



# PRODUCT MANUAL

for the

## IonSwift™ MAX-200 Column

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## **Product Manual**

### **For**

**Thermo Scientific Dionex IonSwift™ MAX-200 Capillary Column**  
(0.25 x 250 mm, P/N 075889)

**Thermo Scientific Dionex IonSwift™ MAX-200G Capillary Guard Column**  
(0.25 x 50 mm, P/N 075891)

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## SECTION 1 – INTRODUCTION

The Thermo Scientific Dionex IonSwift MAX-200 monolithic column is designed to provide high speed and high-resolution separations of inorganic anions and oxyhalides including fluoride, chlorite, bromate, chloride, nitrite, chlorate, bromide, nitrate, sulfate and phosphate. The selectivity of the Dionex IonSwift MAX-200 guard plus capillary column has been designed to retain fluoride well out of water dip and separate oxyhalides and the common anions using hydroxide gradients. Hydroxide eluent is normally used for gradient elution to minimize the background shift.

Dionex IonSwift MAX-200 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 inorganic anions and oxyhalides. IonSwift is a new generation of separation media, which are uniquely designed and engineered for separation of small molecules.

Dionex IonSwift monoliths have high permeability. The pore volume is about 60% of the column volume, which is much higher than 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 Dionex IonSwift MAX-200 0.25x250 mm column is compiled of anion exchange polymeric monolithic media which provides high speed and high resolution separations for inorganic anions and oxyhalides. The Dionex IonSwift MAX-200 stationary phase is based on hyperbranched anion exchange condensation polymer, electrosatically attached to the monolithic surface. The Dionex IonSwift MAX-200 offers selectivity similar to the Thermo Scientific Dionex IonPac AS19 column. The Dionex IonSwift MAX-200 columns are stable between pH 0 and 14 and are compatible with eluents containing 0-100% organic solvents.

**Table 1**  
**Dionex IonSwift MAX-200 Column Specifications**

Column	Substrate X-Linking (%)	Column Capacity (µeq/column)	Functional Group	Hydrophobicity
Dionex IonSwift MAX-200 (0.25 x 250 mm)	55	0.8	Alkanol quaternary ammonium	Low
Dionex IonSwift MAX-200G (0.25 x 50 mm)	55	0.16	Alkanol quaternary ammonium	Low

**Table 2**  
**Dionex IonSwift MAX-200 Operating Parameters**

Column	Maximum Back Pressure (psi [Mpa])	Standard Flow Rate (mL/min)	Maximum Flow Rate
Dionex IonSwift MAX-200 Capillary Column (0.25 x 250 mm)	< 2200 [15.17]	0.010	0.020
Dionex IonSwift MAX-200G Capillary Guard Column (0.25 x 50 mm)	< 500 [3.45]	0.010	0.020
Dionex IonSwift MAX-200 Capillary + MAX-200G Capillary Guard	<2700 [18.62]	0.010	0.020

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

## SECTION 2 – INSTALLATION

### 2.1. System Void Volume

When using 0.25 mm columns, it is particularly important to minimize system void volume. For best performance, all of the tubing installed between the injection valve and detector should be 0.0025" i.d. PEEK precut tubing (use tubing from kit P/N 072186). The Dionex IonSwift MAX-200 0.25 mm must be used with the Thermo Scientific Dionex ICS-5000 capillary system and precut tubing.

If you need assistance in properly configuring your system contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

### 2.2. System Requirements

#### 2.2.1. System Requirements for 0.25 mm Operation

The Dionex IonSwift MAX-200 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 Dionex ICS-5000 capillary system for the best performance.

### 2.3. Installing the Thermo Scientific Dionex CR-ATC Trap Column for Use with Thermo Scientific Dionex EGC - KOH Cartridge

For Dionex IonSwift MAX-200 applications using a Dionex EGC-KOH cartridge, a Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 072078) should be installed at the Dionex EGC eluent outlet to remove trace level anionic contaminants from the carrier deionized water. See the Dionex CR-TC Product Manual (Document No. 031910) for instructions.

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

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

## 2.4. The Injection Loop

### 2.4.1. The 0.25 mm System Injection Loop, 0.4 $\mu$ L Internal Loop

For most applications on a 0.25 mm capillary system, a 0.4  $\mu$ L 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.

## 2.5. The Dionex IonSwift MAX-200G Guard Column

A Dionex IonSwift MAX-200G Capillary Guard Column is normally used with the Dionex IonSwift MAX-200 Capillary Column. Retention times will increase by approximately 20% for isocratic analysis and by approximately 4-15% for gradient analysis (~4% for a divalent anion such as sulfate and ~15% for a monovalent anion such as nitrate) when a guard column is placed in-line prior to the capillary column. A guard is placed prior to the capillary column to prevent sample contaminants from eluting onto the capillary column. It is easier to clean or replace a guard column than it is a capillary column. Replacing the Dionex IonSwift MAX-200G Capillary Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the Dionex IonSwift MAX-200 Capillary Column.

## 2.6. Eluent Storage

Dionex IonSwift MAX-200 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).

## 2.7. Dionex Anion Capillary Electrolytic Suppressor Requirements

A Dionex Anion Capillary Electrolytic Suppressor (Dionex ACES 300) should be used for 0.25 mm applications. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all Dionex ACES 300 modes of operation.



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

### NOTE

When installing a Dionex IonSwift MAX-200 0.25 mm Capillary Column, use a Dionex ACES 300 (P/N 072052).

For detailed information on the operation of the Dionex Anion Capillary Electrolytic Suppressor, see Document No. 065386.

## 2.8. Detector Requirements

See Appendix C, "Comparison of Ion Chromatography Systems," for 0.25 mm system detector, cell and thermal stabilizer requirements.

## 2.9. Using a Dionex Eluent Generator with a Dionex IonSwift MAX-200

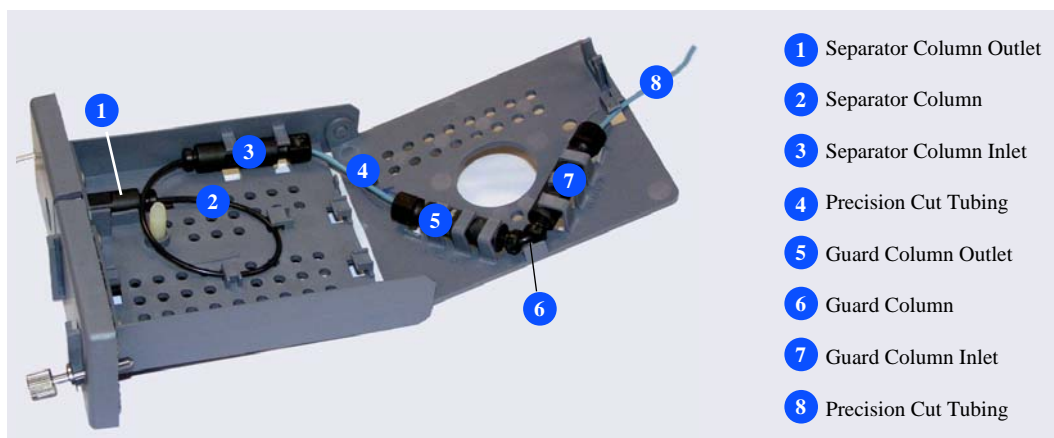
Please refer to the Dionex EGC manual, Document No. 065018, for information on the operation of the Dionex EGC.



