



PRODUCT MANUAL

for the

IonSwift™ MAX-100 Column

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Product Manual

For

IonSwift™ MAX-100 Analytical Column

(1 x 250 mm, P/N 071279)

IonSwift™ MAX-100 Capillary Column

(0.25 x 250 mm, P/N 074246)

IonSwift™ MAX-100G Guard Column

(1 x 50 mm, P/N 071280)

IonSwift™ MAX-100G Capillary Guard Column

(0.25 x 50 mm, P/N 074247)

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SECTION 1 – INTRODUCTION

IonSwift MAX-100 monolithic columns are designed to provide high speed and high-resolution separations of inorganic and organic anions using hydroxide gradients. Hydroxide is normally used for gradient elution to minimize the background shift. By using a hydroxide gradient, strongly retained trivalent ions, such as phosphate and citrate, are efficiently eluted in the same run that also gives baseline resolution of the weakly retained monovalent anions such as fluoride, acetate, formate and butyrate.

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. Its unique morphology, pore structure and pore size distribution offers the optimum performance for fast separation of organic and inorganic anions. IonSwift is a new generation of separation media, which are uniquely designed and engineered for separation of small molecules.

IonSwift monoliths have high permeability. The pore volume is about 60% of the column volume, which is much higher than the porous beads. There are two types of pores: large pores (approximately a micron or larger) for eluent to flow through and small pores (ten to hundreds of nanometers) where most of the separation occurs. These large pores allow the eluent to flow through with moderate back pressure, and allow higher flow rates for faster separations.

The IonSwift MAX-100 1x250mm and 0.25x250mm column is compiled of anion exchange polymeric monolithic media which provide high speed and high resolution separation for a wide range of inorganic and organic acid anions. The IonSwift MAX-100 offers selectivity similar to IonPac AS11-HC. IonSwift MAX-100 columns are stable between pH 0 and 14 and are compatible with eluents containing 0-100% organic solvents.

Table 1
IonSwift MAX-100 Column Specifications

Column	Substrate X-Linking	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
MAX-100 (1 x 250 mm)	55%	60	7.5	12	Alkanol quaternary ammonium	Medium-Low
MAX-100G (1 x 50 mm)	55%	60	7.5	2.4	Alkanol quaternary ammonium	Medium-Low
MAX-100 (0.25 x 250 mm)	55%	60	7.5	0.80	Alkanol quaternary ammonium	Medium-Low
MAX-100G (0.25 x 50 mm)	55%	60	7.5	0.16	Alkanol quaternary ammonium	Medium-Low

Table 2
IonSwift MAX-100 Operating Parameters

Column	Maximum Back Pressure psi (Mpa)	Standard Flow Rate mL/min	Maximum Flow Rate
IonSwift MAX-100 Analytical Column (1 x 250 mm)	< 1800 (12.41)	0.20	0.30
IonSwift MAX-100G Guard Column (1 x 50 mm)	< 500 (3.45)	0.20	0.30
IonSwift MAX-100 Capillary Column (0.25 x 250 mm)	< 1800 (12.41)	0.012	0.020
IonSwift MAX-100G Capillary Guard Column (0.25 x 50 mm)	< 500 (3.45)	0.012	0.020

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 – COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

The proper configuration of 1 mm system injection volume, mass loading, system void volume and flow rate is based on the ratio of the 1 mm to 0.25 mm column cross-sectional area which is a factor of 1/16.



IonSwift MAX-100 1 mm column can be run on a system optimized for the 2 mm application.

NOTE

CONFIGURATION	1 mm	0.25 mm
Eluent Flow Rate	0.2 mL/min	0.012 µL/min
SRS	ASRS 300 (2 mm) (P/N 064555)	ACES 300 (P/N 072052)
NOTE <i>Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven.</i>		
Injection Loop	2 - 10 µL (typical) Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.	0.4 µL (typical; internal)
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the 2 mm GM-4 Mixer (P/N 049135).	Use only in an IC system equipped for capillary analysis.
Pumps	ICS Series (Analytical) Systems equipped with the appropriate mixer. Recommended Mixer GM-4	Use only a pump designed for capillary flow rates such as the ICS-5000 capillary pump.
NOTE: Use of an EGC-KOH cartridge <i>(P/N 074532 or 072076 in conjunction with a CR-ATC P/N 060477 or 072078) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.</i>		
Chromatographic Module	A thermally controlled column oven such as the ICS Series.	A thermally controlled column compartment such as the ICS-5000 DC or IC-Cube.
Detectors	ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 ICS CD Cell	Use only a conductivity detector designed for capillary flow rates such as the ICS-5000 Capillary CD.

Table 3
Tubing Back Pressure

Color	Dionex P/N	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure Psi/ft. at 1 mL/min	Back Pressure Psi/ft. at 0.25 mL/min	Back Pressure Psi/cm. at 1 mL/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	2.44	0.609	0.081
Black	042690	0.010	0.025	0.015	6.96	1.740	0.232
Red	044221	0.005	0.013	0.004	111.4	27.84	3.71
Yellow	049715	0.003	0.008	0.001	859.3	214.8	28.6
Light Blue	071870	0.0025	0.006	0.0009	1766.0	441.0	58.0

SECTION 3 – INSTALLATION

3.1. System Void Volume

When using 1 mm or 0.25 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" (P/N 044221) i.d. PEEK tubing for the 1mm column and be 0.0025" (P/N 071870) i.d. PEEK tubing for the 0.25mm column. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers. IonSwift MAX-100 1 mm can be run on a 2 mm system without any further optimization. However, the MAX-100 0.25 mm must be used with ICS-5000 capillary system.

If you need assistance in properly configuring your system contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

3.2. System Requirements

3.2.1. System Requirements for 1 mm Operation

The IonSwift MAX-100 1 mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. For isocratic and gradient analyses, a microbore pump should be employed. It is recommended to run the 1mm column on the system optimized for the 2mm operation using 0.005" tubing.

3.2.2. System Requirements for 0.25 mm Operation

The IonSwift MAX-100 0.25 mm Guard and Capillary Columns are designed to run on a capillary Dionex Ion Chromatograph equipped with suppressed conductivity detection. It is recommended to run the capillary column only on the ICS-5000 capillary system for the best performance.

3.3. Installing the CR-ATC Trap Column for Use with EGC - KOH Cartridge

For IonSwift MAX-100 applications using an EGC-KOH cartridge, a CR-ATC Continuously Regenerated Trap Column (P/N 060477 or 072078) should be installed at the EGC eluent outlet to remove trace level anionic contaminants from the carrier deionized water. See the CR-TC Product Manual (Document No. 031910) for instructions.

As an alternative, the ATC-HC Trap Column (P/N 059604) should be installed between the pump outlet and the inlet of the EluGen Cartridge in the EG Module to remove anionic contaminants from the carrier deionized water. See the ATC-HC Product Manual (Document No. 032697) for instructions.

The ATC-HC (P/N 059604) and Trap Column will require off-line regeneration. To use the ATC-HC or Anion Trap Column, refer to the Product Manual.

3.4. The Injection Loop

3.4.1. The 1 mm System Injection Loop, 2 - 10 µL

For most applications on a 1 mm analytical system, a 2 - 10 µL injection loop is sufficient. Generally, you should not inject more than 8 nanomoles of total anion concentration onto a 1 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 (<10 µL) of the loop volume used with a 4 mm analytical system.

3.4.2. The 0.25 mm System Injection Loop, 0.4µL Internal Loop

For most applications on a 0.25 mm analytical system, a 0.4 µL injection loop is sufficient. Generally, you should not inject more than 0.5 nanomoles of total anion concentration onto the 0.25 mm capillary 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.

3.5. The IonSwift MAX-100G Guard Column

An IonSwift MAX-100G Guard or Capillary Guard Column is normally used with the IonSwift MAX-100 Analytical or Capillary Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical or capillary column. A guard is placed prior to the analytical or capillary column to prevent sample contaminants from eluting onto the analytical or capillary column. It is easier to clean or replace a guard column than it is an analytical or capillary column. Replacing the MAX-100G Guard or Capillary Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the MAX-100 Analytical or Capillary Column.

3.6. Eluent Storage

IonSwift MAX-100 columns are designed to be used with hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

3.7. Anion Self-Regenerating Suppressor and Anion Capillary Electrolytic Suppressor Requirements

An Anion Self-Regenerating Suppressor should be used for 1 mm applications that require suppressed conductivity detection. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all ASRS 300 modes of operation. An Anion Capillary Electrolytic Suppressor should be used for 0.25 mm applications.



