

# Dionex IonPac AS23-4µm

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# **Product Manual**

# for

# Dionex IonPac AG23-4µm Guard Column

(4 × 50 mm, P/N 302556) (2 × 50 mm, P/N 302558)

# Dionex IonPac AS23-4µm Analytical Column

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Safety and special notices include the following:



Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.



Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.



Indicates information of general interest.

### **IMPORTANT**

Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

Tip

Highlights helpful information that can make a task easier.

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# 1. Introduction

The Thermo Scientific<sup>TM</sup> Dionex<sup>TM</sup> IonPac<sup>TM</sup> AS23-4µm Analytical Column in combination with the Dionex IonPac AG23-4µm Guard Column is designed for the analysis of inorganic anions and oxyhalides including bromate, chlorite, and chlorate in environmental samples. The selectivity of the Dionex IonPac AS23-4µm Column has been designed to retain fluoride well out of the water dip (system dip) and to isocratically separate common anions and oxyhalides. In comparison to the Dionex IonPac AS23 column, the Dionex IonPac AS23-4µm column exhibits higher efficiency while maintaining the same selectivity. This is due to the reduced particle size of the 4µm column. The peak efficiencies produced by the Dionex IonPac AS23-4µm are much higher (50%) than larger particle columns, producing better resolution and easier peak integration. The high capacity and high efficiency of the Dionex IonPac AS23-4µm column allows improved resolution of these analytes in complex matrices. The smaller particle size resin produces operating pressures over 3000 psi, and therefore a high pressure IC system capable of operating up to 5000 psi is required to take advantage of the high resolution and high peak efficiency separations.

The Dionex IonPac AS23-4 $\mu$ m column is available in standard bore (4 mm i.d.) and microbore (2 mm i.d.) formats, both in 250 mm lengths for samples with higher ionic strength matrices. The Dionex IonPac AS23-4 $\mu$ m is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 – 100% in concentration. The Dionex IonPac AS23-4 $\mu$ m column has nominal efficiency of at least 15000 plates/column for sulfate using standard operating conditions.

Table 1 Packing Specifications

Column	Nominal Particle Diameter, µm	Substrate X-linking, %	Column Capacity, µeq/column	Functional Group	Hydrophobicity
Dionex IonPac AS23-4µm 4 × 250 mm	4	55	320	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG23-4µm 4 × 50 mm	11	55	6	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS23-4µm 2 × 250 mm	4	55	80	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG23-4μm 2 × 50 mm	11	55	1.5	Alkanol quaternary ammonium	Ultralow

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

**Table 2** Operating Parameters

Column	Typical Back Pressure psi (MPa <sup>a</sup> ), 30°C <sup>b</sup>	Standard Flow Rate, mL/min	Maximum Flow Rate, mL/min <sup>c</sup>
Dionex IonPac AS23-4µm 4 mm Analytical	≤ 3000 (20.68)	1.0	1.5
Dionex IonPac AG23-4µm 4 mm Guard	≤ 300 (2.07)	1.0	1.5
Dionex IonPac AS23-4µm + Dionex IonPac AG23-4µm 4 mm columns	<b>≤ 3300 (22.75)</b>	1.0	1.5
Dionex IonPac AS23-4µm 2 mm Analytical	≤ 3000 (20.68)	0.25	0.4
Dionex IonPac AG23-4µm 2 mm Guard	≤ 300 (2.07)	0.25	0.4
Dionex IonPac AS23-4µm + Dionex IonPac AG23-4µm 2 mm columns	<b>≤ 3300 (22.75)</b>	0.25	0.4

<sup>&</sup>lt;sup>a</sup> Note: 1 MPa = 145.04psi

<sup>&</sup>lt;sup>c</sup> In all cases, flow rate should not result in pressure over 5000 psi.



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



For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

<sup>&</sup>lt;sup>b</sup> Total backpressure at standard flow rates.

# 2. Installation



Read the instrument manuals. This manual assumes that you are using Thermo Scientific Dionex instrumentation and are familiar with the installation and operation of the Thermo Scientific Dionex Ion Chromatograph (IC). If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis.

The proper configuration of an Ion Chromatography System (ICS) is dependent on column format. Although they can typically use the same system, the use of a 2 mm or 4 mm column requires different set up to ensure maximum performance. The selected format and analysis type 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 or multi-eluent proportioning capabilities. For high pressure applications, the use of high pressure fittings is recommended.

# 2.1 The Thermo Scientific Dionex High Pressure Ion Chromatography Systems

A minimum of a Dionex High Pressure Ion Chromatography System (HPIC) is recommended when running Dionex IonPac  $4\mu m$  columns due to the higher backpressures generated at typical operational flow rates. Systems should have the capability to operate up to at least 5000 psi. Standard IC systems, with an upper limit of 3000 psi, are insufficient for proper column operation. All systems should allow 100% metal-free operation to prevent column damage. This includes pump heads and all flow paths.



Care should always be taken not to exceed the maximum operating pressure of the system component. ICS systems with lower backpressure capabilities are not recommended as reduced flow rates may result in loss of performance.



Contact your local representative for information on how to customize your system to your application needs.

