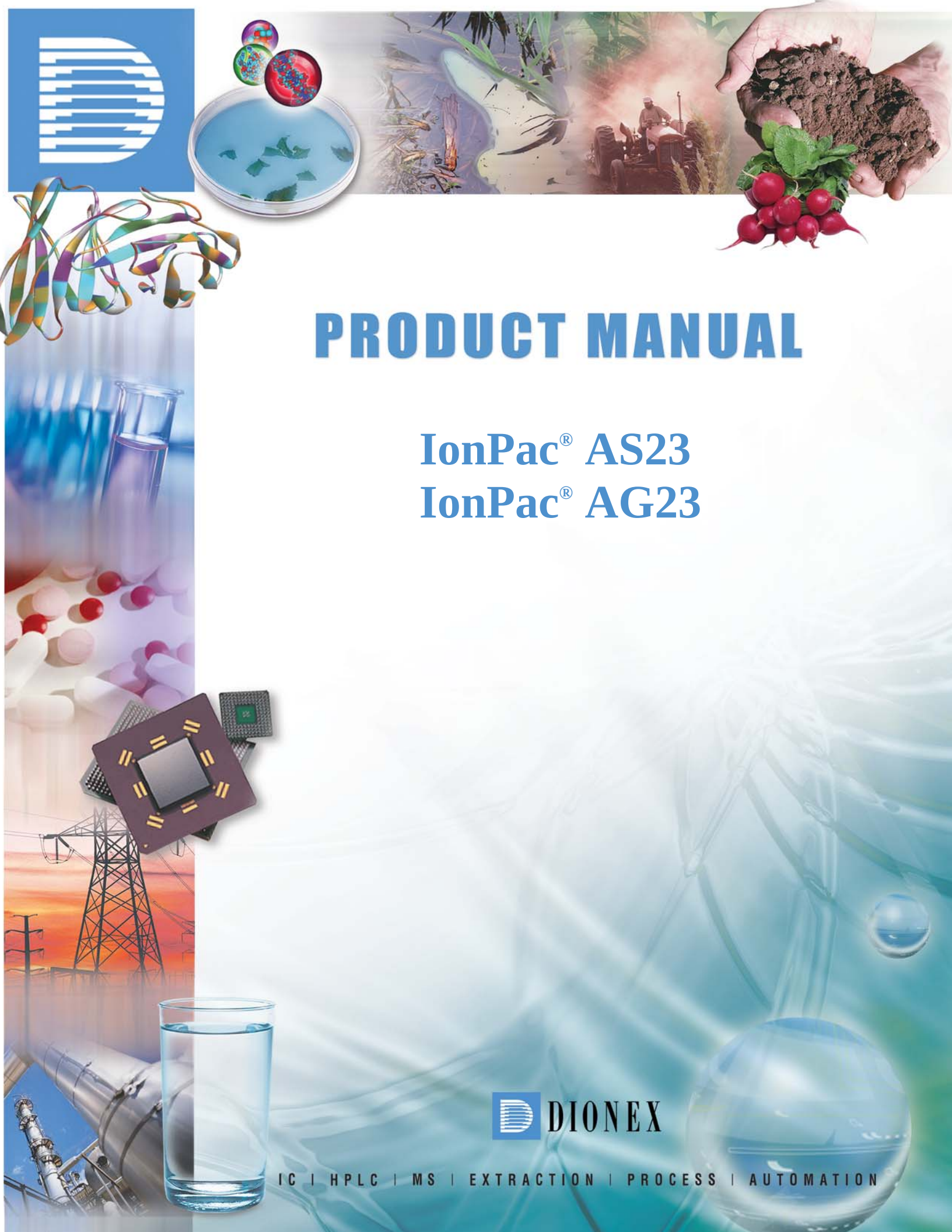

PRODUCT MANUAL

IonPac® AS23
IonPac® AG23

DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

IONPAC® AG23 GUARD COLUMNS

(4 x 50 mm, P/N 064147)

(2 x 50 mm, P/N 064143)

IONPAC® AS23 ANALYTICAL COLUMNS

(4 x 250 mm, P/N 064149)

(2 x 250 mm, P/N 064145)

Document No. 065120
Revision 02
7 April 2006

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SECTION 1 - INTRODUCTION TO IONPAC AS23/AG23 CHROMATOGRAPHY

The IonPac® AS23 Analytical Column in combination with the AG23 Guard Column is designed for the analysis of inorganic anions and oxyhalides including bromate, chlorite, and chlorate. The selectivity of the IonPac AS23 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 and oxyhalides. The AS23 is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS23 can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor ULTRA (ASRS ULTRA II). The IonPac AS23 has nominal efficiency for sulfate using standard operating conditions of at least 9,000 plates/column.

Table 1
IonPac AS23/AG23 Packing Specifications

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
AS23 4 x 250 mm	6.0	55	320	Alkyl/Alkanol quaternary ammonium	Low
AG23 4 x 50 mm	11.0	55	6	Alkyl/Alkanol quaternary ammonium	Low
AS23 2 x 250 mm	6.0	55	80.0	Alkyl/Alkanol quaternary ammonium	Low
AG23 2 x 50 mm	11.0	55	1.5	Alkyl/Alkanol quaternary ammonium	Low

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

Table 2
AS23/AG23 Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS23 4-mm Analytical	≤ 1800 (12.41)	1.0	2.0
AG23 4-mm Guard	≤ 300 (2.07)	1.0	2.0
AS23 + AG23 4-mm columns	**≤ 2100 (14.48)**	**1.0**	**2.0**
AS23 2-mm Analytical	≤ 1800 (12.41)	0.25	0.5
AG23 2-mm Guard	≤ 300 (2.07)	0.25	0.5
AS23 + AG23 2-mm columns	**≤ 2100 (14.48)**	**0.25**	**0.5**

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 AS23 2-mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a pump with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a microbore pump (1/16" pistons) is recommended.