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

NOTE

If you are installing an IonSwift MAX-100 0.25 mm Capillary Column, use an ACES 300 (P/N 072052).
If you are installing an IonSwift MAX-100 1 mm Analytical Column, use an ASRS 300 2 mm, (P/N 064555).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see Document No. 031956, the "Product Manual for the Anion Self-Regenerating Suppressor 300, the ASRS 300." For detailed information on the operation of the Anion Capillary Electrolytic Suppressor, see Document No. 065386.

3.8. Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS 300) may be used instead of an ASRS 300 for applications that require suppressed conductivity detection. Use an AMMS 300 (P/N 064559) with the IonSwift MAX-100 1 mm Analytical Column. It is compatible with all solvents and concentrations with which the systems and columns are compatible. For 1 mm operation, use the AMMS 300 2 mm (P/N 064559).

For detailed information on the operation of the Anion MicroMembrane Suppressor, see Document No. 031727, the "Product Manual for the Anion MicroMembrane Suppressor 300, the AMMS 300".

3.9. Using Displacement Chemical Regeneration (DCR) with the Chemical Suppression Mode

Dionex recommends using the Displacement Chemical Regeneration (DCR) Mode for chemical suppression using sulfuric acid and the Anion MicroMembrane Suppressor (AMMS 300). See the DCR kit manual, Document P/N 031664, for details.



Use proper safety precautions in handling acids and bases.

SAFETY

3.10. Detector Requirements

See Section 2, "Comparison of Ion Chromatography Systems," for 1 mm and 0.25 mm system detector, cell and thermal stabilizer requirements.

3.11. Using an Eluent Generator with MAX-100

Please refer to the EGC manual, Document No. 065018, for information on the operation of the EGC.

3.12. Installation of the Capillary Column

Before installing the new separator column, tear off the column label and slide it into the holder on the front of the cartridge (see Figure 1).

For reference, Figure 1 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 2 shows the column cartridge after installation of only a capillary separator column.

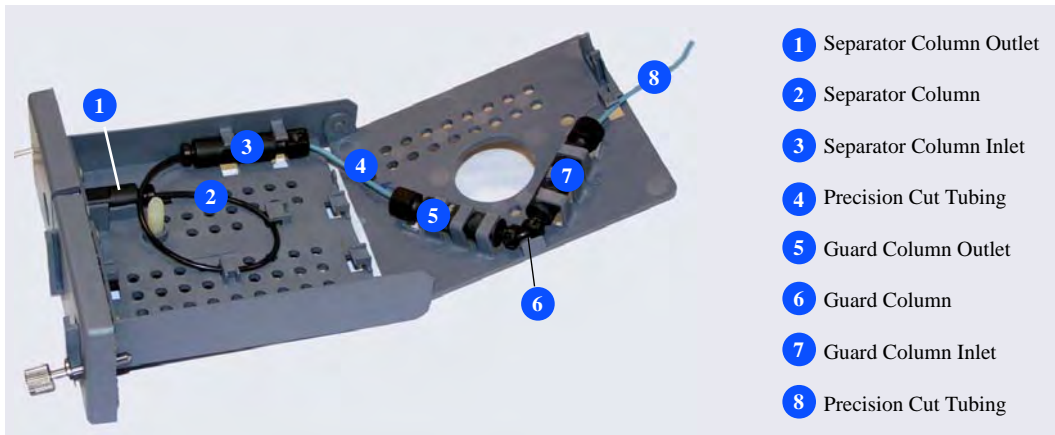


Figure 1
Capillary and Capillary Guard Columns Installed in Column Cartridge

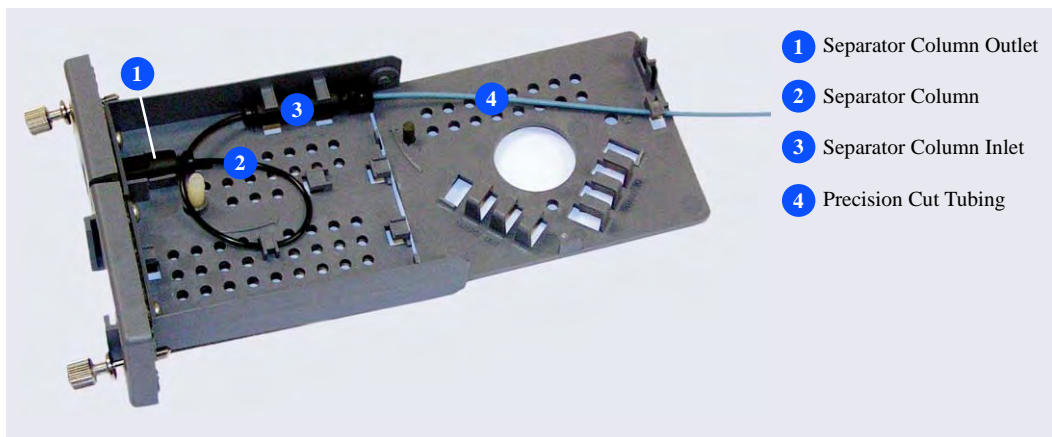


Figure 2
Capillary Column Only Installed in Column Cartridge

1. Locate the IC Cube Tubing Kit (P/N 072186) that is shipped with the IC Cube. The tubing kit includes the following items:

Table 4
Contents of the IC Cube Tubing Kit (P/N 072186)

Part	Length / Quantity	Part Number	Used To Connect...
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue	65 mm (2.56 in)	072188	50 mm guard column outlet to 250 mm separator column inlet
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	115 mm (4.53 in)	072189	Guard column inlet to injection valve
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue	75 mm (2.93 in)	074603	35 mm guard column outlet to 150 mm separator column inlet
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	210 mm (8.27 in)	072187	Separator column inlet to injection valve (if a guard column is not present)
0.25-mm (0.010-in) ID PEEK tubing, black	610 mm (24 in)	042690	EG degas cartridge REGEN OUT to waste (if an EG is not present)
Fitting bolt, 10-32 hex double-cone (smaller), black	3	072949	Connect precision cut 0.062-mm (0.0025-in) ID PEEK tubing
Fitting bolt, 10-32 double-cone (larger), black	1	043275	Connect 0.25-mm (0.010-in) ID PEEK tubing (black)
Ferrule fitting, 10-32 double-cone, tan	4	043276	Use with both sizes of fitting bolts

2. Refer to the following figures for the precision cut tubing required for your configuration:

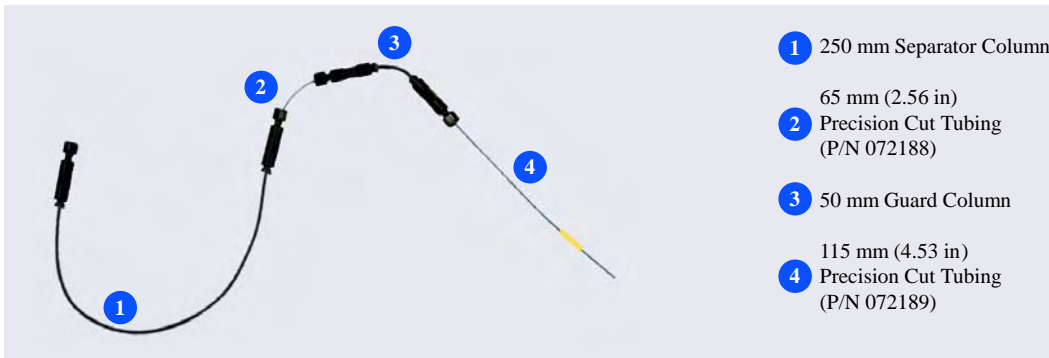


Figure 3
Tubing Connections for 250-mm Capillary Column and 50-mm Capillary Guard Column



Figure 4
Tubing Connections for Capillary Column Only

- Lift up the lid of the column cartridge to open it.
- Remove the fitting plug from the outlet fitting on the separator column. Orient the fitting with a flat side up (see Figure 5) and push the fitting into the opening at the front of the column cartridge until it stops.



Figure 5
Column Outlet Fitting Installed in Column Cartridge

- Coil the capillary column tubing inside the cartridge as shown in Figure 1 or Figure 2. Secure the column tubing and the inlet fitting in the clips on the column cartridge.
- Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
- Route the capillary guard column inlet tubing (if used) or the capillary column inlet tubing through the clip on the top edge of the column cartridge lid.
- Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see Figure 6).



