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# **PRODUCT MANUAL**

IONPAC® AG2 GUARD COLUMN (4 x 50 mm, P/N SP4834) IONPAC® AG3 GUARD COLUMN (4 x 50 mm, P/N 043182) IONPAC® AG4 GUARD COLUMN (4 x 50 mm, P/N 035310) IONPAC® AG5 GUARD COLUMN (4 x 50 mm, P/N 035396) IONPAC® CG2 GUARD COLUMN (4 x 50 mm, P/N 035370)

(4 x 50 mm, P/N 035370) IONPAC® AS2 ANALYTICAL COLUMN (4 x 250 mm, P/N SP4831) IONPAC® AS3 ANALYTICAL COLUMN (4 x 250 mm, P/N 043181) IONPAC® AS4 ANALYTICAL COLUMN (4 x 250 mm, P/N 035311) IONPAC® AS5 ANALYTICAL COLUMN (4 x 250 mm, P/N 035395) IONPAC® CS2 ANALYTICAL COLUMN (4 x 250 mm, P/N 035371)

#### QUICKSTART STEPS AND LINKS Click blue text below to get started.

See Section 2, "Operation". Note operation precautions and chemical purity requirements.
See "Quality Assurance Reports". Run the Production Test Chromatogram as a system check.
See "Column Care" for column cleanup and long-term storage recommendations.

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# **SECTION 1 - INTRODUCTION**

# 1.1 Overview

This manual describes how to prepare this column for operation. The initial start-up procedure usually consists of installing a column in the Chromatography Module, flushing it with deionized water to remove the storage solution, and equilibrating it with eluent.

# 1.2 Chemicals

The chemicals used to make eluents, standards, regenerants and post-column reagents must be of very high purity in order to obtain reliable, consistent and accurate analytical results. Before starting an analysis, consult Section 2.2, "Chemicals Required," for chemical purity requirements.

# 1.3 Column Storage

If a column will not be used within one week after testing, prepare it for long-term storage. Flush the column first with deionized water and then with the storage solution specified in Section 3, "Standard Conditions," of this manual. Cap both ends securely, using the plugs supplied with the column.

# 1.4 Recommended Operating Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. See Table 2, "Standard Flow Rates and Back Pressures," for standard and maximum back pressures.

# 1.5 Applications

For help in selecting the correct column for your application, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

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 - OPERATION**

# 2.1 General Operation Precautions

CAUTION <5% Organic Solvent in the Eluent Filter and Degas Eluents Filter Samples

## 2.2 Chemicals Required

Obtaining reliable, consistent and accurate results requires eluents that are free of ionic and spectrophotometric 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.

#### 2.2.1 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.

#### 2.2.2 Solvents

## CAUTION

#### Eluents used on columns containing low cross-linked resins must contain less than 5% organic solvents.

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

#### 2.2.3 Deionized Water

The deionized water used to prepare eluents should be **Degassed 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. Finally, thoroughly degas all deionized water prior to preparing any eluents.

# **SECTION 3 - STANDARD CONDITIONS**

# 3.1 Standard Reagents

# Table 1

# Storage Solutions, Eluents and Regenerants

Column	Storage	Standard	Standard
	Solution	Eluent	Regenerant

# ANION ANALYTICAL AND GUARD COLUMNS

IonPac <sup>®</sup> AS2/AG2	100 mM NaOH	3 mM Na <sub>2</sub> CO <sub>3</sub> /2 mM NaOH	10X Eluent Concentration
IonPac AS3/AG3	100 mM NaOH	2.2 mM Na <sub>2</sub> CO <sub>3</sub> /2.8 mM NaHCO <sub>3</sub>	10X Eluent Concentration
IonPac AS4/AG4	100 mM NaOH	2.25 mM Na <sub>2</sub> CO <sub>3</sub> /2.8 mM NaHCO <sub>3</sub>	10X Eluent Concentration
IonPac AS5/AG5	100 mM NaOH	2.8 mM $Na_2CO_3/2.2$ mM $NaHCO_3$	10X Eluent Concentration