# 2.2 System Requirements

The Dionex IonPac Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. We recommend the use of ferrules and fittings rated with a pressure of >5000 psi. The use of precut tubing, complete with high pressure fitting and ferrules, is recommended for easier installation.

### 2.2.1 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" i.d. PEEK tubing. In order to reduce system backpressure at higher flow rates, 0.010" i.d. PEEK tubing may be used for connections *before* the injection valve but peak efficiency will be compromised if used for post injector connections, which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

# 2.3 Column Start-Up

The column is shipped using 100 mM sodium bicarbonate as the storage solution.

Prepare the eluent shown on the Quality Assurance Report. To remove the storage solution, flush the column to waste with the QAR eluent for at least 30 minutes before attaching the column outlet to the suppressor. Install the column in the column module and test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

**IMPORTANT** 

When making any tubing connections, first turn off the pump. This will avoid any slippage of the ferrule under high pressure conditions.

### 2.4 Eluents

### 2.4.1 Preparation of Eluent Stock Solution Concentrates

A. Sodium Carbonate/Bicarbonate Eluent Concentrate: The Dionex AS23 Sodium Carbonate/Bicarbonate Eluent Concentrate (0.45 M Na<sub>2</sub>CO<sub>3</sub>/0.08 M NaHCO<sub>3</sub>), P/N 064161, can be used to prepare eluents for the Dionex IonPac AS23-4μm column.

To make the eluent concentrate from reagents, 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 mL of deionized water with a specific resistance of 18.2 megohm-cm in a 1 L volumetric flask. Dilute to a final volume of 1,000 mL.

B. 0.5 M Sodium Carbonate (Na<sub>2</sub>CO<sub>3</sub>) Concentrate: The Dionex Sodium Carbonate Anion Eluent Concentrate, 0.5 M, 500 mL is available by ordering P/N 037162.

To make this eluent concentrate from reagents, thoroughly dissolve 26.49 g of Na<sub>2</sub>CO<sub>3</sub> in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex 0.5 M 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. Do not dry sodium carbonate at excessive temperatures (> 110°C) as this will increase the pH of the salt.

C. 0.5 M Sodium Bicarbonate (NaHCO<sub>3</sub>) Concentrate: The Dionex Sodium Bicarbonate Anion Eluent Concentrate, 0.5 M, 500 mL, is available by ordering P/N 037163.

To make this concentrate from reagents, thoroughly dissolve 21.00 g of NaHCO<sub>3</sub> in 400 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to a final volume of 500 mL.

### 2.4.2 Eluent Preparation

### 2.4.2.1 Eluent: 4.5 mM Sodium Carbonate/0.8 mM Sodium Bicarbonate

A. Using Dionex AS23 Eluent Concentrate:

By Weight: Weigh 988.0 g of deionized water and add 10.5 g of the Dionex AS23 Eluent Concentrate.

<u>By Volume</u>: To make 1 liter of eluent, pipet 10 mL of the Dionex AS23 Eluent Concentrate into a 1 L volumetric flask and dilute to a final volume of 1 L using deionized water.

B. Using 0.5 M Na<sub>2</sub>CO<sub>3</sub> and 0.5 M NaHCO<sub>3</sub> Concentrates
By Weight: Weigh 987.42 g of deionized water and add 9.45 g of 0.5 M Na<sub>2</sub>CO<sub>3</sub> plus 1.68 g of 0.5 M NaHCO<sub>3</sub>.

By Volume: Prepare the eluent by pipetting 9.0 mL of 0.5 M Na<sub>2</sub>CO<sub>3</sub> plus 1.6 mL of 0.5 M NaHCO<sub>3</sub> into a 1 L volumetric flask. Use degassed, deionized water with a specific resistance of 18.2 megohm-cm to dilute the concentrate to a final volume of 1,000 mL.



It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of  $CO_2$  will affect retention times and selectivity.

### 2.4.3 Eluent Generation

### 2.4.3.1 Using the Dionex EGC-Carbonate Eluent Generator

The Dionex IonPac AS23-4µm column is recommended for use with Dionex systems equipped with a Dionex Eluent Generator. The Dionex Eluent Generator is used to automatically produce carbonate and bicarbonate eluents from deionized water. For more information, see the manual for the Dionex Eluent Generator Cartridges Product Manual, Document No. 065018.

It is recommended that Dionex IonPac columns are used with Thermo Scientific Dionex HPIC Systems equipped with a Dionex Eluent Generator (Dionex EG). The use of eluent generation provides a more stable baseline, improved performance and increased reproducibility over the use of manually prepared eluents.



Only Dionex EG Cartridges (Dionex EGCs) rated for 5,000 psi or higher should be used when running Dionex IonPac 4µm columns due to the higher backpressures generated at typical operational flow rates.

### 2.5 Dionex IonPac Guard Columns

A Dionex IonPac Guard Column is normally used with the Dionex IonPac Analytical Column. A guard is placed in front of the analytical column to prevent sample contaminants from damaging the analytical column. It is easier to clean or replace the guard column than it is the analytical column. Placing a guard column in front of the analytical column will cause retention times to increase by approximately 2-3% under isocratic test conditions. Replacing the guard column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the analytical column.

