Dionex IonPac CS16-4µm and Dionex IonPac CS16-Fast-4µm

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Thermo SCIENTIFIC

Product Manual for

Dionex IonPac CS16-4µm Analytical Column

088584 IP CS16-4µm 4×250 mm
088582 IP CS16-4µm 2×250 mm
088599 IP CS16-Fast-4µm 4×150 mm
088601 IP CS16-Fast-4µm 2×150 mm

Dionex IonPac CS16-4µm Capillary Column

088615 IP CS16-4µm 0.4×250 mm
088641 IP CS16-Fast-4µm 0.4×150 mm

Dionex IonPac CG16-4µm Guard Column

088585 IP CG16-4µm 4×50 mm
088583 IP CG16-4µm 2×50 mm
088600 IP CG16-Fast-4µm 4×30 mm
088602 IP CG16-Fast-4µm 2×30 mm

Dionex IonPac CG16-4µm Capillary Guard Column

088616 IP CG16-4µm 0.4×50 mm
088642 IP CG16-Fast-4µm, 0.4×35 mm

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

Revision History:

Revision 01, September, 2015, Original Publication.

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™ CS16-4µm and CS16-Fast-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. Due to its smaller particle size, the IonPac™ CS16-4µm and CS16-Fast-4µm column offers higher peak efficiencies than its predecessor, the IonPac™ CS16 column. It is offered in 4 mm, 2 mm, and capillary (0.4 mm) i.d. formats, and in 250 mm lengths (Thermo Scientific™ Dionex™ IonPac™ CS16-4µm) as well as 150 mm length (Thermo Scientific™ Dionex™ IonPac™ CS16-Fast-4µm) for faster operation. Each specific column separator format has its own guard column format.

NOTE

The standard flow rates used with the IonPac CS16-4µm and CS16-Fast-4µm analytical formats are lower than those used for the IonPac CS16 analytical formats.

The IonPac CS16-4µm and CS16-Fast-4µm 4-mm and 2-mm Analytical Columns and the CS16-4µm and CS16-Fast-4µm 0.4 mm Capillary Columns have been designed specifically for the analysis of alkali metals, alkaline earth metals, and ammonium at diverse concentration ratios. Similar to the IonPac CS16, the CS16-4µm and CS16-Fast-4µm stationary phase is a high-capacity weak cation exchanger functionalized with carboxylic acid groups having a high selectivity for hydronium ion. It has both cation exchange and reverse phase properties. The CS16-4µm and CS16-Fast-4µm are solvent-compatible with 100% aqueous eluents and 100% acetonitrile without loss of performance.

The Dionex IonPac CS16-4µm and CS16-Fast-4µm stationary phase has a higher cation exchange capacity per gram of resin than the Thermo Scientific Dionex IonPac CS12A, CS15, CS17, CS18, CS19 and the Thermo Scientific Dionex IonPac CS19-4µm columns. It has the same cation exchange capacity per gram of resin as the IonPac CS16 column, but because the column i.d. of the CS16-4µm and the CS16-Fast-4µm is smaller than its counterpart CS16 column (for the analytical formats), the total meq/column is smaller for these. Performance as to cation exchange capacity is maintained by using a smaller sample injection volume.

An advantage of having a smaller i.d. column format is that operational flow rate is reduced. Thus, consumables (eluent generator cartridge, cation trap column) should last longer before needing replacement; waste is reduced; and it is possible to use higher MSA concentrations as the eluent generator will support it and the suppressor will require lower currents.

The Dionex IonPac CS16 column and the Dionex IonPac CS16-4µm and CS16-Fast-4µm column chemistries are identical, providing the same selectivity. The only difference is the substrate particle size and the column format (length and i.d.). The Dionex IonPac CS16-4µm, having a smaller particle size, gives about 50% higher peak efficiencies than its 250 mm long CS16 counterparts. 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 CS16 column that happens to have much better peak efficiencies than its minimum specification, may perform as well as or better than a Dionex IonPac CS16-4µm column that is at the specification limits. The IonPac CS16-4µm columns offer a much higher peak efficiency specification than the standard IonPac CS16 column.

Due to its shorter length (150 mm long), the Dionex IonPac CS16-Fast-4µm columns have proportionately lower peak efficiency, lower backpressure, and shorter run times than the Dionex IonPac CS16-4µm (250 mm long).

CAUTION

Do not use alcohols in the eluent.
Formation of esters will occur in the column packing.
This can significantly reduce the column capacity for cation exchange.

The Dionex IonPac CS16-4µm and CS16-Fast-4µm column can be used without loss of performance up to 70 °C.