## CATIONANALYTICALANDGUARDCOLUMNS

IonPac CS2/CG2	100 mM NaOH	10 mM Oxalic/7.5 mM Citric Acids	10X Eluent Concentration
		(pH 4.35)	

# 3.2 Standard Flow Rates and Back Pressures

Table 2
<b>Standard Flow Rates and Back Pressures</b>

Column	Standard Flow Rate	Expected Back Pressure	Maximum Back Pressure
Anion Analytical and Guard Columns			
IonPac AS2/AG2 IonPac AS3/AG3 IonPac AS4/AG4 IonPac AS5/AG5	3.0 mL/min 3.0 mL/min 2.0 mL/min 2.0 mL/min	600 psi/200 psi 600 psi/200 psi 1,000 psi/300 psi 1,000 psi/300 psi	1,500 psi 1,500 psi 2,000 psi 2,000 psi
Cation Analytical and Guard Columns			
IonPac CS2/CG2	2.3 mL/min	650 psi/150 psi	1,500 psi

# SECTION 4-INITIAL START-UP PROCEDURES

For the initial start-up of both anion and cation analytical columns, prepare the eluent listed on the test chromatogram, set the pump to the proper flow rate and perform the following initial start-up procedures. In the case of a guard or suppressor column, use the test conditions on the production test chromatogram for the analytical column required for the application.

- A. Connect the column directly to the pump with a short piece of tubing.
- B. Direct the effluent of the column to a separate waste container.
- C. Pump 10 mL (30 mL for packed-bed suppressors) of deionized water through the column at the standard flow rate listed in Table 2, "Standard Flow Rates and Back Pressures."
- D. Assemble the system (trap, concentrator, guard, analytical and suppressor columns depending on the application).
- E. Pump 10 mL of the standard production test eluent that is listed on the Analytical Column Test Chromatogram, through the System to equilibrate the columns.
- F. Perform the Production Test Chromatogram to test the column and system performance. Note that guard and analytical columns are fully equilibrated when successive runs have the same solute retention times.

# NOTE IonPac Fast Anion Analytical Column

# You must use a 20 µL injection loop with the IonPac Fast Anion to prevent excessive system band broadening and column overloading.

Maintain the eluent at between pH 2 and 11. Do not use hydroxide eluents.

# SECTION 5-TROUBLESHOOTING GUIDE

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

# 5.1 High Back Pressure

# 5.1.1 Finding the Source of High System Pressure

Total system pressure when using specific DIONEX columns is flow rate dependent. Refer to Table 2, "Standard Flow Rates and Back Pressures," for specific flow rates and back pressures for the column system that your are operating. If the system pressure is more than 20% higher than the total back pressure estimated from the back pressures listed in Table 2, "Standard Flow Rates and Back Pressures," it is advisable to find out what is causing the high system pressure. The system should be used with a High-Pressure In-Line Filter (P/N 035331) for the eluents which is positioned between the 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.** Find out what part of the system is causing the high pressure. It could be a piece of tubing that has plugged or whose walls are collapsed, an injection valve with a plugged port, a column with particulates plugging the bed support, a plugged High-Pressure In-Line Filter, the suppressor or the detector cell.

To find out 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 the system's components (injection valve, column(s), suppressor and detector) one by one, while watching the system pressure. The pressure should increase up to a maximum when the column(s) are connected. This maximum system pressure can be estimated from the data in Table 2, "Standard Flow Rates and Back Pressures." No other components should add more than 100 psi of pressure. If high system back pressure is found to be due to a specific system component, refer to the appropriate manual for cleanup or replacement of problem component.

# 5.1.2 Replacing Column Bed Support Assemblies

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

- A. Disconnect the column from the system.
- B. Carefully unscrew the inlet (top) column fitting, using 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. Part numbers for bed supports and other DIONEX components can be found in, "DIONEX Product Selection Guide."