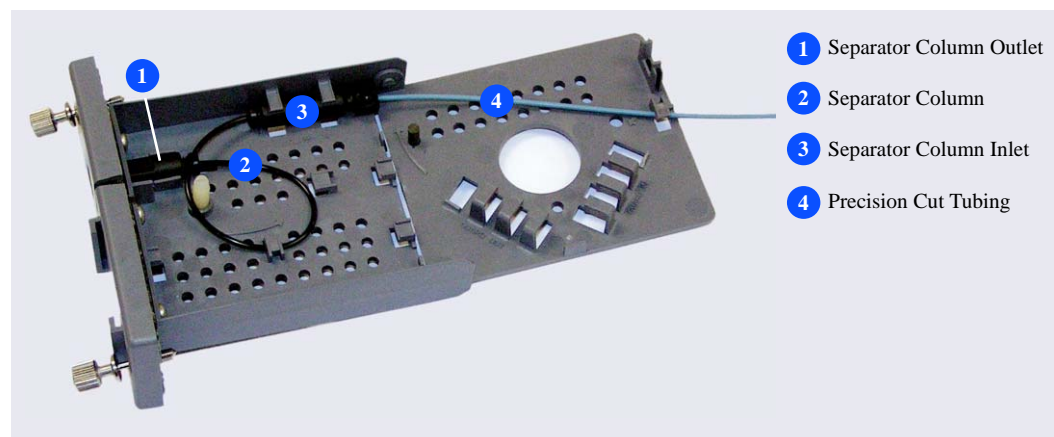
## 2.10. Installation of the Capillary Column

Before installing the new separator column, cut off the column label and slide it into the holder on the front of the cartridge (see Figure 6).

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**



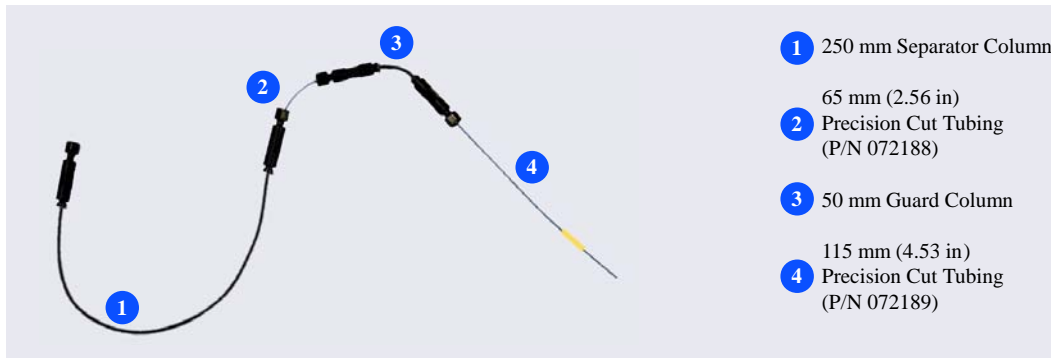
**Figure 2**  
**Capillary Column Only Installed in Column Cartridge**

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

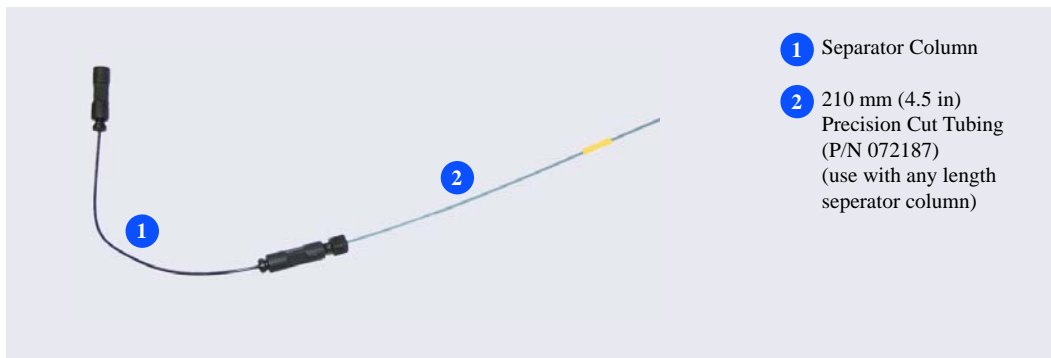
**Table 3**  
**Contents of the Dionex IC Cube Tubing Kit (P/N 072186)**

<b>Part</b>	<b>Length / Quantity</b>	<b>Part Number</b>	<b>Used To Connect...</b>
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, light 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, light 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, light 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, light 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**

3. Lift up the lid of the column cartridge to open it.
4. 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**

5. 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.
6. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
7. 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.
8. 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 3 – OPERATION

### 3.1. General Operating Conditions

Sample Volume:	0.4 µL Loop (internal)
Column:	Dionex IonSwift MAX-200G, MAX-200 Capillary Column, 0.25 mm Capillary Guard Column
Eluent:	2 mM for 0.1 minutes; 2 to 10 mM in 9.9 minutes; 10 mM to 50 mM KOH in 5 minutes Hold for 5 minutes at 50 mM KOH (See QAR)
Eluent Flow Rate:	0.010 mL/min
Dionex SRS Suppressor:	Dionex Anion Capillary Electrolytic Suppressor, Dionex ACES 300 (Capillary) AutoSuppression Recycle Mode for aqueous gradients AutoSuppression External Water Mode for eluents with solvent
Expected Background Conductivity:	< 1 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

### 3.2. Dionex IonSwift MAX-200 Operation Precautions



CAUTION

**Filter and Degas Eluents**  
**Filter Samples**  
**Eluent pH between 0 and 14**  
**Sample pH between 0 and 14**  
**0.020 mL/min Maximum Flow Rate**  
**Maximum Operating Pressure = 3,000 psi (20.68 MPa)**

### 3.3. Chemical Purity Requirements

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

#### 3.3.1. Inorganic Chemicals

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

#### 3.3.2. Deionized Water

The deionized water used to prepare eluents should be Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 µm. Bottled HPLC-Grade Water should not be used since most bottled water contains an unacceptable level of ionic impurities.

### 3.3.3. Solvents

Solvents can be added to the ionic eluents used with Dionex IonSwift MAX-200 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 Thermo Fisher Scientific, we have obtained consistent results using 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 maximum back pressure limit for the Dionex IonSwift MAX-200 columns is 3,000 psi (20.68 MPa).

The Dionex IonSwift MAX-200 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 4**  
**HPLC Solvents for Use with Dionex IonSwift MAX-200 Columns**

<b>Solvent</b>	<b>Maximum Operating Concentration</b>
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 Dionex Anion Capillary Electrolytic Suppressor (Dionex ACES 300) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.*

### 3.4. Making Eluents that Contain Solvents

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



#### 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**

### **3.5. Sample Concentration**

The Thermo Scientific Dionex IonSwift MAC-100 (P/N 074702) or the Thermo Scientific Dionex IonSwift MAX-200 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 3, “Operation,” of the Dionex MAC-100 Column Product Manual (Document No. 065387). These techniques can also be applied to the Dionex IonSwift MAX-200G Guard column.