# 2.6 Sample Concentrators

The function of a concentrator column is to strip ions of interest from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the concentrator column, lowering detection limits by 2-5 orders of magnitude. The concentrator column is used in lieu of the sample loop at the start of the analysis.

Dionex Concentrator columns or the Dionex IonPac Guard Column can be used for trace anion concentration work with Dionex IonPac columns. A pump is used to load the sample onto the concentrator column in the OPPOSITE direction of the eluent flow. Once concentration is complete the eluent flow is then directed through the concentrator to the analytical column. When using concentration techniques, care should be taken not to overload the concentrator column by concentrating an excessive amount of sample. If an excessive amount of sample is used inaccurate results may be obtained. It is possible during the concentration step for the stronger binding polyvalent ions to elute the weakly retained ions from the concentrator column. For more detailed information on sample concentration techniques for high sensitivity work and a detailed discussion of concentration techniques refer to the appropriate concentrator manual for your application.



Dionex IonPac Concentrator Columns are designed for use with specific eluent systems. Use only concentrator columns designed for the eluent system you are using.

### 2.7 The Injection Loop

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

For most applications on a 2 mm analytical system, a  $2-15~\mu L$  injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. Install an injection loop one-fourth or less (<15  $\mu L$ ) of the loop volume used with a 4 mm analytical system (Appendix B, "System Configuration").

### 2.7.2 The 4 mm System Injection Loop, 10 - 50 µL

For most applications on a 4 mm analytical system, a  $10-50~\mu L$  injection loop is sufficient. Generally, you should not inject more than 50 nanomoles of any one analyte onto the 4 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. For typical drinking water samples, you can inject up to  $200~\mu L$ .

# 2.8 Eluent Storage

Dionex IonPac AS23-4µm 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).



It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of CO<sub>2</sub> will affect retention times and selectivity..

# 2.9 Dionex Anion Electrolytically Regenerated Suppressor (Dionex AERS 500 Carbonate) Requirements

A Dionex Anion Electrolytically Regenerated 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 Dionex AERS 500 Carbonate Anion Electrolytically Regenerated Suppressor modes of operation.



The Dionex AERS 500 Carbonate suppressor does not support the chemical regeneration mode. The Dionex Anion Chemically Regenerated Suppressor (ACRS 500) is recommended for chemical regeneration mode.



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

For Dionex IonPac AS23-4µm 4 mm Analytical Column, use a Dionex AERS 500 Carbonate (4 mm, P/N 085029).

For Dionex IonPac AS23-4 $\mu$ m 2 mm Analytical Column, use a Dionex AERS 500 Carbonate (2 mm, P/N 085028).

For detailed information on the operation of the Dionex Anion Electrolytically Regenerated Suppressor, see Document No. 031956, the "Thermo Scientific Dionex ERS 500 Suppressor Product Manual".

# 2.10 Dionex Chemically Regenerated Suppressor Requirements

A Thermo Scientific Dionex Anion Chemically Regenerated Suppressor (Dionex ACRS 500) may be used instead of a Dionex AERS 500 suppressor (4 mm) for applications that require suppressed conductivity detection. Use a Dionex ACRS 500 4 mm (P/N 085090) with the Dionex IonPac AS23-4 $\mu$ m 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 Dionex ACRS 500 2 mm (P/N 085091).

For detailed information on the operation of the Dionex Anion Chemically Regenerated Suppressor (ACRS 500), see Document No. 031727, the "Product Manual for the Dionex Chemically Regenerated Suppressor 500".

# 2.11 Using Dionex Displacement Chemical Regeneration (DCR) with the Chemical Suppression Mode

The Dionex Displacement Chemical Regeneration (Dionex DCR) Mode is recommended for chemical suppression using sulfuric acid and the Dionex Anion Chemically Regenerated Suppressor (Dionex ACRS 500). See the Dionex DCR kit manual, Document No. 031664, for details.



Use proper safety precautions in handling acids and bases.

# 3. Operation

## 3.1 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: Dionex IonPac AS23-4µm 2 mm Analytical Column + Dionex IonPac

AG23-4µm 2 mm Guard Column

4 mm: Dionex IonPac AS23-4µm 4 mm Analytical Column + Dionex IonPac

AG23-4µm 4 mm Guard Column

Eluent: 4.5 mM Na<sub>2</sub>CO<sub>3</sub>/0.8 mM NaHCO<sub>3</sub>

Temperature: 30 °C

Eluent Flow Rate: 2 mm: 0.25 mL/min 4 mm: 1.0 mL/min

ERS Suppressor: Dionex Anion Electrolytically Regenerated Suppressor, Dionex AERS 500 Carbonate

(2 or 4 mm)

AutoSuppression Recycle Mode

Expected Background

Conductivity: 18-22 µS

Long-term Storage Solution (> 1 week): 100 mM Sodium Bicarbonate

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

# 3.2 Dionex IonPac AS23-4µm Column Operation Precautions



Filter and Degas Eluents Filter Samples Eluent pH between 0 and 14 Sample pH between 0 and 14 0.4 mL/min Maximum Flow Rate for 2 mm Columns

0.4 mL/min Maximum Flow Rate for 2 mm Columns
1.5 mL/min Maximum Flow Rate for 4 mm Columns
Maximum Operating Pressure = 5,000 psi (34.47 MPa)

# 3.3 Chemical Purity Requirements

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

### 3.3.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label. Occasionally, batches of sodium carbonate are produced with low concentrations of residual hydroxide impurity. Use of such reagent can adversely affect the resolution of phosphate and sulfate. Use of Dionex 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.