3.1.2 System Requirements for 4-mm Operation

The IonPac AS23 4-mm Guard and Analytical Columns are designed to run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a pump with a standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons).

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

3.2 The Sample Concentrator

For 2-mm or 4-mm concentrator work, use the IonPac AG23 Guard Column when a single piston pump is used for sample delivery. Use the Trace Anion Concentrator Low Pressure Column (TAC-LP1, P/N 046026) or Trace Anion Concentrator Ultra Low Pressure Column (TAC-ULP1, P/N 061400) when the sample is delivered with a syringe or with an autosampler. Alternatively, use the Ultra Trace Anion Concentrator Low Pressure Column (UTAC-LP1, P/N 063079), Ultra Trace Anion Concentrator Ultra Low Pressure Column (UTAC-ULP1, P/N 063475), or Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459). The TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, or the IonPac AG23 Guard Column can be used for trace anion concentration work. The concentrator column is used in lieu of the sample loop. Pump the sample onto the concentrator column in the **OPPOSITE** direction of the eluent flow.

When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample as this can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column.

The function of the TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, or the AG23 Guard Column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" all anionic analyte species onto the TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, or the AG23 leading to a lowering of detection limits by 2–5 orders of magnitude. The unique advantage to the analytical chemist of the TAC-LP1, TAC-ULP1, UTAC-LP1, UTAC-ULP1, UTAC-XLP1, or the AG23 in these applications is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

For a detailed discussion of anion concentration techniques, refer to Section 3, "Operation," of the Trace Anion Concentrator (TAC-LP1 and TAC-ULP1) Column Product Manual (Document No. 034972) or Section 3, "Operation," of the Ultra Trace Anion Concentrator (UTAC-XP1, UTAC-ULP1, and UTAC-XLP1) Column Product Manual (Document No. 065091).

3.3 The Injection Loop

3.3.1 The 2-mm System Injection Loop, 2 - 15 µL

For most applications on a 2-mm analytical system, a 2 - 15 µL injection loop is sufficient. 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 AS23 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4-mm analytical system (Section 2, "Comparison of Ion Chromatography Systems").

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

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop 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 200 µL.

3.4 THE IONPAC AG23 GUARD COLUMN

An IonPac AG23 Guard Column is normally used with the IonPac AS23 Analytical Column. Retention times will increase by approximately 2% 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 AG23 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS23 Analytical Column.

3.5 Eluent Storage

IonPac AS23 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).

3.6 Anion Self-Regenerating Suppressor (ASRS ULTRA II) 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 II modes of operation.

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

If you are installing an IonPac AS23 4-mm Analytical Column, use an ASRS ULTRA II (4-mm, P/N 053946).
If you are installing an IonPac AS23 2-mm Analytical Column, use an ASRS ULTRA II (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 II (ASRS ULTRA II.)"

3.7 Anion Atlas Electrolytic Suppressor (AAES) Requirements

An Atlas Anion Electrolytic Suppressor (AAES) may be used instead of an ASRS ULTRA II for applications that require suppressed conductivity detection. The AAES (P/N 056116) can be used for AS23 2-mm and 4-mm applications using eluents up to 25 µeq/min.

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

3.8 Anion MicroMembrane Suppressor (AMMS III) Requirements

An Anion MicroMembrane Suppressor (AMMS III) may be used instead of an ASRS ULTRA II (4-mm) for applications that require suppressed conductivity detection. Use an AMMS III (P/N 056750) with the IonPac AS23 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."

3.9 Using AutoRegen with the ASRS ULTRA II 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).

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

Dionex recommends using the Displacement Chemical Regeneration (DCR) Mode for chemical suppression using sulfuric acid (H_2SO_4) 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.

3.11 Detector Requirements

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

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	2-mm: 5 µL Loop + 0.8 µL Injection valve dead volume 4-mm: 25 µL Loop + 0.8 µL Injection valve dead volume
Column:	2-mm: AS23 2-mm Analytical Column + AG23 2-mm Guard Column 4-mm: AS23 4-mm Analytical Column + AG23 4-mm Guard Column
Eluent:	4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	2-mm: 0.25 mL/min 4-mm: 1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (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_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	20-22 µS
Storage Solution:	Eluent

4.2 IonPac AS23 Operation Precautions

CAUTIONS
Filter and Degas Eluents
Filter Samples
Eluent pH between 0 and 14
Sample pH between 0 and 14
0.5 mL/min Maximum Flow Rate for 2-mm Columns
2.0 mL/min Maximum Flow Rate for 4-mm Columns
Maximum Operating Pressure = 3,000 psi (20.68 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 AS23 Eluent Concentrate (P/N 064161) 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 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.

4.3.3 Solvents

Solvents can be added to the ionic eluents used with IonPac AS23 columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the IonPac AS23 columns is 3,000 psi (20.68 MPa).

The AS23 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 1
HPLC Solvents for Use with IonPac AS23 Columns

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

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

*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 II) 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.***

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

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

SAFETY **NEVER ADD THE ACETONITRILE DIRECTLY TO THE BASIC CARBONATE OR HYDROXIDE ELUENT SOLUTIONS.**

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 II (ASRS ULTRA II) see Document No. 031727, the "Product Manual for the Anion MicroMembrane Suppressor III, the AMMS III."