NOTE

If the columns are installed correctly, the cartridge lid snaps closed easily. If the lid does not close easily, do not force it. Open the lid and verify that the columns and tubing are installed correctly and secured in the clips.



Figure 6
Column Cartridge Closed

SECTION 4 – OPERATION

4.1. General Operating Conditions

Sample Volume:	1 mm: 2.5 μ L Loop + 0.8 μ L Injection valve dead volume 0.25 mm: 0.4 μ L Loop (internal)
Column:	1 mm: MAX-100 1 mm Analytical Column + MAX-100G 1 mm Guard Column 0.25 mm: MAX-100 0.25 mm Capillary Column + MAX-100G 0.25 mm Capillary Guard Column
Eluent:	25 mM KOH (for test chromatogram)
Eluent Flow Rate:	1 mm: 0.2 mL/min 0.25 mm: 0.012 mL/min
SRS Suppressor:	1 mm: Anion Self-Regenerating Suppressor 300, ASRS 300 (2 mm) 0.25 mm: Anion Capillary Electrolytic Suppressor, ACES 300 (Capillary) AutoSuppression Recycle Mode for aqueous gradients AutoSuppression External Water Mode for eluents with solvent
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300 (2 mm)
MMS Regenerant:	50 mN H ₂ SO ₄
Expected Background Conductivity:	< 3 μ S
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2. IonSwift MAX-100 Operation Precautions



CAUTION

Filter and Degas Eluents

Filter Samples

Eluent pH between 0 and 14

Sample pH between 0 and 14

0.30 mL/min Maximum Flow Rate for 1 mm Columns

0.020 mL/min Maximum Flow Rate for 0.25 mm Columns

Maximum Operating Pressure = 3,000 psi (20.68 MPa)

4.3. Chemical Purity Requirements

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

4.3.1. Inorganic Chemicals

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

4.3.2. Deionized Water

The deionized water used to prepare eluents should be Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 μ 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.

4.3.3. Solvents

Solvents can be added to the ionic eluents used with IonSwift MAX-100 columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the IonSwift MAX-100 columns is 3,000 psi (20.68 MPa).

The MAX-100 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 5
HPLC Solvents for Use with IonSwift MAX-100 Columns

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

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



CAUTION

The Anion Self-Regenerating Anion Suppressor and Anion Capillary Electrolytic Suppressor (ASRS 300 and ACES 300) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

4.4. Making Eluents that Contain Solvents

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 90% acetonitrile, prepare the eluent by adding 900 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.



NOTE

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

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 bottle.

WARNING

4.5. Sample Concentration

The Low Pressure Trace Anion Concentrator, TAC-LP1 (P/N 046026), IonSwift MAC-100 (P/N 074702) or the IonSwift MAX-100 Guard Column can be used for trace anion concentration work required in high purity water analysis. The function of a concentrator column in these applications is to strip ions 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. Pump the sample onto the concentrator column in the **OPPOSITE** direction of the eluent flow.

When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column. For more detailed information on sample concentration techniques for high sensitivity work refer to Section 4, “Operation,” of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column Product Manual (Document No. 034972). These techniques can also be applied to the IonSwift MAX-100G Guard column and IonSwift MAC-100 Concentrator Column.

SECTION 5 – EXAMPLE APPLICATIONS

5.1. Recommendations for Optimum System Performance

The chromatograms in this section were obtained using columns that reproduced the Production test Chromatogram (see Section 5.2, “Production Test Chromatogram”) on optimized Ion Chromatographs (see Section 3, “Installation”). Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components and laboratory temperatures.

- 1. The IonSwift MAX-100 is designed to perform analyses of large numbers of anions of varying valencies through gradient elution.** In any type of gradient elution system it is important to use eluents that produce a minimum shift in baseline conductivity during the run, as well as a fast equilibration time from one run to the next. Because sodium and potassium hydroxide are converted to water in the suppressor, they are the best choice for an eluent. As long as the capacity of the suppressor is not exceeded, the eluent hydroxide concentration has little effect on background conductivity. For example, a gradient run could begin at a few mM KOH and end at 100 mM KOH, with only a resulting 1 to 3 μ S total baseline change.
- 2. Ensure that your system is properly configured.** It is very important that applications run on 1 mm columns utilize the proper pump configuration (see Section 2, “Comparison of 1 mm and 0.25 mm Ion Chromatography Systems”) and have all system void volumes minimized. Fluctuations in operating temperature can affect the retention time and resolution of analytes and should be controlled.
- 3. Ensure that adequate equilibration time is allowed between runs.** If downward shift in baseline is observed during the isocratic section of the chromatogram, increase the equilibration time.
- 4. Ensure that all of the eluents have been made from high purity reagents and deionized water.** All water used in the preparation of eluents should be degassed, deionized water. For chemical purity requirements see Section 4.3, “Chemical Purity Requirements.”
- 5. The addition of chromate to the sample will help stabilize organic acids.** If your sample or standard contains organic acids, adding chromate (about 10 mg/L) will help stabilize them from bacterial degradation at room temperature.
- 6. Install a CR-ATC Continuously Regenerated Trap Column (P/N 060477 or 072078), or an ATC-HC (P/N 059604 to minimize the baseline shift and to improve retention time reproducibility of analytes when doing gradient chromatography and to keep baseline shift to a minimum.** For detailed information on CR-ATC, refer to CR-TC product manual (P/N 031910).
- 7. Use a guard column to protect the analytical column.** If column performance deteriorates and it is determined that the guard and analytical columns has been fouled, refer to the column cleanup protocols in, “Column Care.”
- 8. You can increase the sensitivity of your system by using sample concentration techniques (see Section 4.5, “Sample Concentration”).**



NOTE

Carbon dioxide readily dissolves in dilute basic solutions, forming carbonate. Carbonate contamination of eluents can affect the retention times of the anions being analyzed. Eluents should be maintained under an inert helium atmosphere to avoid carbonate contamination.

5.2. Production Test Chromatograms

Gradient elution of common anions on the IonSwift MAX-100 Analytical and Capillary Columns have been optimized utilizing a hydroxide eluent. By using this eluent, common inorganic anions can be used to test the performance of the MAX-100 Column. To guarantee that all IonSwift MAX-100 Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. An operating temperature of 35°C is used to ensure reproducible resolution and retention.

5.2.1. Production Test Chromatogram: IonSwift MAX-100 0.25 mm

Column: IonSwift MAX-100 0.25 x 250 mm
 Eluent: 0.2mM KOH for 0.1 minute, 0.2mM to 2mM KOH in 1.9 minutes,
 2mM to 15mM KOH in 6 minutes, 15mM to 35mM KOH in 4 minutes,
 Hold for 3 minutes at 35mM KOH
 Flow Rate: 12µL/min
 Inj. Volume: 0.4µL
 Temperature: 35 °C
 Detection: Suppressed Conductivity
 Suppressor: Anion Capillary Electrolytic Suppressor, ACES 300
 Applied Current: 10mA

Peaks:	mg/L
1. Fluoride	0.4
2. Chloride	1
3. Nitrite	2
4. Bromide	4
5. Nitrate	4
6. Carbonate	-
7. Sulfate	2
8. Phosphate	6