### 3.3.2 Deionized Water

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

#### 3.3.3 Solvents

Solvents can be added to the ionic eluents used with Dionex IonPac AS23-4µm column 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 Thermo Fisher Scientific, we have obtained consistent results using Optima<sup>TM</sup> Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent, and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the Dionex IonPac AS23-4µm column is 5,000 psi (34.47 MPa).

The Dionex IonPac AS23-4 $\mu$ m column can withstand common HPLC solvents in a concentration range of 0 - 100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 3 HPLC Solvents for Use with Dionex IonPac AS23-4µm Column

Solvent	Maximum Operating Concentration
Acetonitrile	100%
Methanol	100%
2-Propanol	100%
Tetrahydrofuran	20% *
* Higher concentration ma	y only be used for limited
duration applications su	ch as column clean up
at pressures	< 4000 psi



The Dionex Anion Electrolytically Regenerated Suppressor (Dionex AERS 500 Carbonate) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

## 3.4 Making Eluents that Contain Solvents

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. 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 or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.



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



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



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



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

# 4. Example Applications

The chromatograms in this section were obtained using columns that reproduced the Quality Assurance Report on an optimized Ion Chromatograph. 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.

Ensure that your system is properly configured and 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 with a specific resistance of 18.2 megohm-cm. For chemical purity requirements, see Section 3.3, "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 analytical column has been fouled, refer to the column cleanup protocols in Appendix A, "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 2.6, "Sample Concentrators").

# 4.1 Isocratic Elution Using the Dionex IonPac AS23-4µm Column With and Without a Guard Column

Isocratic elution of inorganic anions and oxyhalides using the Dionex IonPac AS23-4 $\mu$ m Analytical Column has been optimized utilizing a carbonate/bicarbonate eluent as shown in figure 1 and figure 2. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. The Dionex IonPac AS23-4 $\mu$ m Analytical Column should always be used with the Dionex IonPac AG23-4 $\mu$ m Guard Column. Note that the Dionex IonPac AG23-4 $\mu$ m guard column is packed with a microporous resin of proportionally lower capacity and retention times will increase by approximately 2-3% when a guard column is placed in-line prior to the analytical column.

Figure 1 Isocratic Elution of Inorganic Anions and Oxyhalides Using the Dionex IonPac AS23-4µm 4mm Column: With and Without Guard Column

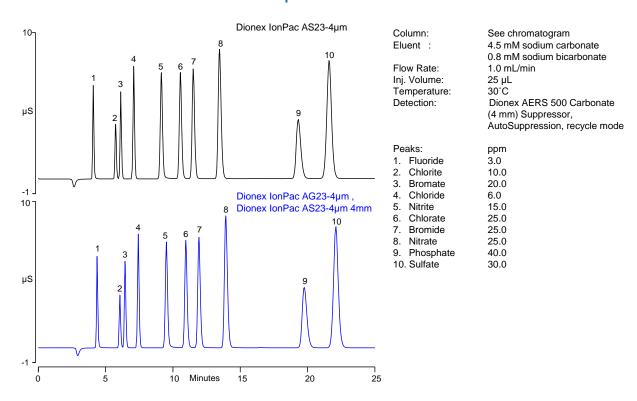
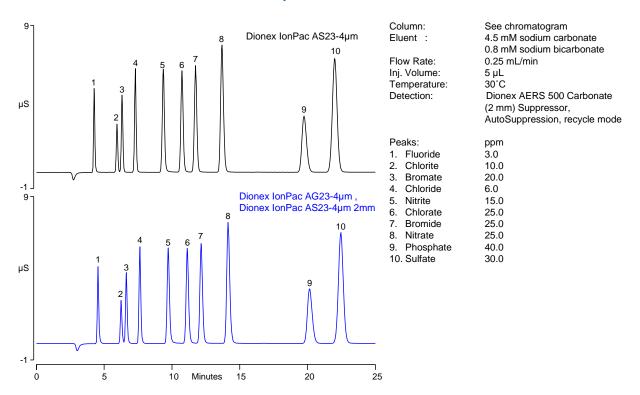
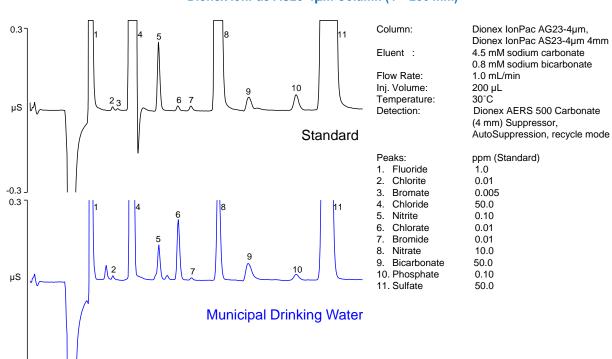