## 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.

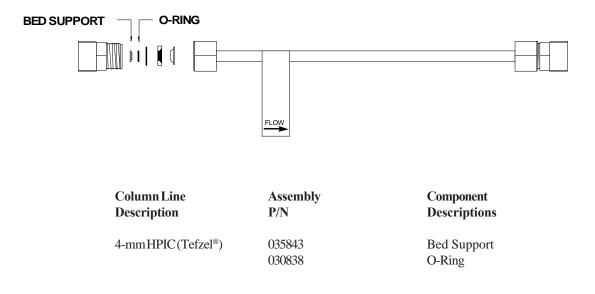
- **E** Screw the end fitting back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in x lb). If the column continues to leak, contact your nearest DIONEX Office (see, "DIONEX Worldwide Offices").
- F. Reconnect the column to the system and resume operation.

NOTE

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

# 5.1.3 Bed Support Descriptions

The following bed support assemblies are supplied with the different DIONEX Column Lines:





Column Line Description	Assembly P/N	Component Descriptions
4-mm IonPac		
(1/4-28 Liquid Line Fitting)	042310	Bed Support Assembly
	041375	Bed Support
	039835	Seal Washer
4-mm IonPac		
(10-32 Liquid Line Fitting)	042955	Bed Support Assembly
	041375	Bed Support
	042956	Seal Washer

# 5.2 High Background or Noise

In a properly working system, the background conductivity level for the standard eluent system is shown in Table 1, "Storage Solutions, Eluents and Regenerants." If the background conductivity is higher than listed in Table 1, "Storage Solutions, Eluents and Regenerants," check for eluent and system contamination as follows.

# **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 Anion Trap Column

If you are doing gradient analysis, an Anion Trap Column (ATC) should be installed between the pressure transducer on the gradient pump the injection valve. If it has not been installed, install one and watch the background conductivity. If the background conductivity decreases, the ATC is trapping contaminants from the eluent. The eluents probably have too many impurities (see Section 5.2.1, "Preparation of Eluents").

If the ATC is already installed, remove it. If the background conductivity decreases, the ATC is the source of the high background conductivity.

- A. Disconnect the ATC from the injection valve and direct the outlet to waste.
- **B.** Flush the ATC with 200 mL of 70 mM Na<sub>2</sub> $B_4O_7$  to remove any trapped impurities.
- C. Equilibrate the ATC with the strongest eluent used during the gradient run at the flow rate specified in Table 2, "Standard Flow Rates and Back Pressures."
- D. If the problem persists, replace the ATC.

## 5.2.3 A Contaminated Guard or Analytical Column

Remove the guard and analytical columns from the system. Is the background conductivity still high? If the column is the cause of the high background conductivity, clean the column as instructed in Section 6.3, "Column Cleanup."

# 5.2.4 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the suppressor and 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.

# 5.2.5 A Contaminated Self-Regenerating Suppressor (SRS<sup>™</sup>) or the MicroMembrane<sup>™</sup> Suppressor (MMS)

If the above items have been checked and the problem persists, the SRS or the MMS is probably causing the problem.

- A. Check the regenerant flow rate at the REGEN OUT port of the SRS or the MMS. For the example isocratic applications, this flow rate should be 3 5 mL/min in the Chemical Suppression Mode of operation.
- **B.** Check the eluent flow rate. The eluent flow rate for most 4-mm applications should be 2.0 mL/min. Refer to the Anion Self-Regenerating Suppressor Product Manual (Document No. 031367), the Cation Self-Regenerating Suppressor Product Manual (Document No. 031370), the Anion MicroMembrane Suppressor Product Manual (Document No. 031728) or the Cation MicroMembrane Suppressor Product Manual (Document No. 031728) to ensure that the eluent is within suppressible limits.
- C. If you are using an AutoRegen<sup>®</sup> 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 Regenerant Cartridge Regenerant Cartridge. Refer to the "AutoRegen Regenerant Cartridge Reference" in Pump approximately 200 mL of Regenerant reservoir. If the background conductivity is high after placing the AutoRegen Regenerant Cartridge Reference and to replace the AutoRegen Regenerant Cartridge. Refer to the "AutoRegen Regenerant Cartridge Refill Product Manual" (Document No. 032852) for assistance.