SECTION 5 - EXAMPLE APPLICATIONS

The chromatograms in this section were obtained using columns that reproduced the Production test Chromatogram (see Section 5.3, "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."

5.1 Preparation of Eluent Stock Solution Concentrates

A. AS23 Sodium Carbonate/Bicarbonate Eluent Concentrate (0.45 M Na2CO3/0.08 M NaHCO3)

Order DIONEX P/N 064161
or
Thoroughly dissolve 47.7 g of sodium carbonate (MW 106.00 g/mole) plus 6.72 g sodium bicarbonate (MW 84.00 g/mole) in 700 L of deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask.
Dilute to a final volume of 1,000 mL.

B. 0.5 M Sodium Carbonate (Na_2CO_3) Concentrate

Order Dionex P/N 037162
or
Thoroughly dissolve 26.49 g of Na_2CO_3 in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

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.

C. 0.5 M Sodium Bicarbonate ($NaHCO_3$) Concentrate

Order Dionex P/N 037163
or
Thoroughly dissolve 21.00 g of $NaHCO_3$ in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

5.2 Eluent Preparation

Eluent: 4.5 mM Sodium Carbonate/0.8 mM Sodium bicarbonate

A. Using AS23 Eluent Concentrate
By Weight: Weigh 988.0 g of deionized water and add 10.5 g of the AS23 Eluent Concentrate.
By Volume: To make 1 liter of eluent, pipet 10 mL of the AS23 Eluent Concentrate into a 1 L volumentric flask and dilute to a final volume of 1 L using deionized water.

B. Using 0.5 M Na_2CO_3 and 0.5 M $NaHCO_3$ Concentrates
By Weight: Weigh 987.42 g of deionized water and add 9.45 g of 0.5 M Na_2CO_3 plus 1.68 g of 0.5 M $NaHCO_3$.
By Volume: Prepare the eluent by pipetting 9.0 mL of 0.5 M Na_2CO_3 plus 1.6 mL of 0.5 M $NaHCO_3$ into a 1 L volumetric flask. Use degassed, deionized water with a specific resistance of 18.2 megohm-cm to dilute the concentrate to a final volume of 1,000 mL.

5.3 Production Test Chromatograms

Isocratic elution of inorganic anions and oxyhalides on the IonPac AS23 Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. The IonPac AS23 Analytical Column should always be used with the IonPac AG23 Guard Column. To guarantee that all IonPac AS23 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Volume:	4-mm: 25 µL Loop + 0.8 µL Injection valve dead volume
Column:	See Chromatogram
Eluent:	4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (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:	20 - 22 µS
Storage Solution:	Eluent