Figure 2 Isocratic Elution of Inorganic Anions and Oxyhalides Using the Dionex IonPac AS23-4µm 2mm Column: With and Without Guard Column



# 4.2 Analysis of Simulated Drinking Water and Municipal Drinking Water

The following examples demonstrate the separation of inorganic anions and oxyhalides in simulated drinking water and municipal drinking water using the Dionex IonPac AS23-4 $\mu$ m column. Figure 3 shows a 200  $\mu$ L injection on the Dionex IonPac AS23-4 $\mu$ m 4 mm column and figure 4 shows a 50  $\mu$ L injection on the Dionex IonPac AS23-4 $\mu$ m 2 mm column



20

25

Minutes

15

10

5

Figure 3 Separation of Inorganic Anions and Oxyhalides in Drinking Water using the Dionex IonPac AS23-4µm Column (4 × 250 mm)

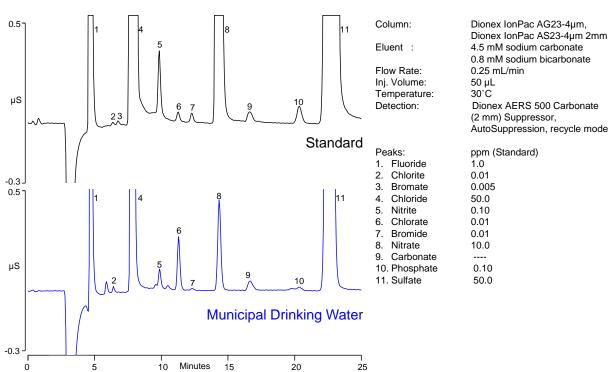
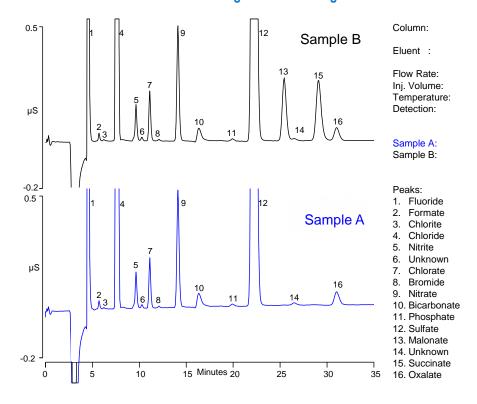


Figure 4 Separation of Inorganic Anions and Oxyhalides in Drinking Water using the Dionex IonPac AS23-4µm Column (2 × 250 mm)

# 4.3 Separation of Anions in Municipal Drinking Water Spiked with Surrogate Anions

The chromatograms in figures 5 show the analysis of a drinking water sample spiked with 1 ppm malonate and succinate using the Dionex IonPac AS23- $4\mu$ m 4 mm column. Notice the excellent separation of surrogate anions from sulfate.

Figure 5 Separation of Anions in Municipal Drinking Water With and Without Surrogate Anions using the Dionex IonPac AS23-4µm Column (4 × 250 mm)



Dionex IonPac AG23-4µm,
Dionex IonPac AS23-4µm 4mm
4.5 mM sodium carbonate
0.8 mM sodium bicarbonate
1.0 mL/min
200 µL
30°C
Dionex AERS 500 Carbonate
(4 mm) Suppressor,
AutoSuppression, recycle mode
Municipal Drinking Water
Municipal Drinking Water

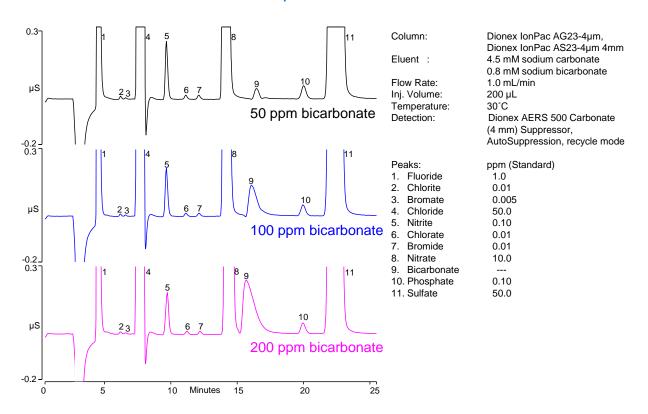
Spiked with 1ppm Malonate

and Succinate

# 4.4 Analysis of Simulated Drinking Water with Low to High Levels of Bicarbonate

The following examples demonstrate the separation of inorganic anions and oxyhalides in a simulated drinking water sample spiked with different levels of bicarbonate using the Dionex IonPac AS23-4 $\mu$ m 4mm column. Figure 6 shows a 200  $\mu$ L injection on the Dionex IonPac AS23-4 $\mu$ m 4 mm column. Notice the effect on resolution of nitrate and bicarbonate with 200 ppm of bicarbonate in the standard. When there are high levels of bicarbonate in the sample, be sure to either dilute the sample or reduce the sample loop size to avoid the bicarbonate peak merging under the nitrate peak.