The Dionex IonPac CS16-4µm and CS16-Fast-4µm column can be washed with up to 1 M acid concentration. The Dionex IonPac CS16-4µm and CS16-Fast-4µm column should not be used with basic eluents. The column backpressure increases too much, disrupting the packing. The Dionex IonPac CS16-4µm and CS16-Fast-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 CS16-4µm and CS16-Fast-4µm capillary column (0.4 mm i.d.) 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. When the column is operated at its highest flow rate (10 µL/minute), the eluent generator can still provide a maximum methanesulfonic acid (MSA) concentration of 200 mM MSA, allowing faster 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 cations, where fast flow rates and high MSA eluent concentrations are required, the capillary Dionex IonPac CS16-4µm column is recommended over the 2 and 4 mm column formats.

The Dionex IonPac CG16-4µm guard column is made with the same resin as the analytical CS16-4µm columns.

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 4200 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. 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 40˚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.

Applications: Over the years, Thermo Fisher Scientific Dionex has developed several polymeric cation exchange columns specifically for the determination of amines, such as the Thermo Scientific Dionex IonPac CS17, CS18, CS19 and the CS19-4µm columns. For amine applications it is therefore highly recommended that you look into these columns first. The IonPac® CS16-4µm and CS16-Fast-4µm columns have been designed specifically for the analysis of alkali metals, alkaline earth metals, and ammonium at ***diverse concentration ratios***. If the matrix is such that a high capacity column is needed, if the sample has high-to-low concentration ratios of adjacent peaks, or if a different selectivity is needed to resolve a certain pair of amines, the IonPac CS16-4µm or CS16-Fast-4µm column could be the column of choice.

Similarly to the IonPac CS16, due to its high cation exchange capacity the CS16-4µm and CS16-Fast-4µm column can tolerate up to 100 mM hydronium ion (pH 1.0) in the sample with minimum loss of performance. Other lower capacity carboxylic acid functionalized columns can tolerate up to 20 mM acid in the sample.

Samples of lower pH can be pretreated before injection with an OnGuard II A cartridge in the bicarbonate form. Anions in the sample will be exchanged for the bicarbonate in the OnGuard resin. The bicarbonate ions neutralize the hydronium ions in the sample.

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 CS16-4µm/CG16-4µm and CS16-Fast-4µm/CG16-Fast-4µm Packing Specifications

Column	Nominal Particle Diameter µm	Substrate	Column Capacity µeq/column	Functional Group	Hydrophobicity
Dionex IonPac CS16-4µm Capillary Column 0.4 × 250 mm	4	Macroporous	50	Carboxylic acid	Medium
Dionex IonPac CG16-4µm Capillary Guard Column 0.4 × 50 mm	4	Macroporous	10	Carboxylic acid	Medium
Dionex IonPac CS16-4µm Analytical Column 2 × 250 mm	4	Macroporous	1340	Carboxylic acid	Medium
Dionex IonPac CG16-4µm Guard Column 2 × 50 mm	4	Macroporous	270	Carboxylic acid	Medium
Dionex IonPac CS16-4µm Analytical Column 4 × 250 mm	4	Macroporous	5370	Carboxylic acid	Medium
Dionex IonPac CG16-4µm Guard Column 4 × 50 mm	4	Macroporous	1070	Carboxylic acid	Medium
Dionex IonPac CS16-Fast-4µm Capillary Column 0.4 × 150 mm	4	Macroporous	30	Carboxylic acid	Medium
Dionex IonPac CG16-Fast-4µm Capillary Guard Column 0.4 × 35 mm	4	Macroporous	5	Carboxylic acid	Medium
Dionex IonPac CS16-Fast-4µm Analytical Column 2 × 150 mm	4	Macroporous	800	Carboxylic acid	Medium
Dionex IonPac CG16-Fast-4µm Guard Column 2 × 30 mm	4	Macroporous	160	Carboxylic acid	Medium
Dionex IonPac CS16-Fast-4µm Analytical Column 4 × 150 mm	4	Macroporous	3220	Carboxylic acid	Medium
Dionex IonPac CG16-Fast-4µm Guard Column 4 × 30mm	4	Macroporous	650	Carboxylic acid	Medium

Table 2. Dionex IonPac CS16-4µm/CG16-4µm Operating Parameters for 250 mm and 50 mm formats

Column	Typical Back Pressure at Standard Flow Rate psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate* mL/min
Dionex IonPac CS16-4µm 0.4 x 250 mm Capillary Column	≤ 2,600 (17.93)	0.006	0.010
Dionex IonPac CG16-4µm 0.4 x 50 mm Capillary Guard Column	≤600 (4.14)	0.006	0.010
Dionex IonPac CS16-4µm + CG16-4µm 0.4 mm Capillary and Capillary Guard Columns, 250 & 50 mm	≤ 3,200 (22.07)	0.006	0.010
Dionex IonPac CS16-4µm 2 x 250 mm Analytical Column	≤ 2,600 (17.93)	0.16	0.25
Dionex IonPac CG16-4µm 2 x 50 mm Guard Column	≤ 600 (4.14)	0.16	0.25
Dionex IonPac CS16-4µm + CG16-4µm 2 mm Analytical and Guard Columns, 250 & 50 mm	≤ 3,200 (22.07)	0.16	0.25
Dionex IonPac CS16-4µm 4 x 250 mm Analytical Column	≤ 2,600 (17.93)	0.64	1.0
Dionex IonPac CG16-4µm 4 x 50 mm Guard Column	≤ 600 (4.14)	0.64	1.0
Dionex IonPac CS16-4µm + CG16-4µm 4 mm Analytical and Guard Columns, 250 & 50 mm	≤ 3,200 (22.07)	0.64	1.0

