PRODUCT MANUAL

for

IonSwift™ MONOLITH ANION CONCENTRATOR (MAC)

Now sold under the
Thermo Scientific brand

Thermo SCIENTIFIC

DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL
for

IonSwift™
MONOLITH ANION CONCENTRATOR (MAC)

MAC-100, 0.5 x 80mm (P/N 074702)
MAC-200, 0.75 x 80mm (P/N 075461)

Dionex® Corporation, 2010
Document No. 065387
Revision 01
October 2010

TABLE OF CONTENTS

IMPORTANT INFORMATION

Several icons are used throughout this document to emphasize important points. The symbols are shown below, along with the purpose of the information.

SAFETY ***Safety information can help prevent bodily harm.***

WARNING ***Warning information can help prevent equipment harm.***

CAUTION ***Caution information can help prevent problems.***

NOTE ***Note information can help with tips for improved use.***

SECTION 1 – INTRODUCTION

The IonSwift Monolith Anion Concentrator (MAC-100 and MAC-200) columns are designed to support ICS-5000 Capillary systems with very low dead volume. The function of the MAC column is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the MAC concentrator, leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the MAC column to the analytical chemist is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment. IonSwift MAC-100 and MAC-200 columns are designed to eliminate or minimize the sulfate blanks during trace level analysis of anions in matrices, such as high purity water. Another unique feature of the IonSwift MAC-100 and MAC-200 columns is that they offer direct connection, as a loop, to the injection valve.

IonSwift media is based on polymeric monoliths prepared by an in-situ polymerization process. The monolith is a single cylindrical polymer rod containing an uninterrupted, interconnected network of through pores, which are also called channels. The open spaces between the large aggregates form large flow-through channels allowing flow without high back pressure. The spaces among the smaller globules are the open or through-pores allowing fast access of the samples to the functionalized surface of the media. IonSwift monoliths have very high permeability and the pore volume is about 65% of the column volume. The monolith substrate is grafted with carboxylate functionality for further agglomeration with an anion exchange latex that has been completely aminated. The latex is a polyvinyl benzyl based polymer and carries the actual anion exchange sites. Due to the highly cross-linked structure, the monolith is solvent compatible.

The MAC-100 (0.5 x 80 mm) and MAC-200 (0.75 x 80 mm) can be used for trace anion analysis. The MAC-200 (0.75 x 80 mm) column is recommended for 2D applications for trace bromate or perchlorate. The capacity of the MAC-100 is approximately 0.17 µeq/column with a void volume of approximately 10 µL. The capacity of the MAC-200 is approximately 0.24 µeq/column with a void volume of approximately 23 µL. The physical rigidity of the IonSwift allows the MAC-100 and MAC-200 columns to be used at pressures up to 3,000 psi (20.7 MPa). The MAC-100 and MAC-200 can be readily converted between the base and the salt form without significant changes in the operating pressure.

The recommended maximum flow rate is 0.2 mL/min for MAC-100 and 1.5 mL/min for MAC-200. The backpressure generated by the MAC-100 is less than 60 psi at 0.12 mL/min. The MAC-200 is less than 60 psi at 1.0 mL/min. The MAC columns can be used with hydroxide, carbonate eluents, and borate eluents, with or without solvent, to concentrate samples on either 1 mm, 0.4mm or 0.25mm analytical systems.

WARNING

Always use the high pressure pulse damper (Dionex P/N 043945) after the AXP pump to ensure the concentrator column does not get exposed to pump pulsations. It is possible to damage the monolith when exposed to high pump pulsations.

Assistance is available for any problem during the shipment or operation of Dionex instrumentation, columns, and consumables 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" on the Dionex Reference Library CD-ROM.

