Thermo Scientific

Dionex IonPac CS19-4µm Column

Product Manual

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Thermo SCIENTIFIC

Product Manual

for

Dionex IonPac CS19-4µm Analytical Column
2 × 250 mm, P/N 078836
4 × 250 mm, P/N 078837

Dionex IonPac CS19-4µm Capillary Column
0.4 × 250 mm, P/N 078835

Dionex IonPac CG19-4µm Guard Column
2 × 50 mm, P/N 078839
4 × 50 mm, P/N 078840

Dionex IonPac CG19-4µm Capillary Guard Column
0.4 × 50 mm, P/N 078838

For Research Use Only. Not for use in diagnostic procedures.

Revision History:

Revision 01, February, 2014, Original Publication.

Revision 02, July, 2014, Updated Appendix D – Column Quick Start Procedure.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

Contents

1. Introduction

The Thermo Scientific™ Dionex™ IonPac™ CS19-4µm column is used with suppressed conductivity detection for the analyses of the common inorganic cations (Lithium, Sodium, Ammonium, Potassium, Magnesium, and Calcium) as well as small polar amines. Its selectivity is particularly useful in the analysis of small, hydrophilic amines such as ethanolamines, methylamines, ethylamines and the biogenic amines.

The Dionex IonPac CS19-4µm stationary phase has a higher cation exchange capacity than the Thermo Scientific Dionex IonPac CS17 and the Thermo Scientific Dionex IonPac CS18 columns. Its cation exchange selectivity is similar to that of the Dionex IonPac CS18 column but has superior peak efficiency and higher capacity. The Dionex IonPac CS19 column and the Dionex IonPac CS19-4µm column chemistries are identical, providing the same selectivity. The only difference is the substrate particle size. The Dionex IonPac CS19-4µm column, having a smaller particle size, gives about 30% higher peak efficiencies. This is reflected in the column specifications for the two products. It should be noted that there could be an overlap of performance between the two products: a highly efficient Dionex IonPac CS19 column that happens to have much better peak efficiencies than its minimum specification, may perform as well as or better than a Dionex IonPac CS19-4µm column that is at the specification limits.

The Dionex IonPac CS19-4µm columns use a supermacroporous polymeric substrate functionalized with carboxylic acid groups. It is compatible with up to 10% organic solvents (such as acetonitrile and acetone). Isopropyl alcohol (IPA) should be avoided as an eluent component because it will cause very high backpressure in the Dionex IonPac CS19-4µm column. IPA, however, can be used to clean the column at very low flow rates or can be present in the sample matrix. The Dionex IonPac CS19-4µm column can be used without loss of performance up to 30 °C.

The Dionex IonPac CS19-4µm column can be washed with up to 1 M acid concentration. The Dionex IonPac CS19-4µm column should not be used with basic eluents. The column backpressure increases too much, disrupting the packing. The Dionex IonPac CS19-4µm column can withstand up to 1.5 times its standard flow rate without loss of performance. However, this does not imply that a particular application can be run at this maximum flow rate, because the entire system pressure must be accounted for and must be under 5000 psi.

The Dionex IonPac CS19-4µm capillary column (0.4 × 250 mm) is packed with the same material as the equivalent standard bore version (producing the same performance as a 4 mm column) but requires less eluent consumption, thus reducing operating costs. Furthermore, the capillary column has significantly lower column backpressure than the larger i.d. analytical formats, thus it is possible to operate it at 1.5 times its standard flow rate (i.e. can be operated at 15 µL/minute), provided the total system pressure is below 5000 psi. At this high flow rate for the capillary format, the eluent generator can still provide a maximum methanesulfonic acid (MSA) concentration of 130 mM MSA, allowing fast analysis of polyvalent cations. Another advantage of the capillary format is its superior mass sensitivity of 100 times more than the 4 mm i.d. column format for the same injection volume. Thus, for fast separations of polyvalent amines, where fast flow rates and high MSA eluent concentrations are required, the capillary Dionex IonPac CS19-4µm column is recommended over the 2 and 4 mm column formats.

The Dionex IonPac CG19-4µm guard column is made with microporous polymeric resin. It has the same functionality as the separator resin, but is of much lower capacity and therefore cannot be used to concentrate samples prior to analysis. All of the Dionex IonPac CG19-4µm guard columns are packed with the same resin.

The Thermo Scientific Dionex ICS-5000^{+} Reagent-Free™ HPIC™ system and the Thermo Scientific Dionex ICS-4000 Capillary HPIC system have a maximum total pressure rating of 5000 psi. This includes the columns and all the different components in the system. When this pressure is reached, the system will shut down to avoid damaging the components. When creating a method, it is recommended that total system pressure is below 4500 psi, that way if pressure builds up it will not shut down the system.

Even though the maximum pressure specification is the same for the 3 formats (4, 2 and 0.4 mm), due to its hydrodynamic properties the 0.4 × 250 mm capillary separator actually has the lowest backpressure of the three, and the 4 × 250 mm separator the highest. Pressure limits should be taken into consideration when developing methods that are intended to be applied to all formats.

When developing a method, it should be taken into consideration that if column temperatures below 30˚C are used, the total system pressure will be higher and therefore the ability of using higher flow rates will decrease.

IMPORTANT

Special attention should be paid to the column specifications during the method development process. Customers should expect to receive columns that are at specification limits from time to time. As a good practice, it would be wise either to use such columns for the method development or, at least, model the separation behavior of a given application based on the column specification parameters.

Read the system manuals. This manual assumes that you are familiar with the installation and operation of the Thermo Scientific Dionex Ion Chromatograph (IC). If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis. All instrument manuals are available on the Reference Library CD-ROM supplied with this column.

Table 1. Dionex IonPac CS19-4µm/CG19-4µm Packing Specifications

Column	Nominal Particle Diameter µm	Substrate	Column Capacity µeq/column	Functional Group	Hydrophobicity
Dionex IonPac CS19-4µm Capillary Column 0.4 × 250 mm	4	SMP	24	Carboxylic acid	Medium
Dionex IonPac CG19-4µm Capillary Guard Column 0.4 × 50 mm	8	Microporous	0.5	Carboxylic acid	Medium
Dionex IonPac CS19-4µm Analytical Column 2 × 250 mm	4	SMP	600	Carboxylic acid	Medium
Dionex IonPac CG19-4µm Guard Column 2 × 50 mm	8	Microporous	11	Carboxylic acid	Medium
Dionex IonPac CS19-4µm Analytical Column 4 × 250 mm	4	SMP	2410	Carboxylic acid	Medium
Dionex IonPac CG19-4µm Guard Column 4 × 50 mm	8	Microporous	46	Carboxylic acid	Medium

NOTE: The term SMP stands for Supermacroporous resin.

Table 2. Dionex IonPac CS19-4µm/CG19-4µm Operating Parameters

Column	Typical Back Pressure at Standard Flow Rate psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate* mL/min
Dionex IonPac CS19-4µm 0.4 mm Capillary Column	≤ 2,200 (15.17)	0.010	0.015
Dionex IonPac CG19-4µm 0.4 mm Capillary Guard Column	≤ 200 (1.38)	0.010	0.015
Dionex IonPac CS19-4µm + CG19-4µm 0.4 mm Capillary and Capillary Guard Columns	≤ 2,400 (16.55)	0.010	0.015
Dionex IonPac CS19-4µm 2 mm Analytical Column	≤ 2,500 (17.23)	0.25	0.38
Dionex IonPac CG19-4µm 2 mm Guard Column	≤ 200 (1.38)	0.25	0.38
Dionex IonPac CS19-4µm + CG19-4µm 2 mm Analytical and Guard Columns	≤ 2,700 (18.61)	0.25	0.38
Dionex IonPac CS19-4µm 4 mm Analytical Column	≤ 3,200 (22.06)	1.0	1.5
Dionex IonPac CG19-4µm 4 mm Guard Column	≤ 200 (1.38)	1.0	1.5
Dionex IonPac CS19-4µm + CG19-4µm 4 mm Analytical and Guard Columns	≤ 3,400 (23.44)	1.0	1.5

NOTE: Maximum Flow Rate (mL/min) defined in this context is the maximum flow rate that the column can withstand without loss of performance. This does not necessarily mean that methods can be developed at these column flow rates, as the method flow rate depends on total system pressure being below the maximum system pressure of 5000 psi.

NOTE

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.

2. Ion Chromatography Systems

The proper configuration of an Ion Chromatography System (ICS) in 2 mm or 4 mm format is based on the ratio of the 2 mm to 4 mm column cross-sectional area (a factor of 1/4). The selected format will affect the type of pump recommended. A gradient pump is designed to blend and pump isocratic, linear, or gradient mixtures of up to four mobile phase components at precisely controlled flow rates. An isocratic pump is for applications not requiring gradient and multi-eluent proportioning capabilities. Both are offered in either standard bore or microbore options. The Dionex IonPac CS19-4µm columns consist of 4 µm polymeric particle substrate, which translates into higher peak efficiencies than the Dionex IonPac CS19 columns, but also generates higher column backpressures. Therefore, an IC system capable of running up to 5000 psi, such as the Dionex ICS-5000$^+$ HPIC system is required to operate the analytical columns at their standard flow rates. The Dionex IonPac CS19-4µm capillary column can be operated under 3000 psi at standard flow rates. To take advantage of the faster run times, using 15µL/min, the Dionex ICS-5000$^+$ HPIC system or the Dionex ICS-4000 Capillary HPIC system, capable of operating up to 5000 psi, is required.

- For an ICS in 2 mm format, a high pressure microbore isocratic pump, high pressure standard bore isocratic pump, high pressure microbore gradient pump, or high pressure standard bore gradient pump is recommended.
- For an ICS in 4 mm format, a high pressure standard bore isocratic pump or high pressure standard bore gradient pump is recommended.
- For an ICS in 0.4 mm format, a high pressure capillary IC system such as the Dionex ICS-5000$^+$ HPIC system or the Dionex ICS-4000 HPIC system is recommended.

See Appendix C, "Configuration" for specific recommended settings and parts including pumps, eluent flow rate, Thermo Scientific Dionex Electrolytically Regenerated Suppressor (Dionex ERS), Thermo Scientific Dionex MicroMembrane Suppressor (Dionex MMS), Thermo Scientific Dionex Capillary Electrolytic Suppressor (Dionex CES), injection loop, system void volume, detectors, and tubing back pressure.

3. Installation

3.1 Column Start-Up

The column is shipped using 8 mM Methanesulfonic acid as the storage solution. Prepare the eluent shown on the Quick Start procedure (Document No. 065591, see Appendix D) and follow the Quick Start instructions to hydrate the columns prior to running QAR. After the column gone though Quick Start hydration steps, connect the column to the suppressor and test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

If peak efficiencies or resolution are poorer than the QAR, see Section 6, Troubleshooting for information regarding possible causes and solutions.

IMPORTANT

When making any tubing connections (column installation, replacing tubing etc), it is recommended to make these connections with the pump turned off. This will avoid any slippage of the ferrule under high pressure conditions. For capillary connections, it is recommended to inject water into the cavities of the fluidic system using a syringe or a micropipette with the flow off before joining two components together. This will prevent air from entering the system and result in a faster equilibration.

3.2 Column Storage

For storage of the column, use 8 mM Methanesulfonic acid for the column storage solution. Flush the column at its standard flow rate for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

3.2.1 System Requirements for 0.4 mm Operation

The Dionex IonPac CS19-4µm Capillary Guard and Capillary Columns are designed to be run on a capillary ion chromatograph equipped with suppressed conductivity detection. It is recommended to run the capillary column only on the Dionex ICS-5000$^+$ HPIC system or the Dionex ICS-4000 Capillary HPIC system for best performance. Use only precut 0.4 mm tubing with the capillary systems.

3.2.2 System Requirements for 2 mm and 4 mm Operation

The Dionex IonPac CS19-4µm Guard and Analytical Columns are designed to be run on an ion chromatograph equipped with suppressed conductivity detection with the capability of continuously running at 5000 psi or higher. For best performance, it is recommended to run the analytical column on a system rated 5000 psi or higher such as Dionex ICS-5000$^+$ HPIC system.

3.2.3 System Void Volume

When using 2 mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4 mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" i.d. PEEK tubing, (P/N 044221), for a 2 mm system. For a 4 mm system, 0.010" i.d. PEEK tubing (P/N 042260) is recommended; 0.012" Tefzel tubing may be used, but peak efficiency will be compromised, which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and the tubing must be cut with a straight edge, NOT slanted. Remove all unnecessary switching valves and couplers. Make sure that a 2 mm Gradient Mixer is used (and not a 4 mm Gradient Mixer) when using 2 mm columns. Any void volumes and eddies will result in analyte dispersion, which produces poor peak efficiencies.

For the 0.4 mm capillary columns, only precut capillary tubing should be used with the Dionex ICS-5000$^+$ HPIC system or the Dionex ICS-4000 HPIC Capillary systems. The Dionex ICS-5000$^+$ HPIC system and Dionex ICS-4000 HPIC Capillary systems have preconfigured tubing to minimize the system void volume. The tubing should only be replaced with precut tubing of the same type. It should also be noted that due to system configuration differences, the system void time in the capillary system will typically be longer than that observed with the analytical system at the same linear velocity. Slight modification of retention time in the method may be required to ensure correct peak identification.

3.3 Installing the Dionex CR-CTC Trap Column for Use with Eluent Generation

For Dionex IonPac CS19-4µm 2 mm and 4 mm column applications using eluent generation, a Thermo Scientific Dionex CR-CTC 500 Continuously Regenerated Cation Trap Column (P/N 066262) may be installed at the EGC eluent outlet to remove trace level cationic contaminants such as ammonium from the carrier deionized water. For capillary applications, use the Dionex CR-CTC II Continuously Regenerated Cation Trap Column (Capillary), P/N 072079. See the Dionex CR-TC Product Manual (Document No. 031910) for instructions. The capillary system should only be used with an eluent generator and a Dionex CR-CTC II trap column.

IMPORTANT ***The Dionex IonPac CTC-1 Cation Trap Column cannot be used as it has a maximum operating pressure of 3000 psi.***

3.4 The Injection Loop

3.4.1 The 0.4 mm System **Injection Loop, 0.4 µL Internal Loop**

For most applications on a 0.4 mm capillary system, a 0.4 µL injection loop is sufficient. Generally, you should not inject more than 0.5 nanomoles total cation concentration onto the 0.4 mm capillary column. Injecting a 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. The standard 4 port injection valve must be changed to the 6 port injection valve (P/N 061947), to accommodate the external loop.

3.4.2 The 2 mm System Injection Loop, 2 - 15 µL

For most applications on a 2 mm analytical system, a 2 - 15 µL injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2 mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. The Dionex IonPac CS19-4µm 2 mm column requires a microbore system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4 mm analytical.