## 5.3 Poor Peak Resolution

Poor peak resolution can be due 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 may be due to the 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, 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 tubing with an ID of no greater than 0.012" to make all eluent liquid line connections between the injection valve and the detector cell inlet, and that the tubing lengths are as short as possible. Check for leaks.

## 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 faster than 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 strong 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 ion exchange sites will no longer be available for the sample ions. Polyvalent anions might be concentrating on the column. Refer to Section 6.3, "Column Cleanup," for recommended column cleanup procedures.

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

**D.** Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times. If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see Section 6.3, "Column Cleanup").

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

# 5.3.3 Loss of Front End Resolution

If poor resolutions and efficiencies are observed for the very early eluting peaks near the system void volume compared to the later eluting peaks, check the following:

- **A. Improper eluent concentration may be the problem.** Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity.
- **B.** Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.
- C. Sluggish operation of the injection valve may be the problem. Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.
- **D. Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem.** Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change.

## 5.4 Spurious Peaks

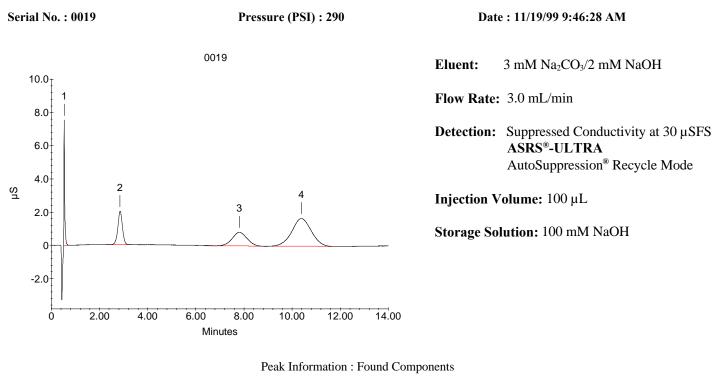
A. The columns may be contaminated. If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, polyvalent anions may contaminate the analytical column. The retention times for the analytes will then decrease and spurious, inefficient (broad) peaks can show up at unexpected times. Clean the column as indicated in Section 6.3, "Column Cleanup".

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix off of DIONEX columns, contact 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. It will happen 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.

If cleaning and retorquing the valve does not help, replace the valve. Consult the nearest DIONEX Office (see, "DIONEX Worldwide Offices") for help in services or replacing a suspect valve.

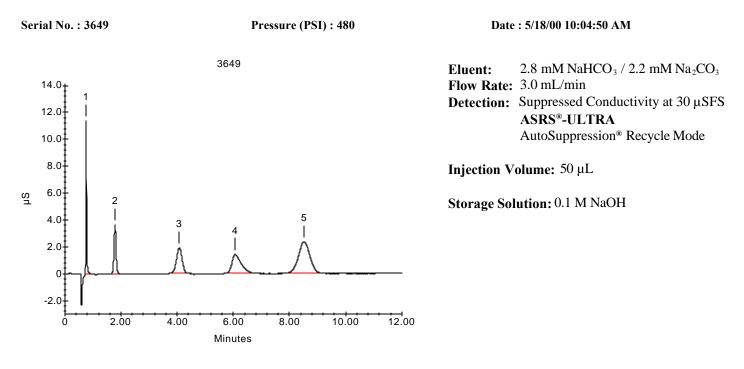
# IonPac® AS2 Analytical (4 x 150 mm) Product No. SP4831



Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	0.53	Fluoride	2.0	1729	3.0	11.73
2	2.85	Chloride	3.0	1088	1.1	6.73
3	7.81	Phosphate	15.0	760	1.0	1.97
4	10.38	Sulfate	15.0	789	1.0	n/a

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# IonPac® AS3 Analytical (4 x 250 mm) Product No. 43181