TABLE 1
IonSwift MAC-100 and MAC-200
Concentrator Column Packing Specifications

Column	Substrate	Substrate X-Linking (%)	Latex Diameter (nm)	Latex X-Linking (%)	Column Capacity (µeq/column)	Functional Group	Hydro-phobicity
MAC-100 0.5 mm	Monolith	55	85	6	0.17	Alkanol quaternary ammonium	Very low
MAC-200 0.75 mm	Monolith	55	85	6	0.24	Alkanol quaternary ammonium	Very low

TABLE 2
IonSwift MAC-100 and MAC-200
Concentrator Column Operating Parameters

Column	Typical Backpressure psi (MPa) at 30 °C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
MAC-100 0.5 mm	≤ 60 (0.413)	0.12	0.2
MAC-200 0.75 mm	≤ 60 (0.413)	1.0	1.5

SECTION 2 – SETUP

2.1. IonSwift MAC-100 and MAC-200 as an Injection Loop

Figure 1 illustrates the recommended setup for the MAC-100 and MAC-200 Concentrators. Note that the concentrator column is connected at the injection valve position 1 and 4. IonSwift MAC-100 and MAC-200 columns offer direct connection as a loop, to the injection valve, without the need for end fittings or couplers; simply use the nuts and ferrules supplied with the concentrator.

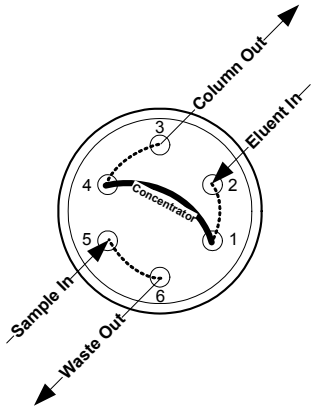


FIGURE 1
Recommended Setup for MAC as an Injection Loop

2.2. Setup for 2D Ion Chromatography System

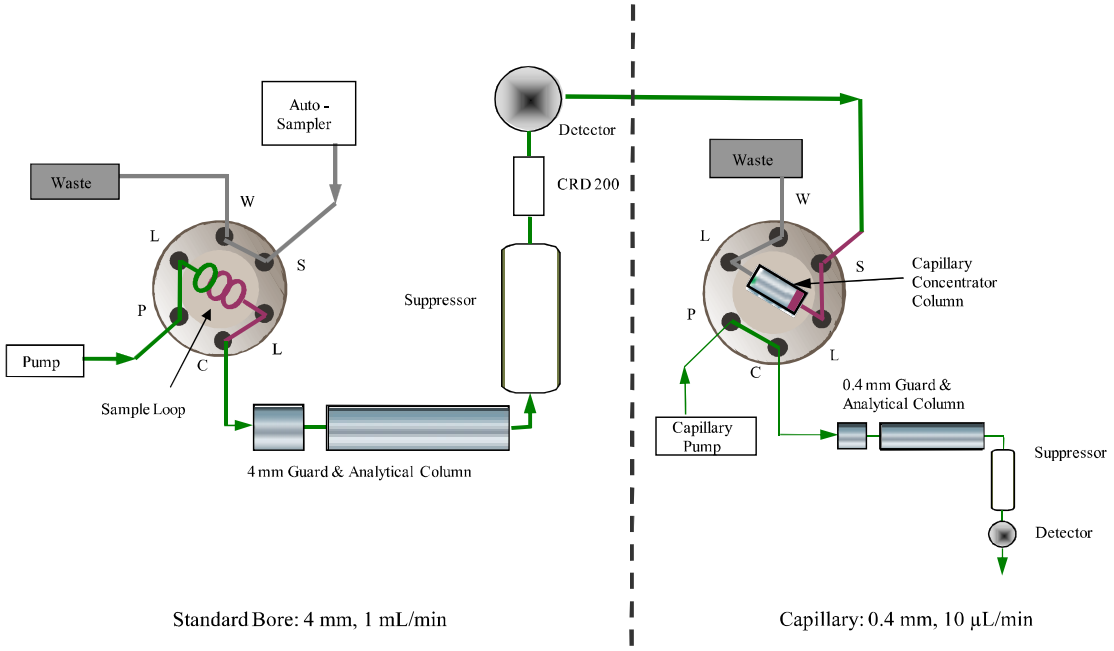


FIGURE 2A – 2D IC
Pre-concentration of Analytes from First Dimension on Concentrator Column (Loading Step)