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

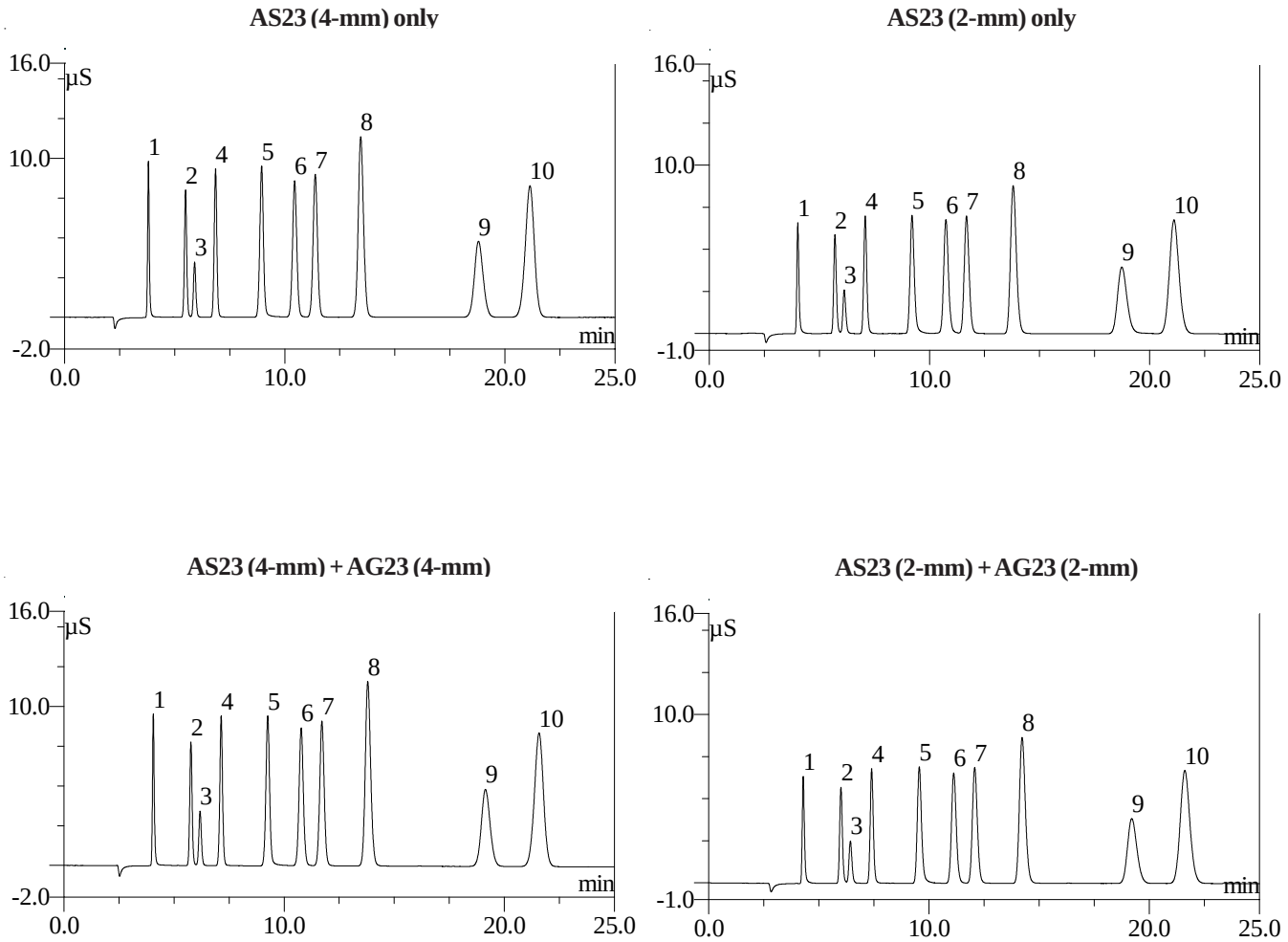

Figure 1
IonPac AS23 Production Test Chromatograms

5.4 Effect of Temperature on the AS23 Selectivity

The following chromatograms demonstrate the effect of temperature on the AS23 selectivity. Monovalent inorganic anions have slightly shorter retention time and divalent inorganic anions have slightly longer retention time as temperature changes from room temperature (21.5 °C) to 35 °C.

Sample Loop Volume:	25 µL
Column:	IonPac AS23 (4-mm) Analytical Column + IonPac AG23 (4-mm) Guard Column
Eluent:	4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Temperature:	See Chromatogram
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, (if eluent suppression required is less than 25 µeq/min)
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-22 µS

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

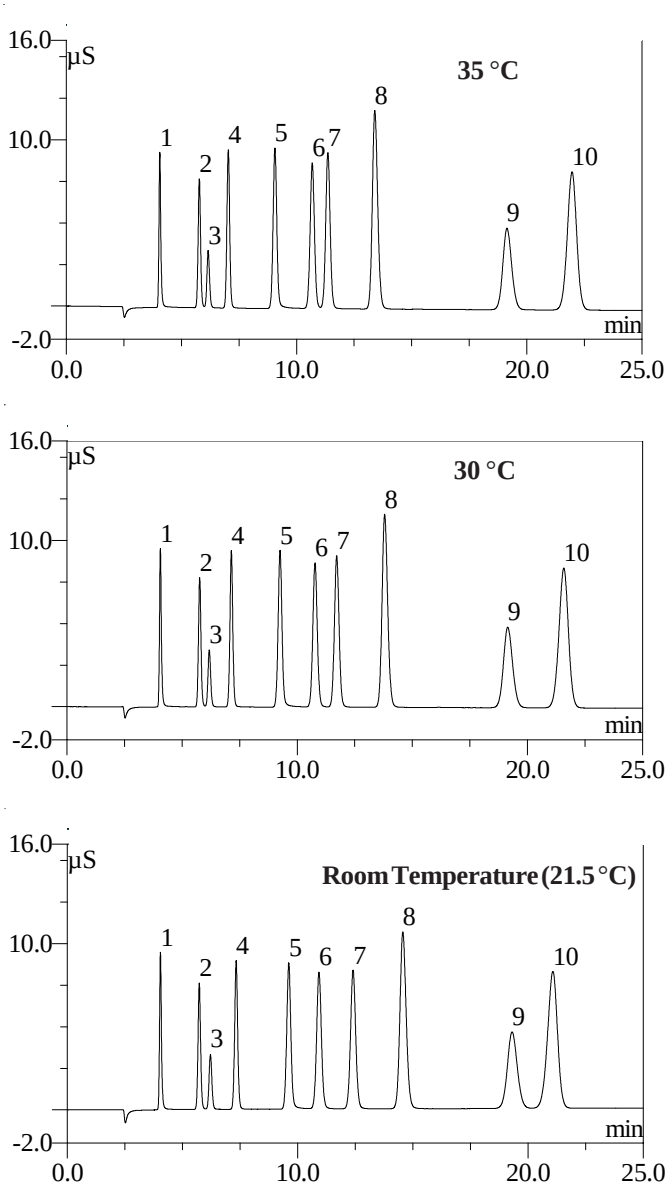

Figure 2
Effect of Temperature in AS23 Selectivity

5.5 Analysis of Simulated Drinking Water

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

Sample Loop Volume:	200 µL
Column:	IonPac AS23 (4-mm) Analytical Column + IonPac AG23 (4-mm) Guard Column
Eluent:	4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	1.0 mL/min.
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (4-mm) AutoSuppression® External 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:	20 - 22 µS

	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.	Chlorate	0.01
7.	Bromide	0.01
8.	Nitrate	10.00
9.	Carbonate	50.00
10.	Phosphate	0.10
11.	Sulfate	50.00