3.4.3 The 4 mm System Injection Loop, 10 - 50 µL

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

3.5 Sample Concentration

Trace cation concentrators are used primarily in high purity water analysis. The function of the trace cation concentrator in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This can be accomplished by replacing the sample loop with the concentrator column, then pumping (and concentrating) large volumes of the sample onto a concentrator column. The sample should be pumped into the concentrator column in the OPPOSITE direction of the eluent flow, otherwise the chromatography will be compromised. This process "concentrates" all cationic analyte species onto the Thermo Scientific Dionex Trace Cation Concentrator column (Dionex TCC-LP1, Dionex TCC-ULP1, Dionex TCC-XLP1) leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the Dionex Trace Cation Concentrator column for the analytical chemist in these applications is the capability of performing routine trace analyses of sample matrix ions at ng/L levels without extensive and laborious sample pretreatment.

Another advantage of the Dionex TCC-LP1, Dionex TCC-ULP1, and Dionex TCC-XLP1 concentrator columns is that because of their low backpressure, samples can be preconcentrated using a hand-held syringe.

The Dionex Trace Cation Concentrator should be used for sample concentration with the Dionex IonPac CS19-4µm 4 mm or the Dionex IonPac CS19-4µm 2 mm Analytical Columns. For trace cation concentration with the Dionex IonPac CS19-4µm 0.4 mm Column, use the Dionex IonSwift MCC-100 Concentrator Column (0.5 × 80 mm, P/N 075462).

IMPORTANT

The Dionex IonPac CG19-4µm Guard/Capillary Guard Column should not be used as a concentrator column, as it has very low cation exchange capacity.

For more detailed information on sample concentration techniques for high sensitivity work and a detailed discussion of cation concentration techniques refer to:

- Section 3, "Operation," of the Thermo Scientific Dionex Trace Cation Concentrator Low Pressure (Dionex TCC-LP1), Dionex Ultra Low Pressure (Dionex TCC-ULP1) and Dionex Extremely Low Pressure (Dionex TCC-XLP1) Column Product Manual (Document No. 034973),
- Section 3, "Operation" of the Thermo Scientific Dionex Monolith Cation Concentrator Column (Dionex IonSwift MCC-100 / Dionex IonSwift MCC-200) Column Manual (Document No. 065411).

WARNING

The Dionex Trace Cation Concentrator (Dionex TCC-2, P/N 043103) should not be used for sample concentration with the Dionex IonPac CS19-4µm column. The Dionex TCC-2 column packing is a strong cation exchange resin functionalized with sulfonic acid. The recommended Dionex IonPac CS19-4µm column eluents will not properly elute ions concentrated on this column.

3.6 Dionex IonPac CG19-4µm Guard/Capillary Guard Column

A Dionex IonPac CG19-4µm Guard/Capillary Guard Column is normally used with the Dionex IonPac CS19-4µm Analytical/Capillary Column. The Dionex IonPac CG19-4µm guard column has a microporous polymeric substrate and is a very low capacity cation exchange column, adding only about 0.5 minutes to the elution time. It should not be used as a concentrator column. A guard column is placed prior to the analytical/capillary column to prevent sample contaminants from eluting onto the analytical/capillary column. Cleaning or replacing a guard column is more economical than replacing an analytical/capillary column. For maximum life of the analytical/capillary column, the guard column should be changed or replaced as part of a regular maintenance schedule or at the first sign of performance deterioration. Use the test chromatogram that is shipped with the analytical/capillary column or the initial application run as a performance benchmark.

3.7 Eluent Storage

Dionex IonPac CS19-4µm columns are designed to be used with acid eluent systems. Storage of the eluent under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents). Eluent storage bottles made of glass should be avoided as sodium contamination will occur.

3.8 Dionex Cation Electrolytically Regenerated Suppressor and Dionex Cation Capillary Electrolytic Suppressor Requirements

A Dionex Cation Electrolytically Regenerated Suppressor (Dionex CERS 500, 2 mm or 4 mm respectively) should be used for 2 mm and 4 mm applications that require suppressed conductivity detection. A Dionex Cation Capillary Electrolytic Suppressor (Dionex CCES 300) should be used for the 0.4 mm capillary applications that require suppressed conductivity. They are 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 CERS 500 and Dionex CCES 300 modes of operation.

Depending on the % level of organic solvent present in the eluent, solvent containing eluents must be used in the External Water Mode using the Dionex CERS 500 or Chemical Suppression Mode using the Dionex Cation MicroMembrane Suppressor (Dionex CMMS 300).

For detailed information on the operation of the Dionex Cation Electrolytically Regenerated Suppressor, see Document No. 031956, “Product Manual for the Thermo Scientific Dionex ERS 500 Suppressor”. For detailed information on the operation of the Dionex Cation Capillary Electrolytic Suppressor, see Document No. 065386, the “Product Manual for the Thermo Scientific Dionex CES 300 Suppressor”. For detailed operation of the Dionex MicroMembrane Suppressor, see Document No. 031727, “Product Manual for the Thermo Scientific Dionex MicroMembrane Suppressor 300”.

For Dionex IonPac CS19-4µm 0.4 mm Capillary Column, use the Dionex CCES 300 (0.4 mm, P/N 072053).
For Dionex IonPac CS19-4µm 4 mm Analytical Column, use the Dionex CERS 500 (4 mm, P/N 082542).
For Dionex IonPac CS19-4µm 2 mm Analytical Column, use the Dionex CERS 500 (2 mm, P/N 082543).

3.9 Installation of the Capillary Column

1. 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).
2. 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. Separator and Guard Columns Installed in Column Cartridge

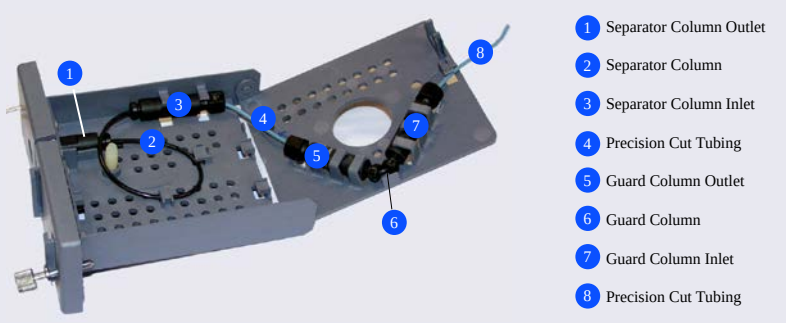
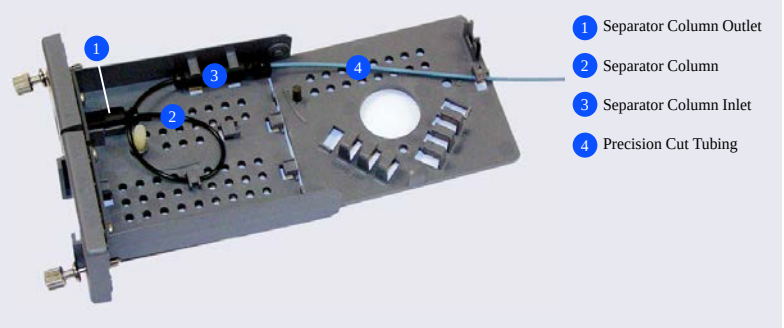


Figure 2. Separator Column Only Installed in Column Cartridge



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

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

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

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

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

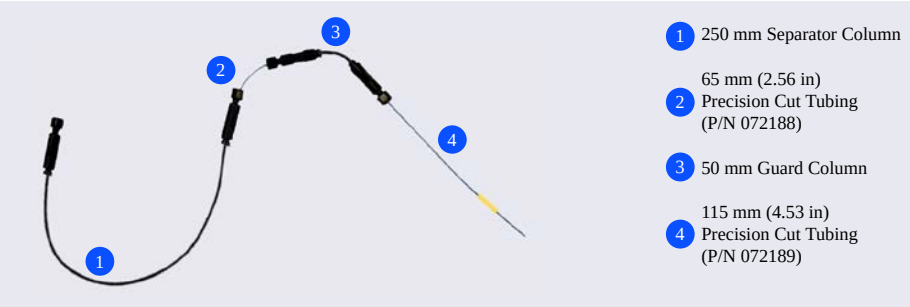
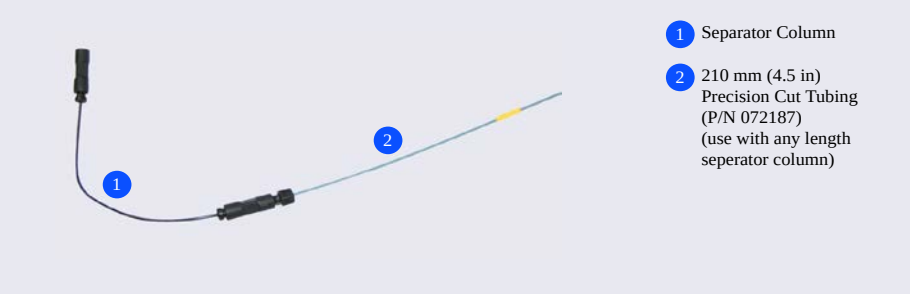
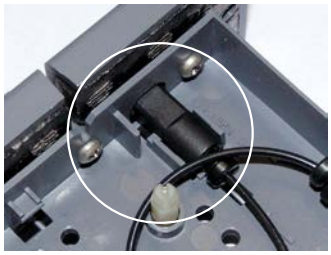


Figure 4. Tubing Connections for Separator Column Only



5. Lift up the lid of the column cartridge to open it.
6. 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



7. Coil the separator 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.
8. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
9. Route the guard column inlet tubing (if used) or the separator column inlet tubing through the clip on the top edge of the column cartridge lid.
10. 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

4. Operation

4.1 General Operating Conditions

Column:	0.4 mm:	Dionex IonPac CS19-4µm 0.4 mm Capillary Column + Dionex IonPac CG19-4µm 0.4 mm Capillary Guard Column
	2 mm:	Dionex IonPac CS19-4µm 2 mm Analytical Column + Dionex IonPac CG19-4µm 2 mm Guard Column
	4 mm:	Dionex IonPac CS19-4µm 4 mm Analytical Column + Dionex IonPac CG19-4µm 4 mm Guard Column
Sample Volume:	0.4 mm:	0.4 µL Loop
	2 mm:	2.5 µL Loop + 0.8 µL Injection valve dead volume
	4 mm:	10 µL Loop + 0.8 µL Injection valve dead volume
Eluent:	8 mM Methanesulfonic acid (MSA)	
Eluent Flow Rate:	0.4 mm:	0.010 mL/min
	2 mm:	0.25 mL/min
	4 mm:	1.0 mL/min
Temperature:	30 °C	
Dionex Electrolytic Suppressors:	Dionex Cation Electrolytically Regenerated Suppressor, Dionex CERS 500 (2 mm or 4 mm) Dionex Cation Capillary Electrolytic Suppressor, Dionex CCES 300 (0.4 mm) AutoSuppression Recycle Mode	
or Dionex Chemical Suppressor:	Dionex Cation MicroMembrane Suppressor, Dionex CMMS 300 (for 2 mm or 4 mm only)	
Dionex CMMS Regenerant:	TBAOH	
Dionex CMMS Mode:	Dionex Displacement Chemical Regeneration (Dionex DCR)	
Expected Background Conductivity:	< 0.3 µS in the suppressed mode	
Storage Solution:	Eluent	

4.2 Dionex IonPac CS19-4µm Column Operation Precautions

CAUTION

- ***Operate below 5,000 psi (34.47 MPa).***
- ***Filter and Degas Eluents.***
- ***Filter Samples.***
- ***Eluent pH range: 0 to 7.***
- ***Organic Solvent: ≤ 10% in eluent or for column clean up (acetonitrile, acetone)***
- ***Maximum Flow Rate: 0.015 mL/min for 0.4 mm columns.***
  0.38 mL/min for 2 mm columns.
  1.5 mL/min for 4 mm columns.
- ***Column Temperature Range: 15 to 30 ºC***

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. Thermo Fisher Scientific cannot guarantee proper column performance when the quality of the chemicals, solvents and water used to prepare eluents has been compromised.

4.3.1 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. Filter water with a 0.2 µm filter. 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.

4.3.2 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. The following chemicals will perform reliably:

A. Use Fluka or Aldrich Methanesulfonic Acid (MSA) (>99% pure) or Thermo Scientific Dionex Methanesulfonic Acid Concentrate (0.4 M) P/N 057562 or Thermo Scientific Dionex Methanesulfonic Acid (15.4 M) P/N 080388.

B. Use Dionex Cation Regenerant Solution, tetrabutylammonium hydroxide (Dionex TBAOH), P/N 039602, to ensure maximum system performance when operating with a Dionex CMMS 300. For the Dionex DCR Mode, use Dionex TBAOH (P/N 057561).

C. Use deionized water with a specific resistance of 18.2 megohm-cm to make all standards and eluents.

4.3.3 Solvents

Solvents can be added to the ionic eluents used with Dionex IonPac CS19-4µm columns to modify the analytes retention in the column, to improve sample solubility, or to clear the column from hydrophobic contaminants. 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 make 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 back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent, the column, the temperature, and the flow rate used. It is recommended to first add 5% solvent to the eluent and rinse the column with it at half the standard flow rate for 15 minutes. The column back pressure will vary as the composition of water-solvent mixture varies. The maximum back pressure limit for the Dionex IonPac CS19-4µm columns is 5,000 psi (34.47 MPa). The Dionex IonPac CS19-4µm column is compatible with the HPLC solvents listed in Table 4, "HPLC Solvents for Use with the Dionex IonPac CS19-4µm Columns." Solvents and water should be premixed in concentrations which allow proper mixing by the 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.

NOTE

At a characteristic concentration range of organic solvent in the eluent, the column back pressure may more than double. If this is the case, you should decrease the eluent flow rate to allow use of the eluent containing solvent in this concentration range.

Table 4. HPLC Solvents for Use with Dionex IonPac CS19-4µm Columns

Solvent	Maximum Operating Concentration
Acetonitrile	10%
Acetone	10%
Alcohol	0%

CAUTION

Do NOT use alcohols as an eluent component with the Dionex IonPac CS19-4µm column. Very high backpressures will be generated and probably disrupt the column packing. Alcohols, however, can be present in the sample matrix.

4.4 Preparation of Eluent Stock Solution Concentrates

Sulfuric acid (H_2SO_4) is very corrosive. Methanesulfonic acid (MSA) is also a corrosive and a strong irritant.

WARNING

Avoid breathing the vapors.
Always use these reagents in a fume hood. Wear gloves and goggles.

4.4.1 1.0 N Methanesulfonic Acid (MSA) Stock Solution

A. 1.0 N methanesulfonic acid stock solution can be prepared as follows:
B. Weigh out 96.10 g of methanesulfonic acid (MSA, > 99%, P/N 033478).
C. Carefully add this amount to a 1-liter volumetric flask containing about 500 mL of deionized water.
D. Dilute to the mark and mix thoroughly.

4.4.2 0.4 N Dionex Methanesulfonic Acid (MSA) Eluent Concentrate

0.4 N Dionex Methanesulfonic Acid Eluent Concentrate (P/N 057562 or package of 4, P/N 057568) is available from Thermo Scientific.