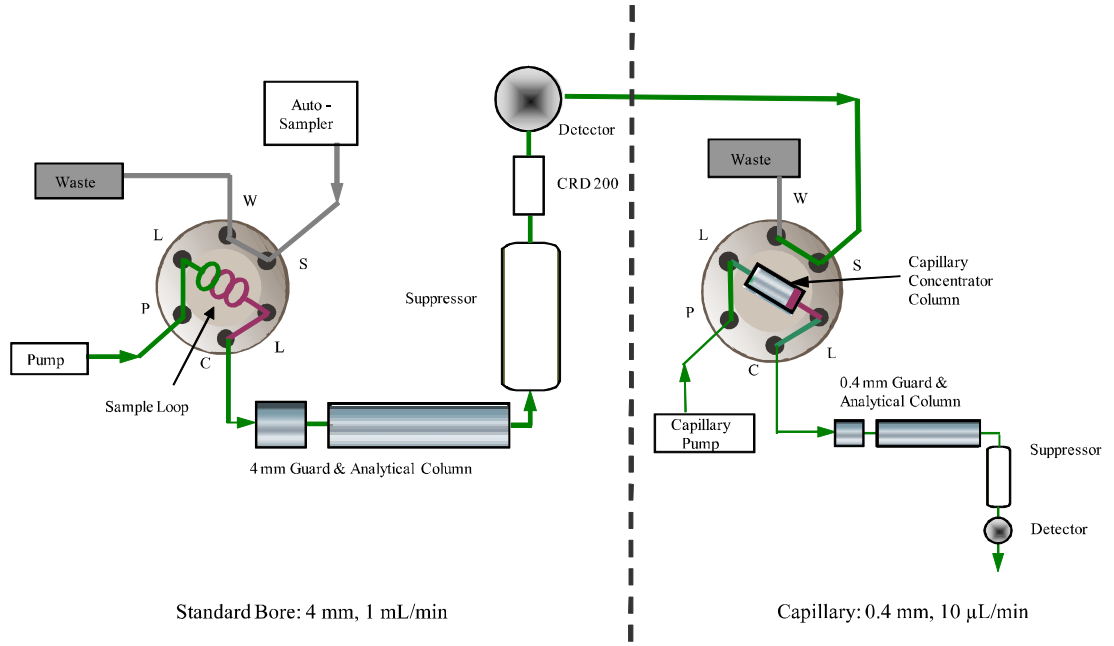


Figure 2B – 2D IC
Elution of Analytes Off of Concentrator Column and Analysis in Second Dimension

SECTION 3 – OPERATION

3.1. Sample Loading

WARNING ***Always use the high pressure pulse damper (Dionex P/N 043945) after the AXP pump to ensure the concentrator column does not get exposed to pump pulsations. It is possible to damage the resin when exposed to high pump pulsations.***

WARNING ***To prevent overloading the MAC-100 and MAC-200, and/or loss of sample analytes, determine the concentration linearity over the desired analytical concentration range. See Section 3.3, "Concentrators Capacity."***

WARNING ***The flow direction during the concentration step is critical. In order to ensure optimal system performance, it is recommended that concentration always be performed in a counter current direction to the eluent flow (See Figure 1 for example).***

After the sample has been loaded onto the MAC-100 and MAC-200, in the direction opposite to the eluent flow, it is then eluted with eluent onto the guard and analytical columns. Loading the sample in the opposite direction to the eluent flow ensures that the analyte ions are concentrated on the outlet of the concentrator column upon loading. Upon injection, the analyte ions are eluted off the concentrator column by the eluent in a tight plug and injected onto the guard and analytical column for further analysis. In this configuration the concentrated ions do not undergo any chromatographic separation in the concentrator column. When injected, all of the ions are rapidly eluted off of the MAC columns, and onto the guard and analytical columns. On the other hand, if the sample is loaded onto the MAC-100 and MAC-200 in the same flow direction as the eluent flow, the anions are concentrated at the inlet of the column rather than at the outlet. When injected, the anions begin chromatographic separation on the concentrator before reaching the guard and analytical columns. Therefore, the retention time of the analytes would be significantly longer than a standard loop injection. Normally the function of the concentrator is to strip the ions of interest from the sample matrix and not to act as an analytical column.
Figure 3 shows the configuration for sample loading using a Rheodyne valve.

