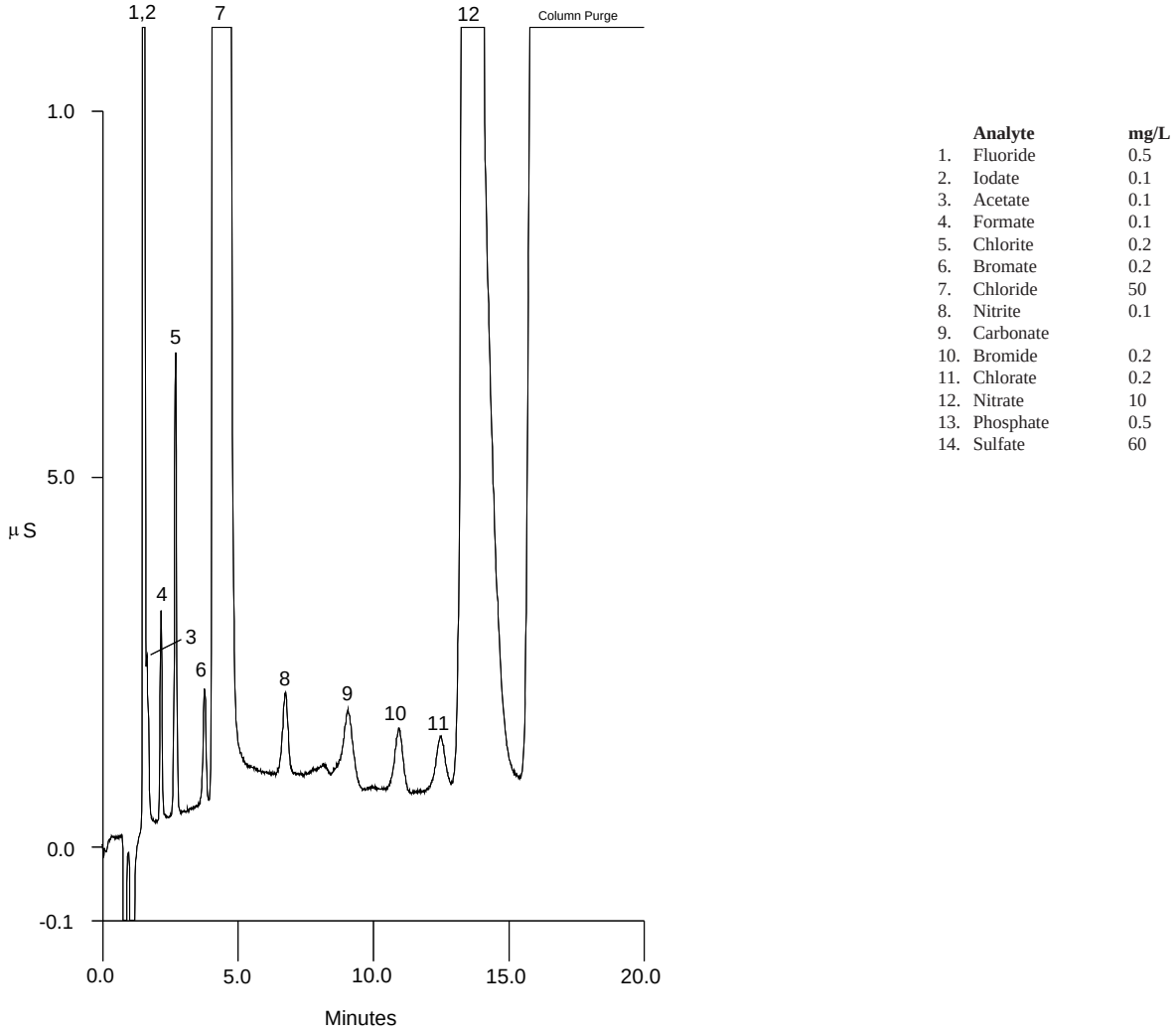
	Analyte	mg/L
1.	Fluoride	0.5
2.	Iodate	0.1
3.	Acetate	0.1
4.	Formate	0.1
5.	Chlorite	1.0
6.	Bromate	1.0
7.	Chloride	1.0
8.	Carbonate	-
9.	Nitrite	0.7
10.	Bromide	2.5
11.	Chlorate	2.0
12.	Nitrate	2.0
13.	Phosphate	2.5
14.	Sulfate	3.0

Figure 4
Anion Separation Using 22 mM Borate Eluent

5.5 Varying the Eluent System - 10 mM Borate with Column Purge

A low concentration of borate is used to improve resolution of early eluting peaks. This is particularly beneficial when quanititating chlorite and bromate at sub-ppm levels in the presence of high amounts of fluoride and chloride. High amounts of nitrate, phosphate, or sulfate can be rapidly removed from the column using a step change to a higher borate concentration. Dilute borate eluents are less affected by CO_2 contamination from the air than dilute carbonate eluents. The Thermal Stabilizer (TS-2, P/N 043117) was incorporated in this analysis for high sensitivity baseline stabilization.