## SECTION 4 – EXAMPLE APPLICATIONS

### 4.1. 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 Dionex 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 Dionex IonSwift MAX-200 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  $\mu\text{S}$  total baseline change.
- 2. Ensure that your system is properly configured.** A capillary system such as the Dionex ICS-5000 must be used for the Dionex IonSwift MAX-200 capillary column. 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 3.3, “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 Dionex CR-ATC Continuously Regenerated Trap Column (P/N 072078), or a Dionex 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 Dionex CR-ATC, refer to the Dionex CR-ATC 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 Appendix B “Column Care.”**
- 8. You can increase the sensitivity of your system by using sample concentration techniques (see Section 3.5, “Sample Concentration”).**



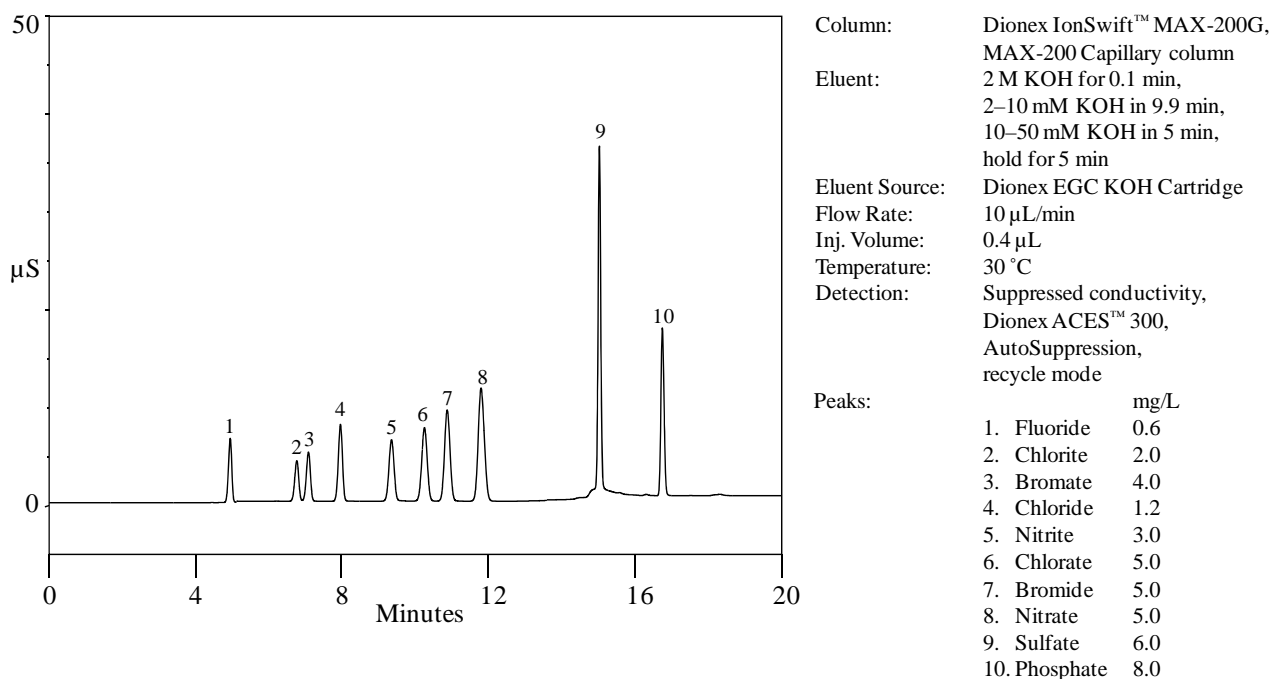
#### NOTE

*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.*



## 4.2. Production Test Chromatograms

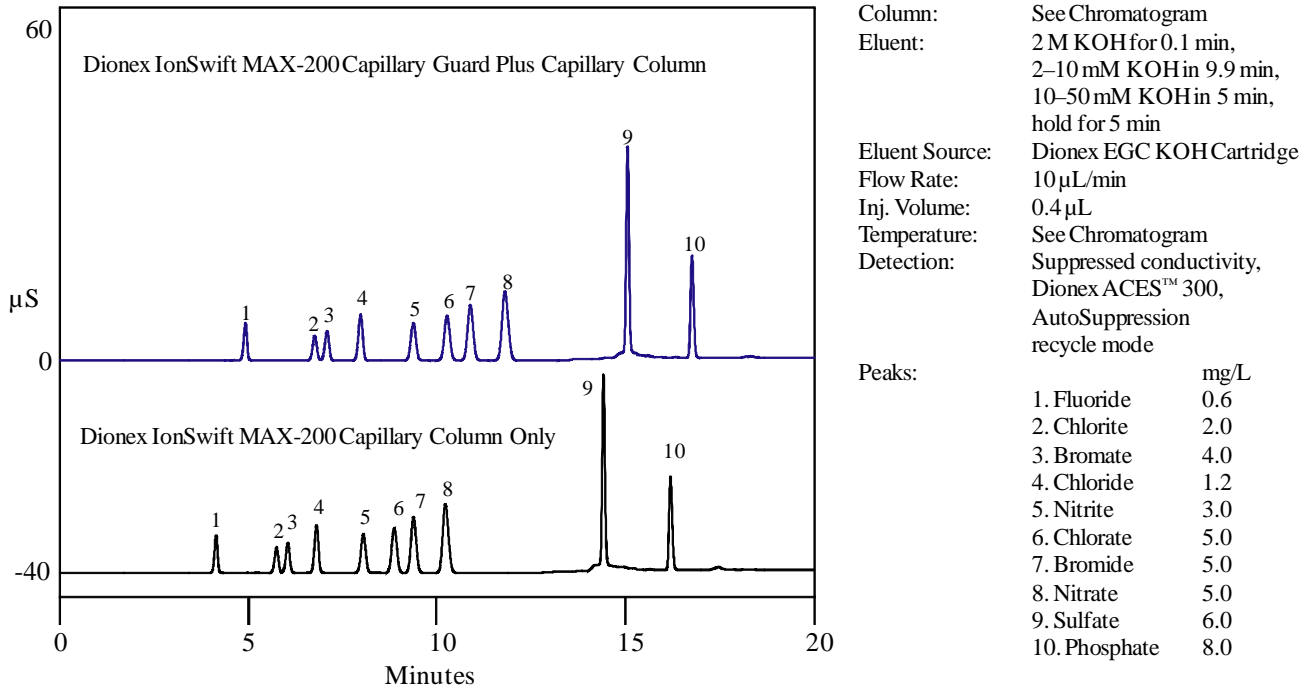
Gradient elution of common anions on the Dionex IonSwift MAX-200 Capillary Column has been optimized utilizing a hydroxide eluent. By using this eluent, common inorganic anions can be used to test the performance of the Dionex IonSwift MAX-200 Column. To guarantee that all Dionex IonSwift MAX-200 Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. An operating temperature of 30°C is used to ensure reproducible resolution and retention.