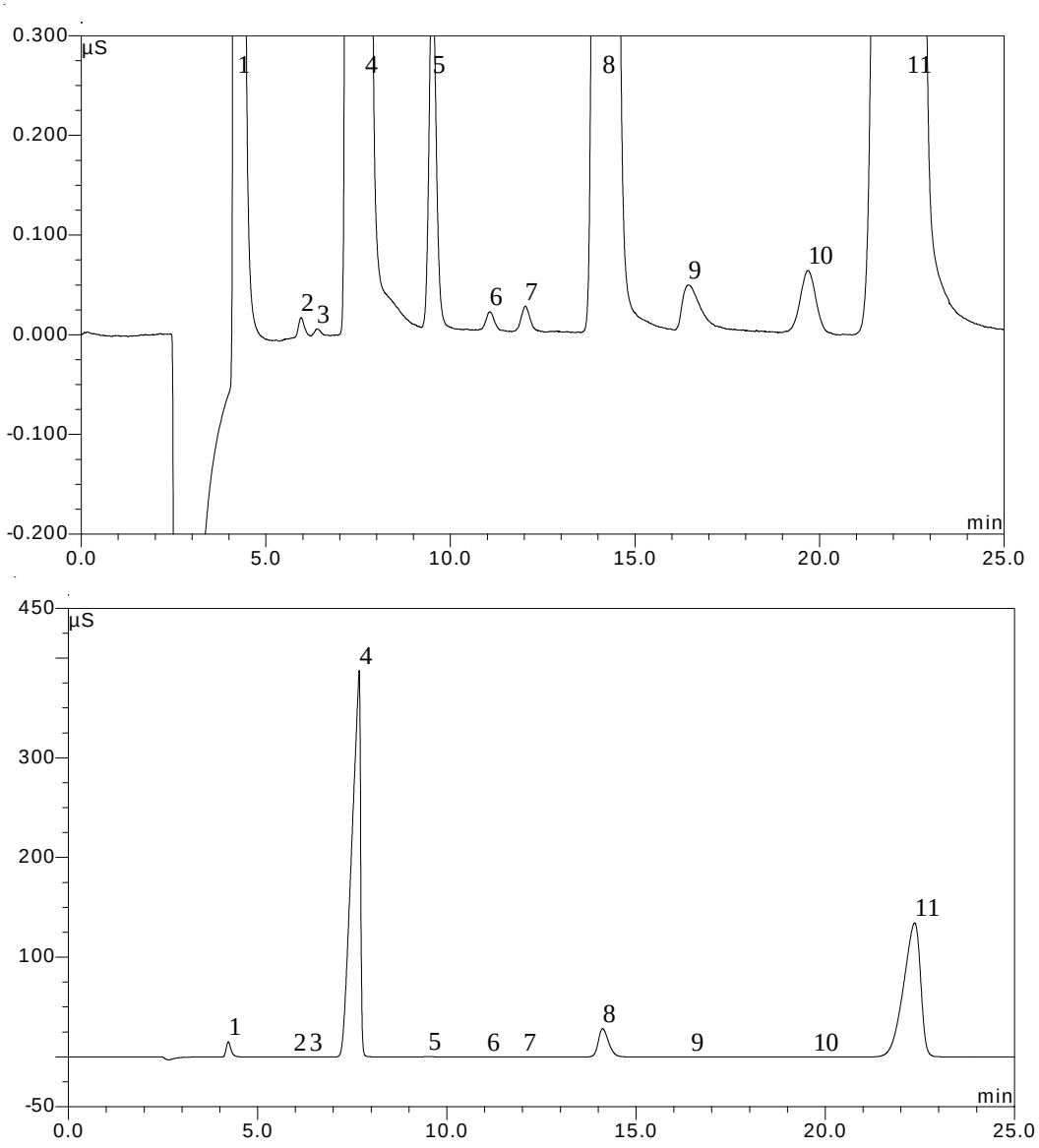

Figure 3
Separation of Simulated Drinking Water on AS23

5.6 Separation of Anions in Municipal Drinking Water Spiked with Surrogate Anions

The following chromatogram shows the analysis of a drinking water sample spiked with 1 ppm Malonate and Succinate using the IonPac AS23 column and a 200 µL injection loop. Notice the excellent separation of surrogate anions from sulfate.

Sample Loop Volume:	200 µL
Column:	IonPac AS23 (4-mm) Analytical Column + IonPac AG23 (4-mm) Guard Column
Eluent:	4.5 mM Na_2CO_3 0.8 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II AutoSuppression Recycle Mode
or AES Suppressor:	Atlas Anion Electrolytic Suppressor, AAES, (if eluent suppression required is less than 25 µeq/min)
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	20-23 µS

	Analyte
1.	Fluoride
2.	Formate
3.	Chlorite
4.	Chloride
5.	Nitrite
6.	*unknown*
7.	Chlorate
8.	Bromide
9.	Nitrate
10.	Carbonate
11.	Phosphate
12.	Sulfate
13.	Malonate
14.	Succinate
15.	Oxalate