Sample Loop Volume:	50 µL
Analytical Column:	IonPac AS9-SC Analytical Column
Eluent:	10 mM H_3BO_3/10 mM $Na_2B_4O_7$ with step change at 15.1 minutes to
Purge:	50 mM H_3BO_3/50 mM $Na_2B_4O_7$
Eluent Flow Rate:	2.0 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:	25-50 mN H_2SO_4
Expected Background Conductivity:	3-4 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Bicarbonate
Short-term Storage Solution (< 1 week):	Eluent



	Analyte	mg/L
1.	Fluoride	0.5
2.	Iodate	0.1
3.	Acetate	0.1
4.	Formate	0.1
5.	Chlorite	0.2
6.	Bromate	0.2
7.	Chloride	50
8.	Nitrite	0.1
9.	Carbonate	
10.	Bromide	0.2
11.	Chlorate	0.2
12.	Nitrate	10
13.	Phosphate	0.5
14.	Sulfate	60

Figure 5
Anion Separation using 10 mM Borate Eluent with Column Purge after Nitrate

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS9-SC 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 Worldwide Office nearest you (see, DIONEX Worldwide Offices).

6.1 High Backpressure

6.1.1 Finding the Source of High System Pressure

Total system pressure when using the IonPac AG9-SC (4-mm) Guard and AS9-SC (4-mm) Analytical Columns at 1.0 mL/min should also be less than 1,500 psi when using the test chromatogram conditions. Refer to Section 3.4, Eluent Preparation, to see how solvent concentration can affect the column operating pressure. If the system pressure is higher than 2,000 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 035331) 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 of 1,500 psi when the column(s) are connected. The Anion MicroMembrane Suppressor may add up to 100 psi. No other components should add more than 100 psi of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high backpressure, 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. Using two open end wrenches, carefully unscrew the inlet (top) column fitting.

C. Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you **DO NOT SCRATCH THE WALLS OF THE END FITTING.** Discard the old bed support assembly.

D. Place a new bed support assembly into the end fitting. Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

Part	IonPac AS9-SC 4-mm Columns (P/N)
Analytical Column	043185
Guard Column	043186
Bed Support Assembly	042955
End Fitting	052809

CAUTION

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

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

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

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
1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$	14 - 18 µS
5 mM $Na_2B_4O_7$	3 - 4 µS
10 mM $Na_2B_4O_7$	4 - 6 µS
22 mM $Na_2B_4O_7$	5 - 7 µS

6.2.1 Preparation of Eluents

A. Make sure that the eluents and the regenerant are made correctly.

B. Make sure that the eluents are made from chemicals with the recommended purity.

C. Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

6.2.2 Borate Eluent Precautions

When using the borate eluent a few precautions should be kept in mind.

Baseline Stability

In order to have the expected baseline stability, the Anion MicroMembrane Suppressor III, AMMS III or the ASRS-ULTRA, must be reliably removing all the cations (e.g., sodium) when using the standard carbonate eluent, the AMMS III or the ASRS-ULTRA has only to remove 5.3 mM sodium. However, when using a 22 mM borate eluent, the AMMS III or the ASRS-ULTRA has to remove 44 mM sodium. The AMMS III or the ASRS-ULTRA is fully capable of removing this level of sodium, but only if it is functioning at optimum efficiency. If it is not, then a high

and varying baseline will result. In this case the AMMS III or the ASRS-ULTRA will need to be serviced as outlined in the AMMS III manual (see Document No. 031727, Product Manual for the Anion MicroMembrane Suppressor III, the AMMS III or Document No. 031367, the "Product Manual for the Anion Self-Regenerating Suppressor-ULTRA, the ASRS-ULTRA.")