**Figure 7**  
Separation of Oxyhalides and the Inorganic Anions Using Hydroxide Gradient

### 4.3. Separation of Oxyhalides and Inorganic Anions Using the Dionex IonSwift MAX-200 Column with and without the MAX-200G Guard Column

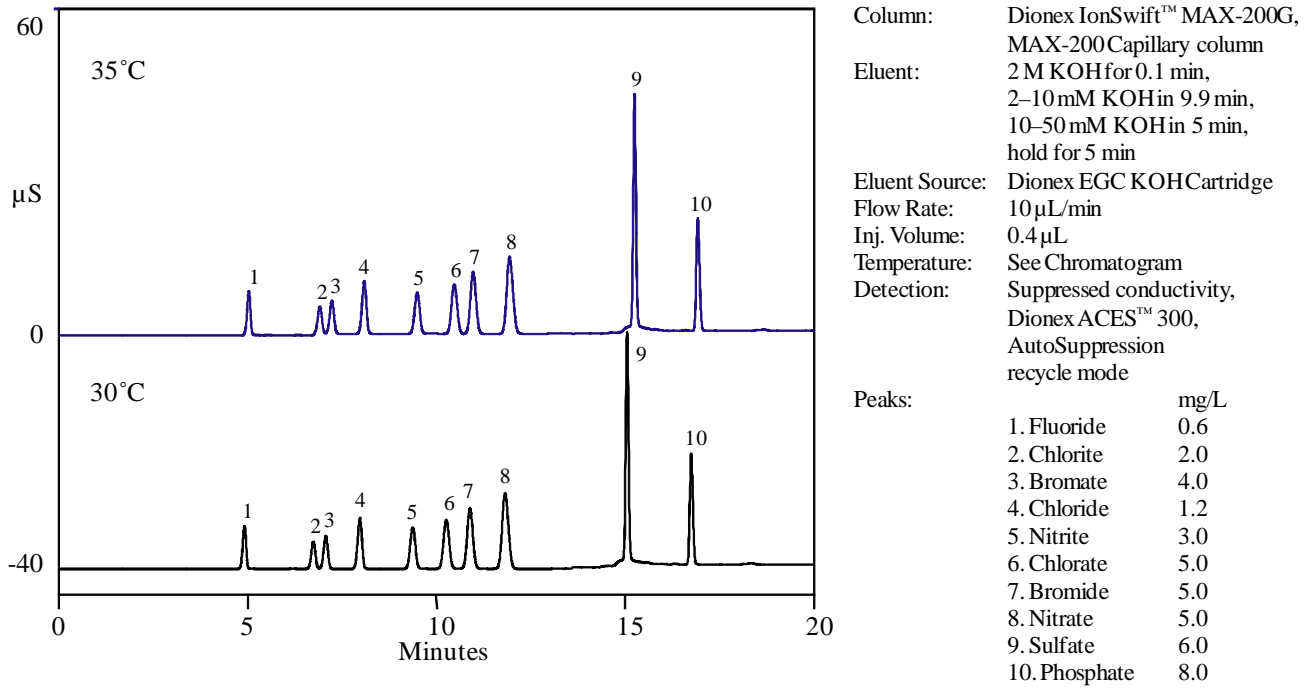
The chromatograms below compare the separation of inorganic anions and oxyhalides on the Dionex IonSwift MAX-200 column with and without the use of the Dionex IonSwift MAX-200G Guard column. Retention times will increase by approximately 20% for isocratic analysis and by approximately 4-15% for gradient analysis (~4% for a divalent anion such as sulfate and ~15% for a monovalent anion such as nitrate) when a guard column is placed in-line prior to the capillary column. A guard is placed prior to the capillary column to prevent sample contaminants from eluting onto the capillary column.



**Figure 8**  
**Comparison of the Dionex IonSwift MAX-200 with and without the Dionex IonSwift MAX-200 Guard column**

#### 4.4. Effect of Temperature on the Separation of Oxyhalides and Inorganic Anions Using the Dionex IonSwift MAX-200 Capillary Column

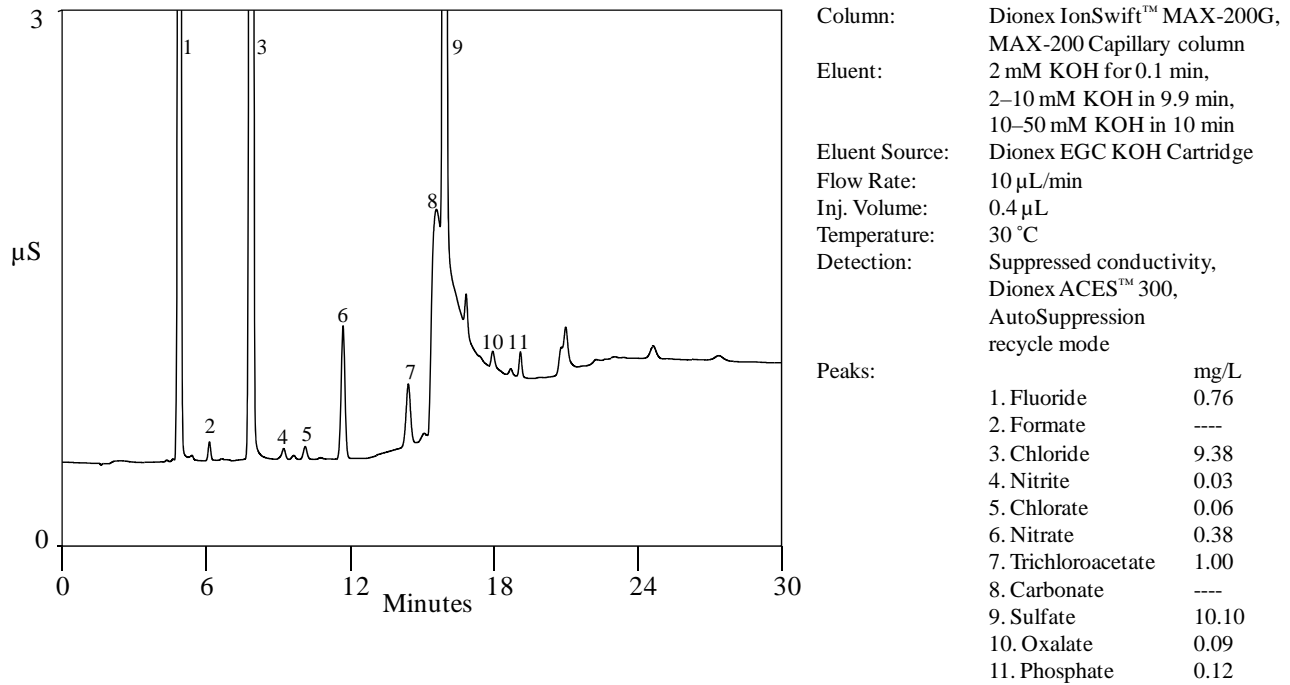
The recommended operating temperature for the Dionex IonSwift MAX-200 column is 30° C. However, if necessary, it is possible to operate this column at 35°C, but the resolution of chlorate and bromide decreases at 35°C as compared to 30°C.