4.4.3 1.0 N Sulfuric Acid Stock Solution

For manually prepared eluents, sulfuric acid can be used instead of methanesulfonic acid. Note that the in the Applications section of this manual mM (millimolar) amounts of methanesulfonic acid are used. If you are going to use sulfuric acid instead, make sure then to use an equivalent Normal concentration to the Molar concentration of methanesulfonic acid.

Calculate the amount (in grams) of concentrated sulfuric acid (H_2SO_4) that you need to add to a 1 liter volumetric flask by using the % H_2SO_4 composition stated on the label of the particular bottle of H_2SO_4 you are using. For example, if the H_2SO_4 concentration is 98%, you need to weigh out 50.04 grams of concentrated H_2SO_4. Carefully add this amount of H_2SO_4 to a 1- liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm. Dilute to the 1 liter mark and mix thoroughly.

In other words:

1M H_2SO_4 = 2.0 N H_2SO_4

FW of H_2SO_4 = 98.08 g

H_2SO_4 concentration = 98%

Therefore, to prepare 1 L of a 1 N H_2SO_4 solution, weigh out:

$$\frac{1\ \text{liter}}{1\ \text{mole}} \times \frac{98.08\ \text{g}}{2\ \text{Eq}} \times \frac{1\ \text{mole}}{1\ \text{liter}} \times \frac{1\ \text{mole}}{98\ \text{g}} \times 100\ \text{g} = 50.04\ \text{g}$$

4.4.4 Eluent Preparation

Eluent: X mN Sulfuric Acid (H_2SO_4) or Methanesulfonic acid (MSA)

Using the table below, pipet X.0 mL of the 1.0 N H_2SO_4 or 1.0 N MSA eluent concentrate (see Section 4.4, “Preparation of Eluent Stock Solution Concentrates”) into a 1-L volumetric flask. Dilute to 1-L using deionized water with a specific resistance of 18.2 megohm-cm. Degas the eluent.

Table 5. mN Eluents from Stock Solutions

MSA/H_2SO_4 mN	#mL
4	4.0
10	10.0
16	16.0
18	18.0
20	20.0
22	22.0
24	24.0
30	30.0
40	40.0
100	100.0

4.5 Making and Using Eluents that Contain Solvents

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 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.

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 10% acetonitrile, prepare the eluent by adding 100 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water at a time to the acetonitrile in the reservoir and fill it up to the 1 liter mark. 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.

Avoid creating high viscosity pressure fronts that may disrupt the column packing when the eluent solvent component is added or changed. To do this, equilibrate the column at half its standard flow rate for approximately 10 minutes with an eluent containing only 5% of the current solvent type. Exchange this eluent for an eluent with 5% of the new solvent type and then equilibrate the column and allow the system to stabilize (approximately 10 minutes). Next run a 15-minute gradient from 5% of the new solvent type to the highest percentage that will be used during the new analysis protocol. Adjust the flow rate so that it does not exceed the maximum pressure limit at any point.

Properly equilibrate the column when changing to a solvent-free eluent system after using eluents containing solvent. First equilibrate the column with 1 to 5 percent of the current solvent for approximately 5 minutes. Next run a 10-minute gradient from the eluent with 1 to 5 percent of the current solvent to the new solvent free aqueous eluent.

Depending on the % level of organic solvent present in the eluent, solvent containing eluents must be used in the External Water Mode using the Dionex CERS 500 or Chemical Suppression Mode using the Dionex Cation MicroMembrane Suppressor (Dionex CMMS 300).

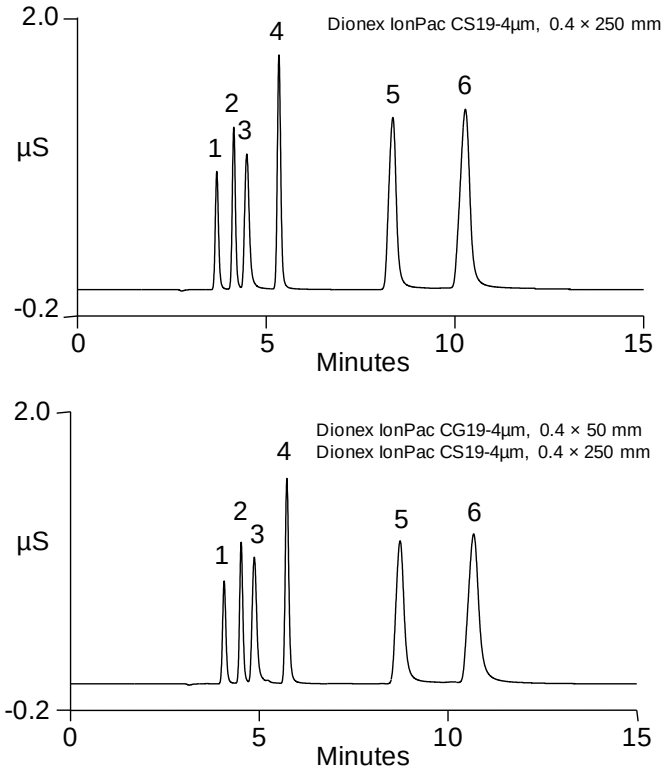
Consult the appropriate suppressor manual for the suppressor operation when organic solvent is present in the eluent.

5. Example Applications

5.1 Isocratic Elution of Six Common Cations using the Dionex IonPac CS19-4µm Column with and without Dionex IonPac CG19-4µm Guard Column

The chromatograms below show the separation of the common cations plus ammonium ion using the Dionex IonPac CS19-4µm column with and without a Dionex IonPac CG19-4µm guard column. As can be seen, the Dionex IonPac CG19-4µm guard column has much lower capacity per gram of resin than the Dionex IonPac CS19-4µm separator column, and adds only about 0.5 minutes to the total retention time when it is used. It is purposely made of lower cation exchange capacity so that its pressure contribution is small (should be less than 200 psi).

Figure 7. Isocratic Elution of Six Common Cations using the Dionex IonPac CS19-4µm Column (0.4 × 250 mm), with and without Dionex IonPac CG19-4µm Guard Column (0.4 × 50 mm)

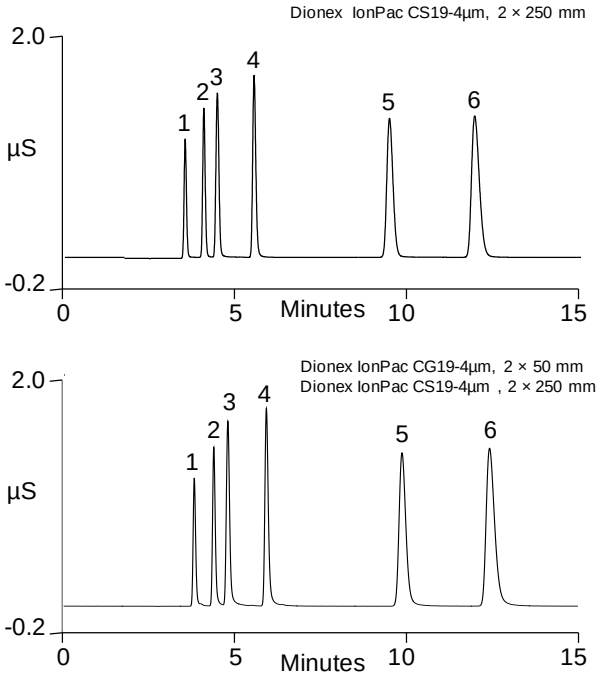


Eluent:	8 mM MSA
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1. Lithium	0.063 mg/L
2. Sodium	0.25
3. Ammonium	0.313
4. Potassium	0.625
5. Magnesium	0.313
6. Calcium	0.625

Figure 8. Isocratic Elution of Six Common Cations using the Dionex IonPac CS19-4µm Column (2 × 250 mm), with and without Dionex IonPac CG19-4µm Guard Column (2 × 50 mm)

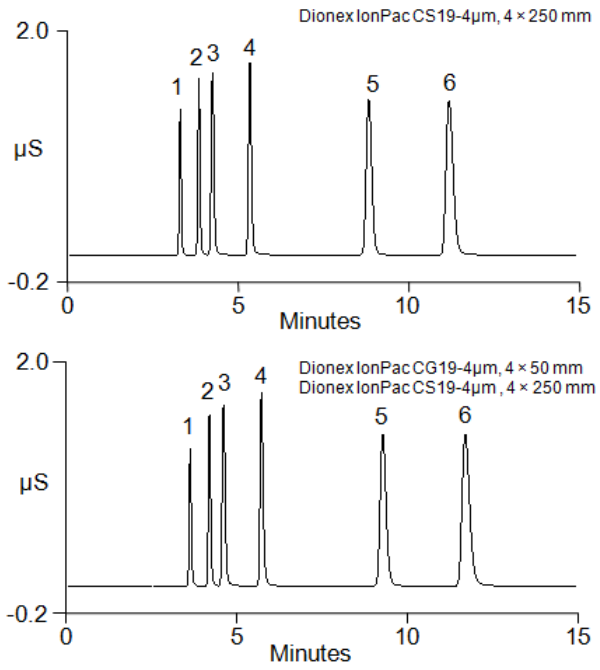


Eluent:	8 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.25 mL/min
Inj. Volume:	2.5 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2 mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.25 mg/L
2.	Sodium	1.00
3.	Ammonium	1.25
4.	Potassium	2.50
5.	Magnesium	1.25
6.	Calcium	2.50

Figure 9. Isocratic Elution of Six Common Cations using the Dionex IonPac CS19-4µm Column (4 × 250 mm), with and without Dionex IonPac CG19-4µm Guard Column (4 × 50 mm)



Eluent:	8 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	1.0 mL/min
Inj. Volume:	10 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4 mm, AutoSuppression, recycle mode

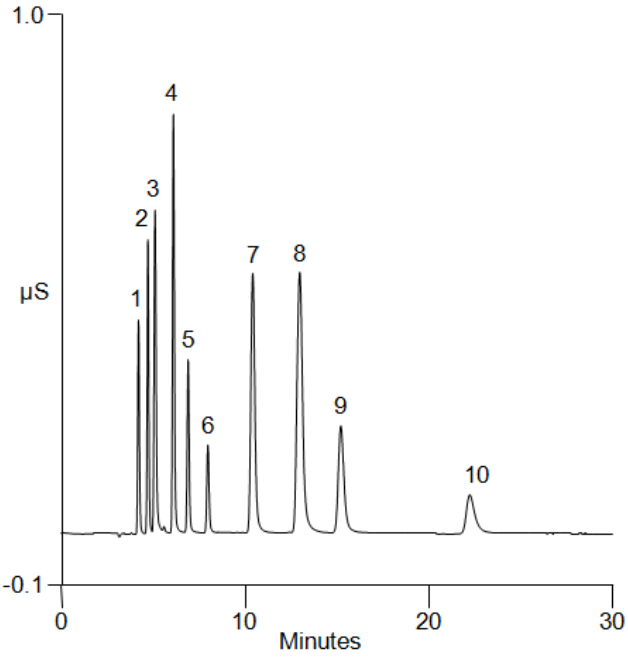
Peaks:

1.	Lithium	0.25 mg/L
2.	Sodium	1.00
3.	Ammonium	1.25
4.	Potassium	2.50
5.	Magnesium	1.25
6.	Calcium	2.50

5.2 Isocratic Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CS19-4µm Column

The chromatograms below show the separation of Group I and Group II cations plus ammonium using an isocratic eluent of 7 mM methanesulfonic acid concentration; the monovalent cations elute first followed by divalent cations. Under isocratic conditions at the standard flow rates shown below, the analysis can take almost 30 minutes.

Figure 10. Isocratic Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column



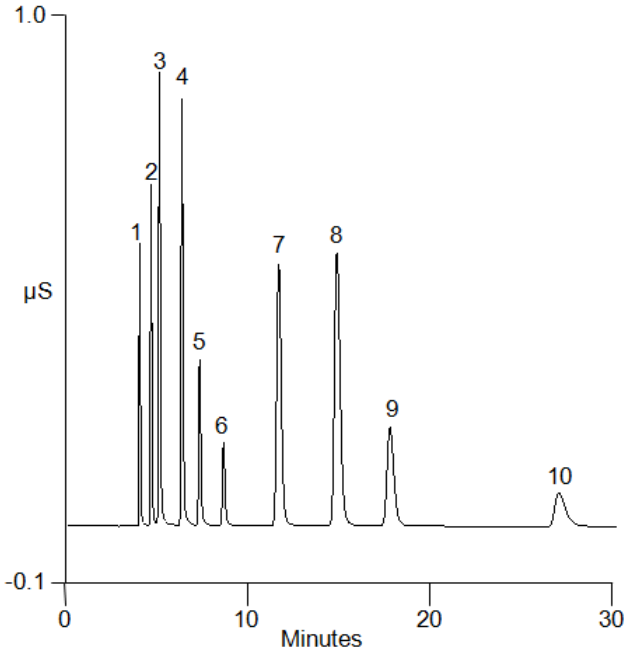
Column:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	7 mM MSA
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.038 mg/L*
2.	Sodium	0.15
3.	Ammonium	0.188
4.	Potassium	0.375
5.	Rubidium	0.375
6.	Cesium	0.375
7.	Magnesium	0.188
8.	Calcium	0.375
9.	Strontium	0.375
10.	Barium	0.375

*Concentrations are approximate.

Figure 11. Isocratic Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm 2 mm Column



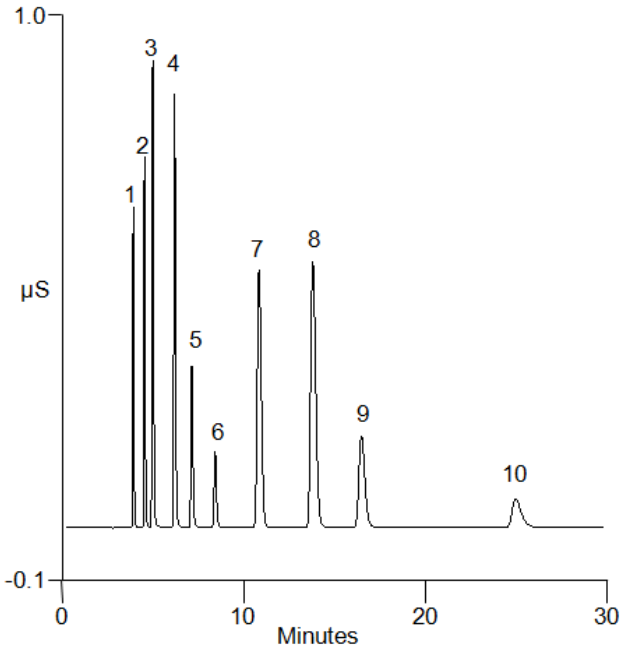
Column:	Dionex IonPac CG19-4µm/CS19-4µm (2 × 250 mm)
Eluent:	7 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.25 mL/min
Inj. Volume:	2.5 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2 mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.15 mg/L *
2.	Sodium	0.60
3.	Ammonium	0.75
4.	Potassium	1.5
5.	Rubidium	1.5
6.	Cesium	1.5
7.	Magnesium	0.75
8.	Calcium	1.5
9.	Strontium	1.5
10.	Barium	1.5

*Concentrations are approximate.