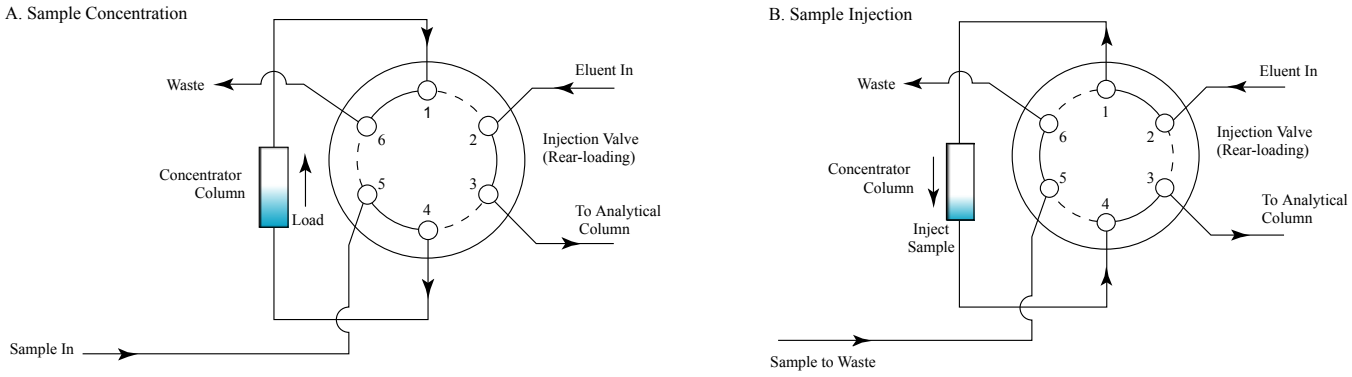


FIGURE 3
Process of Ion Chromatography Trace Enrichment

3.2. Reagent and Sample Handling

The following sections focus on critical points that must be followed when using the MAC-100 and MAC-200 concentrator columns. Proper consideration of these points will enable the analyst to obtain accurate and reproducible results at trace analyte levels.

3.2.1. Water Quality

All water used in the preparation of standards and eluents must be deionized water with a specific resistance of 18.2 megohm-cm. The quality of the dilution water must be determined by Ion Chromatography since even deionized water with a specific resistance of 18.2 megohm-cm may contain trace levels of the ions of interest. To do this, analyze the water in exactly the same manner as the sample.

3.2.2. Sample Collection and Storage

CAUTION

Never use plastic syringes with rubber pistons for any loading of trace ions. These materials cause non-reproducible results. It is recommended to wear gloves when performing trace analysis.

At trace analyte concentration levels (µg/L), chances of contamination during collection or storage are high. Every container and every procedural step constitutes a potential source of contamination. Polystyrene containers with leak-tight caps can be used to store 1 to 5 µg/L levels of inorganic and organic anions for up to 8 days. Recommended storage vessels are Corning tissue culture flasks. The following procedure should be used for storage of µg/L level samples.

A. Rinse the polystyrene container and cap twice with deionized water having a specific resistance of 18.2 megohm-cm. Fill the container until it overflows, cap it securely, and soak for 4 hours.

B. Empty the container and refill it with deionized water having a specific resistance of 18.2 megohm-cm. Cap the container securely. It should remain filled at least 24 hours before sample collection.

C. Empty the container and rinse it twice with the sample to be collected. Fill the container with the sample until it overflows and then cap the container securely. Be sure that the sample line does not touch the container.

3.2.3. Standards

It is good practice to run standards at the beginning, middle, and end of each day to ensure constant instrument response. Because external standard quantification is used, it is critical that standard solutions are correctly prepared.

A. 1,000 mg/L (1000 ppm) stock standard solutions should be prepared by accurately weighing amounts of salts as described in the instrument manual. These solutions are stable over a period of several months.

B. 1 mg/L stock standard solutions may be prepared by diluting 1 mL of 1,000 mg/L stock standard to 1,000 mL in a volumetric flask. These solutions should then be transferred to clean polystyrene containers. They may be stored for up to one month.

C. 1 µg/L working standard solutions may be prepared by diluting 1 mL of the 1 mg/L stock standard to 1,000 mL. These working standards are stored in polystyrene containers. They are stable up to 8 days, but Dionex recommends daily preparation since standard response is critical in the analysis.