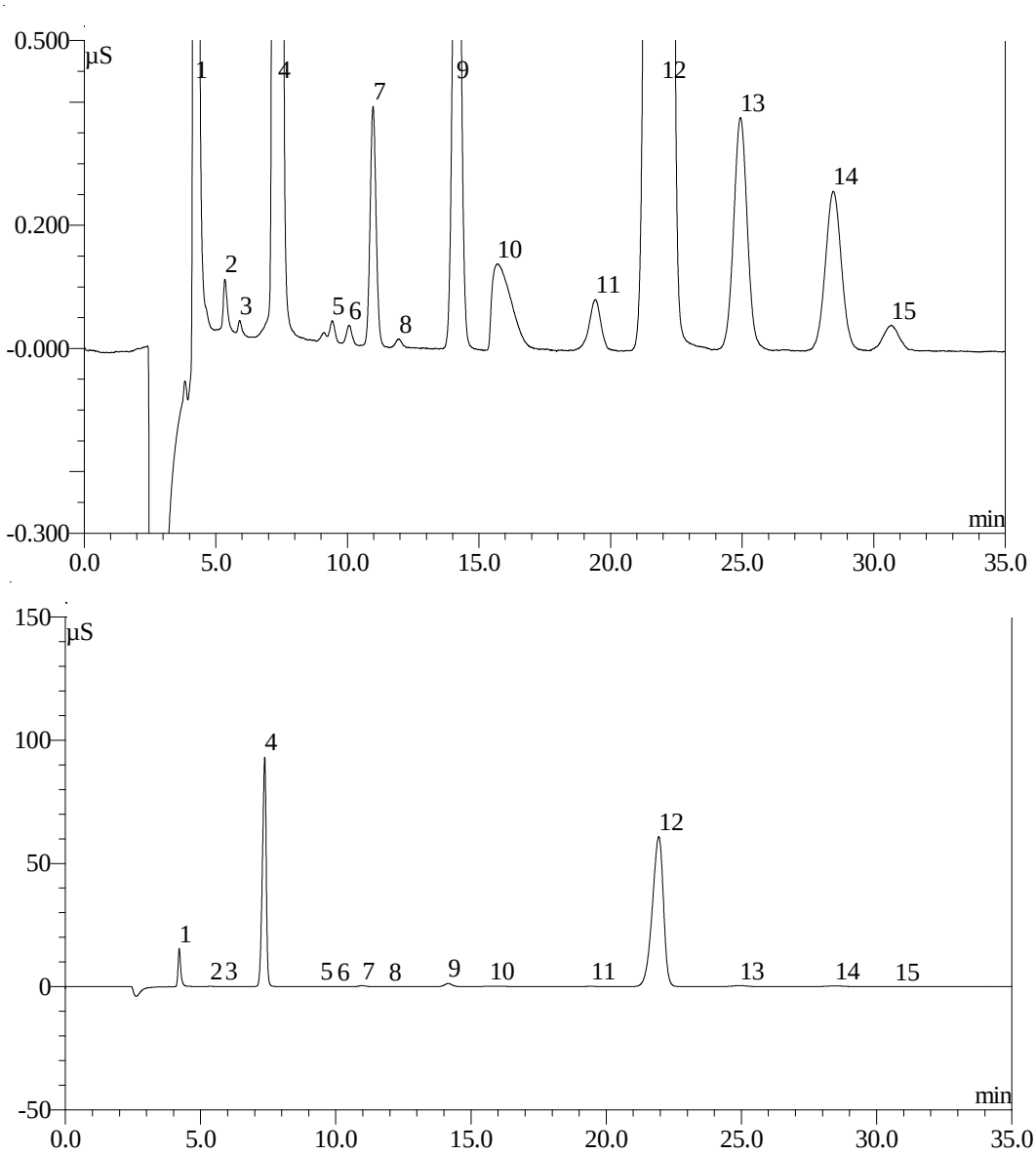

Figure 4
Separation of Anions in Municipal Drinking Water on AS23

5.7 Analysis of Waste Water with the IonPac AS23 Column

The following chromatogram shows the analysis of a waste water sample using the IonPac AS23 column. Notice that 2.5 µL injection loop is used for this application due to the high ionic strength of the waste water sample..

Sample Loop Volume:	2.5 µL
Column:	IonPac AS23 (4-mm) Analytical Column + IonPac AG23 (4-mm) Guard Column
Eluent:	4.5 mM Na_2CO_3 0.8 mM $NaHCO_3$
Temperature:	30 °C
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II AutoSuppression Recycle Mode

	Analyte	mg/L (ppm)
1.	Acetate	877.8
2.	Formate	183.2
3.	Chloride	61.8
4.	Nitrite	0.6
5.	Chlorate	213.8
6.	Nitrate	1.5
7.	Phosphate	1.4
8.	Sulfate	1211.1
9.	Unkknown	NQ