Carbonate Peak

When using a carbonate eluent, carbonate in the sample is not normally evident in the chromatogram. However, when using other eluents (e.g., borate) a carbonate peak will be observed. The elution time of this peak will depend upon the pH of the eluent. The higher the pH, the higher the carbonate/bicarbonate ratio. The divalent carbonate form tends to elute later then the bicarbonate form. The converse is true as the pH is lowered. The borate eluent described in this manual is an equimolar solution of sodium tetraborate and boric acid. The boric acid is added to lower the tetraborate eluent pH. This places the carbonate peak between chloride and nitrite (see Figure 5, Anion Separation using 22 mM Borate Eluent) when using a 22 mM borate eluent formulation. When using a more dilute eluent the carbonate is retained longer since it is divalent. This explains why the carbonate peak elutes after the nitrite peak when using the 10 mM borate eluent formulation. Since the carbonate peak is reduced by almost two orders of magnitude during passage through the AMMS III or the ASRS-ULTRA, this method is not useful for carbonate quantification.

Eluent Impurities

Use an Anion Trap Column (ATC-3, P/N 059660) to retard divalent anion contaminants (e.g., sulfate) in dilute eluents (e.g., 10 mM borate) so that they do not concentrate on the analytical column when the dilute eluent is being run through the column and then elute as sharp bands when the following strong eluent (e.g., 50 mM borate) is pumped through the column. Dilute eluent contaminants eluting as sharp bands in the strong eluent can interfere with the quantification of sample divalent anions (see Figure 6, Anion Separation using 10 mM Borate Eluent with Column Purge after Nitrate). If the tetraborate salts or deionized water are contaminated (e.g., with sulfate) this will prolong the reequilibration process from the strong eluent back to the dilute eluent.

6.2.3 A Contaminated Guard or Analytical Column

Remove the IonPac AG9-SC Guard and AS9-SC 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-SC 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 Care."

6.2.4 A Contaminated Anion Trap Column, ATC-3

When doing gradient analysis, has the Anion Trap Column, 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-3 is trapping contaminants from the eluent. The eluents probably have too many impurities (see items 1 - 3 above).

If the ATC-3 is already installed, remove it. Is the background conductivity still high? If the background conductivity decreases, the ATC-3 is the source of the high background conductivity.

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

B. Flush the ATC-3 with 200 mL of 70 mM $Na_2B_4O_7$ at 2.0 mL/min on a 4-mm system.

C. Equilibrate the ATC-3 with the strongest eluent used during the gradient run. Use a flow rate of 2.0 mL/min on a 4-mm system.

D. If the problem persists, replace the ATC-3.

6.2.5 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the columns and the suppressor. Pump deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

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 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 Care,” for recommended column cleanup procedures.

   Possible sources of column contamination are impurities in chemicals and in the deionized water used for eluents or components of the sample matrix.. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of at least 18.2 megohm-cm.

D. Diluting the eluent will improve peak resolution, but will also increase the analytes’ retention times. If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see “Column Care”).

   After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column. If you need assistance in solving resolution problems, contact the nearest DIONEX Worldwide Office (see, DIONEX Worldwide Offices)

6.2.6 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 **REGEN IN** port.

E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.2 M oxalic acid. Pump this solution through the ASRS-ULTRA (4-mm) at 1-2 mL/min for 30 minutes.

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.

6.2.7 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 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 MMS.

2. If the background conductivity is low when freshly prepared regenerant is run through the 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.

6.2.8 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_2SO_4/90% acetonitrile. H_2SO_4/acetonitrile solutions are not stable during long term storage so this cleanup solution must be made immediately before each column cleanup. Alternatively, it can be proportioned from 1 bottle containing 1.0 M H_2SO_4 and another bottle containing 100% acetonitrile. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 1.0 mL/min for 60 minutes.

NOTE

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

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 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.

6.4 Poor Peak Resolution

Poor peak resolution can be due any or all of the following factors.

6.4.1 Loss of Column Efficiency

A. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.010" to make all eluent liquid line connections between the injection valve and the detector cell inlet, and that the tubing lengths are as short as possible. Check for leaks.

B. Check to see if headspace has developed in the guard or analytical column (e.g., due to improper use of the column such as submitting it to high pressures). Remove the column's top end fitting (see Section 5.1.2, Replacing Column Bed Support Assemblies). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

6.4.2 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. Ensure that 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.

6.5 Spurious Peaks

A. If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, polyvalent anions may contaminate the analytical column. The retention times for the analytes will then decrease and spurious, inefficient (broad) peaks can show up at unexpected times. Clean the column as indicated in "Column Care."

B. If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS9-SC columns, contact the nearest DIONEX Worldwide Office (see, DIONEX Worldwide Offices).

C. 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.

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-SC columns is 4,000 psi.

COLUMN START-UP

The column is shipped in 2.0 mM Na_2CO_3/0.75 mM $NaHCO_3$ (Eluent) storage solution.