3.3. Concentrator Capacity

As in all ion exchange systems, the column has a finite capacity. It can strip a given amount of ions from water. When the capacity of the concentrator is exceeded, the stripping will not be quantitative. This condition is referred to as column overload.

When estimating the capacity of a concentrator, one must remember that the column is used in a dynamic state where the liquid containing the analytes is flowing over the monolith at a finite rate. This reduces the capacity somewhat since the analyte ions have less time to interact with the monolith surface.

Low concentrator column capacity creates the following practical implications.

A. Trace analysis of an analyte is difficult in the presence of µg/L concentrations of species which exhibit higher or similar affinities for the monolith. If the dynamic column capacity is exceeded, high affinity ions will displace the analytes on the ion exchange sites and result in their elution to waste during the loading process.

B. Conversely, qualitative analysis of ions with higher affinities for the monolith in the presence of high concentrations of ions with low affinities is possible. Again, the key to successful analysis is that the ionic content of the high affinity ion to be quantitated may not exceed the effective column capacity.

C. Do not dilute samples to be concentrated in eluent because the eluent ions elute the ions of interest.

D. A plot of response versus volume should be generated using a simulated sample representative of the sample of interest for the determination of the maximum amount of sample that can be quantitatively loaded. The point in the graph where the plot deviates from linearity represents the maximum volume that can be concentrated. For practical purposes, the volume concentrated for a series of samples should be 75% of this value. This will ensure that there is a safety margin built into the concentration process in case a sample in a series of concentration experiments has a slightly higher ionic concentration.

3.3.1. Determination of the Concentrator Column Breakthrough Volume

The breakthrough volume of an analyte ion is that volume of sample which causes an ion of interest to be eluted from, rather than retained or concentrated on, the concentrator column.

The breakthrough volume for a concentrator column is usually defined as the volume of sample necessary to elute the most weakly retained ions of interest in the sample. The more strongly retained ions in the sample, such as sulfate, can elute the more weakly retained ions in the sample, such as fluoride.

It is also possible for a high concentration of a weakly retained ion such as chloride to elute a more strongly retained ion present at low concentration. This can occur if one is attempting to concentrate trace ions in a concentrated matrix.

The breakthrough is dependent upon several factors:

A. The volume of sample loaded.

B. The rate at which the sample is loaded.

C. The pH of the sample.

D. The ionic strength of the sample.

E. The amount and capacity of resin in the column.

The breakthrough volume is determined as follows:

A. Prepare 1 L of a solution that closely simulates the type of sample to be analyzed. For example, if the sample contains high levels of sulfate, the simulated sample should also contain sulfate. The sulfate ion will act as an eluent.

B. Prepare a 1 mg/L standard of the first eluting ion of interest (e.g., Fluoride).

C. Setup the Ion Chromatograph.

D. Equilibrate the MAC-100 and MAC-200 with the eluent to be used in the analysis. Set the flow rate necessary to achieve a stable baseline and wash the column in this manner for at least 10 minutes.

E. Switch to the simulated sample as an eluent. Without delay, manually inject 50 µL of the 1 mg/L standard.

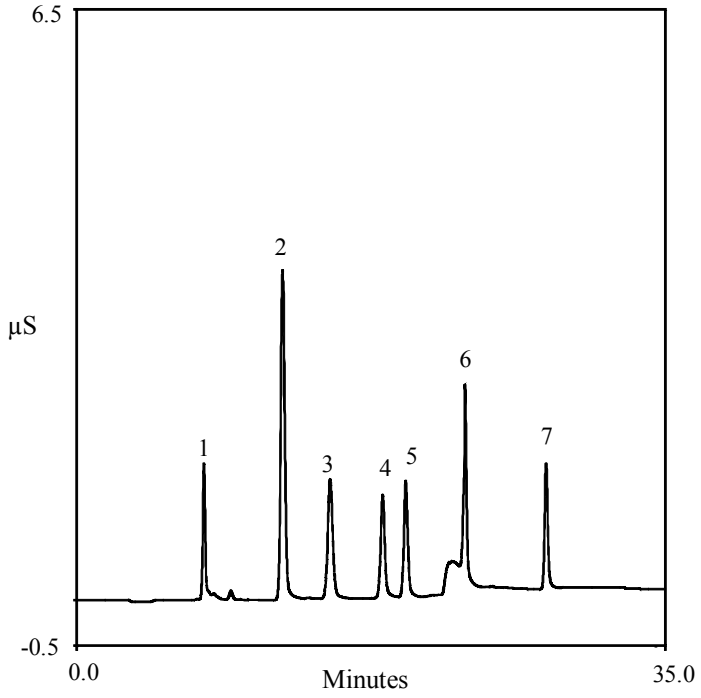
F. Record the resulting chromatogram and calculate the breakthrough volume.