NOTE: Maximum Flow Rate (mL/min) defined in this context is the maximum flow rate that the column can withstand without loss of performance.

Table 3. Dionex IonPac CS16-Fast-4µm/CG16-Fast-4µm Operating Parameters for 150 mm and 30 mm formats

Column	Typical Back Pressure at Standard Flow Rate psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate* mL/min
Dionex IonPac CS16-Fast-4µm 0.4 x 150 mm Capillary Column	≤ 1,560 (10.75)	0.006	0.010
Dionex IonPac CG16-Fast-4µm 0.4 x 35 mm** Capillary Guard Column	≤450 (3.10)	0.006	0.010
Dionex IonPac CS16-Fast-4µm + CG16-4µm 0.4 mm Capillary and Capillary Guard Columns, 150 & 35 mm	≤ 2,010 (13.86)	0.006	0.010
Dionex IonPac CS16-Fast-4µm 2 x 150 mm Analytical Column	≤ 1,560 (10.75)	0.16	0.25
Dionex IonPac CG16-Fast-4µm 2 x 30 mm Guard Column	≤360 (2.48)	0.16	0.25
Dionex IonPac CS16-Fast-4µm + CG16-Fast-4µm 2 mm Analytical and Guard Columns, 150 & 30 mm	≤ 1,920 (13.24)	0.16	0.25
Dionex IonPac CS16-Fast-4µm 4 x 150 mm Analytical Column	≤ 1,560 (10.75)	0.64	1.0
Dionex IonPac CG16-Fast-4µm 4 x 30 mm Guard Column	≤360 (2.48)	0.64	1.0
Dionex IonPac CS16-Fast-4µm + CG16-Fast-4µm 4 mm Analytical and Guard Columns, 150 & 30 mm	≤ 1,920 (13.24)	0.64	1.0

NOTE: Maximum Flow Rate (mL/min) defined in this context is the maximum flow rate that the column can withstand without loss of performance.
****NOTE: Due to hardware limitations, the capillary guard column is 35 mm long instead of 30 mm.***

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 CS16-4µm columns consist of 4 µm polymeric particle substrate, which translates into higher peak efficiencies than the Dionex IonPac CS16 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 comfortably operate the analytical columns and their guards at their standard and maximum recommended flow rates. Similarly, a Dionex ICS-5000$^+$ Capillary HPIC system is required to comfortably operate the capillary columns and their guards.

- 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 is recommended.

The Dionex IonPac CS16-Fast-4µm columns, being of shorter length (150 mm), have lower backpressures and can be operated as well in an IC system capable of running up to 3000 psi, such as the Dionex ICS-3000 system. However, if higher operational flow rates are needed, the Dionex ICS-5000$^+$ HPIC system should be used for these as well.

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 30 mM Methanesulfonic acid as the storage solution. Prepare the eluent shown on the Quick Start procedure (see Appendix D) and follow the Quick Start instructions to hydrate the columns prior to running QAR. After the column has gone through 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.

For columns that have not been used for some time, peak efficiencies may start lower than the QAR but will improve as the column is run. The column may need to be run overnight under QAR conditions for optimum peak efficiencies.

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 30 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 CS16-4µm and CS16-Fast-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 0.062 mm tubing with the capillary systems.

3.2.2 System Requirements for 2 mm and 4 mm Operation

The Dionex IonPac CS16-4µm 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.

The Dionex IonPac CS16-Fast-4µm columns, being of shorter length (150 mm), have lower backpressures and can be operated as well in an IC system capable of running up to 3000 psi, such as the Dionex ICS-3000 system. However, if higher operational flow rates are needed, the Dionex ICS-5000$^+$ HPIC system should be used for these as well.

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 042690) 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 CS16-4µm and CS16-Fast-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 075551) 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 Continuously Regenerated Cation Trap Column (Capillary), P/N 072079. See the Dionex CR-TC Product Manual (Document No. 079684) for instructions. The capillary system should only be used with an eluent generator and a Dionex CR-CTC (Capillary) 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. Dilution of the sample should be done in those cases. 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 CS16-4µm and CS16-Fast-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 CS16-4µm and the Dionex IonPac CS16-Fast-4µm 4 mm and 2 mm Analytical Columns. For trace cation concentration with the Dionex IonPac CS16-4µm and the CS16-Fast-4µm 0.4 mm Column, use the Dionex IonSwift MCC-100 Concentrator Column (0.5 × 80 mm, P/N 075462).