Figure 12. Isocratic Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm 4 mm Column



Column:	Dionex IonPac CG19-4µm/CS19-4µm (4 × 250 mm)
Eluent:	7 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	1.0 mL/min
Inj. Volume:	10 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4 mm, AutoSuppression, recycle mode

Peaks:

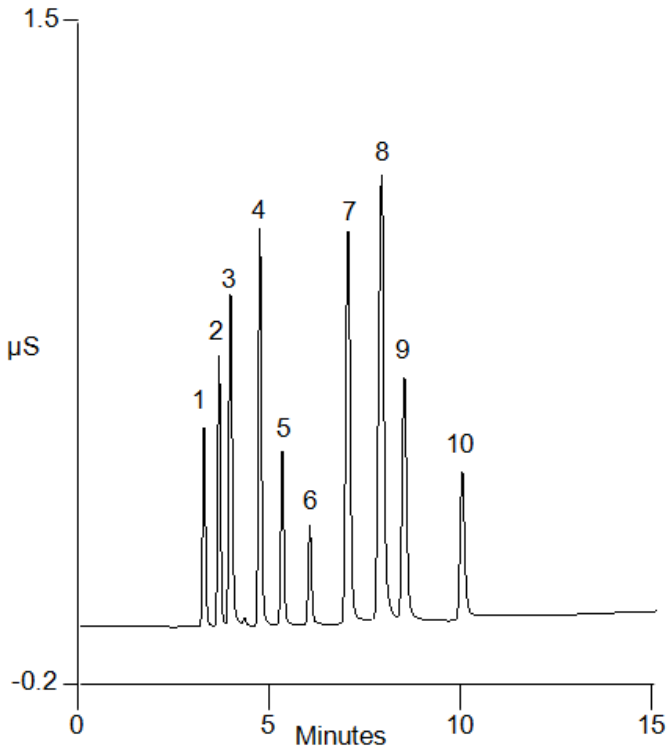
1.	Lithium	0.15 mg/L*
2.	Sodium	0.60
3.	Ammonium	0.75
4.	Potassium	1.5
5.	Rubidium	1.5
6.	Cesium	1.5
7.	Magnesium	0.75
8.	Calcium	1.5
9.	Strontium	1.5
10.	Barium	1.5

*Concentrations are approximate.

5.3 Fast Gradient Elution of Group I & Group II Cations plus Ammonium using the Dionex IonPac CS19-4µm Column

The Group I and Group II cations plus ammonium can be separated faster using a gradient eluent at an increased flow rate. The total system pressure must be kept below 5000 psi by adjusting the flow rate. In the examples below an increased flow rate combined with a gradient eluent reduces the run time from almost 30 minutes to about 10 minutes.

Figure 13. Fast Gradient Elution of Group I and Group II Cations Plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column

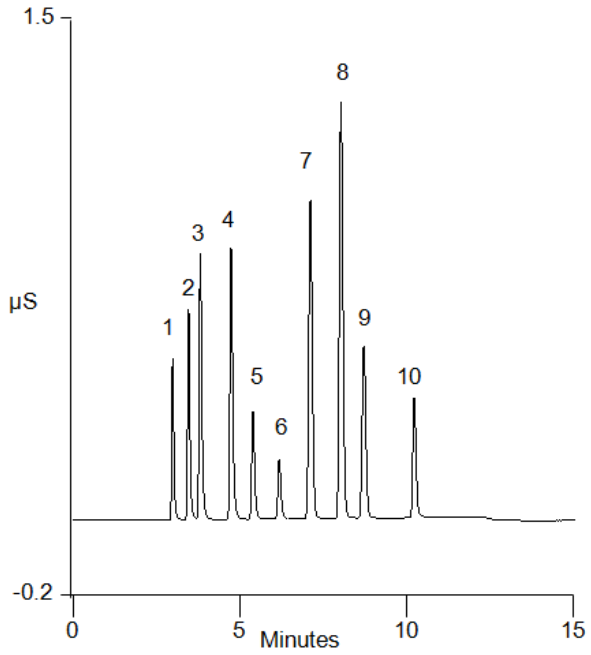


Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	7 mM MSA, gradient to 14 mM MSA from 1 to 6 min, gradient to 20 mM MSA at 7 min, isocratic to 9 min, back to 7 mM MSA at 9.1 min.
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	13 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:	
1. Lithium	0.038 mg/L*
2. Sodium	0.15
3. Ammonium	0.188
4. Potassium	0.375
5. Rubidium	0.375
6. Cesium	0.375
7. Magnesium	0.188
8. Calcium	0.375
9. Strontium	0.375
10. Barium	0.375

*Concentrations are approximate.

Figure 14. Fast Gradient Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm 2 mm Column

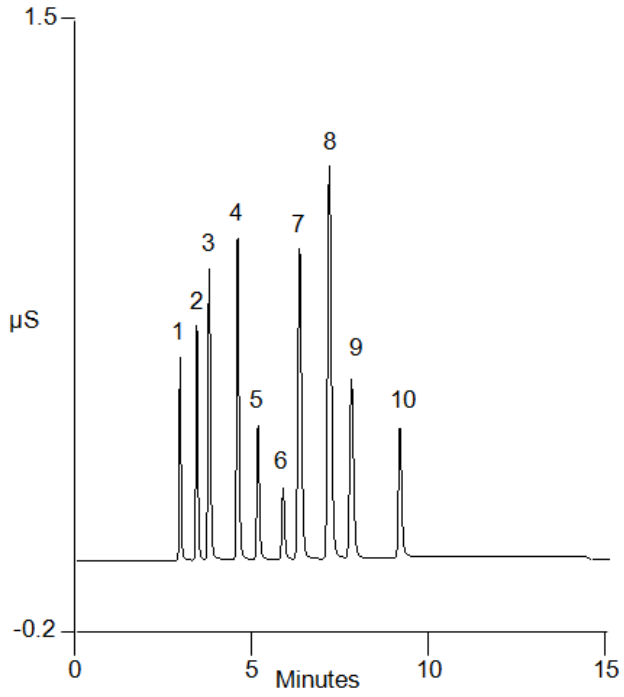


Columns:	Dionex IonPac CG19-4µm/CS19-4µm (2 × 250 mm)
Eluent:	7 mM MSA, gradient to 14 mM MSA from 1 to 6 min, gradient to 20 mM MSA at 7 min, isocratic to 9 min, back to 7 mM MSA at 9.1 min.
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.325 mL/min
Inj. Volume:	2.5 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2 mm, AutoSuppression, recycle mode

Peaks:	
1. Lithium	0.15 mg/L*
2. Sodium	0.60
3. Ammonium	0.75
4. Potassium	1.5
5. Rubidium	1.5
6. Cesium	1.5
7. Magnesium	0.75
8. Calcium	1.5
9. Strontium	1.5
10. Barium	1.5

*Concentrations are approximate.

Figure 15. Fast Gradient Elution of Group I and Group II Cations plus Ammonium using the Dionex IonPac CG19-4µm/CS19-4µm 4 mm Column



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (4 × 250 mm)
Eluent:	7 mM MSA, gradient to 14 mM MSA from 1 to 6 min, gradient to 20 mM MSA at 7 min, isocratic to 9 min, back to 7 mM MSA at 9.1 min.
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	1.3 mL/min
Inj. Volume:	10 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4 mm, AutoSuppression, recycle mode

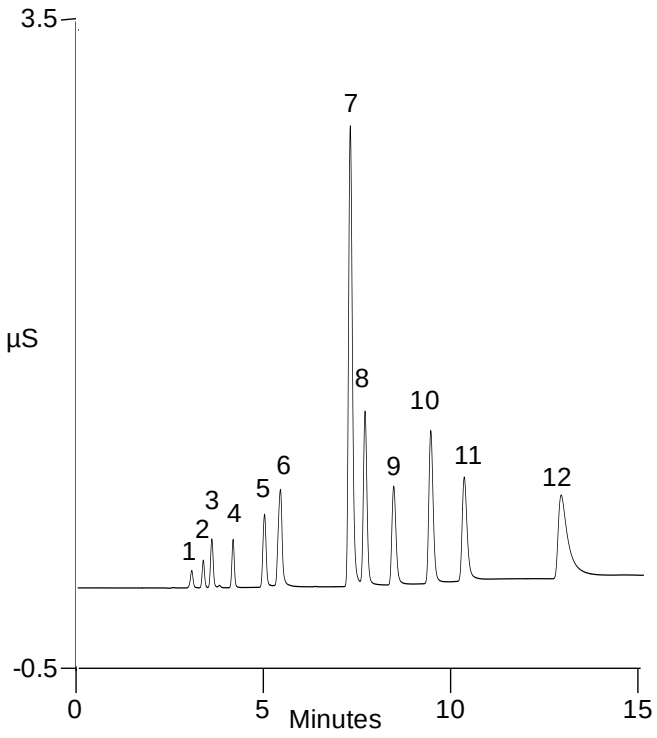
Peaks:	
1. Lithium	0.15 mg/L*
2. Sodium	0.60
3. Ammonium	0.75
4. Potassium	1.5
5. Rubidium	1.5
6. Cesium	1.5
7. Magnesium	0.75
8. Calcium	1.5
9. Strontium	1.5
10. Barium	1.5

*Concentrations are approximate.

5.4 Fast Gradient Elution of Cations plus Ammonium and Biogenic Amines using the Dionex IonPac CS19-4µm Column

This example shows the separation of the six common cations plus biogenic amines, which are of interest in the Food Industry. Histamine, for example, is formed by bacterial decomposition of histidine, and is important in the analysis of wine. The freshness of seafood and meat products is determined by the amounts of biogenic amines present. Figure 16 below shows an organic-solvent-free gradient eluent used at a slightly increased flow rate to elute the common cations and biogenic amines, including the polyvalents, spermidine and spermine, in less than 15 minutes. The same eluent and flow rate can be scaled for either the capillary or standard bore system.

Figure 16. Gradient Separation of Six Common Cations plus Biogenic Amines using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column (0.4 × 250 mm)



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	9 mM MSA, gradient to 65 mM MSA from 0.1 to 10.7 min, isocratic to 12.8 min, back to 9 mM MSA at 12.9 min
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	13 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

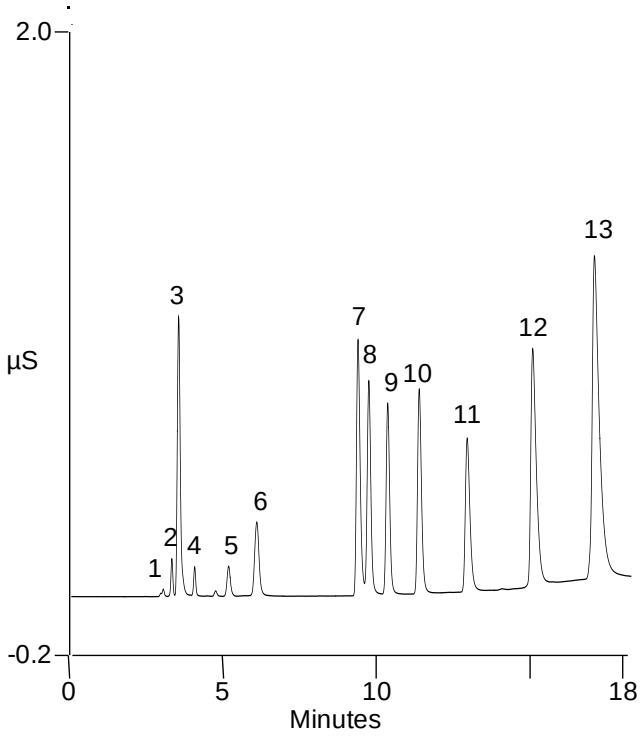
Peaks:	
1. Lithium	0.013 mg/L*
2. Sodium	0.05
3. Ammonium	0.063
4. Potassium	0.125
5. Magnesium	0.063
6. Calcium	0.125
7. Putrescine	1.875
8. Cadaverine	1.125
9. Histamine	1.625
10. Agmatine	1.25
11. Spermidine	0.75
12. Spermine	0.375

*Concentrations are approximate.

5.5 Fast Gradient Separation of Cations plus Ammonium and Diamines using the Dionex IonPac CS19-4µm Column

Gradient elution combined with a faster flow rate can also be applied to the analysis of cations plus ammonium and diamines. Figure 17 below shows the separation of common cations from 7 diamines using the Dionex IonPac CS19-4µm Capillary Column. The same eluent and flow rate can be scaled up for either the microbore or standard bore system.

Figure 17. Gradient Elution of Common Cations plus Diamines using the Dionex IonPac CG19-4µm/CS19-4µm Column (0.4 × 250 mm)



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	10 mM MSA, gradient to 35 mM MSA from 2.3 to 10.4 min, gradient to 65 mM MSA at 12.7 min, isocratic to 20.8 min, back to 10 mM MSA at 20.9 min.
Eluent Source:	EGC-MSA (Capillary) cartridge
Flow Rate:	13 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks :		
1.	Lithium	0.005 mg/L*
2.	Sodium	0.02
3.	Ammonium	>>0.03
4.	Potassium	0.05
5.	Magnesium	0.03
6.	Calcium	0.05
7.	Ehtylenediamine	0.50
8.	1,4-Butanediamine	0.50
9.	1,5-Pentanediamine	0.50
10.	1,6-Hexanediamine	0.50
11.	1,7-Heptanediamine	0.50
12.	1,8-Octanediamine	0.50
13.	1,9-Nonanediamine	1.50

*Concentrations are approximate.

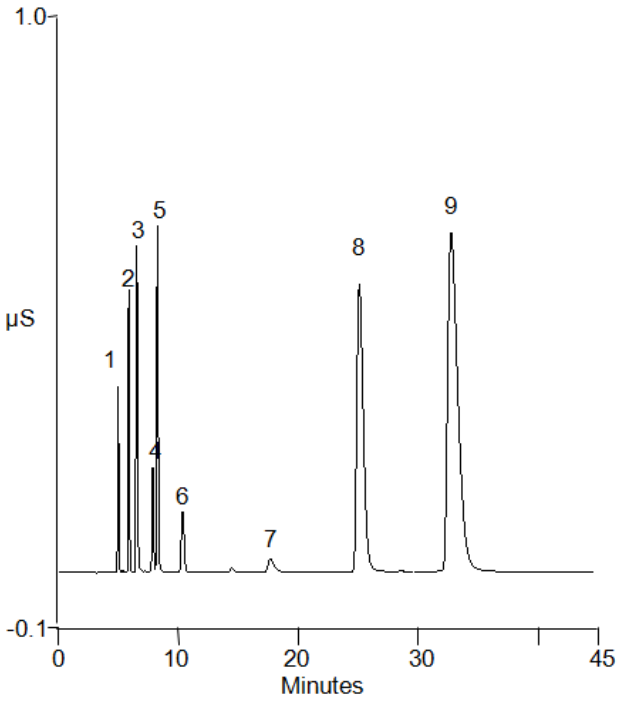
5.6 Separation of Six Common Cations plus Ethylamines using the Dionex IonPac CS19-4µm Column

The chromatograms below show the separation of cations plus ammonium and ethylamines using different operating conditions. Figure 18 shows the separation using the Dionex IonPac CS19-4µm capillary column with an isocratic eluent at the standard flow rate and 30 °C. Under these conditions scaled up to a microbore column, the separation can take almost 45 minutes as shown in Figure 19, which uses the Dionex IonPac CS19-4µm 2mm column.