G. For practical purposes, the volume concentrated should be below 75% of the breakthrough volume.

SECTION 4 – EXAMPLE APPLICATIONS

4.1. Separation of Inorganic Anions at Trace Concentrations on an IonPac AS19 Capillary Column with a 200 µL Injection

Column:	IonPac AS19 Capillary Column (0.4 × 250 mm)
Concentrator Column:	IonSwift MAC-100 Concentrator (0.5 x 80 mm)
Eluent Source:	Capillary EGC-KOH cartridge
Eluent:	10 mM KOH (0 to 10 minutes), 10 to 50 mM KOH (10 to 25 minutes), 50 mM KOH (25 to 30 minutes), 10 mM (30 to 35 minutes)
Flow Rate:	0.010 mL/min
Temperature:	30 °C
Suppressor:	Electrolytic capillary anion suppressor (ACES 300)
Detection:	Suppressed conductivity
Injection Volume:	200 µL



Peak	(µg/L)
1. Fluoride	1.0
2. Chloride	5.0
3. Nitrite	5.0
4. Bromide	5.0
5. Nitrate	5.0
6. Sulfate	10
7. Phosphate	5.0

FIGURE 4
Separation of Inorganic Anions at Trace Concentrations on an IonPac AS19 Capillary Column with 200 µL Injection

4.2. Determination of Trace Bromate in a Bottled Water Sample Using a 2-D Capillary RFIC System

A. First-Dimension Conditions

Column:	IonPac AG19, AS19 Analytical Column, 4 mm
Flow Rate:	1.0 mL/min
Eluent Source:	EGC-KOH Cartridge
Eluent:	10 mM KOH (0 to 12 minutes), 65 mM KOH (12 to 35 minutes), and 10 mM KOH (35 to 40 minutes)
Suppressor:	ASRS 300, 4-mm
Inj. Volume:	1000 µL
Temperature:	30 °C

B. Second-Dimension Conditions

Column:	IonPac AS20 Capillary Column (0.4 x 250 mm)
Concentrator Column:	IonSwift MAC-200 concentrator (0.75 x 80 mm), 2500 µL of 1st dimension suppressed effluent (7.5 to 10 minutes)
Flow Rate:	0.010 mL/min
Eluent Source:	Capillary EGC-KOH Cartridge
Eluent:	8 mM KOH (0 to 12 minutes), 8 to 65 mM KOH (12 to 35 minutes), and 8 mM KOH (35 to 40 minutes)
Suppressor:	Capillary Anion Electrolytic Suppressor (ACES 300)
Temperature:	30 °C