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 CS16-4µm or CS16-Fast-4µm column. The Dionex TCC-2 column packing is a strong cation exchange resin functionalized with sulfonic acid. The recommended Dionex IonPac CS16-4µm and CS16-Fast-4µm column eluents will not properly elute ions concentrated on this column.

3.6 Dionex IonPac CG16-4µm and CG16-Fast-4µm Guard/Capillary Guard Column

A Dionex IonPac CG16-4µm and CG16-Fast-4µm Guard/Capillary Guard Column is normally used with the Dionex IonPac CS16-4µm and CS16-Fast-4µm Analytical/Capillary Column respectively. The Dionex IonPac CG16-4µm and CG16-Fast-4µm guard column is packed with the same resin as the separator 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 CS16-4µm and CS16-Fast-4µm columns are designed to be used with acid eluent systems. If manually prepared eluents are used (instead of Electrolytically Generated), it is recommended that storage of the eluent be under a helium atmosphere to ensure contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain organic 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 CS16-4µm and CS16-Fast-4µm 0.4 mm Capillary Column, use the Dionex CCES 300 (0.4 mm, P/N 072053).
For Dionex IonPac CS16-4µm and CS16-Fast-4µm 4 mm Analytical Column, use the Dionex CERS 500 (4 mm, P/N 082542).
For Dionex IonPac CS16-4µm and CS16-Fast-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

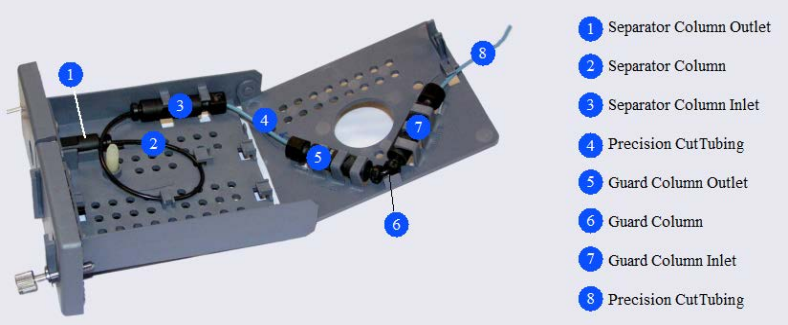
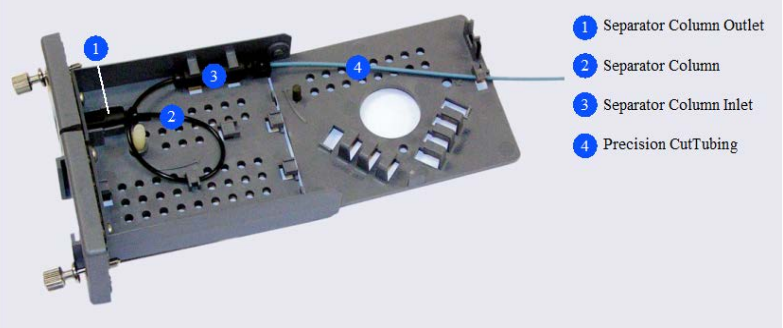


Figure 2 Separator Column Only Installed in Column Cartridge



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	125 mm (4.92 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)
60-082647	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)
044221	0.125-mm (0.005-in) ID PEEK tubing, red	610 mm (24 in)	Injection valve to waste
074449	Fitting bolt, 10-32 hex double-cone, blue	7	Connect precision cut 0.062 mm (0.0025-in) ID PEEK tubing
074373	Ferrule fitting, 10-32 double-cone, blue	7	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