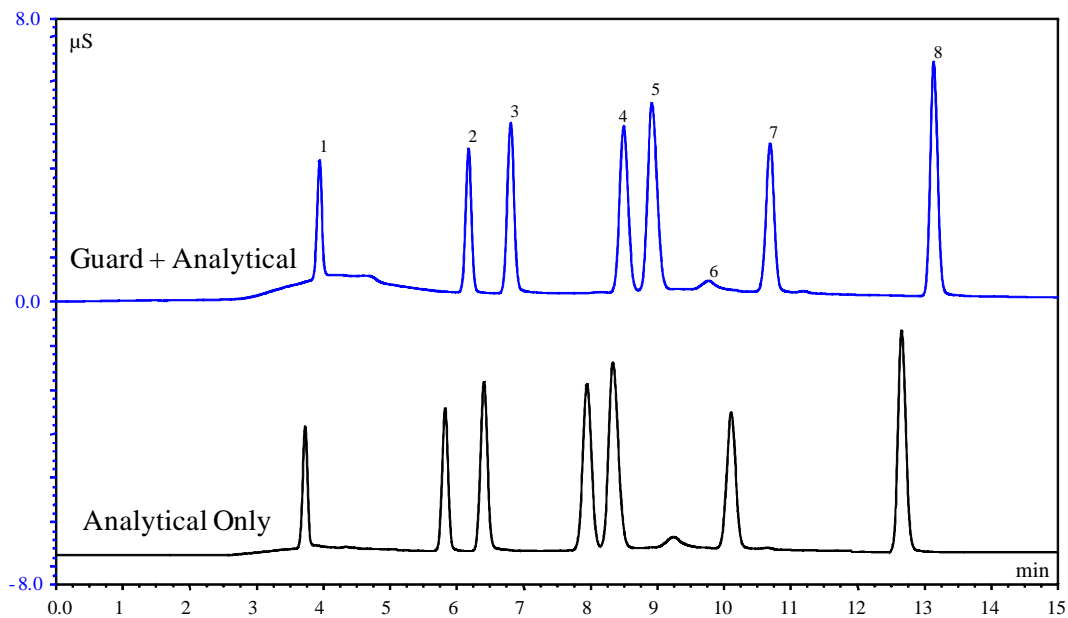


Figure 7
Production Test Chromatograms: MAX-100 0.25mm

5.2.2. Production Test Chromatogram: IonSwift MAX-100 1 mm

Column: IonSwift MAX-100 1 x 250 mm
 Eluent: 0.2mM KOH for 0.1 minute, 0.2mM to 2mM KOH in 1.9 minutes,
 2mM to 15mM KOH in 6 minutes, 15mM to 35mM KOH in 4 minutes,
 Hold for 3 minutes at 35mM KOH
 Flow Rate: 0.2mL/min
 Inj. Volume: 2.5µL
 Temperature: 35 °C
 Detection: Suppressed Conductivity
 Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (2 mm)
 Applied Current: 18 mA

Peaks:	mg/L
1. Fluoride	2.0
2. Chloride	5.0
3. Nitrite	10.0
4. Bromide	20.0
5. Nitrate	20.0
6. Carbonate	-
7. Sulfate	10.0
8. Phosphate	30.0

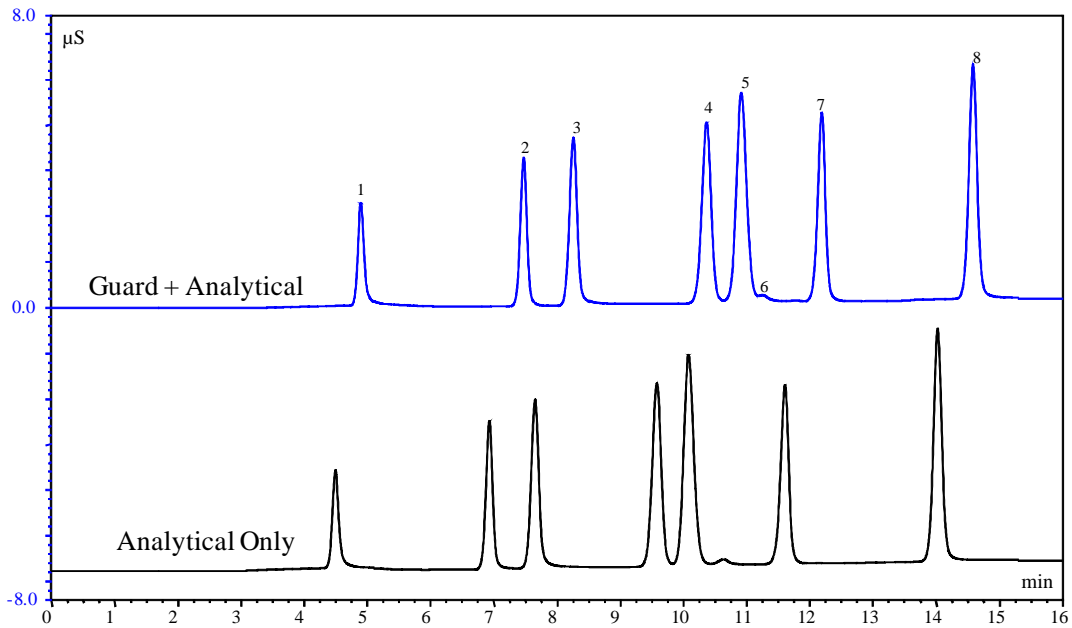


Figure 8
Production Test Chromatograms: MAX-100 1mm

5.3. Optimization of Gradient Program Based on the Column Capacity Using MAX-100 1mm

Gradient program 1 was optimized using MAX-100 1mm analytical column only. The MAX-100 guard column adds about 20% of the column capacity and when the gradient method was kept constant, carbonate eluted too close to nitrate. By adding 20% time to each of the gradient steps (see gradient program 2), the carbonate can be moved out and away from the nitrate peak.

This comparison demonstrates that if the resolution of two peaks is affected due to different capacity of the column (in this case column capacity was increased by placing the guard column in-line), one can adjust the gradient program by whatever the increase is in the column capacity to get desired resolution of closely placed peak pairs with different valency.

Column:	IonSwift MAX-100 1 x 250 mm (A+G)	Peaks:	mg/L
Gradient Program 1:	0.2mM KOH for 0.1 minute, 0.2mM to 2mM KOH in 1.9 minutes, 2mM to 15mM KOH in 6 minutes, 15mM to 35mM KOH in 4 minutes, Hold for 3 minutes at 35mM KOH	1. Fluoride	2.0
Gradient Program 2:	0.2mM KOH for 0.1 minute, 0.2mM to 2mM KOH in 2.28 minutes, 2mM to 15mM KOH in 7.2 minutes, 15mM to 35mM KOH in 4.8 minutes, Hold for 3 minutes at 35mM KOH	2. Chloride	5
Flow Rate:	0.2mL/min	3. Nitrite	10
Inj. Volume:	2.5µL	4. Bromide	20
Temperature:	35 °C	5. Nitrate	20
Detection:	Suppressed Conductivity	6. Carbonate	--
Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 (2 mm)	7. Sulfate	10
Applied Current:	18mA	8. Phosphate	30

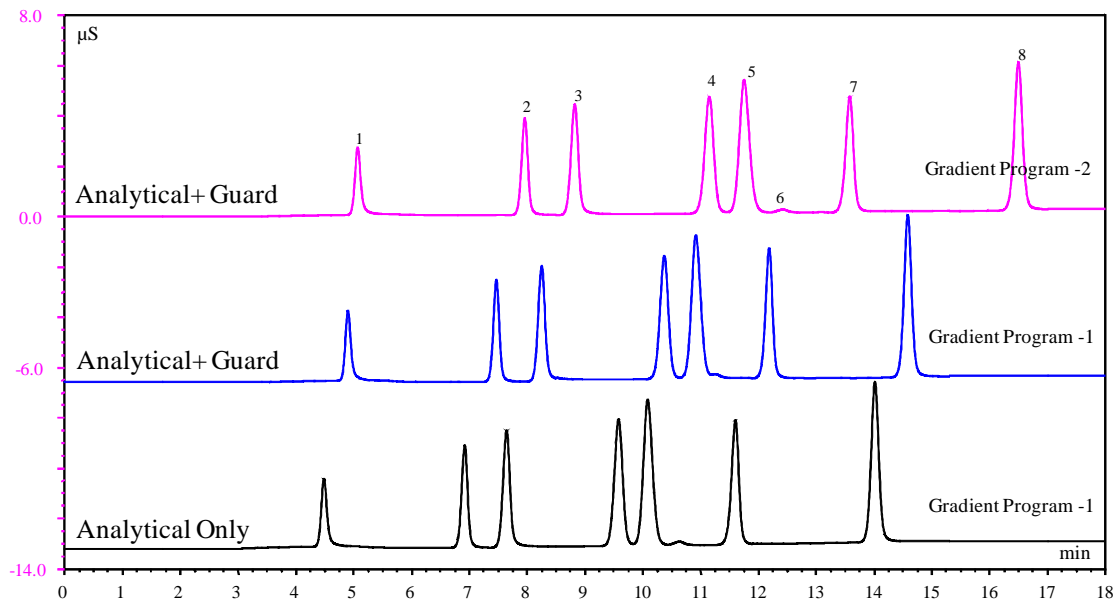


Figure 9
Optimization of Gradient Program Based on the Column Capacity Using MAX-100 1mm

5.4. Gradient Elution of Large Numbers of Inorganic Anions and Organic Acids Using a KOH Gradient

A large number of inorganic anions and organic acid anions can be separated on the IonSwift MAX-100 using gradient elution. The potassium hydroxide gradient is optimized in order to elute mono-, di-, and trivalent organic acid anions in a single run. The starting eluent in the beginning of the gradient has a low concentration allowing fluoride to elute after the void volume and also separates several weakly retained monovalent organic acids. The hydroxide concentration in the later part of the gradient elutes polyvalent ions such as trivalent phosphate, citrate, and cis- and trans-aconitate.