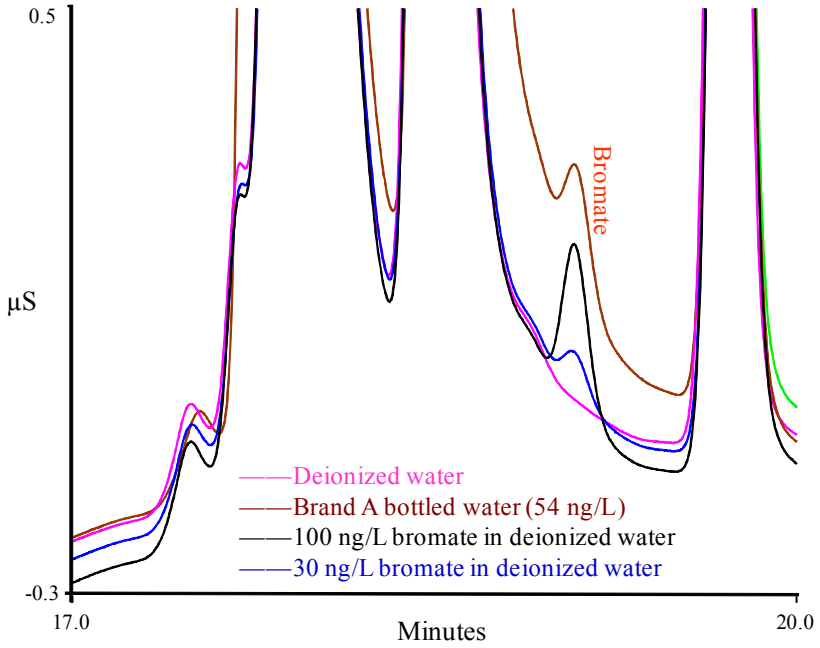


FIGURE 5
Determination of Trace Bromate in a Bottled Water Sample Using a 2-D Capillary RFIC System

SECTION 5 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the MAC-100 and MAC-200 columns. For more information on problems that originate with the Ion Chromatograph, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call your nearest Dionex Regional Office (see, "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM).

5.1. High Backpressure from a Contaminated Column Inlet

If the MAC-100 and MAC-200 column displays high backpressure, the in-let of the column may be contaminated.
A contaminated in-let may also lead to loss of peak asymmetry.

A. Disconnect the column from the system. Rinse the concentrator column for about one hour in reverse flow direction with 50-100mM KOH.

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

5.2. High Background, Noise, or Baseline Instability

Normally, problems such as high background, noise, or baseline instability will not be attributable to the MAC-100 and MAC-200 column. These problems usually originate in either the analytical column or the post-column detection chemistry. Before checking the MAC-100 and MAC-200 as the source of system background noise, consult the appropriate troubleshooting sections in the Column Product Manual, the Ion Chromatograph Operator's Manual, the ACES 300 Manual and the Detector Manual.

If the source of the high background noise is isolated to the MAC-100 or MAC-200 column, then proceed with the following steps.

A. Be sure that the MAC column is not leaking.

B. Be sure that the eluents are correctly formulated.

C. Be sure that the eluents are made from chemicals with the recommended purity (see Section 3, "Operation").

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

5.3. Poor Peak Shape

In some instances, poor peak shape in Ion Chromatography may be caused by a contaminated MAC column. To clean the MAC Column, see, "Column Cleanup of Polyvalent Anions and Base-Soluble Contaminants" in the Appendix (Column Care).

When pursuing pre-concentration with a pump, ensure that the pump has a pulse damper installed. Failure to dampen the pump pulsations may result in damage to the MAC columns.

APPENDIX A
COLUMN CARE

A.1 Recommended Operating Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the IonSwift MAC-100 and MAC-200 columns is 3,000 psi (20.7 MPa).

A.2 Column Start-up

1. Recommended Start-Up Flow Rates:
   MAC-100 (0.5 mm ID) concentrator use a flow rate of <0.05 mL/min (50 µL/min)
   MAC-200 (0.75 mm ID) concentrator use a flow rate of ≤0.5 mL/min
2. Concentrator Conditioning:
   Use the guidelines below to determine the proper start-up conditions. To properly condition the concentrator it is recommended to pump at least 10 column volumes (CV) through the concentrator.

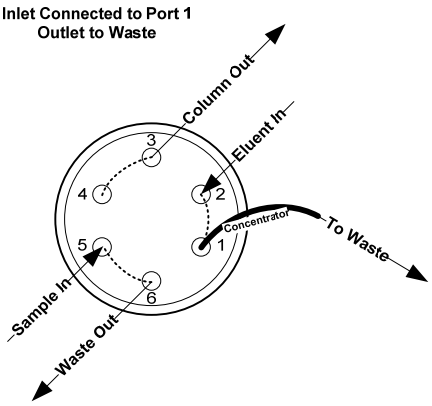


Figure A

Injection Valve MUST be in the INJECT position

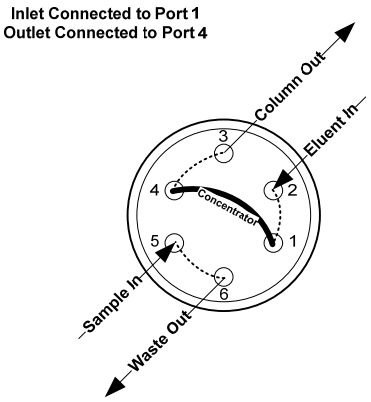


Figure B

MAC-100 (0.5 mm):