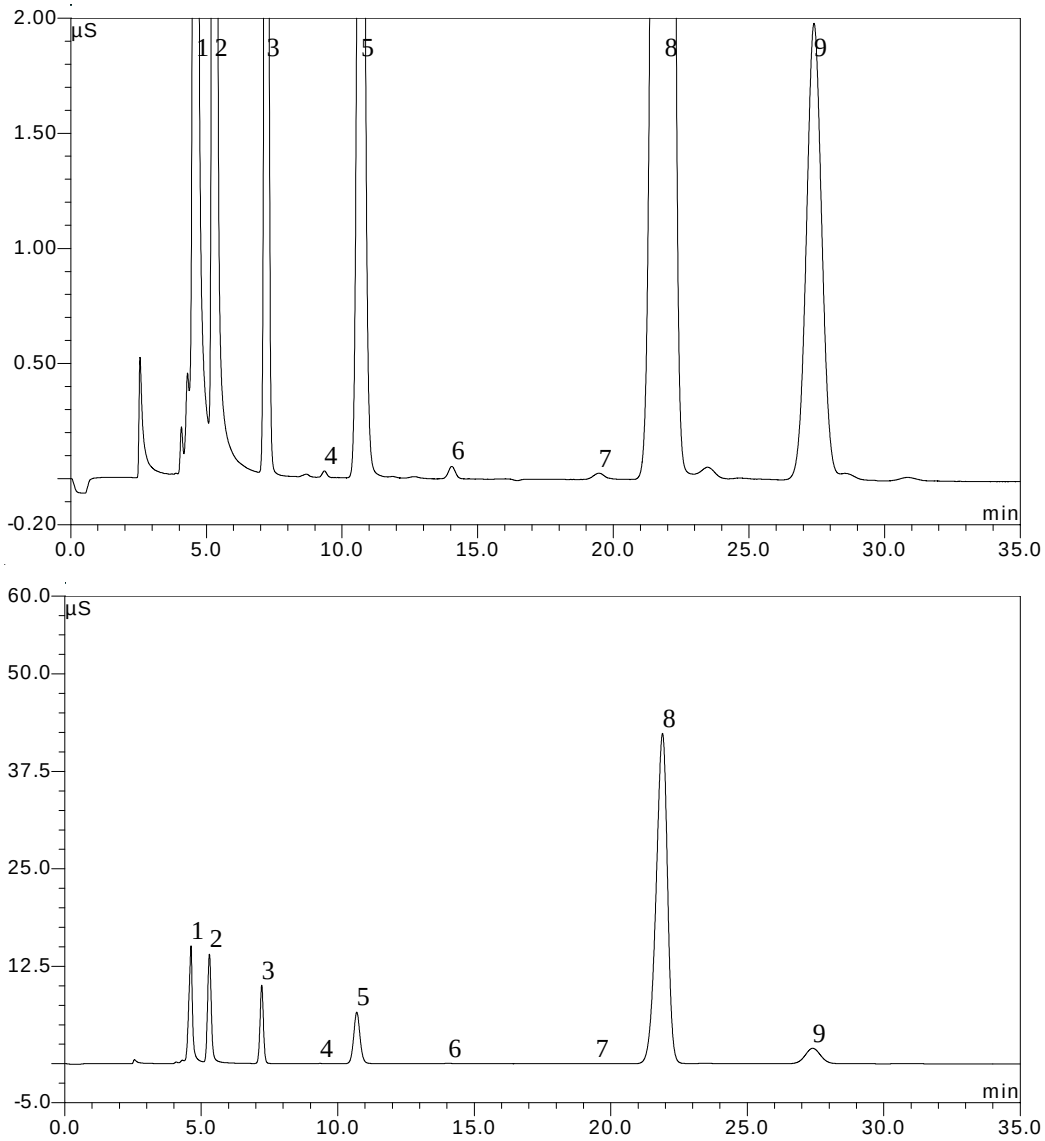

Figure 5
Analysis of Waste Water with AS23

5.8 Gradient Separation of Environmental Anions with the IonPac AS23 Column

The following chromatogram demonstrates that the IonPac AS23 is a highly hydroxide-selective column. Notice the separation of a variety of environmental anions using the IonPac AS23 column and a potassium hydroxide gradient.

Sample Loop Volume:	10 µL
Column:	IonPac AS23 (4-mm) Analytical Column + IonPac AG23 (4-mm) Guard Column
Eluent:	Potassium Hydroxide: 5 mM from 0 to 5 min, 5-30 nM from 5 to 15 min, 30-40 mM from 15 to 30 min.
Eluent Source:	EGC II KOH with CR-ATC
Temperature:	30 °C
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II AutoSuppression Recycle Mode

	Analyte	mg/L (ppm)
1.	Fluoride	2
2.	Acetate	10
3.	Butyrate	10
4.	Formate	10
5.	Chlorite	10
6.	Bromate	10
7.	Chloride	5
8.	Nitrite	10
9.	Chlorate	10
10.	Bromide	10
11.	Nitrate	10
12.	Carbonate	20
13.	Sulfate	10
14.	Selenate	10
15.	Oxalate	10
16.	Phthalate	20
17.	Phosphate	20
18.	Chromate	20
19.	Iodide	20
20.	Arsenate	20
21.	Citrate	20
22.	Thiocyanate	20
23.	Perchlorate	30

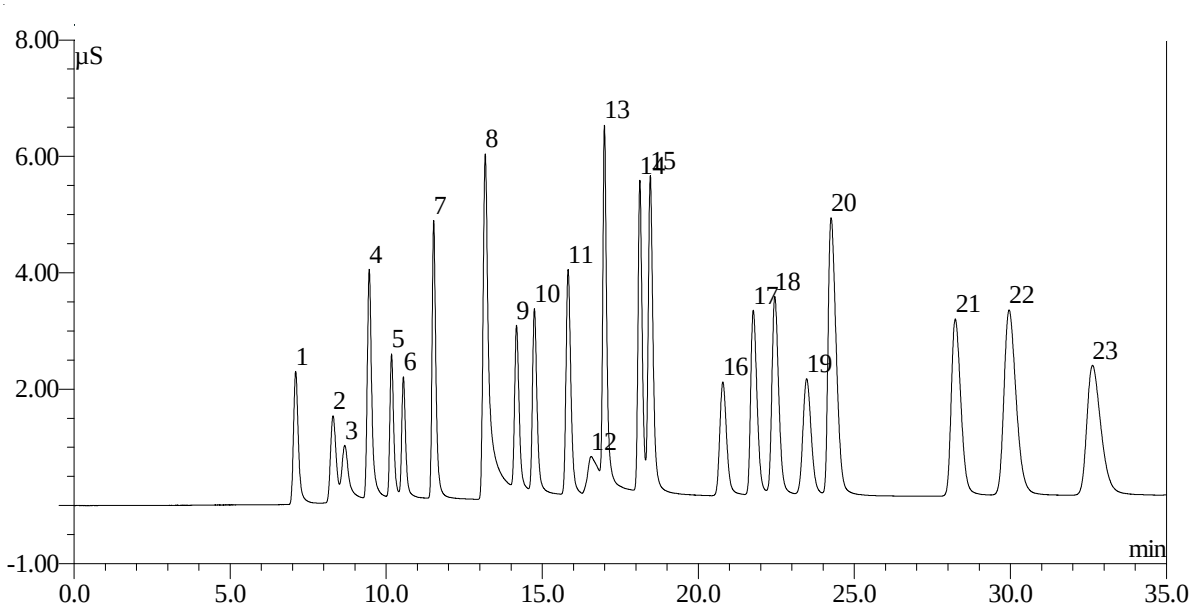

Figure 6
Separation of Environmental Anions with AS23

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS23 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 2
AS23/AG23 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 AG23 (4-mm) Guard Column plus the AS23 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,100 psi. If the system pressure is higher than 2,100 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 AS23/AG23 Operating Back Pressures").