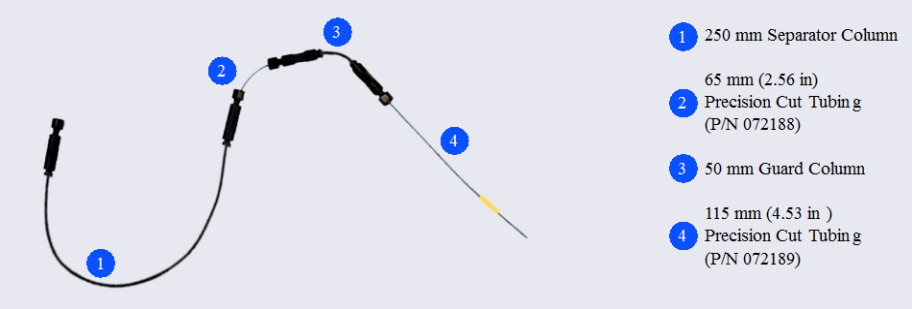
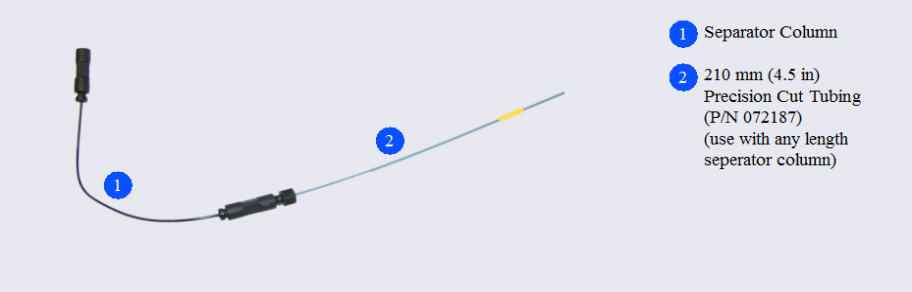
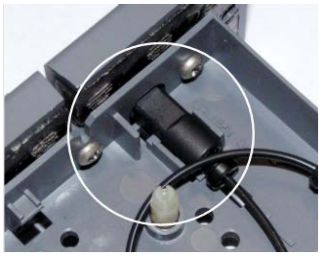


Figure 4 Tubing Connections for Separator Column Only



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 CS16-4µm 0.4 mm Capillary Column + Dionex IonPac CG16-4µm 0.4 mm Capillary Guard Column
	0.4 mm:	Dionex IonPac CS16-Fast-4µm 0.4 mm Capillary Column + Dionex IonPac CG16-Fast-4µm 0.4 mm Capillary Guard Column
	2 mm:	Dionex IonPac CS16-4µm 2 mm Analytical Column + Dionex IonPac CG16-4µm 2 mm Guard Column
	2 mm:	Dionex IonPac CS16-Fast-4µm 2 mm Analytical Column + Dionex IonPac CG16-Fast-4µm 2 mm Guard Column
	4 mm:	Dionex IonPac CS16-4µm 4 mm Analytical Column + Dionex IonPac CG16-4µm 4 mm Guard Column
	4 mm:	Dionex IonPac CS16-Fast-4µm 4 mm Analytical Column + Dionex IonPac CG16-Fast-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:		30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.4 mm:	0.006 mL/min
	2 mm:	0.16 mL/min
	4 mm:	0.64 mL/min
Temperature:		40 °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 CS16-4µm and CS16-Fast-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: 100% for column clean up (acetonitrile, acetone).***
- ***Maximum Flow Rate: 0.010 mL/min for 0.4 mm columns.***
  0.25 mL/min for 2 mm columns.
  1.0 mL/min for 4 mm columns.
- ***Column Temperature Range: 15 to 70 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

Organic solvents can be added to the ionic eluents used with Dionex IonPac CS16-4µm and CS16-Fast-4µm columns to modify the analytes retention in the column, to improve sample solubility, or to clear the column from hydrophobic contaminants. The organic 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 organic solvents available be used. Currently, several manufacturers make ultrahigh purity organic solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity organic 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 an organic 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 CS16-4µm and CS16-Fast-4µm columns is 5,000 psi (34.47 MPa). The Dionex IonPac CS16-4µm and CS16-Fast-4µm column is compatible with the HPLC solvents listed in Table 4, "HPLC Solvents for Use with the Dionex IonPac CS16-4µm and CS16-Fast-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.
It is recommended to reduce by half the standard flow rate for the format when the eluent contains an organic solvent.

Table 4. HPLC Solvents for Use with Dionex IonPac CS16-4µm and CS16-Fast-4µm Columns

Solvent	Maximum Operating Concentration
Acetonitrile	100%
Acetone	100%
Alcohol	0%

CAUTION

Do NOT use alcohols as an eluent component with the Dionex IonPac CS16-4µm and CS16-4µm 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 057558) 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 organic 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 organic solvent. First equilibrate the column with 5 percent of the current solvent for approximately 5 minutes. Next run a 10-minute gradient from the eluent with 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).