5.4.1. IonSwift MAX-100 0.25 x 250 mm

Column: IonSwift MAX-100 0.25 x 250 mm (Capillary + Capillary Guard)
 Eluent: 0.1 mM for 0.1 min
 0.1 mM to 2 mM KOH in 5 min,
 2 mM to 25 mM in 15 min,
 25 mM to 65 mM KOH in 10 min
 Flow Rate: 0.012 mL/min
 Suppressor: ACES® 300
 Inj. Volume: 0.4 µL
 Temperature: 35 °C
 Detection: Suppressed Conductivity
 Suppressor: Anion Capillary Electrolytic Suppressor, ACES 300
 Applied Current: 15mA

Peaks:	mg/L
1. Fluoride	0.6
2. Acetate	2
3. Formate	2
4. Butyrate	2
5. Galacturonate	2
6. Chloride	1
7. Nitrite	2
8. Bromide	2
9. Nitrate	2
10. Carbonate	4
11. Sulfate	2
12. Oxalate	2
13. Tungstate	4
14. Phosphate	4
15. Chromate	4
16. Citrate	4
17. Isocitrate	4
18. cis-Aconitate	--
19. trans-Aconitate	4

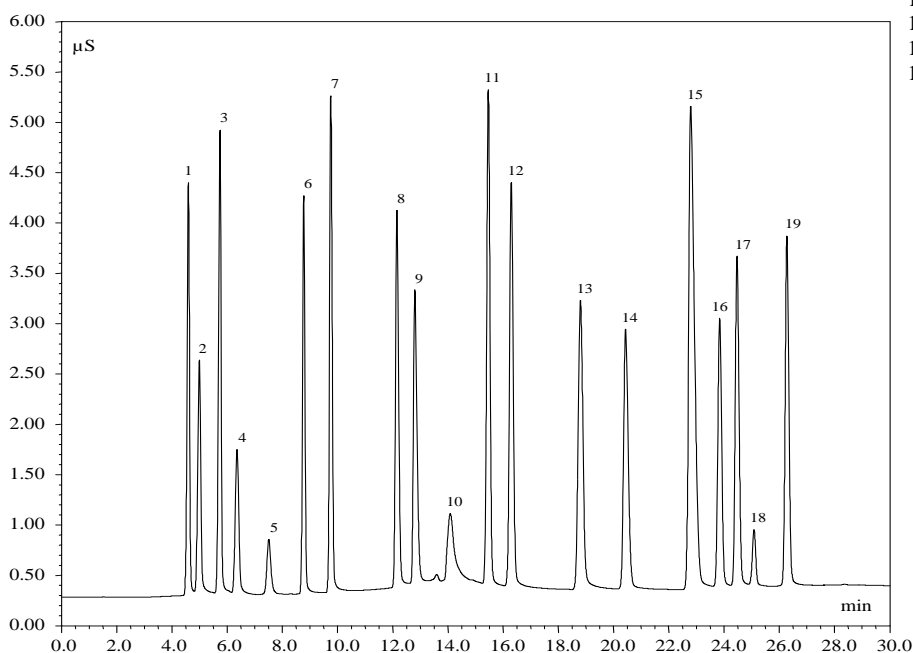


Figure 10
Gradient Elution of Large Numbers of
Inorganic anion and Organic Anion Acids Using KOH Gradient

5.4.2. IonSwift MAX-100 1 x 250 mm

Column : IonSwift MAX-100 1 x 250 mm (A+G)
 Eluent: 0.1mM for 0.1minutes
 0.1 mM to 2 mM KOH in 5 min,
 2 mM to 25 mM in 15 min,
 25 mM to 65 mM KOH in 10 min
 Flow Rate : 0.2 mL/min
 Detection: Suppressed Conductivity
 Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (2 mm)
 Applied Current: 18 mA
 Inj. Volume: 2.5 µL
 Temperature: 35 °C

Peaks:	mg/L
1. Fluoride	3
2. Acetate	10
3. Formate	10
4. Butyrate	10
5. Galacturonate	10
6. Chloride	5
7. Nitrite	10
8. Bromide	10
9. Nitrate	10
10. Carbonate	20
11. Sulfate	10
12. Oxalate	10
13. Tungstate	20
14. Phosphate	20
15. Chromate	20
16. Citrate	20
17. Isocitrate	20
18. cis-Aconitate	-
19. trans-Aconitate	20

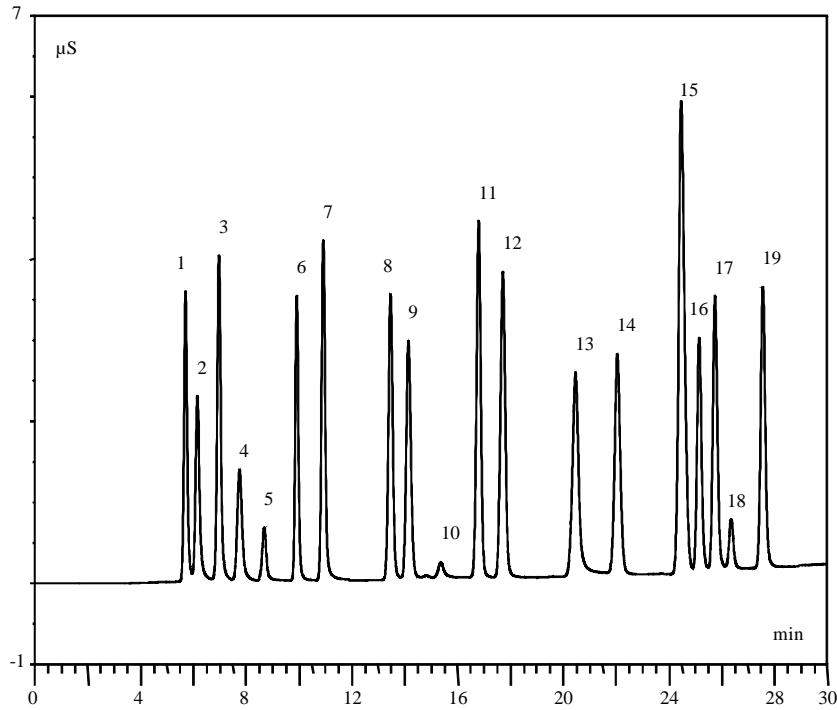


Figure 11
Gradient Elution of Large Numbers of
Inorganic anion and Organic Anion Acids Using KOH Gradient and MAX-100 1mm

5.5. Refinery Amine Scrubbing Solution Using IonSwift MAX-100 1 x 250 mm Column

Figure 12 uses an optimized potassium hydroxide gradient for analysis of a refinery scrubbing sample. The MAX-100 column has a high capacity which allows injection of concentrated samples for determination of trace components.

Column: IonSwift MAX-100 1 x 250 mm
 Eluent: 0.1mM KOH for 0.1min
 0.1 mM to 2 mM KOH for 5 min,
 2–25 mM KOH in 15 min,
 25–65 mM KOH in 10 min

Flow Rate: 0.2 mL/min

Detection: Suppressed Conductivity

Suppressor: Anion Self-Regenerating Suppressor 300, ASRS 300 (2 mm)

Applied Current: 41 mA

Inj. Volume: 10 μ L

Sample Dilution: 1:1000

Temperature: 35 °C

Peaks:	mg/L
1. Acetate	1.9
2. Formate	10.4
3. Chloride	0.2
4. Sulfate	1.2
5. Oxalate	0.02
6. Phosphate	0.5
7. Thiosulfate	0.04
8. Thiocyanate	3.9