The Anion Self-Regenerating Suppressor ULTRA II 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 3
Typical AS23/AG23 Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS23 4-mm Analytical	1800(12.41)	1.0
AG23 4-mm Guard	300 (2.07)	1.0
AS23 + AG23 4-mm columns	**2100 (14.47)**	**1.0**
AS23 2-mm Analytical	1800(12.41)	0.25
AG23 2-mm Guard	300 (2.07)	0.25
AS23 + AG23 2-mm columns	**2100 (14.47)**	**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 AS23 4-mm Columns (P/N)	IonPac AS23 2-mm Columns (P/N)
Analytical Column	064149	064145
Guard Column	064147	064143
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
4.5 mM Na_2CO_3 /0.8 mM $NaHCO_3$	20 - 22 µS

6.2.1 Preparation of Eluents

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

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

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

6.2.2 A Contaminated Guard or Analytical Column

Remove the IonPac AG23 Guard and AS23 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 AG23 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 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

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

This section describes routine cleanup procedures for the Anion Self-Regenerating Suppressors (ASRS ULTRA II) 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 II 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 II. 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 II **REGEN IN** port.

D. Disconnect the liquid line from the ASRS ULTRA II **ELUENT OUT** port to the cell at the cell fitting and reconnect it to the **REGEN IN** port.

E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.2 M oxalic acid. Pump this solution through the ASRS-ULTRA (4-mm) at 1-2 mL/min for 30 minutes. For 2-mm systems pump this solution through the ASRS-ULTRA (2-mm) at 0.25-0.50 mL/min for 30 minutes.

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 II 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 II 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 1.2 mL/min. Refer to the Anion MicroMembrane Suppressor Product Manual (Document No. 034449-02) for assistance in determining that the eluent is within suppressible limits.

C. If you are using an AutoRegen Accessory with the SRS (in the Chemical Suppression Mode) or the MMS, prepare fresh regenerant solution. Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.

1. **If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge, you probably need to clean or replace your SRS or MMS.**

2. If the background conductivity is low when freshly prepared regenerant is run through the SRS or MMS without an AutoRegen Accessory in-line, test the AutoRegen Regenerant Cartridge to see if it is **expended.** Connect the freshly prepared regenerant to the AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is high after placing the AutoRegen Accessory in-line, you probably need to replace the AutoRegen Regenerant Cartridge. Refer to the "AutoRegen Regenerant Cartridge Refill Product Manual" (Document No. 032852) for assistance.

6.2.7 A Contaminated Anion Atlas Electrolytic Suppressor, AAES

Metal Contaminants or Precipitates

A. Turn off the power to the AAES.

B. Disconnect the analytical (and guard) column(s) from the injection valve and the AAES. Refer to the specific analytical column Product Manual for column cleanup procedures.

C. If you are running in the **AutoSuppression External Water Mode**, turn off the external water and disconnect the external water line from the AAES **REGEN IN** port.

D. Disconnect the liquid line from the AAES **ELUENT OUT** port to the cell at the cell fitting and reconnect it to the **REGEN IN** port.

E. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.5 M oxalic acid. Pass 60 mL of this solution through the AAES using the Trap Column / Suppressor Clean-up Kit (P/N 059649) or pump this solution through the AAES at 2.0 mL/min for 30 minutes.

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 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 AS23 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 AS23 Eluent Concentrate (P/N 064161).
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 Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS23 columns is 3,000 psi (20.68 MPa).

A.2 Column Start-Up

The column is shipped using the column test eluent as the storage solution.

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

A.3 Column Storage

For both short-term and long-term storage, use column test eluent for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution (eluent). Cap both ends securely, using the plugs supplied with the column.

A.4 Column Cleanup

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

WARNING

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

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

A.4.2 Column Cleanup Procedure

Use the following cleanup procedures to clean the AG23 and AS23.

a) Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in, "Choosing the Appropriate Cleanup Solution".
b) Disconnect the ASRS ULTRA II or AMMS III from the IonPac AS23 Analytical Column.
c) 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.
d) Double check that the eluent flows in the direction designated on each of the column labels.

CAUTION

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

e) Set the pump flow rate to 1.0 mL/min for an AS23 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS23 2-mm Analytical or Guard Column.
f) Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.
g) Pump the cleanup solution through the column for at least 60 minutes.
h) Rinse the column for 10 minutes with deionized water before pumping eluent over the column.
i) Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.
j) Reconnect the ASRS ULTRA II or AMMS III to the AS23 Analytical Column
k) Place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

APPENDIX B - CONFIGURATION

Table 1
Configuration

CONFIGURATION	2-mm	4-mm
Eluent Flow Rate	0.30 mL/min	1.20 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
	Rheodyne Microinjection Valve (P/N 044697) for full loop injections <15 µL.	
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the 2-mm GM-4 Mixer (P/N 049135).	Minimize dead volume. Switching valves, couplers can be used. Use the GM-2 , GM-3 or recommended gradient mixers.
Pumps	Use the DP/SP/GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography.	Use the DP/SP/GP40/GP50/IP20/IP25 in Standard-Bore Configuration. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP50. Note: The GP40 has an active mixer.
Detectors	AD20 Cell (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL) (P/N 043120) DC/CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 (P/N 044130) or Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-2 (P/N 043117) is optimized for 2-mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi	AD25 Cell (10-mm, 9 µL, P/N 049393) VDM-2 Cell (6-mm, 10 µL) (P/N 043113) DC/CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 (P/N 044130) or Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-1 or TS-2 (P/N 043117) is optimized for 4-mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi

Table 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