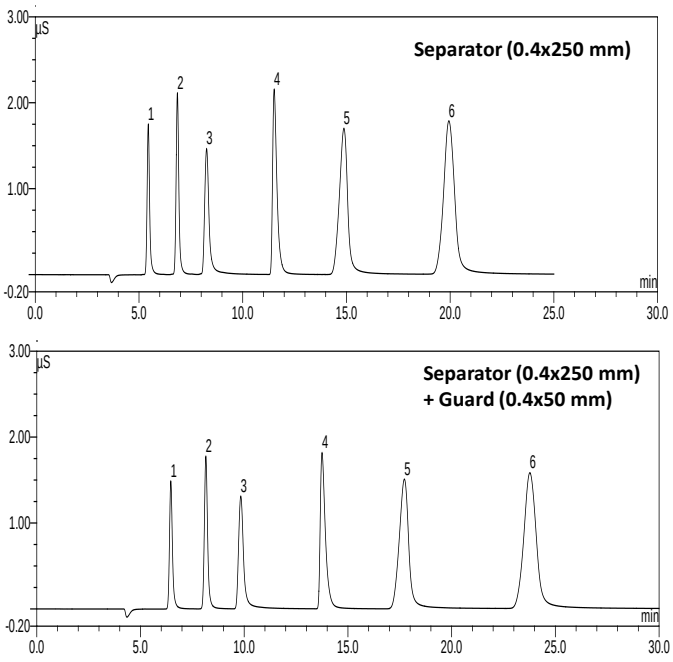
Consult the appropriate suppressor manual for the suppressor operation when organic solvent is present in the eluent.

5. Example Applications

5.1 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-4µm Column with and without Dionex IonPac CG16-4µm Guard Column

The chromatograms below show the separation of the common cations plus ammonium ion using the Dionex IonPac CS16-4µm column with and without a Dionex IonPac CG16-4µm guard column. The Dionex IonPac CG16-4µm guard column has the same capacity per gram of resin than the Dionex IonPac CS16-4µm separator column. In the case of the 250 mm length separator, using the 50 mm long CG16-4µm column (of the same i.d. as the separator) adds about 5 minutes to the total retention time.

Figure 7 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-4µm Column (0.4 × 250 mm), with and without Dionex IonPac CG16-4µm Guard Column (0.4 × 50 mm)

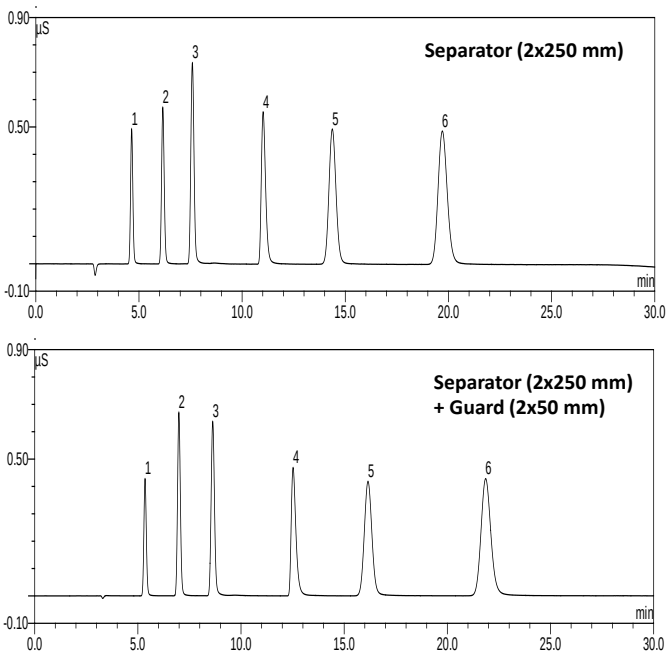


Columns:	Dionex IonPac CS16-4µm & Dionex IonPac CG16-4µm (0.4 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	6 µL/min
Inj. Volume:	400 nL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

Figure 8 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-4µm Column (2 × 250 mm), with and without Dionex IonPac CG16-4µm Guard Column (2 × 50 mm)

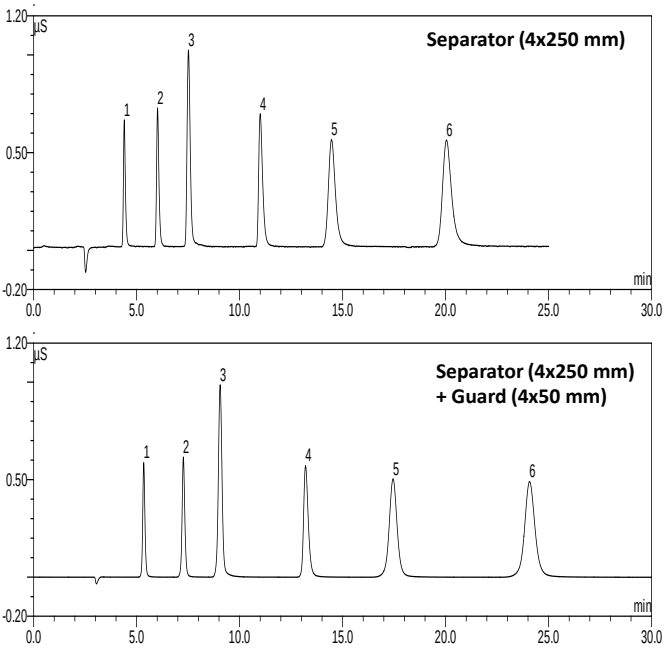


Columns:	Dionex IonPac CS16-4µm & Dionex IonPac CG16-4µm (2 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