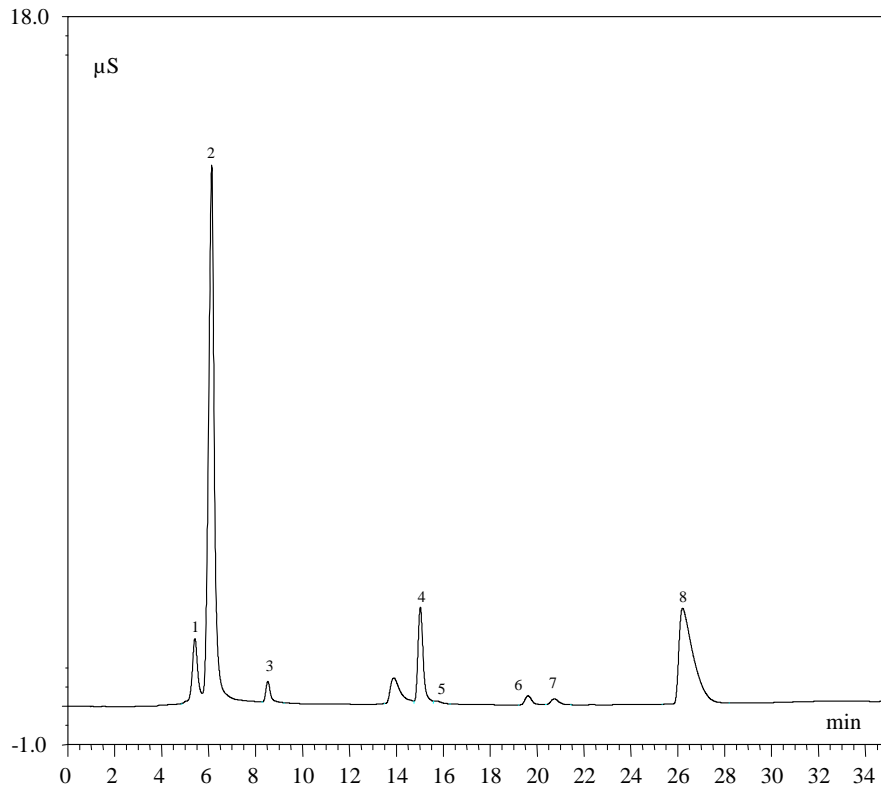


Figure 12
Refinery Amine Scrubbing Solution

5.6. Analysis of Apple Juice Using IonSwift MAX-100 0.25 x 250mm and ICS-5000 Capillary System

Figure 13 uses an optimized potassium hydroxide gradient for analysis of apple juice samples. All three juice samples were diluted 1:40 with deionized water and filtered through a 0.45 mm syringe filter. Note that under these conditions, malic and succinic acid co-elute and can be separated using methanol in the gradient.

Column: IonSwift MAX-100 0.25 x 250 mm (Capillary + Capillary Guard)
 Eluent: 0.1 mM for 0.1 min
 0.1 mM 2 mM KOH for 5 min,
 2–25 mM KOH in 15 min,
 25–65 mM KOH in 10 min
 Flow Rate: 12 µL/min
 Inj. Volume: 0.4 µL
 Sample Dilution: 1:40
 Temperature: 35 °C
 Detection: Suppressed Conductivity
 Suppressor: Anion Capillary Electrolytic Suppressor, ACES 300
 Applied Current: 15 mA

- Peaks:**
1. Quinate
 2. Lactate
 3. Propionate
 4. Formate
 5. Galacturonate
 6. Chloride
 7. Succinate + Malate*
 8. Sulfate
 9. Oxalate
 10. Fumarate
 11. Phosphate
 12. Citrate
 13. Isocitrate
 14. cis-Aconitate

* Succinate and Malate can be resolved using organic solvent.

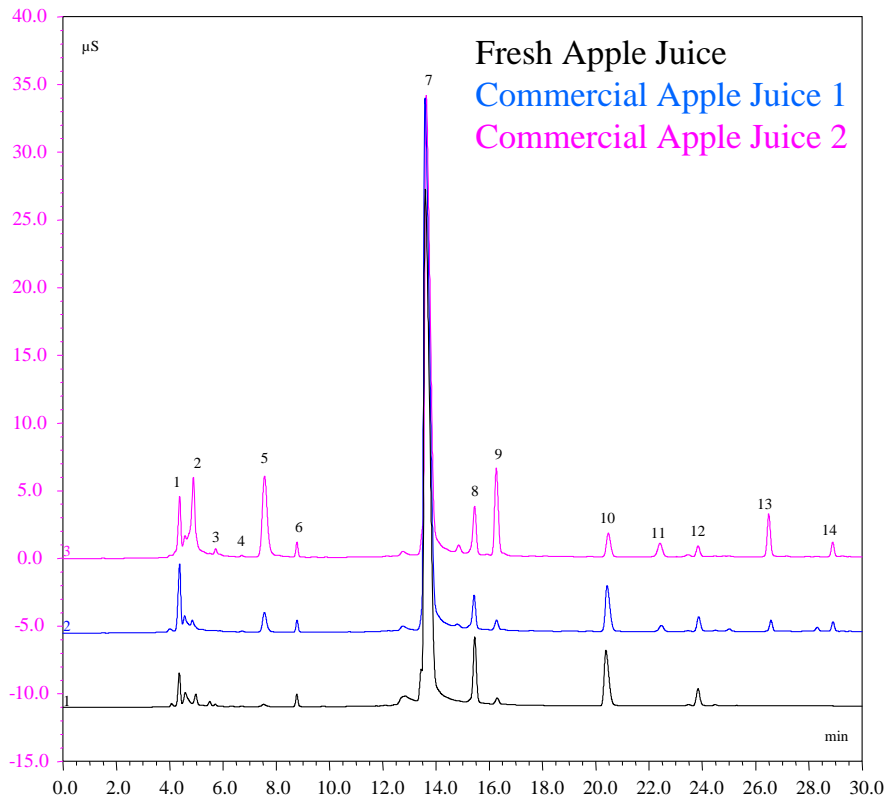


Figure 13
Analysis of Apple Juice

5.7. Analysis of Apple Juice Using two Different Flow Rates

Figure 14 uses an optimized potassium hydroxide gradient for analysis of apple juice samples. Note that by increasing the flow rate by ~30% , the analysis time can be reduced by ~30%

Column: IonSwift MAX-100 0.25 x 250 mm (A+G)
 Eluent A: 0.1-1 mM KOH for 7 min,
 2–25 mM KOH in 15 min,
 25–65 mM KOH in 10 min
 Eluent B: 0.11 mM KOH for 4.5 min,
 2–25 mM KOH in 5.4 min,
 25–65 mM KOH in 10 min
 Flow Rate A: 12 µL/min
 Flow Rate B: 18 µL/min
 Detection: Suppressed Conductivity
 Suppressor: Anion Capillary Electrolytic Suppressor, ACES 300
 Applied Current: 15 mA
 Inj. Volume: 0.4 µL
 Sample: Apple Juice (1:40 Dilution)
 Temperature: 35 °C

Peaks:

1. Quinate
2. Acetate
3. Galacturonate
4. Chloride
5. Succinate + Malate*
6. Sulfate
7. Oxalate
8. Fumarate
9. Phosphate
10. Citrate
11. Isocitrate
12. cis-Aconitate

* Succinate and Malate can be resolved using organic solvent

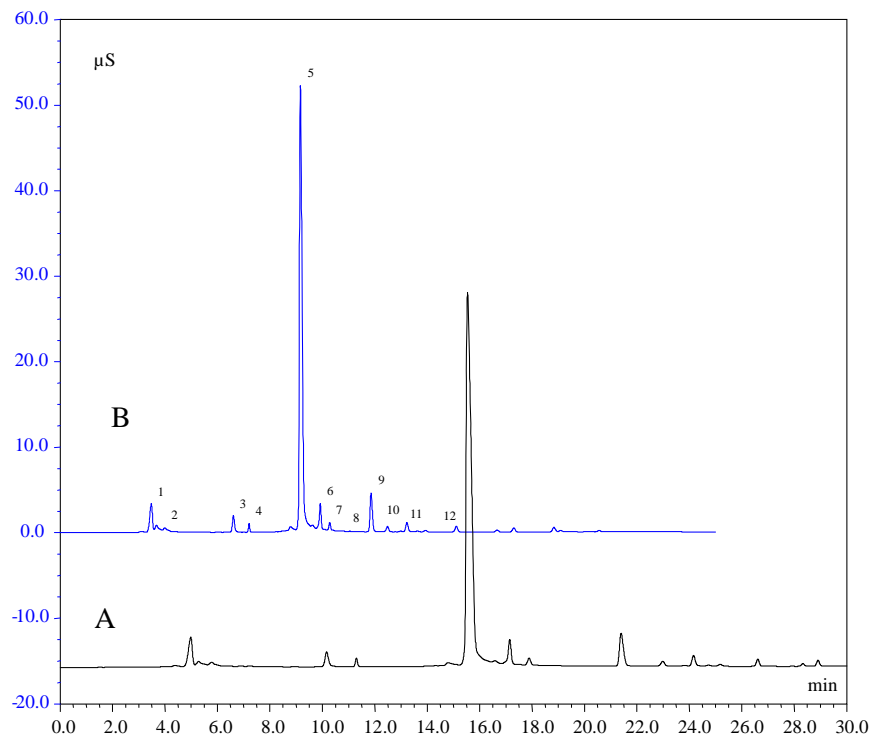


Figure 14
Analysis of Apple Juice
With Two Different Flow Rates

5.8. Analysis of Beer Sample Using IonSwift MAX-100 0.25x250mm and ICS-5000 Capillary System

Figure 15 uses an optimized potassium hydroxide gradient for the analysis of beer samples. Beer samples were diluted 1:40 with deionized water. Note that under these conditions, malic and succinic acid co-elute and can be separated using methanol in the gradient. Also, the samples were spiked with 1 ppm of butyrate to show that it is well separated from formic and pyruvic acid using an aqueous eluent.