Lowering the temperature and using a gradient eluent will decrease the run time as shown in Figure 20. The effect of temperature on potassium is stronger than for the other monovalent cations, thus as the column temperature is lowered potassium is retained longer than ethylamine and their resolution is greatly improved at 15 ˚C. At the lower temperature, an impurity peak, (peak #7) which was hidden under diethylamine, is now resolved from it. As expected, the total system pressure increases as the column temperature is decreased.

In Figure 21, a gradient eluent, reduced temperature and the maximum flow rate for the Dionex IonPac CS19-4µm capillary column were combined to shorten the total run time. When developing a method, make sure the total system pressure is well within 5000 psi maximum operational pressure of the system by decreasing the flow rate if necessary. Higher resolution among peaks may allow for the higher concentrations to be injected and still produce good chromatography. The sample injected here is the same as injected in the 4 mm analytical format, where normally it is desirable to inject ¼ of the 4 mm sample concentration. Note the gradient times were adjusted to accommodate the faster flow rate and maintain the same separation as the standard flow rate.

Figure 18. Isocratic Elution of Six Common Cations plus Ethylamines using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column



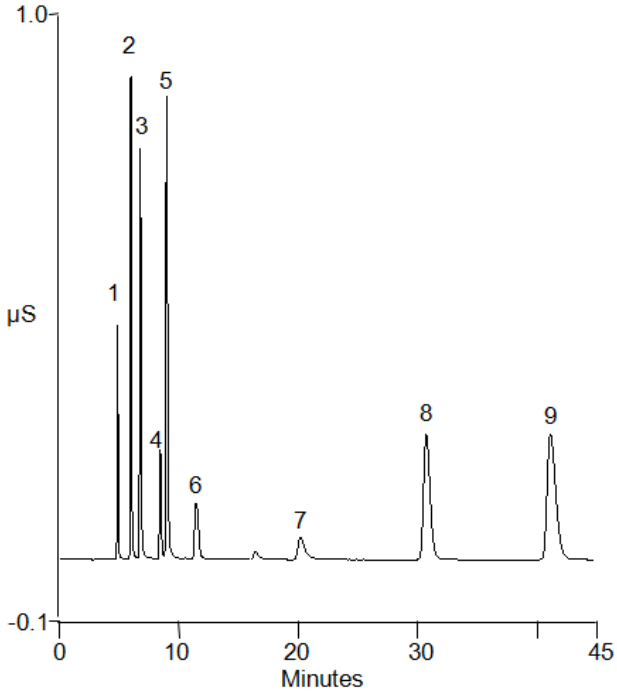
Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	4 mM MSA
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.03 mg/L*
2.	Sodium	0.13
3.	Ammonium	0.16
4.	Ethylamine	0.18
5.	Potassium	0.31
6.	Diethylamine	0.25
7.	Triethylamine	0.50
8.	Magnesium	0.16
9.	Calcium	0.31

*Concentrations are approximate.

Figure 19. Isocratic Elution of Six Common Cations plus Ethylamines using the Dionex IonPac CG19-4µm/CS19-4µm Microbore Column



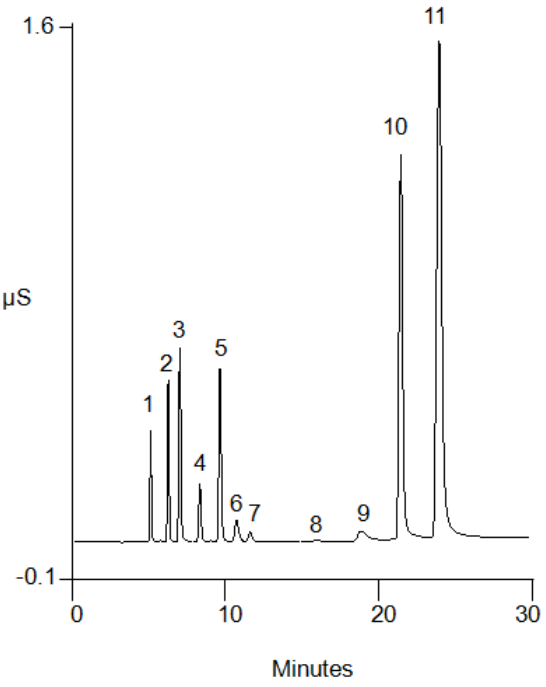
Columns:	Dionex IonPac CG19-4µm/CS19-4µm (2 × 250 mm)
Eluent:	4 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.25 mL/min
Inj. Volume:	2.5 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2 mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.13 mg/L*
2.	Sodium	0.50
3.	Ammonium	0.62
4.	Ethylamine	0.70
5.	Potassium	1.25
6.	Diethylamine	1.00
7.	Triethylamine	2.00
8.	Magnesium	0.62
9.	Calcium	1.25

*Concentrations are approximate.

Figure 20. Gradient Elution of Six Common Cations plus Ethylamines at 15 °C using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column



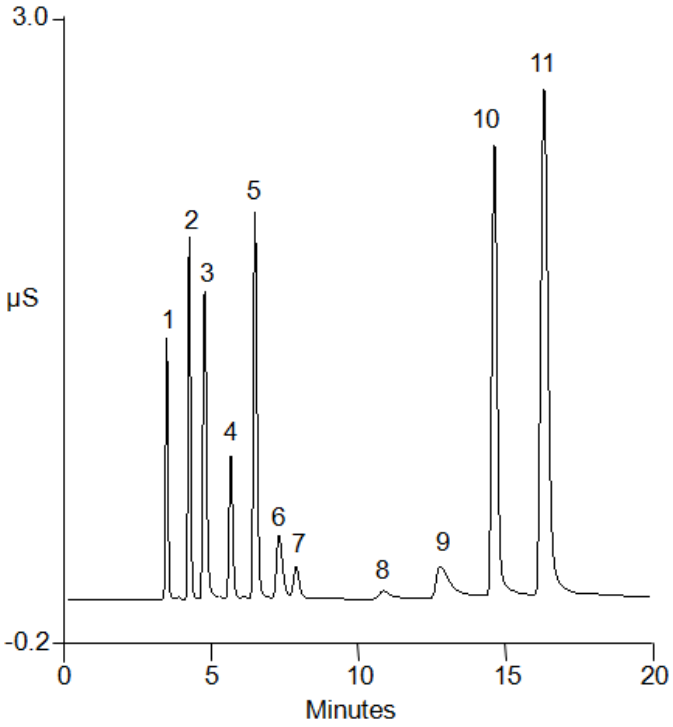
Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	Isocratic 4 mM MSA to 11.5 min, gradient to 8 mM MSA at 17.2 min, isocratic to 26.1 min, back to 4 mM MSA at 26.2 min
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.03 mg/L*
2.	Sodium	0.13
3.	Ammonium	0.16
4.	Ethylamine	0.18
5.	Potassium	0.31
6.	Diethylamine	0.25
7.	Impurity	-
8.	Impurity	-
9.	Triethylamine	0.50
10.	Magnesium	0.16
11.	Calcium	0.31

*Concentrations are approximate.

Figure 21. Gradient elution combined with a faster flow rate and lower temperature reduce the run time to less than 20 minutes for the Six Common Cations plus Ethylamines using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	Isocratic 4 mM MSA to 7.7 min, gradient to 8 mM MSA at 11.5 min, isocratic to 17.4 min, back to 4 mM MSA at 17.5 min
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	15 µL/min
Inj. Volume:	0.4 µL
Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

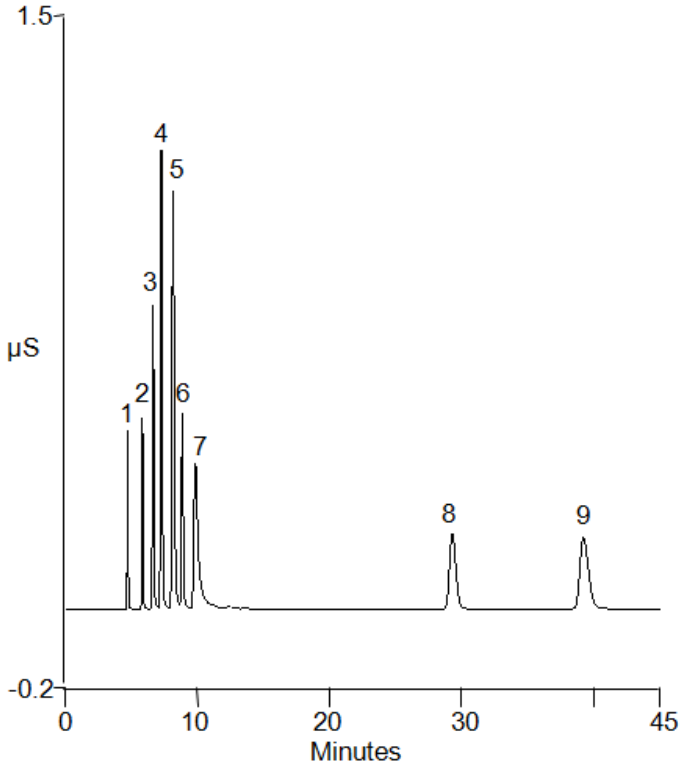
Peaks:	
1. Lithium	0.13 mg/L*
2. Sodium	0.50
3. Ammonium	0.62
4. Ethylamine	0.70
5. Potassium	1.25
6. Diethylamine	1.00
7. Impurity	-
8. Impurity	-
9. Triethylamine	2.00
10. Magnesium	0.62
11. Calcium	1.25

*Concentrations are approximate.

5.7 Separation of Six Common Cations plus Ethanolamines using the Dionex IonPac CS19-4µm Column

The separation of common cations plus ammonium and ethanolamines using the Dionex IonPac CS19-4µm column is shown in Figure 22. Under these isocratic conditions at the standard flow rate, the first several peaks elute closely and the run time is almost 45 minutes. As shown in Figure 23, using an optimized gradient eluent combined with a faster flow rate, the first seven peaks are baseline resolved with the last two analytes eluting within 30 minutes.

Figure 22. Isocratic Elution of Six Common Cations plus Ethanolamines using the Dionex IonPac CG19-4µm/CS19-4µm Column (4 × 250 mm)



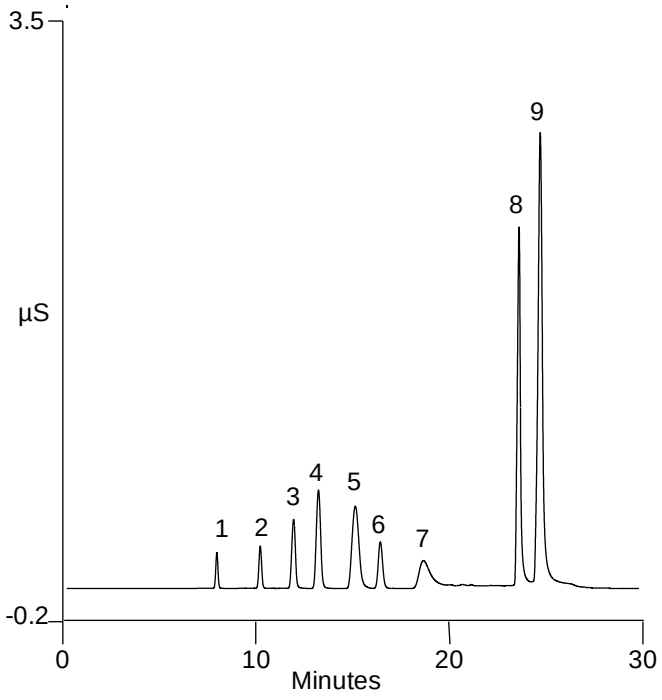
Columns:	Dionex IonPac CG19-4µm/CS19-4µm (4 × 250 mm)
Eluent:	Isocratic 4 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	1.0 mL/min
Inj. Volume:	10 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4 mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.125 mg/L*
2.	Sodium	0.5
3.	Ammonium	0.62
4.	Ethanolamine	5.0
5.	Diethanolmine	10.0
6.	Potassium	1.25
7.	Triethanolamine	40.0
8.	Magnesium	0.62
9.	Calcium	1.25

*Concentrations are approximate.

Figure 23. Gradient Elution of Common Cations plus Ethanolamines using the Dionex IonPac CG19-4µm/CS19-4µm Column (0.4 × 250 mm)



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	Isocratic 1.5 mM MSA to 13.8 min, gradient to 11 mM MSA at 17.3 min, isocratic to 21.9 min, back to 1.5 mM MSA at 22 min
Eluent Source:	Dionex EGC-MSA (Capillary) cartridge
Flow Rate:	13 µL/min
Inj. Volume:	0.4 µL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

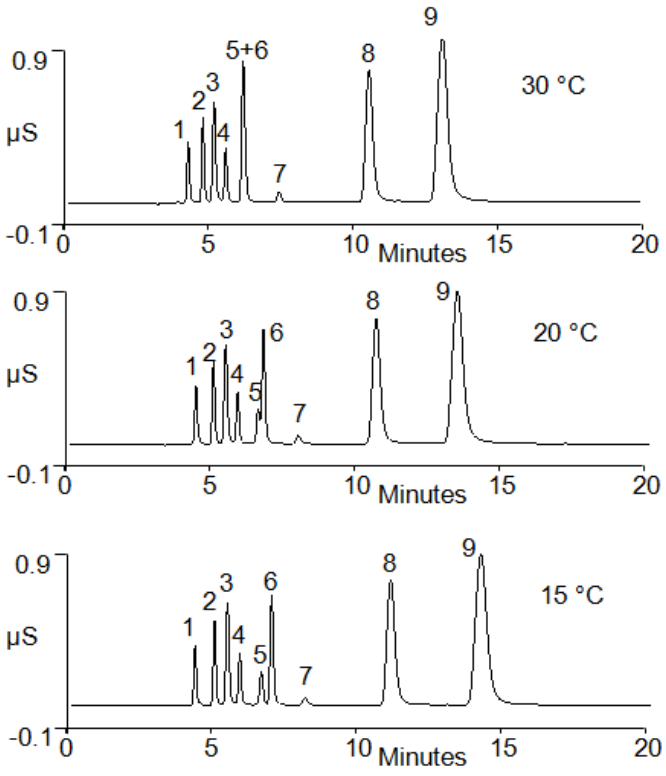
1. Lithium	0.03 mg/L*
2. Sodium	0.13
3. Ammonium	0.16
4. Ethanolamine	1.25
5. Diethanolmine	2.50
6. Potassium	0.31
7. Triethanolamine	10.00
8. Magnesium	0.16
9. Calcium	0.31

*Concentrations are approximate.