**Figure 9**  
**Effect of Temperature on the Dionex IonSwift MAX-200 Capillary Column**

#### 4.5. Inorganic Anions and Organic Acids in Drinking Water Spiked with Trichloroacetate Separated Using the Dionex IonSwift MAX-200 Capillary Column

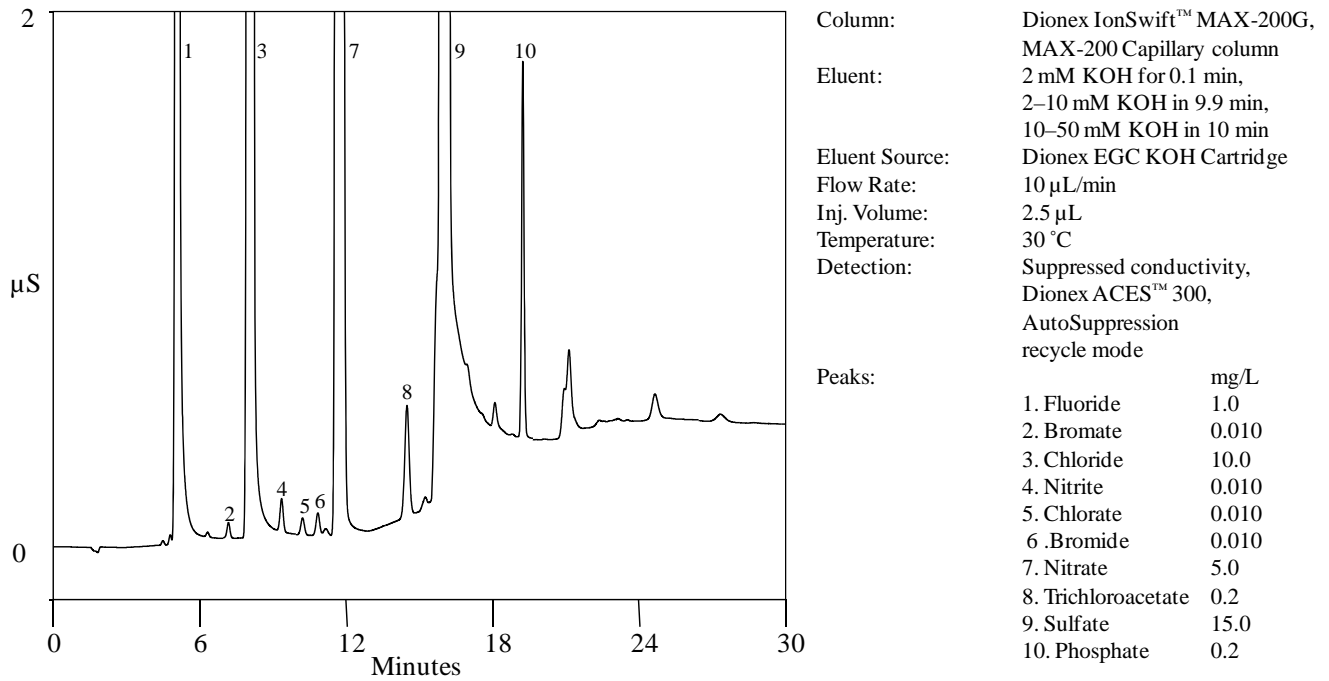
This slide shows analysis of municipal drinking water sample spiked with 1ppm of surrogate anion, trichloroacetic acid. Note that the Dionex Carbonate Removal Device (Dionex CRD) can be used to remove some interference of carbonate from the sulfate peak.



**Figure 10**  
**Analysis of Municipal Drinking Water Using the Dionex IonSwift MAX-200 Capillary Column**

### 4.6. Analysis of Simulated Drinking Water Using the Dionex IonSwift MAX-200 Capillary Column

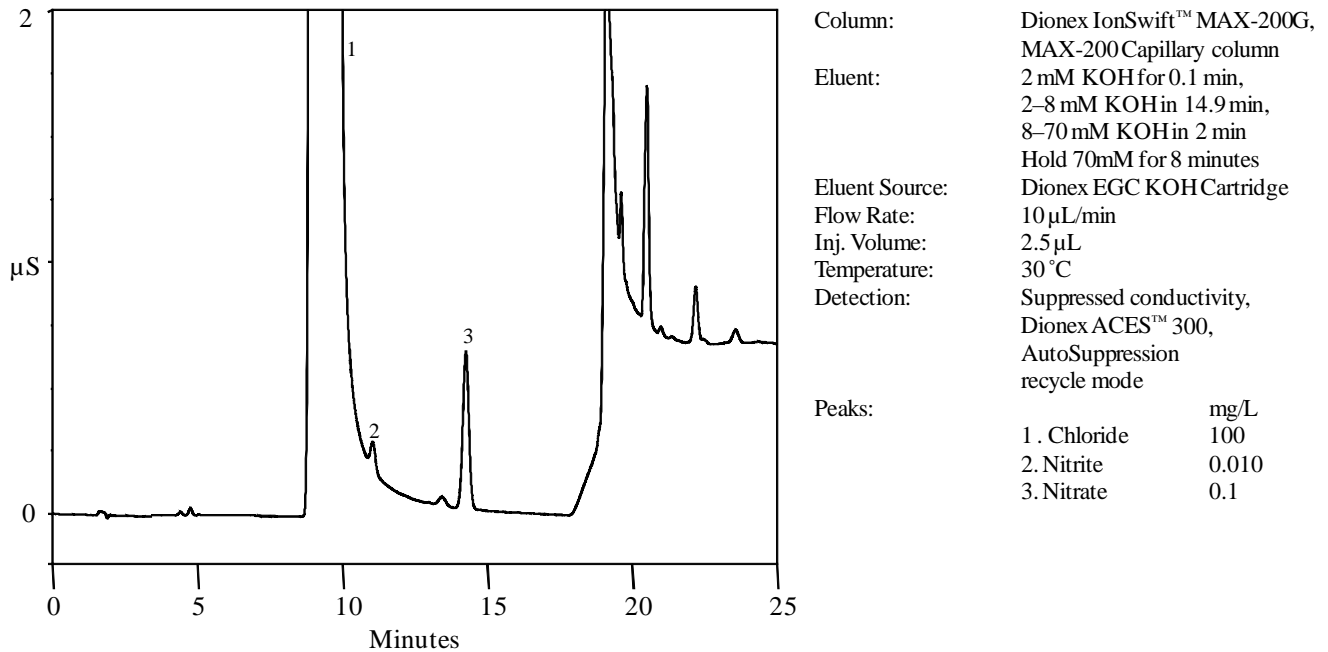
This example shows the analysis of inorganic anions and oxyhalides in simulated drinking water. A low level of bromate, 10 ppb, can be easily seen in the sample containing high levels of chloride, nitrate and sulfate using a 2.5 µL injection volume. For increased sensitivity, a larger volume can be injected. The sample was also spiked with the surrogate anion, trichloroacetate.