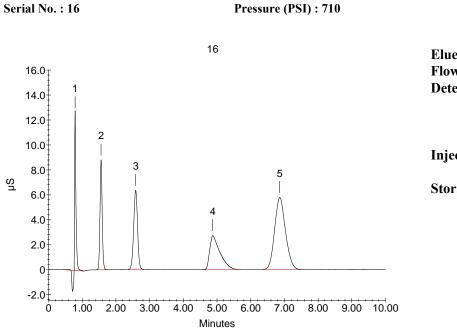


Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	0.76	Fluoride	2.0	4500	1.6	11.72
2	1.79	Chloride	3.0	3009	1.1	9.21
3	4.08	Phosphate	15.0	1961	1.0	4.13
4	6.07	Nitrate	10.0	1635	2.1	3.64
5	8.52	Sulfate	15.0	2076	1.0	n/a

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# IonPac® AS4 Analytical (4 x 250 mm) Product No. 35311



Date : 9/7/00 8:15:02 AM

Eluent:2.8 mM NaHCO3 / 2.2 mM Na2CO3Flow Rate:2.0 mL/minDetection:Suppressed Conductivity at 30 μSFS<br/>ASRS®-ULTRA<br/>AutoSuppression® Recycle Mode

**Injection Volume:** 50 µL

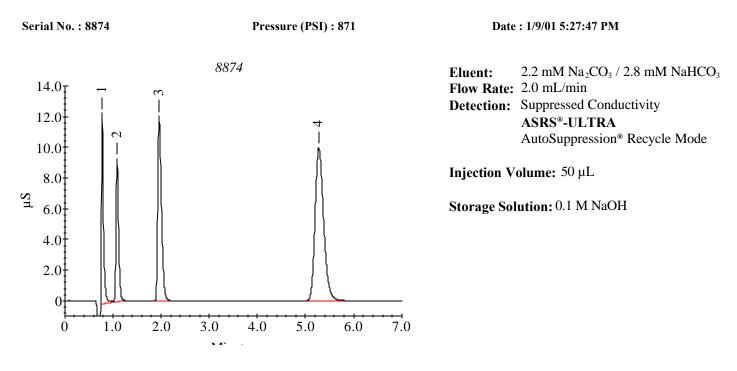
Storage Solution: 0.1 M NaOH

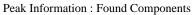
Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	0.79	Fluoride	5.0	2550	2.6	8.42
2	1.56	Chloride	20.0	2625	1.1	6.21
3	2.59	Phosphate	10.0	2437	0.9	6.09
4	4.87	Nitrate	15.0	1285	2.9	3.51
5	6.86	Sulfate	25.0	2149	1.2	n/a

File Name : C:\PEAKNET\DATA\EXAMPLES\35311 AS4\_A006.DXD

# IonPac® AS5 Analytical (4 x 250 mm) Product No. 35395

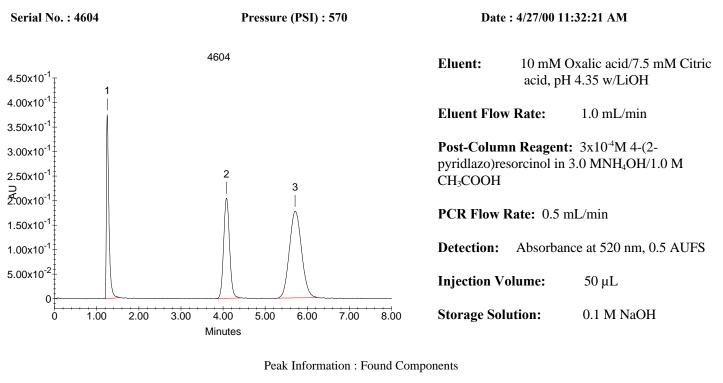




Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	0.77	Fluoride	2.0	2071	3.0	3.70
2	1.09	Chloride	3.0	1817	1.5	7.19
3	1.96	Nitrate	10.0	3065	1.8	14.13
4	5.27	Sulfate	15.0	4122	1.5	n/a