Column: IonSwift MAX-100 0.25 x 250 mm (Capillary + Capillary Guard)
 Eluent: 0.1 for 0.1 min
 0.1 mM 2mM KOH for 5 min,
 2–25 mM KOH in 15 min,
 25–65 mM KOH in 10 min
 Flow Rate: 12 µL/min
 Detection: Suppressed Conductivity
 Suppressor: Anion Capillary Electrolytic Suppressor, ACES 300
 Applied Current: 15 mA
 Inj. Volume: 0.4 µL
 Sample Dilution: 1:40
 Temperature: 35 °C

Peaks:

1. Lactate
2. Acetate
3. Formate
4. Butyrate (1ppm spiked)
5. Pyruvic
6. Chloride
7. Succinate + Malate*
8. Sulfate
9. Oxalate
10. Fumarate
11. Phosphate
12. Citrate
13. Isocitrate
14. trans-Aconitate

* Succinate and Malate can be resolved using organic solvent

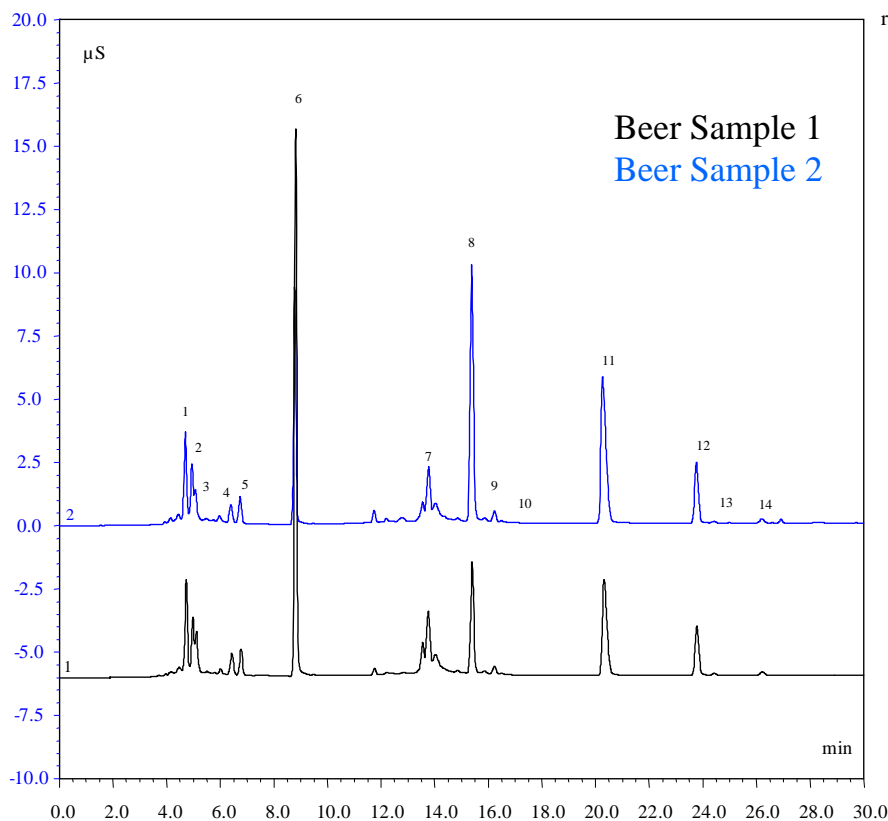


Figure 15
Analysis of Beer Sample
Using IonSwift MAX-100 0.25x250 mm and
ICS-5000 Capillary System

SECTION 6 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonSwift MAX-100 columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the Dionex Office nearest you (see, "Dionex Worldwide Offices").

Table 6
MAX-100 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Other System Components	Replace Unplug, Replace	6.1.2 Component Manual
High Background Conductivity	Contaminated ATC	Clean Column	6.2.2
	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.3
	Contaminated ASRS or AMMS Contaminated Hardware	Clean Suppressor Clean Component	6.2.4, Component Manual Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Check the tubing connection to ensure it is butting in properly for all the column and injection valve connections	6.3.1.A, Component Manual
	Column Headspace	Replace Column	6.3.1.B
Short Retention Times	Flow Rate Too Fast	Recalibrate Pump	6.3.2.A
	Conc. Incorrect Eluents	Remake Eluents	6.3.2.B
	Column Contamination	Clean Column	6.3.2.C, 6.3.2.D
Poor Front End Resolution	Conc. Incorrect Eluents	Remake Eluents	6.3.3.A
	Column Overloading	Reduce Sample Size	6.3.3.B, 3.4.1, 3.4.2
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual
	Large System Void Volumes	Replumb System	6.3.3.D, Component Manual
Spurious Peaks	Sample Contaminated	Pretreat Samples	6.3.4.A, 6.3.4.B
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual

6.1. High Back Pressure

6.1.1. Finding the Source of High System Pressure

Total system pressure for the IonSwift MAX-100 (0.25 mm) Capillary Guard Column plus the MAX-100 (0.25 mm) Capillary Column when using the test chromatogram conditions should be equal or less than 1900 psi. If the system pressure is higher than 1900 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated.

- A. **Make sure that the pump is set to the correct eluent flow rate.** Higher than recommended eluent flow rates will cause higher pressure. Measure the pump flow rate if necessary with a stop watch and graduated cylinder.
- B. **Determine which part of the system is causing the high pressure.** High pressure could be due to a plugged tubing or tubing with collapsed walls, an injection valve with a clogged port, a column with particulates clogging the bed support, a clogged High-Pressure In-Line Filter, the suppressor or the detector cell.

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi. Continue adding system components (injection valve, column(s), suppressor and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 7, "Typical MAX-100 Operating Back Pressures").

The Anion Self-Regenerating Suppressor 300 with backpressure loops may add up to 100 psi (0.69 MPa). No other components should add more than 100 psi (0.69 MPa) of pressure. Refer to the appropriate manual for cleanup or replacement of the problem component.

Table 7
Typical MAX-100 Operating Back Pressures

Column	Typical Back Pressure psi (Mpa)	Standard Flow Rate mL/min	Maximum Flow Rate
IonSwift MAX-100 Analytical Column (1 x 250 mm)	< 1800 (12.41)	0.20	0.30
IonSwift MAX-100G Guard Column (1 x 50 mm)	< 500 (3.45)	0.20	0.30
IonSwift MAX-100 Capillary Column (0.25 x 250 mm)	< 1800 (12.41)	0.012	0.020
IonSwift MAX-100G Capillary Guard Column (0.25 x 50 mm)	< 500 (23.45)	0.012	0.020

6.2. High Background

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

<u>ELUENT</u>	<u>EXPECTED BACKGROUND CONDUCTIVITY</u>
1.0 mM KOH	0.3 - 0.5 μ S
60 mM KOH	0.5-2 μ S
60 mM KOH/15% CH ₃ OH	2.0 - 3.0 μ S

6.2.1. Preparation of Eluents

- A. **Make sure that the eluents and the regenerant are made correctly.**
- B. **Make sure that the eluents are made from chemicals with the recommended purity.**
- C. **Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.**

6.2.2. A Contaminated Trap Column, CR-ATC, ATC-HC, or ATC-3

For RFIC-EG operation, use a CR-ATC Trap Column. Install a CR-TC Anion Trap Column (P/N 060477 or 072078) if using an Eluent Generator with EGC-KOH cartridge. If the CR-ATC becomes contaminated, please refer to Section 6, Clean-Up, in the CR-ATC manual (P/N 031910).

Remove the IonSwift MAX-100 (Capillary) Guard and MAX-100 Capillary or 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 MAX-100 at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in "Column Cleanup" (see, "Column Care").

6.2.3. Contaminated Hardware

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

6.2.4. A Contaminated ASRS 300, ACES 300 or AMMS 300 Suppressor

If the above items have been checked and the problem persists, the Suppressor is probably causing the problem.