Peaks:

1. Lithium 0.10 mg/L
2. Sodium 0.40
3. Ammonium 0.50
4. Potassium 1.00
5. Magnesium 0.50
6. Calcium 1.00

Figure 9 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-4µm Column (4 × 250 mm), with and without Dionex IonPac CG16-4µm Guard Column (4 × 50 mm)



Columns:	Dionex IonPac CS16-4µm & Dionex IonPac CG16-4µm (4 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.64 mL/min
Inj. Volume:	15 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4-mm, AutoSuppression, recycle mode

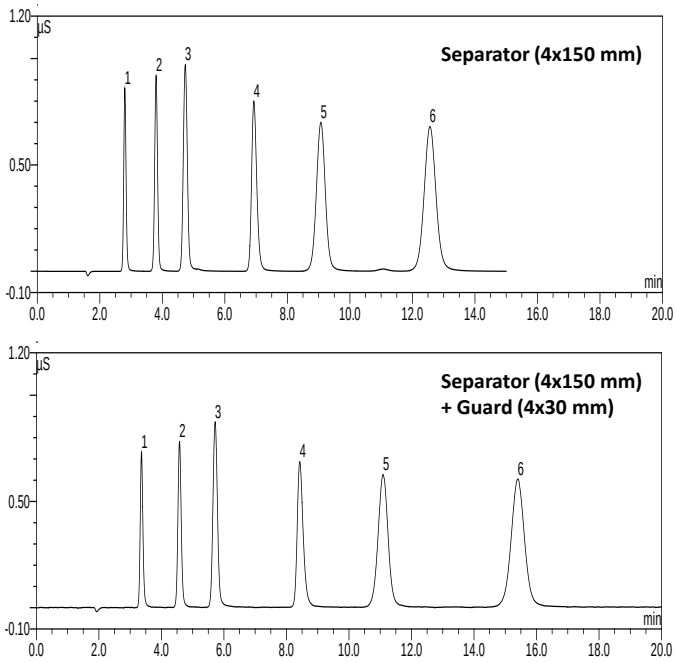
Peaks:

1. Lithium 0.10 mg/L
2. Sodium 0.40
3. Ammonium 0.50
4. Potassium 1.00
5. Magnesium 0.50
6. Calcium 1.00

5.2 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-Fast-4µm Column with and without Dionex IonPac CG16-Fast-4µm Guard Column

Similarly, in the case of the 150 mm length separator, the Dionex IonPac CG16-Fast-4µm guard column has the same capacity per gram of resin as the Dionex IonPac CS16-Fast-4µm separator column. In the case of the 4-mm and 2-mm i.d. separator columns, because the IonPac CG16-Fast-4µm length is only 30 mm (1/5 of the separator length), it adds about 3 minutes to the total retention time when it is used. In the case of the capillary IonPac CS16-Fast-4µm separator column, due to physical restrictions its guard length needs to be 35 mm long, adding about 4 minutes to the total retention time. The purpose of these shorter columns is to provide faster analysis times where very high resolution among adjacent analyte pairs is not as critical, as their concentration ratios are not as disparate.

Figure 10 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-Fast-4µm Column (4 × 150 mm), with and without Dionex IonPac CG16-Fast-4µm Guard Column (4 × 30 mm)

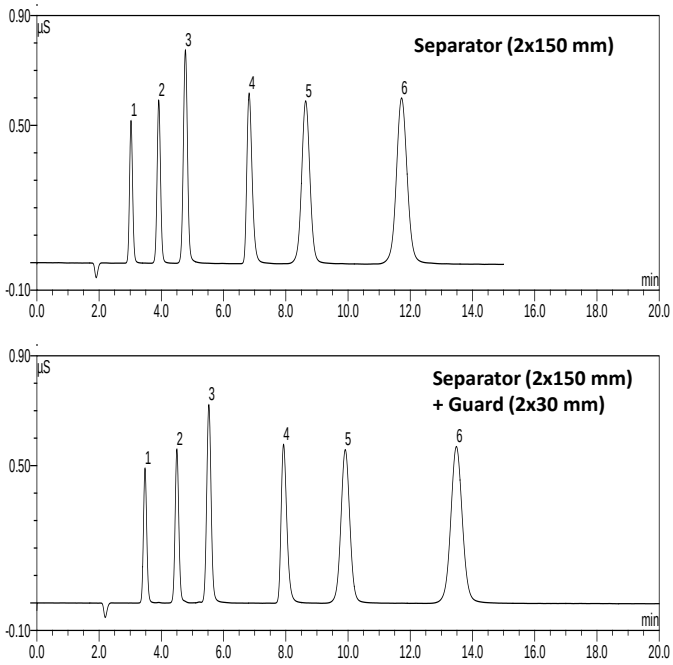


Columns:	Dionex IonPac CS16-Fast-4µm & Dionex IonPac CG16-Fast-4µm (4 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.64 mL/min
Inj. Volume:	10 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4-mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