File Name : C:\PEAKNET\DATA\EXAMPLES\35395 AS5 4MM\_011.DXD

# IonPac® CS2 Analytical (4 x 250 mm) Product No. 35371



Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	1.25	Copper	1.0	2158	2.5	15.41
2	4.08	Zinc	2.0	3922	1.2	4.09
3	5.71	Cobalt	2.0	1801	1.2	n/a

#### File Name : C:\PEAKNET\DATA\EXAMPLES\35371 CS2 4MM\_B010.DXD

# COLUMN CARE

# **RECOMMENDED OPERATION PRESSURES**

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the columns covered by this manual are listed in Table 2, "Standard Flow Rates and Back Pressures."

# COLUMN STORAGE

For short-term storage, the strongest eluent in use can be used as the storage solution. For long-term storage, use the storage solution listed in Table 1, "Storage Solutions, Eluents and Regenerants." Flush the column for 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

# COLUMN CLEANUP

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

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

## CAUTION

Eluents used on columns containing low cross-linked resins must contain less than 5% organic solvents.

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# **BASE-SOLUBLE CONTAMINANTS**

This procedure is used to elute anionic contaminants from anion exchange columns. Typical eluents are formulated from carbonate/bicarbonate mixtures, borate and hydroxide.

- A. Prepare a 500 mL solution of 10X eluent concentrate. See Table 1, "Storage Solutions, Eluents and Regenerants."
- **B.** Disconnect the suppressor from the analytical column. If your system is configured with both a guard column and an analytical column, place the guard column after the analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels. Direct the effluent from the outlet line of the guard column to a separate waste container.

## CAUTION

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

## C. Set the pump flow rate to that listed in Table 2, "Standard Flow Rates and Back Pressures."

- D. If your eluent contains a solvent that is not compatible with the 10X eluent concentrate, rinse the column for 15 minutes with deionized water before pumping the 10X eluent concentrate over the column.
- E. Pump the 10X eluent concentrate solution through the column for 30-60 minutes.
- F. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.
- G. Equilibrate the column(s) with eluent before resuming normal operation.

# ACID-SOLUBLE CONTAMINANTS

This procedure is used to elute cationic contaminants from cation exchange columns. Typical eluents are formulated from hydrochloric acid.

- A. Prepare a 500 mL solution of 10X eluent concentrate. See Table 1, "Storage Solutions, Eluents and Regenerants."
- **B.** Disconnect the suppressor from the analytical column. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels. Direct the effluent from the outlet line of the guard column to a separate waste container.

## CAUTION

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

## C. Set the pump flow rate to that listed in Table 2, "Standard Flow Rates and Back Pressures."

- D. If your eluent contains a solvent that is not compatible with the 10X eluent concentrate, rinse the column for 15 minutes with deionized water before pumping the 10X eluent concentrate over the column.
- E. Pump 10X eluent concentrate solution through the column for 30-60 minutes.
- F. Reconnect the suppressor to the analytical column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.
- G. Equilibrate the column(s) with eluent before resuming normal operation.

# **ORGANIC CONTAMINANTS**

# CAUTION

Eluents used on columns containing low cross-linked resins must contain less than 5% organic solvents.

#### A. Prepare a 500 mL solution of 5% acetonitrile in deionized water having a specific resistance of 18.2 megohm-cm.

**B.** Disconnect the suppressor from the analytical column. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels. Direct the effluent from the outlet line of the guard column to a separate waste container.

## CAUTION

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

## C. Set the pump flow rate to that listed in Table 2, "Standard Flow Rates and Back Pressures."

D. If your eluent contains a solvent that is not compatible with 5% acetonitrile, rinse the column for 15 minutes with deionized water before pumping the 5% acetonitrile over the column.

#### E. Pump a 5% acetonitrile solution through the column for 30-60 minutes.

- F. If your eluent contains a solvent that is not compatible with 5% acetonitrile, rinse the column for 15 minutes with deionized water before pumping eluent over the column.
- G. Reconnect the suppressor column to the analytical column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.
- H. Equilibrate the column(s) with eluent before resuming normal operation.