5.8 Isocratic Separation of Six Common Cations plus Methylamines using the Dionex IonPac CS19-4µm Capillary Column

Figure 24 below shows the isocratic separation of common cations plus ammonium and methylamines at various temperatures. The effect of temperature on potassium is higher than for the other monovalent cations. At the standard operating temperature of 30 ˚C, dimethylamine and potassium co-elute. Temperature has a larger effect on the elution of potassium than it has for dimethylamine. At a lower column temperature, potassium is retained longer than dimethylamine, and their resolution is achieved at 15 ˚C as shown in the bottom chromatogram and also in Figure 25. As expected, total system pressure increases as the column temperature is decreased (approximately 50 psi per °C). When developing a method, make sure the total system pressure is within the operational pressure limits of the system.

Figure 24. Isocratic Elution of Six Common Cations plus Methylamines using the Dionex IonPac CG19-4µm/CS19-4µm Capillary Column at various temperatures



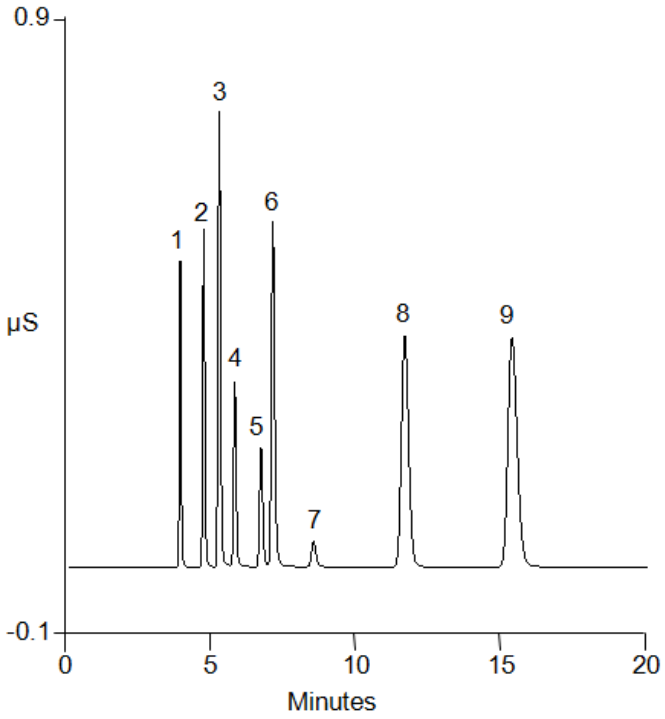
Columns:	Dionex IonPac CG19-4µm/CS19-4µm (0.4 × 250 mm)
Eluent:	Isocratic 7 mM MSA
Eluent Source:	EGC-MSA (Capillary) cartridge
Flow Rate:	10 µL/min
Inj. Volume:	0.4 µL
Temperature:	In chromatogram
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.03 mg/L*
2.	Sodium	0.10
3.	Ammonium	0.13
4.	Methylamine	1.25
5.	Dimethylmine	1.00
6.	Potassium	0.25
7.	Trimethylamine	3.75
8.	Magnesium	0.13
9.	Calcium	0.25

*Concentrations are approximate.

Figure 25. Isocratic Elution of Six Common Cations plus Methylamines using the Dionex IonPac CG19-4µm/CS19-4µm (4 × 250 mm) Column at 15 °C



Columns:	Dionex IonPac CG19-4µm/CS19-4µm (4 × 250 mm)
Eluent:	Isocratic 7 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	1.0 mL/min
Inj. Volume:	10 µL
Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4 mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.10 mg/L*
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Methylamine	5.00
5.	Dimethylmine	4.00
6.	Potassium	1.00
7.	Trimethylamine	15.00
8.	Magnesium	0.50
9.	Calcium	1.00

*Concentrations are approximate.

6. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac CS19-4µm columns. For more information on problems that originate with the Ion Chromatograph (IC) or 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., call the nearest Thermo Fisher Scientific office.

Table 6. Dionex IonPac CS19-4µm/CG19-4µm Column Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown component	Isolate blockage	6.1.1
	Plugged column bed supports	Replace bed supports	6.1.2
	Plugged system hardware	Unplug, Replace	Component manual
High Background Conductivity and/or High Noise			
Improper Suppressor Operation	Dionex CERS 500 or Dionex CCES 300 not suppressing	Check current	6.5, Component manual
		Check REGEN OUT flow	6.5 D, Component manual
		Check for leaks	6.5 B, Component manual
	Dionex CMMS 300 not suppressing	Check regenerant	6.5 D, Component manual
		Check AutoRegen cartridge	6.5 F, Component manual
	Air bubble trapped in Dionex CERS 500 or Dionex CCES 300	Remove bubble by loosening fittings	6.4
Contamination	Bad eluents	Remake eluents	6.2, 6.4, 6.7 A
	Contaminated column	Clean column	6.3.2, Appendix B
	Contaminated suppressor	Clean suppressor	6.3.1, Component manual
Hardware Operation	Proportioning valve	Service valve	Component manual
Poor Efficiency	Large system void volumes	Replumb system	6.6.1 A, Component manual
	Sluggish injection valve	Service valve	6.6.3 B, Component manual
	Contaminated or deformed bed support	Replace bed support	6.1.2
	Column headspace	Replace column	6.6.1 B
	Column overloading	Reduce sample size	3.3
	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	3.3
Fronting Peaks	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	3.3
	Column overload	Reduce sample size	3.3
	Contaminated or deformed bed support	Replace bed support	6.1.2
	Column headspace	Replace column	6.6.1 B
Tailing Peaks	Contaminated suppressor	Clean suppressor	6.3.1, Component Manual
	Column overloading	Reduce sample size	3.3
	Sluggish injection valve	Service valve	6.6.3 B, Component Manual
	Contaminated sample loop	Replace loop	6.3.3
Short Retention Times	Flow rate too fast	Recalibrate pump	6.6.2 A, Component Manual
	First peaks elute too fast	Equilibrate to first eluent	6.6.3 A
	Bad eluents	Remake eluents	6.6.2 B
	Column contamination	Clean column	6.6.2 C. 6.3.2, Appendix B
Spurious Peaks	Eluent contamination	Remake eluents	6.7 A, 6.2, 6.4
	Column contamination	Clean Column	6.3.2 , 6.7 B, Appendix B
	Sluggish injection valve	Service valve	6.7 C, Component Manual
Poor Qualifications of Divalents	Sample loop contamination	Flush, replace	6.3.3
	Suppressor Contamination	Clean Suppressor	6.3.1 , Component Manual

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the Dionex IonPac CG19-4µm Guard/Capillary Guard Column plus the Dionex IonPac CS19-4µm Analytical/Capillary Column when using the test chromatogram conditions should be as indicated in Table 2. If the system pressure is approximately 200 psi higher than this, it is advisable to determine the cause of the high system pressure.

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 (if you are using one), the EGC Cartridge, the RFIC Eluent Degasser, 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 EGC Cartridge and turn the pump on at its standard flow rate (1 mL/min, 0.25 mL/min, or 10 µL/min). Watch the pressure; it should not exceed 50 psi (0.34 MPa). Continue adding system components (EGC cartridge, CR-CTC, RFIC Eluent Degasser, 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/Capillary Guard and Analytical/Capillary columns are connected (see Table 2, “Typical Dionex IonPac CS19-4µm/CG19-4µm Operating Back Pressures”).

   The Dionex EGC-500 cartridge may add up to 400 psi (2.76 MPa) and Cation Electrolytically Regenerated Suppressor 500 (CERS 500) may add up to 100 psi (0.69 MPa) of back pressure. 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.

C. **Make sure your system does not have extra tubing** to increase the back pressure (as needed for the eluent generator to work properly with a low pressure column), left over from a previous set up.

6.1.2 Replacing Column Bed Support Assemblies (2 mm and 4 mm columns only)

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. If the bed support is contaminated and/or deformed, it may be the cause of poor efficiency and/or poor peak shape. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

A. **Disconnect the column from the system**.

B. **Carefully unscrew the inlet (top) column fitting.** Use two open-end wrenches.

C. **Remove the bed support.** Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you do not scratch the walls of the end fitting. Discard the old bed support assembly.

D. **Place a new bed support assembly into the end fitting.** Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

CAUTION

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

Part	Dionex IonPac CS19-4µm 4 mm Columns (P/N)	Dionex IonPac CS19-4µm 2 mm Columns (P/N)	Dionex IonPac CS19-4µm 0.4 mm Columns (P/N)
Analytical or Capillary Column	078837	078836	078835
Guard Column	078840	078839	078838
Bed Support Assembly	042955	044689	N/A
End Fitting	052809	043278	N/A

E. **Screw the end fitting back onto the column.** Tighten it fingertight, then an additional 1/4 turn (25 in × lb). Tighten further only if leaks are observed.

F. **Reconnect the column to the system and resume operation**.

NOTE

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

6.2 Preparation of Eluents

A. Make sure that the eluents and 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.

6.3 Contamination

6.3.1 Suppressor Contamination

A contaminated suppressor could be a cause for high background conductivity due to inadequate eluent suppression, as well as a cause for poor divalent peak efficiencies and high divalent peak asymmetries (i.e. tailing peaks). If tailing peaks are observed, test and clean the suppressor.

A. Testing if the suppressor has been contaminated and is the source of poor divalent peaks chromatography:
    1. Modify the QAR test chromatogram stated conditions for the particular format Dionex IonPac CS19-4µm column so that the suppressor current applied is half of what is stated in the QAR.
    2. Without much delay, inject the QAR standard of the six common cations.
    3. As soon as the six common cations have eluted, repeat the injection under these new conditions. Save the data. If you leave the suppressor too long with this reduced current, the background conductivity will start increasing as the lower current is insufficient to regenerate the suppressor.
    4. Increase the suppressor current to what is stated in the QAR test chromatogram.
    5. Inject the QAR standard of the six common cations.
    6. If the peak efficiencies and asymmetries for magnesium and calcium are worse in step 5 than in step 3, this is an indication that the source is a contaminated suppressor.
B. Cleaning the suppressor:
    1. Remove the suspected suppressor from the system.
    2. With a piece of tubing, connect the Eluent In port of the suppressor to its Regen OUT port.
    3. Connect the Regen In port to a waste line.
    4. Connect the Eluent OUT port to a pump with 0.5 M NaOH eluent. It is highly recommended to use a different pump than the analytical pump you are using for the cation analysis.
    5. Pump 0.5 M NaOH eluent for at least one hour at the “standard” flow rate for the suppressor (10µL/minute for a 0.4 mm Dionex CCES 300 suppressor, 0.25 mL/min for a 2 mm Dionex CERS 500 suppressor, and 1 mL/min for a 4 mm Dionex CERS 500 suppressor). After cleaning the suppressor with 0.5M NaOH, be sure to prime and rinse the pump with di water for 10-15 minutes in order to avoid issues with pump seals when using 0.5M NaOH as a cleaning eluent.
    6. Rinse the suppressor by pumping DI water for 30 minutes at the same flow rate.
    7. The suppressor is now ready to be re-installed and used.

6.3.2 A Contaminated Guard or Analytical/Capillary Column

Determine if the column is contaminated. Column contamination can lead to a loss of column capacity since all of the cation exchange sites will no longer be available for the sample ions. Polyvalent cations may be concentrating on the column over a series of runs. Remove the Dionex IonPac CG19-4µm Guard and Dionex IonPac CS19-4µm Analytical or 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 IonPac CG19-4µm at the first sign of column performance degradation (compared to the original QAR) to eliminate downtime. Clean the column(s) as instructed in, "Column Cleanup" (See Appendix B, "Column Care"). To make sure that contaminated hardware is not causing the high background, use deionized water with a specific resistance of 18.2 megohm-cm as eluent. The background should be less than 1 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

A. **Check for a contaminated Gradient Mixer**. Gradient Mixers in the Gradient Pump Module should be flushed thoroughly to remove any contaminant. Chloride containing eluents should not be pumped through the Dionex CERS 500 suppressor.

B. **Use chemicals and deionized water of the proper purity**. 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. **The system should be as metal-free as possible**. Gripper tubing fittings used in older systems are a potential source for metal contamination of the column. The new Dionex ThermoFlare or PEEK ferrule fittings are preferred. Inspect the eluent pumps periodically for any signs of leakage.

D. **Glass eluent reservoirs can be a source of sodium contamination in the eluent**. Two-liter polyethylene eluent reservoirs (P/N 039163) are preferred.

E. **For EG operation, use a Dionex CR-CTC 500 Trap Column**. Install a Dionex CR-CTC 500 Cation Trap Column (P/N 075551) if using an Eluent Generator with Dionex EGC MSA cartridge. Use the Dionex CR-CTC II (PN 072079) for capillary applications.

6.3.3 Sample Loop and/or Tubing Contamination

Eluents made with deionized water that is contaminated with bacteria and samples such as humic acids and soil extracts can potentially contaminate eluent lines and sample loops. Weak cation exchange sites are created on (or attached to) the tubing. This can happen to either Tefzel or PEEK tubing. Thus, the sample loop itself can act as a concentrator and depending on the pH of the sample or the standard and the way it is introduced; inaccurate readings for divalent analytes on weak cation exchange resins may be observed.

A. Weak Cation Exchangers

Carboxylated resins (used in the Thermo Scientific Dionex IonPac CS12, CS12A, CS14, CS15, CS16, CS17, CS18, CS19 and CS19-4µm) are weak acid cation exchangers. These resins have high selectivity for hydronium ion and are used with weak acid eluents. When the sample pH is high (pH 5), the weak cation exchange sites on the contaminated tubing are ionized and divalent cations are preferentially retained. When the sample pH is low (< pH 4), these sites are protonated by the sample and rendered inactive, so that the divalent quantification is not affected.

B. Testing for Loop Contamination when Using Carboxylated Cation Exchange Columns

A simple test can be performed (when using a column such as the Dionex IonPac CS19-4µm which contains a carboxylated resin) with methanesulfonic acid or sulfuric acid to see if the sample loop has been contaminated:

1. Prepare a standard containing 0.5 ppm of calcium and add a small amount of 0.2 mM sodium hydroxide so that the final pH of the standard is between 6.5 and 7.5.
2. With the sample loop in the load position, flush the loop with just enough standard to rinse and fill the loop (e.g. if the loop is 25 µL, flush it with no more than 100 µL).
3. Run the standard and record the peak area.
4. Repeat steps 2 and 3, but this time flush the loop with about 5 mL of standard.
5. If after repeating steps 2 through 4, the peak area for calcium recorded in 4 is significantly larger than that in 3, then the sample loop is contaminated and acting as a concentrator.
6. Replace the sample loop with new tubing and repeat this test.
7. If there is still a quantification problem, check other components of the system (tubing, injection valve, detector cell) or call your Dionex Products representative.