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

COLUMN STORAGE

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

COLUMN CLEANUP

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

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

BASE-SOLUBLE CONTAMINANTS

WARNING
Do not use the standard NaOH procedure described in other DIONEX documentation for base cleanup of IonPac AS9-SC/AG9-SC Columns. This procedure will destroy IonPac AS9-SC/AG9-SC Columns

A. Prepare a 500 mL solution of 200 mM Na_2CO_3/75 mM $NaHCO_3$ (see Section 4.1, Preparation of Eluent Concentrates).

B. Disconnect the anion suppressor from the IonPac AS9-SC 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. Direct the effluent from the outlet line of the AS9-SC Guard Column to a separate waste container.

CAUTION
When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

C. Set the pump flow rate to 1.0 mL/min.

D. If your eluent contains a solvent that is not compatible with the cleanup solution of 200 mM Na_2CO_3/75 mM $NaHCO_3$, rinse the column for 15 minutes with deionized water before pumping the cleanup solution over the column.

E. Pump the cleanup solution through the column for 30-60 minutes

F. If the application eluent contains a solvent that is not compatible with the cleanup solution, rinse the column for 15 minutes with deionized water. Then pump the application eluent over the column.

G. Reconnect the AMMS III to the AS9-SC Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

H. Equilibrate the column(s) with the application eluent before resuming normal operation.

NOTE
It may not be necessary to use the above cleanup solution. You may find it more beneficial, depending on your sample matrix, to use a 10X concentrate of your sodium carbonate/bicarbonate or sodium borate eluent. Note that the maximum solubility of sodium borate is 300 mM.

METAL CONTAMINANTS

WARNING
Do not use the standard HCl procedure described in other DIONEX documentation for base cleanup of IonPac AS9-SC/AG9-SC Columns. This procedure will destroy IonPac AS9-SC/AG9-SC Columns

A. Prepare a 500 mL cleanup solution of 100 mM oxalic acid/50 mM NaOH (pH 2.5 to 3.0) by dissolving 6.2 g of oxalic acid dihydrate with 4.2 g (2.6 mL) of 50% w/w sodium hydroxide concentrate solution with 400 ml of deionized water having a specific resistance of 18.2 megohm-cm. After thoroughly mixing, dilute to a final volume of 500 mL with deionized water having a specific resistance of 18.2 megohm-cm.

B. Disconnect the anion suppressor from the IonPac AS9-SC 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. Direct the effluent from the outlet line of the AS9-SC Guard Column to a separate waste container.

CAUTION
When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

C. Set the pump flow rate to 1.0 mL/min.

D. If your eluent contains a solvent that is not compatible with the cleanup solution of 100 mM oxalic acid/50 mM NaOH, rinse the column for 15 minutes with deionized water before pumping the cleanup solution over the column.

E. Pump the cleanup solution through the column for 30-60 minutes

F. If the application eluent contains a solvent that is not compatible with the cleanup solution, rinse the column for 15 minutes with deionized water. Then pump the application eluent over the column.

G. Reconnect the AMMS III to the AS9-SC Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

H. Equilibrate the column(s) with the application eluent before resuming normal operation.

ORGANIC CONTAMINANTS

A. Prepare a two bottles with approximately 500 mL each of the following two eluents. In the first bottle (E1) place 100% acetonitrile. In the second bottle (E2) make a 1M solution of NaCl using deionized water having a specific resistance of 18.2 megohm-cm and then adjust the pH of the solution to pH 2.0 using HCl. Because acetonitrile slowly breaks down to form ammonia and acetate in acidic aqueous solutions, the column cleaning solution (80% E1/20% E2) is created by proportionally mixing these two eluents with the gradient pump which then isocratically pumps the resulting cleaning solution over the columns to be cleaned.

B. Disconnect the anion suppressor from the IonPac AS9-SC 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. Direct the effluent from the outlet line of the AS9-SC Guard Column to a separate waste container.

CAUTION

When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

C. Set the pump flow rate to 2.0 mL/min.

D. If the application eluent contains a salt that is not compatible with 80% acetonitrile, rinse the column for 10 minutes with deionized water before pumping the cleaning solution over the column.

E. The cleaning solution is created by proportionally mixing 80% E1 plus 20% E2 with the gradient pump. Isocratically pump this cleaning solution through the column for at least 60 minutes.

F. If the application contains a salt that is not compatible with 80% acetonitrile, rinse the column for 10 minutes with deionized water before pumping the application eluent over the column.

G. Reconnect the anion suppressor to the AS9-SC Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

H. Equilibrate the column(s) with the application eluent before resuming normal operation.