Figure 6 Separation of Inorganic Anions and Oxyhalides in Simulated Drinking Water using the Dionex IonPac AS23-4µm Column: 50, 100 and 200ppm Bicarbonate Spike



# 4.5 Analysis of Waste Water Using the Dionex IonPac AS23-4µm Column

The following chromatograms show the analysis of a waste water sample using the Dionex IonPac AS23-4 $\mu$ m column. Note that smaller sample loops were used for these injections due to the high concentration of various anions in the sample.

Figure 7 Analysis of Waste Water using the Dionex IonPac AS23-4µm 4x250mm Column

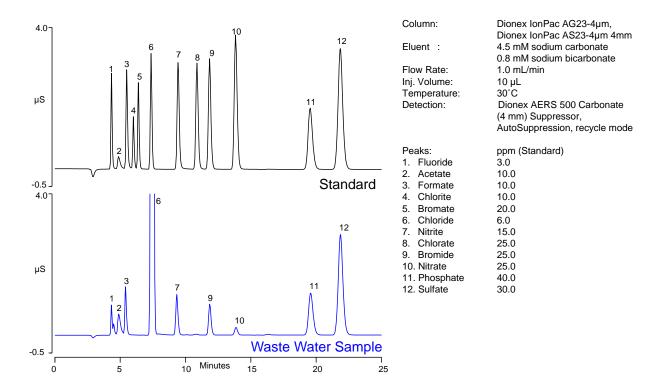
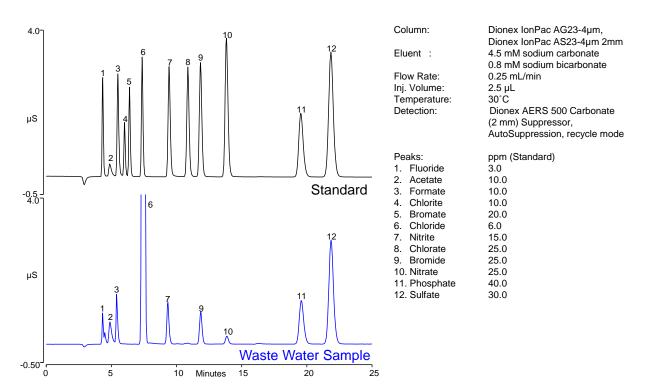


Figure 8 Analysis of Waste Water using the Dionex IonPac AS23-4µm 2x250mm Column



# 5. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac AS23-4µm column. For more information on problems that originate with the Ion Chromatograph (IC) or other consumables such as the suppressor, trap, or concentrator columns, refer to the Troubleshooting Guide in the appropriate product manual.



For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

Table 4 Dionex IonPac AS23-4µm/AG23-4µm Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate blocked component	5.1.1
	Plugged column bed support	Replace bed supports	5.1.2
	Other system components	Unplug, replace	Component Manual
High Background	Contaminated eluents	Remake eluents	5.2, 5.2.1
Conductivity	Contaminated columns	Clean column	5.2.2, A.4
	Contaminated suppressor	Clean suppressor	5.2.4, Component Manual
	Contaminated hardware	Clean component	5.2.3, Component Manual
Poor Resolution	Poor efficiency due to large system void volumes	Replumb system	5.3.1.B, Component Manual
	Column headspace	Replace column	5.3.1.A
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	5.3.5
Short Retention Times	Flow rate too fast	Recalibrate pump	5.3.2.A, Component Manual
	Incorrect eluent conc.	Remake eluents	5.3.2.B
	Column contamination	Clean column	5.3.2.C, 5.3.2.D, A.4
Poor Front End Resolution	Incorrect eluent conc.	Remake eluents	5.3.3.A
	Column overloading	Reduce sample size	5.3.3.B, 2.7
	Sluggish injection valve	Service valve	5.3.3.C, Component Manual
	Large system void volumes	Replumb system	5.3.3.D, Component Manual
Poor Resolution of Only Fluoride and Acetate	Poor plumbing, incorrect eluent	Replumb system, check eluent	5.3.6
Spurious Peaks	Column contaminated	Clean column	5.3.4.A, A.4
	Sluggish injection valve	Service valve	5.3.3.B, Component Manual

# 5 - Troubleshooting

Poor Efficiency	Poor connections	Remake connections	5.3.1.B	

# 5.1 High Back Pressure

### 5.1.1 Finding the Source of High System Pressure

Total system pressure for the Dionex IonPac AG23-4 $\mu$ m Guard Column plus the Dionex IonPac AS23-4 $\mu$ m Analytical Column when using the test chromatogram conditions should be less than 3300 psi at standard flow rate. If the system pressure is higher than 3300 psi, it is advisable to determine the cause of high pressure. The system should be operated with a High-Pressure Inline Filter (P/N 074505) which is positioned between the gradient pump outlet 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 Inline 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 2 Operating Parameters").