**Figure 11**  
**Analysis of a Simulated Drinking Water Sample Using the Dionex IonSwift MAX-200 Capillary Column**

#### 4.7. Analysis of 10,000 to 1 Ratio of Chloride to Nitrite Using the Dionex IonSwift MAX-200 Capillary Column

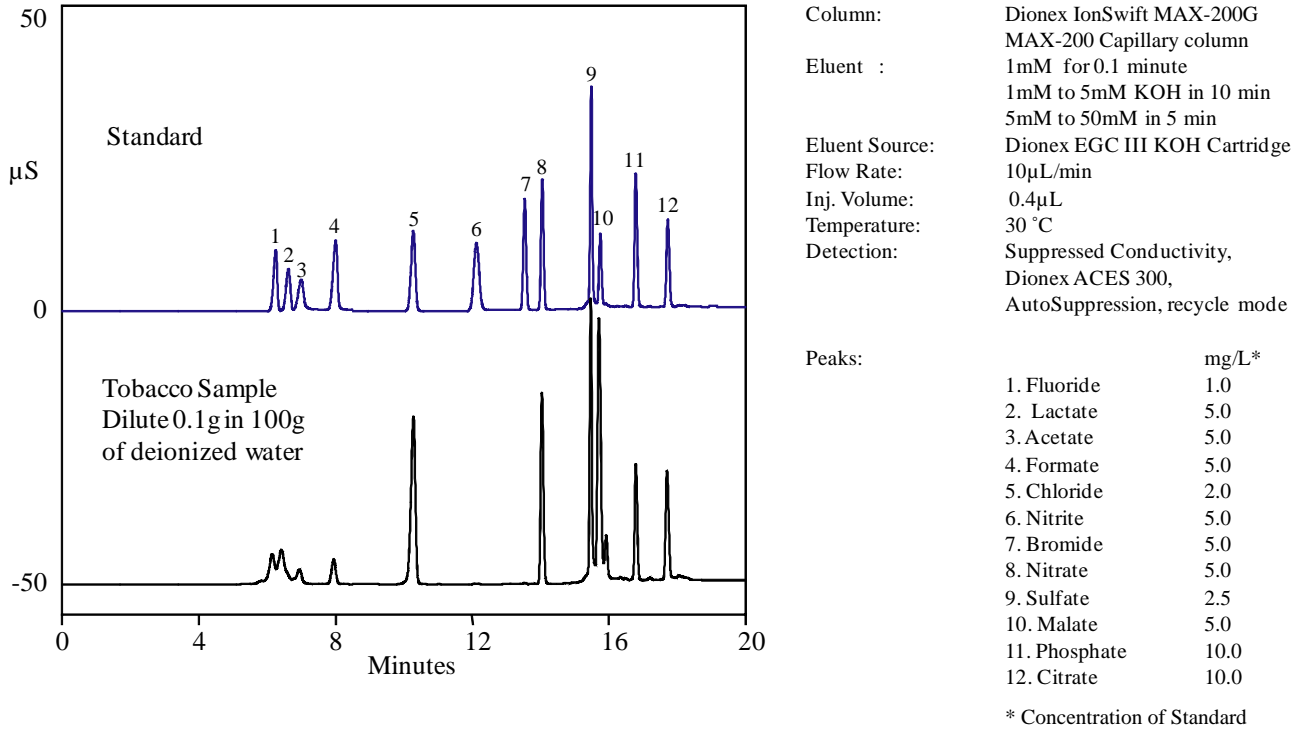
This example shows the analysis of a low level of nitrite in the presence of a high level of chloride. The nitrite, at 10 ppb, can be easily quantitated in the presence of 100 ppm of chloride.



**Figure 12**  
**Analysis of 10,000 to 1 Ratio of Chloride to Nitrite**  
**Using the Dionex IonSwift MAX-200 Capillary Column**

### 4.8. Analysis of a Tobacco Sample Using the Dionex IonSwift MAX-200 Capillary Column

This figure shows the analysis of a tobacco sample containing inorganic anions and several organic acids. The tobacco sample was diluted in deionized water prior to analysis.

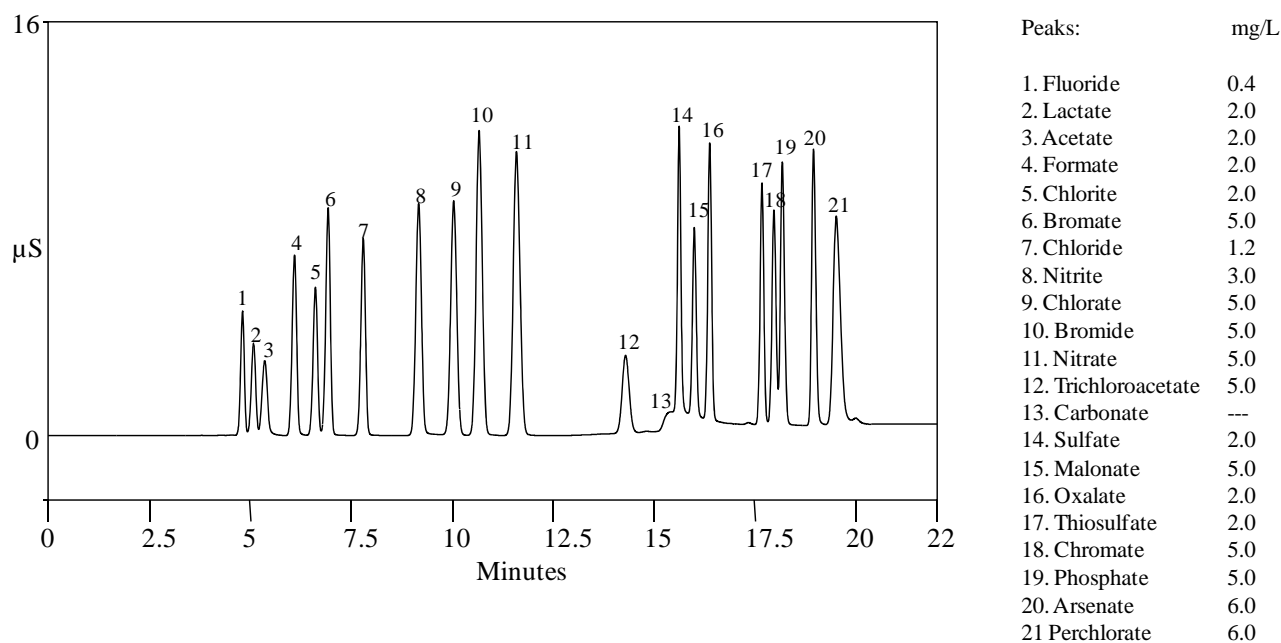


**Figure 13**  
**Analysis of a Tobacco Sample Using the Dionex IonSwift MAX-200 Capillary Column**

#### 4.9. Gradient Elution of Large Numbers of Environmental Anions Using a KOH Gradient

A large number of environmental anions can be separated on the Dionex IonSwift MAX-200 using gradient elution. The potassium hydroxide gradient is optimized in order to elute mono-, di-, and trivalent 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 anions. The hydroxide concentration in the later part of the gradient elutes polyvalent ions such as trivalent phosphate and arsenate.

Column: Dionex IonSwift™ MAX-200G,  
MAX-200 Capillary column  
Eluent: 2 mM KOH for 0.1 min,  
2–10 mM KOH in 9.9 min,  
10–60 mM KOH in 10 min  
Eluent Source: Dionex EGC KOH Cartridge  
Flow Rate: 10 µL/min  
Inj. Volume: 0.4 µL  
Temperature: 30 °C  
Detection: Suppressed conductivity,  
Dionex ACES™ 300, AutoSuppression,  
recycle mode



**Figure 14**  
Analysis of Environmental Anions Using the Dionex IonSwift MAX-200 Capillary Column



## SECTION 5 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonSwift MAX-200 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, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., contact the nearest Thermo Fisher Scientific office.