Step #1 – Removal of Storage Solution: Using the start-up flow rate of 0.05 mL/min pump about 0.75 mL for ~15minutes of 50 mM KOH or 50 mM NaOH through the concentrator to waste. (Figure A)

Step #2 – Concentrator Equilibration: Changing from 50 mM KOH or 50 mM NaOH to the starting eluent will require pumping 0.25 mL of the desired eluent composition through the concentrator for ~5 minutes.

Step #3 – Final Connection: After equilibration, connect the concentrator to Port #4. (Figure B)

Step #4 – Set the flow to the operational flow rate for your analysis, the concentrator is ready to use.

MAC-200 (0.75 mm):

Step #1 – Removal of Storage Solution: Using the start-up flow rate of 0.5 mL/min pump about 7.5 mL for ~15 minutes of 50 mM KOH or 50 mM NaOH through the concentrator to waste. (Figure A)

Step #2 – Concentrator Equilibration: Changing from 50 mM KOH or 50 mM NaOH to the starting eluent will require pumping 2.5 mL of the desired eluent composition through the concentrator for ~5 minutes.

Step #3 – Final Connection: After equilibration, connect the concentrator to Port #4. (Figure B)

Step #4 – Set the flow to the operational flow rate for your analysis, the concentrator is ready to use.

A.3 Column Storage

Using the start-up flow rate, flush the concentrator for a minimum of 10 minutes using the storage solution listed below. Then using the red caps, supplied with the concentrator, fill the caps with storage solution to displace any air and slip a cap on to each end of the column.

- *Short-term storage (<1 week) use the eluent used in your analysis.*
- *Long-term storage (>1 week) use 100 mM Sodium Borate (prepared from $Na_2B_4O_7$•10 H_2O)*

A.4 Column Cleanup of Polyvalent Anions and Base-soluble Contaminants

A. Prepare a 500 mL solution of 0.5 M NaOH.

B. Disconnect the guard, analytical or capillary columns and the suppressor from the injection valve and the Conductivity Module. Disconnect the Gradient Mixer or Anion Trap Column from the Pump. Connect the MAC-100 and MAC-200 column directly to the Pump. Direct the effluent from the MAC directly to a waste container.

C. Set the flow rate to 0.05 mL/min for MAC-100 and 0.5mL/min for MAC-200 column.

D. Pump the 0.5 M NaOH solution through the column for 30-60 minutes.

E. Equilibrate the MAC with eluent for 15 minutes at 0.05 mL/min for MAC-100 and 0.5 mL/min for MAC-200 column before resuming normal operation.

F. Reconnect the anion guard, analytical or capillary column and the suppressor between the injection valve and the Conductivity Module. Reconnect the Anion Trap Column between the Pump and the Injection Valve. Resume operation.

A.5 Column Cleanup of Organic/Anionic Contaminants

A. Prepare a 500 mL solution of 200 mM HCl/80% acetonitrile.

B. Disconnect the guard, analytical or capillary columns and the suppressor from the injection valve and the Conductivity Module. Disconnect the Anion Trap column from the Pump. Connect the MAC-100 or MAC-200 column directly to the Pump. Direct the effluent from the MAC directly to a waste container.

C. Set the flow rate to at 0.05 mL/min for MAC-100 and 0.5mL/min for MAC-200 column. Pump the 200 mM HCl 80% acetonitrile solution through the column for 30-60 minutes.

D. Equilibrate the MAC column with eluent for 30 minutes at 0.05 mL/min for MAC-100 and 0.5mL/min for MAC-200 column before resuming normal operation.

E. Reconnect the Anion Guard, analytical or capillary column and the suppressor between the injection valve and the Conductivity Module. Reconnect the Anion Trap Column between the Pump and the Injection Valve. Resume operation.