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

### 5.1.2 Replacing Column Bed Support Assemblies for 2 mm and 4 mm Columns

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.

Table 5 Ordering Information

Product	Dionex IonPac AS23-4µm 4 mm Columns (P/N)	Dionex IonPac AS23-4µm 2 mm Columns (P/N)
Analytical Column	302555	302557
Guard Column	302556	302558
Bed Support Assembly	042955	044689
End Fitting	052809	043278



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

- E. **Screw the end fitting back onto the column.** Tighten it finger tight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.
- F. Reconnect the column to the system and resume operation.



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

## 5.2 High Background or Noise

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

#### Eluent

#### **Expected Background Conductivity**

4.5 mM Na<sub>2</sub>CO<sub>3</sub> /0.8 mM NaHCO<sub>3</sub>

 $18-22 \mu S$ 

### 5.2.1 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.

### 5.2.2 A Contaminated Guard or Analytical Column

Remove the guard and 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 columns at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in Appendix A, "Column Care".

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

### 5.2.4 A Contaminated Suppressor

If the above items have been checked and the problem persists, the Dionex Anion Electrolytically Regenerated Suppressor or the Dionex Chemically Regenerated Suppressor is probably causing the problem. For details on Dionex Anion Electrolytically Regenerated Suppressor, refer to the Product Manual (Document No. 031956). For details on Dionex Chemically Regenerated Suppressor operation, refer to the Product Manual (Document No. 031727).

### 5.3 Poor Peak Resolution

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

### 5.3.1 Loss of Column Efficiency

- **A.** Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 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.
- B. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4 mm systems or no greater than 0.005" for 2 mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks.

### 5.3.2 Poor Resolution Due to Shortened Retention Times

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

- A. **Check the flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.
- B. Check to see if the eluent compositions and concentrations are correct. An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.
- C. Column contamination can lead to a loss of column capacity. This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to Appendix A "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 Appendix A, "Column Care").



For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

#### 5.3.3 Loss of Front End Resolution

If poor resolution or efficiency is observed for the peaks eluting near the system void volume compared to the later eluting peaks, check the following:

- A. **Improper eluent concentration may be the problem.** Remake the eluent as required for your application. Ensure that the water and chemicals used are of the recommended 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 actuation of the valve and make sure there are no partially plugged port faces. Refer to the valve manual for instructions.
- D. Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem. Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change.

### 5.3.4 Spurious Peaks

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



For assistance, contact Technical Support for Dionex Products. In the U.S. call, 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

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.

### 5.3.5 Poor Resolution of Only Phosphate and Sulfate

#### A. Causes

- 1. Sodium carbonate is contaminated with sodium hydroxide.
- 2. Inadequate equilibration after use of an alkaline buffer or hydroxide eluent.
- 3. Sodium carbonate was dried at temperatures > 110°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.



It is highly recommended to pressurize the eluent with nitrogen or helium to maintain the pH, as any change in pH due to absorption of  $CO_2$  will affect retention times and selectivity.

### 5.3.6 Poor Resolution of Only Fluoride and Acetate

### A. Causes

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

#### B. Action

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

# **Appendix A – Column Care**

### A.1 Recommended Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for Dionex IonPac AS23-4µm column is 5,000 psi (34.47 MPa).

## A.2 Column Start-Up

The column is shipped using 100 mM sodium bicarbonate as the storage solution.

Prepare the eluent shown on the Quality Assurance Report (QAR), install the column in the chromatography module and direct the column effluent to waste for 30 minutes, and then connect to the suppressor. Test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

## A.3 Column Storage

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

## A.4 Column Cleanup

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

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

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 the chromatography still suggests metal contamination after acid treatment, additional 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 non-ionic 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 contentities will decompose in the
	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

- A. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in Section A.4.1, "Choosing the Appropriate Cleanup Solution".
- B. Disconnect the suppressor from the columns and direct the effluent to waste.
- 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.



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 a 4 mm analytical and/or guard column and 0.25 mL/min for a 2 mm analytical and/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. If the column is heavily contaminated, then clean the column for at least four hours or overnight.
- 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. Reinstall the guard column in line between the injection valve and the analytical column and reconnect the analytical column to the suppressor.

# **Appendix B – Additional Information**



For assistance, visit Unity Lab Services online at www.unitylabservices.com From the U.S. call the Customer Care Center for Dionex Products at 1-800-346-6390 Outside the U.S., call the nearest Thermo Fisher Scientific office.

# B.1 Useful Information for Operation of Dionex High Pressure Ion Chromatography Systems

### B.1.1 General Information

Dionex brand PEEK™ tubing is available. Note that tubing from different suppliers varies with respect to color and i.d. Care should be taken to ensure the correct i.d. is being used to avoid problems caused by sample dispersion.

Table 6 Tubing Back Pressures.

Color	Part Number	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure, Psi/ft. at 1mL/min	Back Pressure, Psi/ft. at 0.25mL/min	Back Pressure, Psi/cm. at 1mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642
Light Blue	071870	0.0025	0.006	0.0009	1766.0	441.0	58.0

Note: Blue and Light blue are very similar in color. Additionally, tubing from different suppliers may differ to that stated above.