If you have a divalent quantification problem in your system but you neither have the time nor replacement parts, you can still get accurate results for divalent cations if any one of the following applies:

1. Your application involves high levels of divalent cations e.g. > 5 ppm calcium; the “concentration error” is small percentage-wise.
2. The pH of your samples and standards is < 4.
3. A constant volume of sample (and standard), only slightly larger than the sample loop, is flushed through the loop at a constant sampling flow rate.

6.4 High Background or Noise

In a properly working system, the background conductivity using the operating conditions described in Section 4, "Operation," should be < 0.3 µS with a Dionex suppressor. If the background is low but the system is noisy, an air bubble may be trapped in the suppressor. With the system running, disconnect the **ELUENT OUT** line from the suppressor and apply pressure to the open port with your gloved finger to dislodge a suspected bubble. Reconnect the line. Do not take too long to do this, as the current is still being applied to the Dionex suppressor and the eluent flow is needed to produce regenerant.

A. Check the conductivity flow cell for bubbles. See the conductivity detector manual for details.
   A system with a high background (> 0.5 µS) will probably also have high noise, resulting in increased detection limits.
B. Make sure that the eluents and regenerant are prepared correctly (see Section 6.2, "Eluent Preparation").
C. Determine if the columns or system are contaminated (see Section 6.3, "Contamination").
D. Determine if the Suppressor is the cause of the high background and/or noise. If the above items have been checked and the problem still persists, the suppression system is causing the problem. See Section 6.5, "Suppressor Not Suppressing Properly."

Typical background conductivity levels, in a properly working system, are shown below:

ELUENT	**EXPECTED BACKGROUND CONDUCTIVITY**
22 mN H_2SO_4 or 20 mN Methanesulfonic acid	< 0.5 µS
50 mN H_2SO_4 or Methanesulfonic acid	< 1 µS

6.5 Suppressor Not Suppressing Properly

If the Dionex Cation Electrolytically Regenerated Suppressor, Dionex Cation Capillary Electrolytic Suppressor, or the Dionex Cation MicroMembrane Suppressor is causing the problem, refer to the product manual for detailed troubleshooting assistance.

A. **Check that the Dionex CERS 500 suppressor is not in an alarm state**.

B. **Check for Dionex CERS 500 suppressor leaks**.

C. **Make sure that the correct back pressure tubing is properly installed after the Dionex CERS 500 suppressor**.

D. **Check the regenerant flow rate at the REGEN OUT port of the Dionex CERS 500 suppressor**. Turn the power to the Dionex CERS 500 suppressor off. Measure the regenerant flow rate. If it is being used in the recycle mode, it should be the same flow rate as the eluent (typically 1 mL/min for 4 mm operation or 0.25 mL/min for 2 mm operation or 0.010 mL/min for 0.4 mm operation). If the Dionex CERS 500 suppressor is used in the AutoSuppression External Water Mode, the regenerant flow rate should be 3-5 mL/min (4 mm) or 1-2 mL/min (2 mm).

E. **Check the eluent 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. Refer to the appropriate suppressor product manual for assistance in determining if the eluent is within suppressible limits.

F. **If you are using a Dionex AutoRegen Accessory with the Dionex CMMS suppressor, prepare fresh regenerant solution**. Test both the suppressor and the Dionex AutoRegen Regenerant Cartridge for contamination.

   1. If the background conductivity is high after preparing fresh regenerant and bypassing the Dionex AutoRegen Regenerant Cartridge, you probably need to clean or replace your Dionex CMMS 300.

   2. If the background conductivity is low when freshly prepared regenerant is run through the Dionex CMMS 300 without a Dionex AutoRegen Accessory in-line, test the Dionex AutoRegen Regenerant Cartridge to see if it is expended. Connect the freshly prepared regenerant to the Dionex AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the Dionex AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is high after placing the Dionex AutoRegen Accessory in-line, you probably need to replace the Dionex AutoRegen Regenerant Cartridge. Refer to the "Thermo Scientific Dionex AutoRegen Regenerant Cartridge Refill Product Manual" (Document No. 032852) for assistance.

NOTE

Do not recycle the regenerant through the Dionex Cation Regenerant Cartridge if the eluent contains acetonitrile.

A. Non-linear response or loss of sensitivity. Indications of carbonate contamination are:

1. A higher ammonium peak than should be expected.
2. Dips on either side of an analyte peak's base.

Non-linear response or loss of sensitivity may occur when the suppressor is contaminated with carbonate. This contamination is possibly from dissolved carbon dioxide in the DI water. Degassing will help minimize the presence of carbon dioxide in acidic eluents or in DI water. Note, when pressurizing eluent reservoirs on the system use inert gases such as nitrogen (aqueous applications) or helium.

When the Dionex CERS 500 suppressor is contaminated with carbonate the following treatment is recommended.

1. Push 5 mL of 2 M NaOH (freshly prepared) through the ELUENT IN port and divert a line from the ELUENT OUT port to waste.
2. Push 10 mL of 2 M NaOH (freshly prepared) through the REGEN IN port and divert a line out from the REGEN OUT port to waste.
3. Allow the suppressor to equilibrate for 20 minutes.
4. Repeat steps 1 and 2 with degassed DI water and reinstall the unit on the system.
5. If problem persists repeat steps 1–4.

6.6 Poor Peak Resolution

Poor peak resolution can be due any or all of the following factors.

6.6.1 Loss of Peak Efficiency throughout the Chromatogram

A. **Extra-column effects can result in sample band dispersion, causing loss of peak efficiencies**. Make sure you are using PEEK tubing with an i.d. of no greater than 0.010" for 4 mm systems or 0.005" for 2 mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks. Use only precut capillary tubing for capillary systems.

B. **Check to see if headspace has developed in the guard or analytical column**. This is usually due to improper use of the column such as submitting it to high pressures. Carefully not to disturb the resin bed, remove the column’s top end fitting (see Section 6.1.2, “Replacing Column Bed Support Assemblies”). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

6.6.2 Loss of Resolution throughout the Chromatogram Due to Shortened Retention Times

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

A. **Check the flow rate. See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol**. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

B. **Check to see if the eluent compositions and concentrations are correct**. An eluent with higher than intended concentration will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

C. **Column contamination can lead to a loss of column capacity**. This is because all of the cation exchange sites will no longer be available for the sample ions. For example, polyvalent cations 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.

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

6.6.3 Loss of Early Eluting Peak Resolution

Lack of equilibration to the initial eluent or improperly swept out of void volumes are usually the cause of poor resolution or efficiency of peaks eluting near the system void volume compared to the later eluting peaks.

A. **Be sure that the column is equilibrated to the initial eluent**. Typically, gradient applications require approximately 10 minutes (at standard flow rate conditions for the column format) to equilibrate to the initial eluent. The minimum equilibration time can be determined by making successive runs with increasing equilibration times. The column is equilibrated to the initial eluent when additional equilibration time does not increase the runtime of the first eluting peaks.

B. **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.

C. **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.

6.7 Spurious Peaks

A. **Eluents made with chemicals lacking the required purity will contaminate columns rapidly**. Remake all stock solutions and eluents using chemicals that meet the chemical requirements specified in Section 4.3, “Chemical Purity Requirements.” Clean the column as indicated in “Column Cleanup” (see Appendix B, “Column Care”).

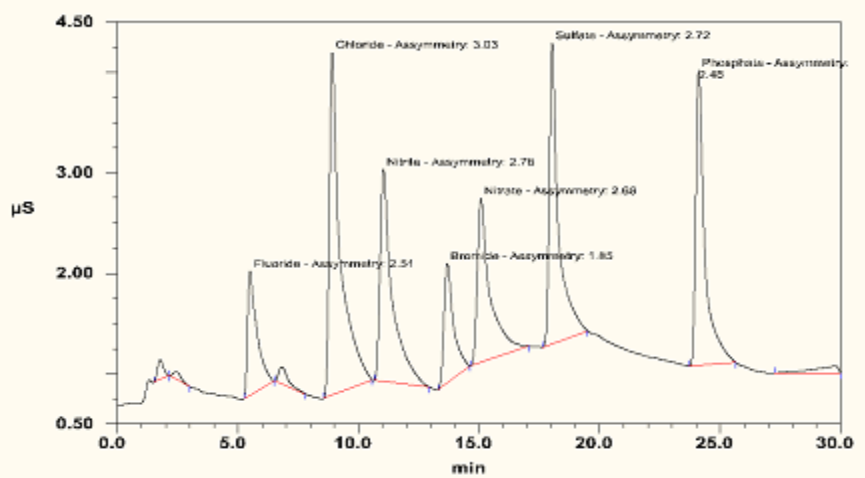
B. **Spurious peaks may be due to column contamination**. If the samples contain an appreciable level of polyvalent cations, polyvalent cations may contaminate the column. As a result, the retention times for the analytes will decrease, and spurious, inefficient peaks can show up at unexpected times. This problem may be solved by increasing the time between analyses or by adding a regeneration step between successive runs to elute polyvalent cationic contaminants off the column before the next sample injection takes place.

C. **An injection valve that needs service may produce baseline upsets**. This baseline upset can show up as one or multiple peaks of varying size(s) and shape(s). Typically this will occur when the particular valve needs to be cleaned or torqued (see the system manual). 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.

6.8 Poor Efficiency Using Capillary Columns

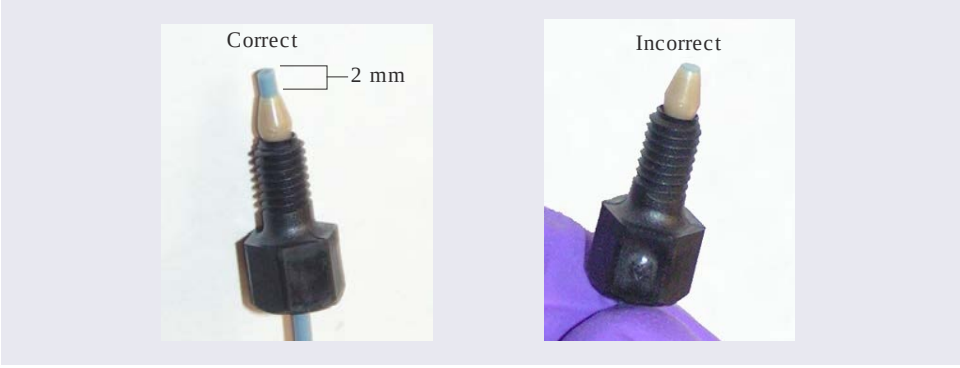
Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks.

Figure 26. 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. Insert the tubing hard and hold it in place while tightening the fitting. Figure 27 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing. If necessary to hold the ferrule and nut securely, turn the pump off while making capillary connections.

Figure 27. Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

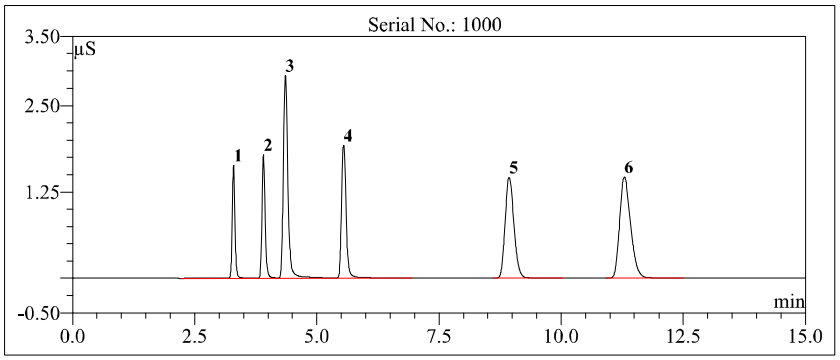


Appendix A – Quality Assurance Reports

Dionex IonPac™ CS19 4µm
Analytical (4 x 250 mm)
Product No. 078837

Date: 03-Sep-13 15:50
Serial No. : 001000
Lot No. : 2013-23-087

Eluent: 8 mM Methanesulfonic acid (MSA)
Eluent Source: Dionex EGC-MSA 500 Cartridge
Eluent Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Self-Regenerating Suppressor (Dionex CERS™ 500 4 mm) AutoSuppression™ Recycle Mode
Applied Current: 24 mA
Injection Volume: 10 µL
Storage Solution: Eluent (8 mM MSA)



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.29	1.2	5.70	16784	0.25
2	Sodium	3.91	1.1	3.16	18407	1.00
3	Ammonium	4.36	1.3	7.23	10729	1.25
4	Potassium	5.55	1.3	13.82	18754	2.50
5	Magnesium	8.94	1.3	6.34	11736	1.25
6	Calcium	11.29	1.5	n.a.	11852	2.50

QA Results:

Analyte	Parameter	Specification	Results
Potassium	Efficiency	>=10800	Passed
Potassium	Asymmetry	1.0-2.0	Passed
Magnesium	Efficiency	>=7650	Passed
Magnesium	Asymmetry	1.0-2.1	Passed
Calcium	Retention Time	10.10-12.30	Passed
	Pressure	<=3630	2842

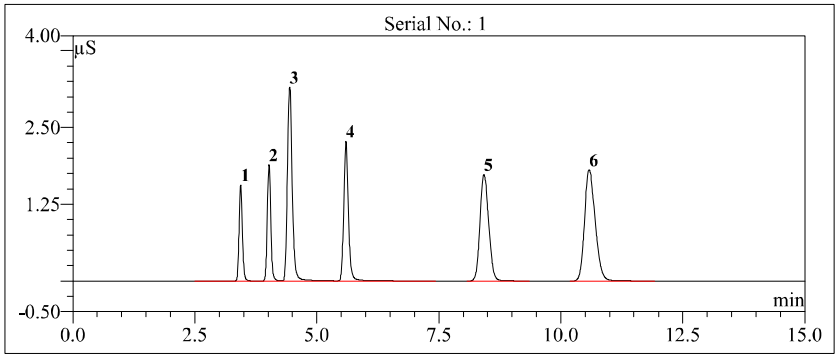
Production Reference:
Datasource: QAR
Directory: Cation\CS19_4µm
Sequence: CS19-4µM_4X250
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS19 4µm
Analytical (2 x 250 mm)
Product No. 078836

Date: 03-Sep-13 13:58
Serial No. : 000001
Lot No. : 2013-23-093

Eluent: 8 mM Methanesulfonic acid (MSA)
Eluent Source: Dionex EGC-MSA 500 Cartridge
Eluent Flow Rate: 0.25 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Self-Regenerating Suppressor (Dionex CERS™ 500 2 mm) AutoSuppression™ Recycle Mode
Applied Current: 6 mA
Injection Volume: 2.5 µL
Storage Solution: Eluent (8 mM MSA)



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.44	1.2	4.58	12564	0.25
2	Sodium	4.02	1.2	2.76	14945	1.00
3	Ammonium	4.44	1.2	6.58	9921	1.25
4	Potassium	5.59	1.3	11.41	16935	2.50
5	Magnesium	8.42	1.3	5.95	10719	1.25
6	Calcium	10.58	1.4	n.a.	11118	2.50

QA Results:

Analyte	Parameter	Specification	Results
Potassium	Efficiency	>=10800	Passed
Potassium	Asymmetry	1.00-2.0	Passed
Magnesium	Efficiency	>=7650	Passed
Magnesium	Asymmetry	1.00-2.1	Passed
Calcium	Retention Time	10.10-12.30	Passed
	Pressure	<=3630	2582

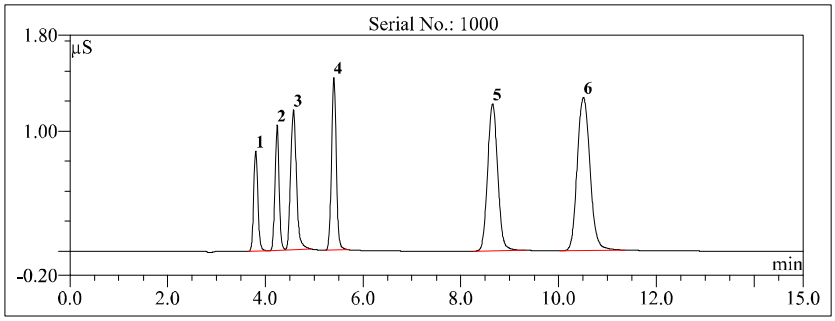
Production Reference:
Datasource: QAR
Directory: Cation\CS19_4µm
Sequence: CS19-4µm_2x250
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS19-4µm
Capillary (0.4 x 250 mm)
Product No. 078835

Date: 15-Feb-14 15:00
Serial No. : 001000
Lot No. : 2013-23-099

Eluent: 8 mM Methanesulfonic acid (MSA)
Eluent Source: Dionex EGC-MSA (Capillary)
Eluent Flow Rate: 10 µL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Capillary Electrolytic Suppressor (Dionex CCES 300)
AutoSuppression™ Recycle Mode
Applied Current: 6 mA
Injection Volume: 0.4 µL
Storage Solution: Eluent (8 mM MSA)



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.80	1.1	2.83	9713	0.06
2	Sodium	4.24	1.0	1.83	12027	0.25
3	Ammonium	4.58	1.1	4.23	7368	0.31
4	Potassium	5.40	1.1	11.79	14936	0.62
5	Magnesium	8.65	1.1	4.37	8462	0.31
6	Calcium	10.51	1.2	n.a.	7801	0.62

QA Results:

Analyte	Parameter	Specification	Results
Potassium	Efficiency	>=9900	Passed
Potassium	Asymmetry	1.00-2.0	Passed
Magnesium	Efficiency	>=6300	Passed
Magnesium	Asymmetry	1.00-2.1	Passed
Calcium	Retention Time	10.10-12.30	Passed
	Pressure	<=3630	1315

Production Reference:
Datasource: QAR
Directory: Cap\CS19-4µm
Sequence: CS19_0p4x250_4µm
Sample No.: 1

6.80 SR11 Build 3161 (184582) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Appendix B – Column Care

B.1 Recommended Operating Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the Dionex IonPac CS19-4µm Analytical, Capillary or Guard Column is 5,000 psi (34.46 MPa).

CAUTION

Do not use alcohols in the eluent.
Do not use the Dionex IonPac CS19-4µm column with basic eluents. This can significantly increase column back pressures.

B.2 Column Start-Up

The column is shipped using 8 mM Methanesulfonic acid as the storage solution. Prepare the eluent shown on the Quick Start procedure (Document No. 065591, see Appendix D) and follow the Quick Start instructions to hydrate the columns prior to running QAR. After the column gone though Quick Start hydration steps, connect the column to the suppressor and test the column performance under the conditions described in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

If peak efficiencies or resolution are poorer than the QAR, see Section 6, Troubleshooting, for information regarding possible causes and solutions.

B.3 Column Storage

For storage of the column, use 8 mM Methanesulfonic acid 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 two general isocratic protocols:

A. Polyvalent cations and acid soluble contaminants or transition metals
B. Hydrophobic cations and organic contaminants.

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 use low eluent flow rate (half of standard flow rate for the particular format), and include short column steps to reduce the solvent content of the eluent to < 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. This intermediate low concentration step will prevent precipitation or high viscosity zones. Avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

I. Column Cleanup Procedure for Polyvalent Cations and Acid-Soluble Contaminants or Transition Metals

A. **Prepare 500 mL of 1 M HCl for the cleanup solution**. Alternatively prepare 500 mM oxalic acid to remove transition metals such as iron or aluminum contamination.

CAUTION

Nitric acid should not be used instead of hydrochloric acid since nitric acid will not effectively remove iron contaminants. Do not clean the column with alcohols or with basic eluents.

B. **Disconnect the Suppressor from the Dionex IonPac CS19-4µm Analytical or Capillary Column**. If your system is configured with both a guard column and an analytical/capillary column, place the guard after the analytical/capillary column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.

NOTE

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

CAUTION

DO NOT pump hydrochloric acid through the Dionex CERS 500 or the CCES 300 suppressor.

C. **Set the pump flow rate to 1.0 mL/min** for a Dionex IonPac CS19-4µm 4 mm Analytical or Guard Column, to 0.25 mL/min for a Dionex IonPac CS19-4µm 2 mm Analytical or Guard Column, and to 0.010 mL/min for a capillary Dionex IonPac CS19-4µm 0.4 mm Analytical or Guard Column.

D. **Rinse the column for 15 minutes with eluent** (8 mM MSA) before pumping the chosen cleanup solution over the column.

E. **Pump the cleanup solution** (1 M HCl or 500 mM oxalic acid) through the column for 60 minutes.

F. **Rinse and equilibrate the column(s)** with 8 mM MSA eluent for at least 60 minutes before resuming normal operation (send effluent to waste).

G. **Reconnect the Suppressor** to the Dionex IonPac CS19-4µm Analytical or Capillary Column and place the guard column in line between the injection valve and the analytical or capillary column if your system was originally configured with a guard column.

H. **Equilibrate the system with eluent** before resuming normal operation.

II. Hydrophobic Cations and Organic Contaminants

A. **Disconnect the analytical/capillary column** from the injection valve and the suppressor. Disconnect the Gradient Mixer or the Dionex Cation Trap from the pump. Connect the Dionex IonPac CS19-4µm Analytical/Capillary Column directly to the pump. Direct the effluent from the analytical/capillary column directly to a waste container.

B. **Set the pump flow rate to 1.0 mL/min** for a Dionex IonPac CS19-4µm 4 mm Analytical or Guard Column, to 0.25 mL/min for a Dionex IonPac CS19-4µm 2 mm Analytical or Guard Column, and to 0.010 mL/min for a capillary Dionex IonPac CS19-4µm 0.4 mm Analytical or Guard Column.

C. **Use the following gradient program** to remove hydrophobic cations and organic contaminants.

Eluent 1: 100 mM HCl
Eluent 2: 90% Acetonitrile in deionized water

Time (min)	**% E1**	**% E2**
0.0	100	0
20.0	90	10
65.0	90	10
75.0	100	0
95.0	100	0

D. **Rinse and equilibrate the column(s)** with 8 mM MSA eluent for at least 60 minutes before resuming normal operation.

E. **Reconnect the Dionex IonPac CS19-4µm column**. Connect the Analytical/Capillary Column outlet to the Suppressor and the inlet to either the Dionex IonPac CG19-4µm Guard Column or the Pump Module.

F. **Equilibrate the column** with eluent before resuming normal operation.

Appendix C – Configuration

C.1 Configuration of Ion Chromatography (IC) Systems

Table 7. Configuration of Ion Chromatography Systems

Condition	2 mm System Operation Summary	4 mm System Operation Summary	0.4 mm System Operation Summary
Eluent Flow Rate	Typically 0.25 mL/min	Typically 1.0 mL/min	Typically 10 µL/min
Dionex Cation Electrolytically Regenerated Suppressor	Dionex CERS 500 2 mm (P/N 082543)	Dionex CERS 500 4 mm (P/N 082542)	Dionex CCES 300 (P/N 072053)
Dionex Cation MicroMembrane Suppressor 300	Dionex CMMS 300 2 mm (P/N 064561)	Dionex CMMS 300 4 mm (P/N 0064560)	N/A
Regenerant Flow Rate	Typically 50-100% of 4 mm System, 2.5 – 4 mL/min	Typically 10-15 mL/min	N/A
Injection Loop	5-25 µL	10-50 µL	0.4 µL
System Void Volume	Eliminate switching valves, couplers and the Dionex GM-3 Gradient Mixer. Use only the Dionex Microbore GM-4 (2 mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the Dionex GM-2, Dionex GM-3, Dionex GM-4 or recommended gradient mixers.	Use only on an IC System equipped for capillary analysis.
Pumps	Use the Dionex ICS-5000$^+$ HPIC system in Microbore Configuration.	Use the Dionex ICS-5000$^+$ HPIC system in Standard bore Configuration.	Use the Dionex ICS-5000$^+$ HPIC or Dionex ICS-4000 HPIC capillary systems.
Note **For Dionex IonPac CS19-4µm 2 mm and 4 mm column applications using eluent generation, a Dionex CR-CTC 500 (P/N 075551) may be installed at the EGC eluent outlet to remove trace level cationic contaminants. For capillary applications, use the Dionex CR-CTC II (Capillary), P/N 072079.**			
Chromatographic Module	A thermally controlled column oven such as the Dionex, ICS-5000$^+$DC	A thermally controlled column oven such as the Dionex ICS-5000$^+$ DC	A thermally controlled column compartment such as the Dionex ICS-5000$^+$ DC equipped with the Dionex IC-Cube.
Detectors	DionexCD20, CD25, CD25A, ED40, ED50 or ED50A Dionex Conductivity Cell with DS3 P/N 044130 or Dionex Conductivity Cell with Dionex P/N 061830 Dionex AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) Ensure 30-40 psi back pressure.	Dionex CD20, CD25, CD25A, ED40, ED50 or ED50A Dionex Conductivity Cell with DS3 P/N 044130 or Dionex Conductivity Cell with Dionex P/N 061830 Dionex AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) Ensure 30-40 psi back pressure.	Use only a conductivity detector designed for capillary flow rates such as the Dionex ICS-5000$^+$ Capillary CD.

C.2 Tubing Back Pressures

Table 8. Tubing Back Pressures for Suppressed IC

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	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642
Light Blue	071870	0.0025	0.006	0.009	1766.0	441.0	58.0

Appendix D – Column Quick Start Procedure

D.1 Overview

The Thermo Scientific™ Dionex™ IonPac™ CS19 and CS19-4µm columns are used with suppressed conductivity detection for the analysis of the common inorganic cations (Lithium, Sodium, Ammonium, Potassium, Magnesium, and Calcium) as well as small polar amines. Conditioning of the column bed by following this Quick Start procedure, is **required prior to initial use and after long-term storage**. The Quick Start procedure will ensure extended column lifetime and reproducibility. This procedure also prevents any storage solution or column residuals from flowing to the suppressor, extending its lifetime and expediting the system equilibration time. For 4 mm and 2 mm columns follow the procedure in section 2. For capillary columns, follow the procedure in section 3. Section 4 describes an alternate conditioning program for capillary columns if an offline pump is available.

WARNING

If your mobile phase composition generates back pressure in excess of the maximum operation pressure, reduce the flow rate to ensure the upstream back pressure of the column is less than the maximum operation pressure.

The maximum pressure limit for the Dionex IonPac CS19 column is 3000 psi (20.7 MPa). The maximum pressure limit for the Dionex IonPac CS19-4µm column is 5000 psi (34.5 MPa).

D.2 Gradient Program for Dionex IonPac CS19 and CS19-4µm 2×250 mm and 4×250 mm columns

a. After connecting the inlet of the column to the IC system in the correct flow direction, direct the outlet of the column to waste.
b. Pump eluent through the column using the conditioning flow rates and gradient program below.
c. Proceed to section 5 to test the column under Quality Assurance Report conditions.

Note: ***If an offline gradient pump is not available, be sure to bypass the EGC and connect the pump directly to the injection valve or column***

Conditioning flow rate for 4 mm columns is 0.5 mL/min.
Conditioning flow rate for 2 mm columns is 0.13 mL/min.

Time	%E1 DI Water	%E2 80 mM MSA	%E3 95% Acetonitrile
0	90	10	0
5	90	10	0
10	80	10	10
60	80	10	10
65	90	10	0
75	90	10	0

D.3 Isocratic Program for Dionex IonPac CS19 and CS19-4µm 0.4×250 mm Column

a. Prepare 8 mM MSA with10% acetonitrile in DI water (50 mL is sufficient).
b. Bypass the EGC and prime the pump using this solution.
NOTE: Do not pump acetonitrile through the EGC. Acetonitrile is not compatible with the EGC.
c. After priming, connect the column directly to the pump and direct the outlet of the column to waste.
d. Pump this solution through the column directly to waste for one hour at 10 µL/min.
e. Disconnect the column and change the 8 mM MSA with10% acetonitrile solution to DI water only.
f. Prime the pump with DI water to remove the acetonitrile and MSA, and then pump DI water through the lines for 60 minutes at 0.1 mL/min..
g. Connect the pump back to the EGC.
h. Reconnect the column to the injection valve directing the column outlet to waste.
i. Using the EGC, pump 8 mM MSA through the column directly to waste for one hour at 10 µL/min.
j. Proceed to section 5 to test the column under Quality Assurance Report conditions.

D.4 Alternate Program for *Dionex IonPac CS19 and CS19-4µm 0.4×250 mm* Column if an offline gradient pump is available

a. Set up the eluent bottles as described in section 2.
b. 8 mM MSA/9.5% acetonitrile eluent: Prime pump with 8 mM MSA (10% of E2) and 9.5% acetonitrile (10% of E3). After priming, pump 10 mL through the mixer at 2 mL/min (rinse out the mixer at standard flow rate). Connect the Dionex IonPac CS19 or CS19-4µm 0.4×250 mm column and wash with this eluent (8 mM MSA/9.5% acetonitrile) for one hour at 10 µL/min. Continue to step b.
c. 8 mM MSA eluent: Remove the capillary column from the pump and prime pump with 8 mM MSA (10% of E2). After priming, pump 10 mL through the mixer at 2 mL/min (rinse out the mixer at standard flow rate). Reconnect the Dionex IonPac CS19 or CS19-4µm 0.4×250 mm and wash with this eluent (8 mM MSA (10% of E2) for one more hour at 10 µL/min.
d. Proceed to section 5 to test the column under Quality Assurance Report conditions.

D.5 Quality Assurance Report

Once the column conditioning is complete, connect the column outlet to the suppressor. The last eluent concentration used in the conditioning step is 8 mM MSA, the same eluent used in the Quality Assurance Report (QAR). Test the column performance under the conditions in the QAR. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Column equilibration is complete when consecutive injections of the standard give reproducible retention times.

D.6 Storage

Store the column in 8mM Methanesulfonic acid and seal both ends immediately with column plugs to avoid drying of the column.

Tip ***For additional information, please refer to the Dionex IonPac CS19 Product Manual (Document No. 065440) or the Dionex IonPac CS19-4µm Product Manual (Document No. 065472).***