**Table 5**  
**Dionex IonSwift MAX-200 Troubleshooting Summary**

Observation	Cause	Action	Reference Section
<b>High Back Pressure</b>	Unknown	Isolate Blocked Component	5.1
	Plugged Column Other System Components	Replace Unplug, Replace	5.1 Component Manual
<b>High Background Conductivity</b>	Contaminated Dionex ATC	Clean Column	5.2.2
	Contaminated Eluents	Remake Eluents	5.2.1
	Contaminated Columns	Clean Column	5.2.2
	Contaminated Suppressor Contaminated Hardware	Clean Suppressor Clean Component	5.2.4, Component Manual 5.2.3 Component Manual
<b>Poor Resolution</b>	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	5.3.1.B, Component Manual
	Column Headspace	Replace Column	5.3.1.A
<b>Short Retention Times</b>	Flow Rate Too Fast	Recalibrate Pump	5.3.2.A
	Conc. Incorrect Eluents	Remake Eluents	5.3.2.C
	Column Contamination	Clean Column	5.3.2.B
<b>Poor Front End Resolution</b>	Conc. Incorrect Eluents	Remake Eluents	5.3.3.A
	Column Overloading	Reduce Sample Size	5.3.3.B, 2.4
	Sluggish Injection Valve	Service Valve	5.3.3.C, Component Manual
	Large System Void Volumes	Replumb System	5.3.3.D, Component Manual
<b>Spurious Peaks</b>	Sample Contaminated	Pretreat Samples	5.3.4.A,
	Sluggish Injection Valve	Service Valve	5.3.3.B, Component Manual

## 5.1. High Back Pressure

### 5.1.1. Finding the Source of High System Pressure

Total system pressure for the Dionex IonSwift MAX-200 (0.25 mm) Capillary Guard Column plus the Dionex IonSwift MAX-200 (0.25 mm) Capillary Column when using the test chromatogram conditions should be equal or less than 2500 psi. If the system pressure is higher than 2500 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a Thermo Scientific Dionex 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 clogged Dionex 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 Capillary columns are connected (see Table 6, “Typical Dionex IonPac MAX-200 Operating Back Pressures”).

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 6**  
**Typical Dionex IonPac MAX-200 Operating Back Pressures**

Column	Typical Back Pressure psi (Mpa)	Standard Flow Rate mL/min	Maximum Flow Rate
Dionex IonSwift MAX-200 Capillary Column (0.25 x 250 mm)	< 2200 [15.17]	0.010	0.020
Dionex IonSwift MAX-200G Capillary Guard Column (0.25 x 50 mm)	< 500 (3.45)	0.010	0.020
Dionex IonSwift MAX-200 Capillary Column + Dionex IonSwift MAX-200G Capillary Guard Column	<2700 [18.62]	0.010	0.020

## 5.2. 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 $\mu$ S
60 mM KOH	0.5-2 $\mu$ S
60 mM KOH/15% CH <sub>3</sub> OH	2.0 - 3.0 $\mu$ S

### 5.2.1. Preparation of Eluents

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

### 5.2.2. A Contaminated Guard, Capillary or Trap Column

Remove the Dionex IonSwift MAX-200 Capillary Guard and Dionex IonSwift MAX-200 Capillary 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 Dionex IonSwift MAX-200 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, Appendix B "Column Care"). If the Dionex CR-ATC becomes contaminated, please refer to Section 6, Clean-Up, in the Dionex CR-ATC manual (P/N 031910).

### 5.2.3. Contaminated Hardware

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

### 5.2.4. A Contaminated Dionex ACES 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 0.25 mm applications should be 0.010 mL/min.. Refer to the Dionex Anion Capillary Electrolytic Suppressor Product Manual (Document No. 065386) for assistance in determining that the eluent is within suppressible limits.
- B. **If the background is very high, (>1,000  $\mu$ S) or the baseline noise is very high, the Dionex ACES 300 may have failed to suppress the eluent.** You may need to replace the Dionex ACES 300 suppressor.
- C. If you are using eluents containing solvents, use Dionex ACES 300 in external water mode and flow rate should be 0.10 mL/min for a Dionex ACES 300.

### 5.3. Poor Peak Resolution

One of the unique features of the Dionex IonPac MAX-200 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.

#### 5.3.1. Loss of Column Efficiency

- A. **Check to see if headspace has developed in the capillary guard or 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 to make all eluent liquid line connections between the injection valve and the detector cell inlet. Check for leaks. Do not cut tubing for 0.25 mm capillary systems; always use the pre-cut tubing provided by Thermo Scientific.

#### 5.3.2. Poor Resolution Due to Shortened Retention Times

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

- A. **Check the flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. **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, Appendix B "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 Appendix B "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 Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., contact the nearest Thermo Fisher Scientific office.

### 5.3.3. Loss of Front End Resolution

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

- A. **Improper eluent concentration may be the problem.** 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 capillary columns may be the problem.** Swap components, one at a time, in the system prior to the capillary column and test for front-end resolution after every system change.

### 5.3.4. Spurious Peaks

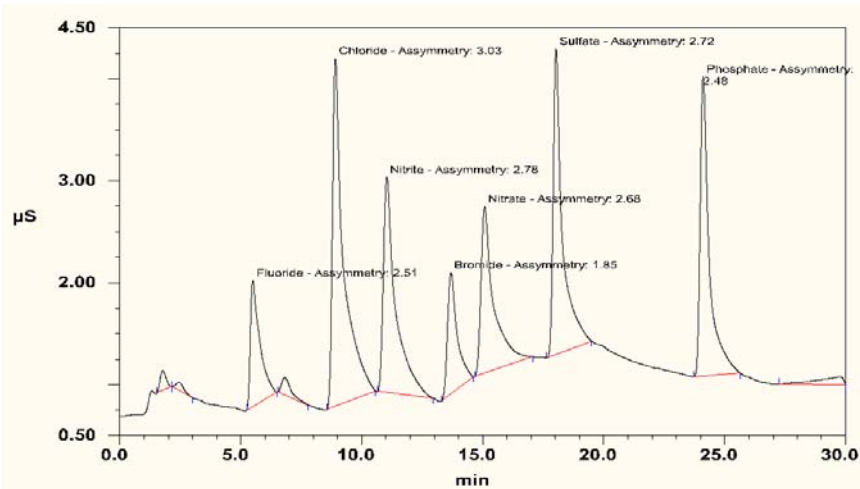
- A. **The columns may be contaminated.** If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, the retention times for the analytes will then decrease and be spurious, inefficient (broad) peaks that can show up at unexpected times. Clean the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

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the Dionex IonSwift MAX-200 columns, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., contact the nearest Thermo Fisher Scientific office.

- 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.