# **Appendix C - Quality Assurance Reports** (QAR)

01-Jun-16 15:33 Dionex IonPacTM AS23-4µm Date: 160616001 Serial No.: Analytical (2 x 250 mm) Device Monitoring Enabled and Viper Fitting Ready Lot No.: 2014-26-159A3 **Product No. 302557** 

Eluent: 4.5 mM Na2CO3/ 0.8 mM NaHCO3

Flow Rate: 0.25 mL/min

30 °C Temperature:

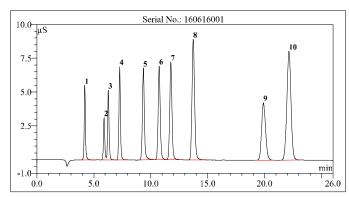
**Detection:** Suppressed Conductivity

Dionex Anion Electrolytically Regenerated Suppressor<sup>TM</sup> Suppressor:

(Dionex AERS 500 Carbonate 2mm) AutoSuppression<sup>TM</sup> Recycle Mode

Applied Current: 5 μL Injection Volume:

100 mM Sodium bicarbonate **Storage Solution:** 



No.	Peak Name	Ret.Time	Asymmetry	Resolution	Efficiency	Concentration
		(min)	(AIA)	(EP)	(EP)	(mg/L)
1	Fluoride	4.21	1.2	8.89	9407	3.0
2	Chlorite	5.91	1.1	1.79	12776	10.0
3	Bromate	6.28	1.1	4.49	13887	20.0
4	Chloride	7.28	1.1	7.81	15977	6.0
5	Nitrite	9.35	1.1	4.39	15240	15.0
6	Chlorate	10.74	1.1	3.02	16816	25.0
7	Bromide	11.77	1.1	5.21	18163	25.0
8	Nitrate	13.73	1.3	11.90	18326	25.0
9	Phosphate	19.90	1.2	3.47	15713	40.0
10	Sulfate	22.14	1.0	n.a.	18142	30.0

### OA Results:

<b>Analyte</b>	<b>Parameter</b>	Specification	Results
Sulfate	Efficiency	>=13500	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	19.85-23.15	Passed
	Pressure	<=3850	3094

Production Reference: Datasource: QAR

Directory: RFID\Anion\AS23-4µm AS23-4µm\_2X250MM

Sequence:

Sample No.: 2 Chromeleon TM Thermo Fisher Scientific 6.80 SR15 Build 4656 (243203)

Device Monitoring Enabled<br/>and Viper Fitting ReadyDionex IonPacTM AS23-4μmDate:<br/>Analytical (4 x 250 mm)26-May-16 07:50Device Monitoring Enabled<br/>and Viper Fitting ReadyAnalytical (4 x 250 mm)Serial No. :160616000Lot No. :01226146

Eluent: 4.5 mM Na2CO3/ 0.8 mM NaHCO3

Flow Rate: 1.0 mL/min Temperature: 30 °C

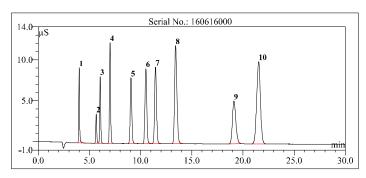
**Detection:** Suppressed Conductivity

Suppressor: Dionex Anion Electrolytically Regenerated Suppressor™

(Dionex AERS 500 Carbonate 4mm) AutoSuppression™ Recycle Mode

Applied Current:32 mAInjection Volume:25 μL

Storage Solution: 100 mM Sodium bicarbonate



No.	Peak Name	Ret.Time	Asymmetry	Resolution	Efficiency	Concentration
		(min)	(AIA)	(EP)	(EP)	(mg/L)
1	Fluoride	4.00	1.3	10.92	14287	3.0
2	Chlorite	5.67	1.2	2.25	17497	10.0
3	Bromate	6.06	1.1	5.15	18910	20.0
4	Chloride	7.02	1.0	8.63	20212	6.0
5	Nitrite	9.06	1.1	4.89	17081	15.0
6	Chlorate	10.51	1.1	2.88	17531	25.0
7	Bromide	11.46	1.1	5.24	18028	25.0
8	Nitrate	13.42	1.3	11.09	17155	25.0
9	Phosphate	19.11	1.4	3.76	15219	40.0
10	Sulfate	21.52	1.0	n.a.	16673	30.0

#### QA Results:

<u>Analyte</u>	<u>Parameter</u>	<b>Specification</b>	Results
Sulfate	Efficiency	>=13500	Passed
Sulfate	Asymmetry	0.9-1.7	Passed
Sulfate	Retention Time	19.85-23.15	Passed
	Pressure	<=3850	2775

Production Reference:

Datasource: QAR

 $\begin{array}{ll} \mbox{Directory:} & \mbox{\_RFID\Anion\AS23-4}\mu\mbox{m} \\ \mbox{Sequence:} & \mbox{AS23-4}\mu\mbox{M\_4X250MM} \end{array}$ 

Sample No.: 2 6.80 SR15 Build 4656 (243203)

Chromeleon Thermo Fisher Scientific