- A. **Check the eluent flow rate.** In general, the eluent flow rate for 1.0 mm applications should be 0.20 mL/min. Refer to the Anion Self-Regenerating Suppressor Product Manual (Document No. 031367) for assistance in determining that the eluent is within suppressible limits. The eluent flow rate for 0.25 mm applications should be 0.012 mL/min.
- B. **If the background is very high, (>1,000 μ S) or the baseline noise is very high, the ASRS or ACES 300 may have failed to suppress the eluent.** You may need to replace the ASRS or ACES 300 suppressor.
- C. If you are using eluents containing solvents, use the ASRS or ACES in external water mode and flow rate should be 3 - 7 mL/min, for an ASRS 300 (2 mm) or 0.10 mL/min for an ACES 300.
- D. **Check the regenerant flow rate at the REGEN OUT port of the AMMS.** For the example isocratic applications, this flow rate should be 3 - 5 mL/min.

6.3. POOR PEAK RESOLUTION

One of the unique features of the MAX-100 is fast equilibration time in gradient applications from the last eluent (high ionic strength) to the first eluent (low ionic strength). The actual equilibration time depends on the ratio of the strongest eluent concentration to the weakest eluent concentration. Typically equilibration times range from 7 to 10 minutes.

6.3.1. Loss of Column Efficiency

- A. **Check to see if headspace has developed in the (capillary) guard or analytical / capillary 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. If the monolith does not fill the column body all the way to the top, it means that the monolith 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 0.0025" for 0.25 mm systems or no greater than 0.005" for 1 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. Do not cut tubing for 0.25 mm capillary systems; always use the pre-cut tubing provided by Dionex.

6.3.2. Poor Resolution Due to Shortened Retention Times

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

- A. **Check the flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. **Check the flow rate of the pump. If the flow rate is higher than the set flow rate, it will cause longer run time as it will dilute the eluent generated by the eluent generator. If the flow rate is lower than the set flow rate, it will cause short run as eluent will be more concentrate than needed for the separation.**
- B. **Column contamination can lead to a loss of column capacity.** This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to "Column Cleanup" (see, "Column Care"), for recommended column cleanup procedures.

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

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

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. No water wash is necessary. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column. If you need assistance in solving resolution problems, contact the North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

6.3.3. Loss of Front End Resolution

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

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

6.3.4. Spurious Peaks

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

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonSwift MAX-100 columns, contact the North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, “Dionex Worldwide Offices”).

- B. **The injection valve may need maintenance.** When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

6.3.5. Poor Efficiency Using Capillary Columns

Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks (see Figure 16).

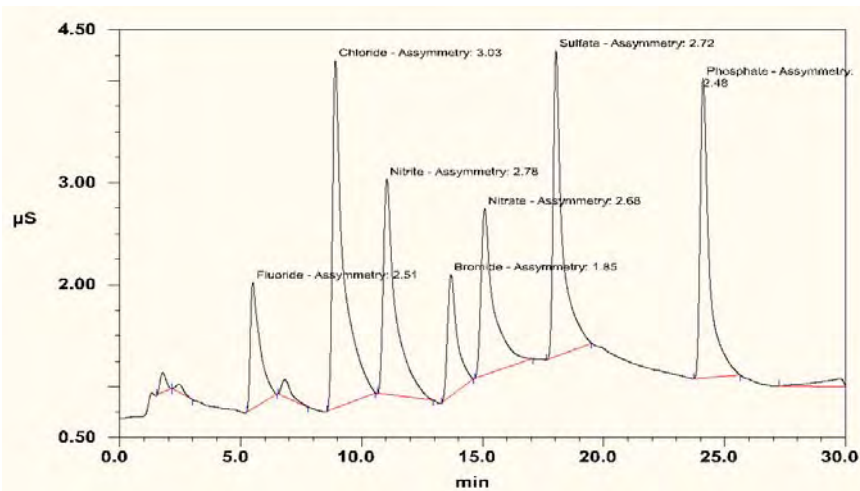


Figure 16
Tailing Peaks Caused by Incorrectly Installed
Capillary Tubing Fittings

When connecting a capillary tube fitting, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing. Figure 17 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

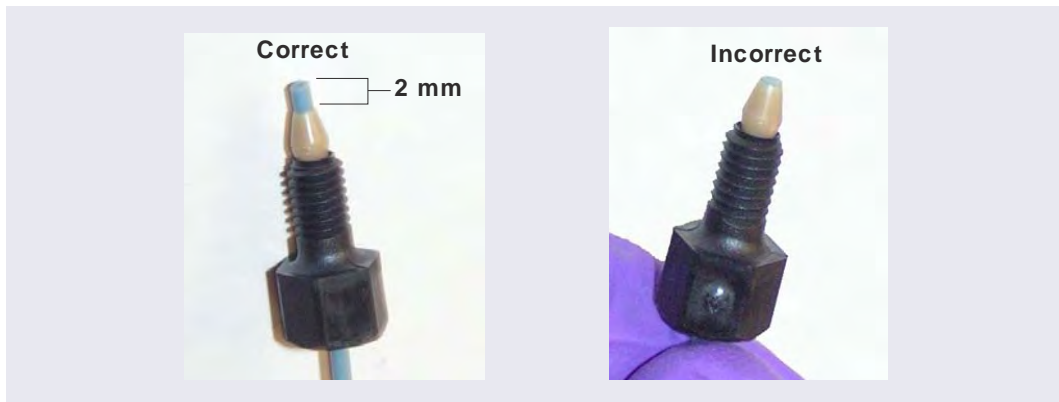


Figure 17
Correct and Incorrect Ferrule and
Fitting Bolt Placement for Capillary Tubing Connections

6.3.6. Installing Capillary Fittings

1. Install the fitting bolt and ferrule onto the tubing. Position the ferrule at least 2 mm (0.1 in) from the end of the tubing.



2. Insert the tubing into the port until it stops.



3. While maintaining pressure on the tubing to keep it in place in the port, tighten the fitting bolt fingertight.



APPENDIX B - COLUMN CARE

B.1 Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonSwift MAX-100 columns is 3,000 psi (20.67 MPa).

B.2 Column Start-Up

The column is shipped using the 100 mM sodium borate as the storage solution. Follow the column Start-Up instructions provided in the IonSwift™ MAX-100 QuickStart (P/N 065308). Prepare the eluent shown on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

B.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), use 100 mM Sodium Borate 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.

B.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 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 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 ≤ 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

B.4.1 Choosing the Appropriate Cleanup Solution

- A. **Concentrated hydroxide solutions** such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.
- B. **Concentrated acid solutions** such as 1 to 3 M HCl, remove high valency hydrophilic ions by ion suppression and elution by the chloride ion.
- C. **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.

Iron contamination of the MAX-100 results in an initial decrease in polyphosphate peak heights. However, successive injections of polyphosphate samples will gradually remove the iron resulting in increasing peak heights. If the eluent is contaminated with iron, polyphosphate peak heights may vary depending on the rate of column contamination versus the rate of column cleaning due to repeated injections of polyphosphate samples.

Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.

D. Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents listed in Table 5, HPLC Solvents for Use with IonSwift MAX-100 Columns.

E. 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. The organic solvent then removes the subsequent nonionic and hydrophobic contamination. See Section D above.

A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

F. Regardless of the cleanup solution chosen, use the following cleanup procedure in Section B.4.2 "Column Cleanup Procedure," to clean the MAX-100.

B.4.2 Column Cleanup Procedure

A. Prepare a 200 mL solution of the appropriate cleanup solution using the guidelines in Section B.4.1, "Choosing the Appropriate Cleanup Solution."

B. Disconnect the ASRS 300, AMMS 300 or ACES 300 from the IonSwift MAX-100 Analytical or Capillary Column. If your system is configured with both a guard column and an analytical, or a capillary column, reverse the order of the guard and capillary or analytic column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.



CAUTION

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

C. Set the pump flow rate to 0.010 mL/min for an MAX-100 0.25 mm Capillary or Capillary Guard Column or set the pump flow rate to 0.1mL/min for an MAX-100 1 mm Analytical or Guard Column.

D. Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.

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

F. Rinse the column for 10 minutes with deionized water before pumping eluent over the column.

G. Equilibrate the column(s) with eluent for at least 30 minutes before resuming normal operation.

H. Reconnect the ASRS 300, AMMS 300 or ACES 300 to the MAX-100 Capillary or Analytical Column and **place the guard (capillary) column in line** between the injection valve and the analytical or capillary column if your system was originally configured with a guard (capillary) column.