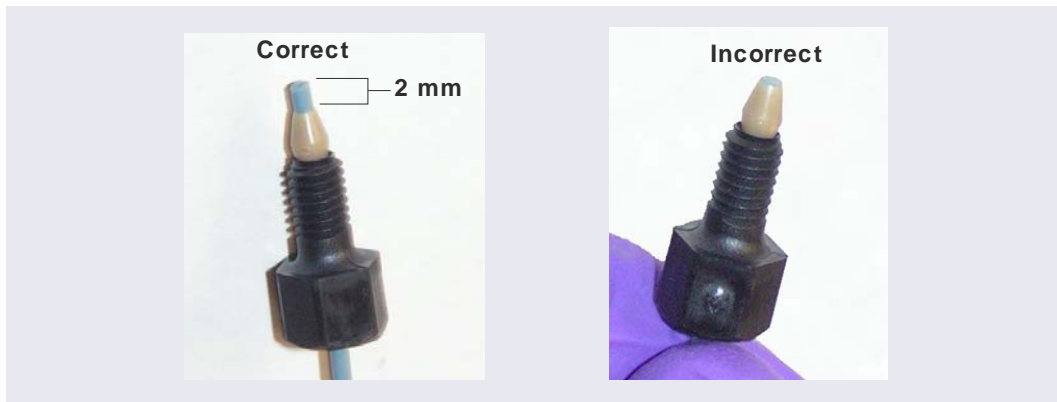
### 5.3.5. Poor Efficiency Using Capillary Columns

Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks (see Figure 15).



**Figure 15**  
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 16 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.



**Figure 16**  
Correct and Incorrect Ferrule and  
Fitting Bolt Placement for Capillary Tubing Connections

### 5.3.6. 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.

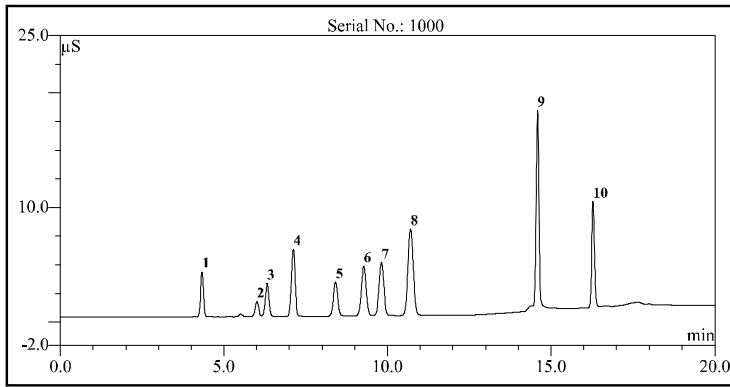


# APPENDIX A – QUALITY ASSURANCE REPORT

**IonSwift™ MAX-200**  
**0.25 x 250 mm**  
**Product No. 075889**

**Date:** 19-Aug-11 13:59  
**Serial No. :** 001000  
**Lot No. :** 133-11-001

**Eluent Source:** EGC-KOH (Capillary)  
**Eluent Gradient:** 2mM KOH for 0.1 minute,  
 2mM to 10mM KOH in 9.9 minutes,  
 10mM to 50mM KOH in 5 minutes,  
 Hold for 5 minutes 50mM KOH  
**Flow Rate:** 10 µL/min  
**Temperature:** 30 °C  
**Detection:** Suppressed Conductivity  
**Suppressor:** Anion Capillary Electrolytic Suppressor (ACES 300)  
**Applied Current:** 10 mA  
**Injection Volume:** 0.4 µL (Internal Loop)  
**Storage Solution:** 100mM Sodium Borate



No.	Peak Name	Ret.Time (min)	Asymmetry (ALA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	4.33	0.9	9.24	11057	0.30
2	Chlorite	6.01	0.9	1.55	14577	0.30
3	Bromate	6.32	0.9	3.87	15172	0.30
4	Chloride	7.12	0.9	5.70	18531	0.60
5	Nitrite	8.41	1.0	3.35	18912	1.50
6	Chlorate	9.28	1.0	1.99	18546	2.50
7	Bromide	9.82	1.0	3.10	20899	2.50
8	Nitrate	10.71	1.1	17.01	19747	2.50
9	Sulfate	14.59	1.0	11.01	145595	3.00
10	Phosphate	16.29	1.1	n.a.	174254	4.00

**QA Results:**

Component	Parameter	Specification	Results
	Pressure	<=2420	1982
Sulfate	Asymmetry	0.9-1.8	Passed
Sulfate	Efficiency	>=72000	Passed
Sulfate	Ret. Time	13.9-16.1	Passed
T1/T2	Ret.Time Ratio	1.76-2.75	Passed

T1 = Ret.Time of Sulfate minus Nitrate  
 T2 = Ret.Time of Phosphate minus Sulfate

*Production Reference:*

Datasource: MonolC  
 Directory: IS\_MAX-200\MAX-200\_0,25mm\_ID  
 Sequence: 075889\_IS\_MAX-200\_0,25X250\_GRAD\_VALIDATION\_2011-001-133  
 Sample No.: 1



## 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 Dionex IonSwift MAX-200 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 Dionex IonSwift™ MAX-200 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 monolith bonding to PEEK walls and irreversibly damage the performance of the column. High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent which may create a neutralization pressure band. The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to  $\leq 5\%$  levels and the ionic strength of the eluent to  $\leq 50$  mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

#### 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 Dionex IonPac MAX-200 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 4, HPLC Solvents for Use with Dionex IonSwift MAX-200 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 Dionex IonSwift MAX-200.**

#### 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 Dionex ACES 300** from the Dionex IonSwift MAX-200 Capillary Column. If your system is configured with both a guard column and a capillary column, reverse the order of the guard and capillary 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 capillary column and a guard capillary column in series, ensure that the guard capillary column is placed after the capillary column in the eluent flow path. Contaminants that have accumulated on the guard capillary column can be eluted onto the 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 a Dionex IonSwift MAX-200 0.25 mm Capillary or Capillary Guard.**

**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 at least 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 Dionex ACES 300** to the Dionex IonSwift MAX-200 Capillary Column and **place the guard capillary column in line** between the injection valve and the capillary column if your system was originally configured with a guard capillary column.

## APPENDIX C – COMPARISON OF CHROMATOGRAPHY SYSTEMS

The proper system configuration is important for the Dionex IonSwift MAX-200 capillary column. A capillary system such as Thermo Fisher Scientific Dionex ICS-5000 must be used for the 0.25mm Dionex IonSwift MAX-200 column.

**Table 1C**  
Comparison Table

<b>CONFIGURATION</b>	<b>0.25 mm</b>
<b>Eluent Flow Rate</b>	0.010 µL/min
<b>SRS</b>	Dionex ACES 300 (P/N 072052)
<i>NOTE: Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven.</i>	
<b>Injection Loop</b>	0.4 µL (typical; internal)
<b>System Void Volume</b>	Use only in an IC system equipped for capillary analysis, such as the Dionex ICS-5000 capillary system.
<b>Pumps</b>	Use only a pump designed for capillary flow rates such as the Dionex ICS-5000 capillary pump.
<i>NOTE: Use of a Dionex EGC-KOH cartridge (P/N 072076 in conjunction with a Dionex CR-ATC P/N 072078) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.</i>	
<b>Chromatographic Module</b>	A thermally controlled column compartment such as the Dionex ICS-5000 DC or Dionex IC-Cube.
<b>Detectors</b>	Use only a conductivity detector designed for capillary flow rates such as the Dionex ICS-5000 Capillary CD.

**Table 2C**  
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