Figure 11 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-Fast-4µm Column (2 × 150 mm), with and without Dionex IonPac CG16-Fast-4µm Guard Column (2 × 30 mm)

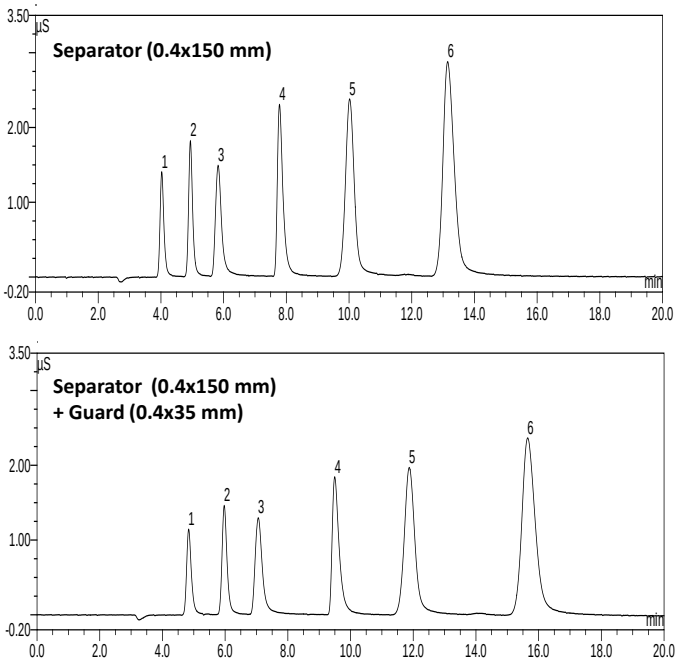


Columns:	Dionex IonPac CS16-Fast-4µm & Dionex IonPac CG16-Fast-4µm (2 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

Figure 12 Isocratic Elution of Six Common Cations using the Dionex IonPac CS16-Fast-4µm Capillary Column (0.4 × 150 mm), with and without Dionex IonPac CG16-Fast-4µm Capillary Guard Column (0.4 × 35 mm)



Columns:	Dionex IonPac CS16-Fast-4µm & Dionex IonPac CG16-Fast-4µm (0.4 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	6 µL/min
Inj. Volume:	400 nL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CCES 300, AutoSuppression, recycle mode

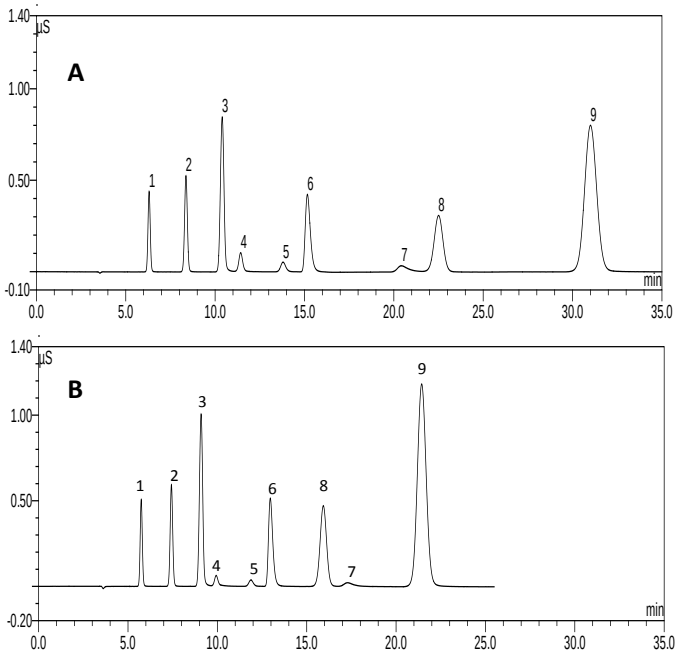
Peaks:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

5.3 Isocratic Separation of Six Common Cations and Methylamines using the Dionex IonPac CS16-4µm Column with the IonPac CG16-4µm Guard Column

Figure 13 shows isocratic separations of the common six cations and methylamines using different concentrations of MSA. Besides attaining a faster run time with the stronger 32 mM MSA eluent, the elution order of trimethylamine and magnesium switches, with magnesium- a divalent cation- eluting sooner. Quantitation is always easier when a higher concentration peak elutes after a low concentration analyte. The effect that the eluent concentration has on a divalent is twice that for a monovalent cation.

Figure 13 CS16-4µm (2x250 mm): Isocratic Separation of Methylamines plus the 6 Common Cations



Columns:	Dionex IonPac CG16-4µm (2x50 mm) & Dionex IonPac CS16-4µm (2x250 mm)
Eluent A:	26 mM MSA
Eluent B:	32 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

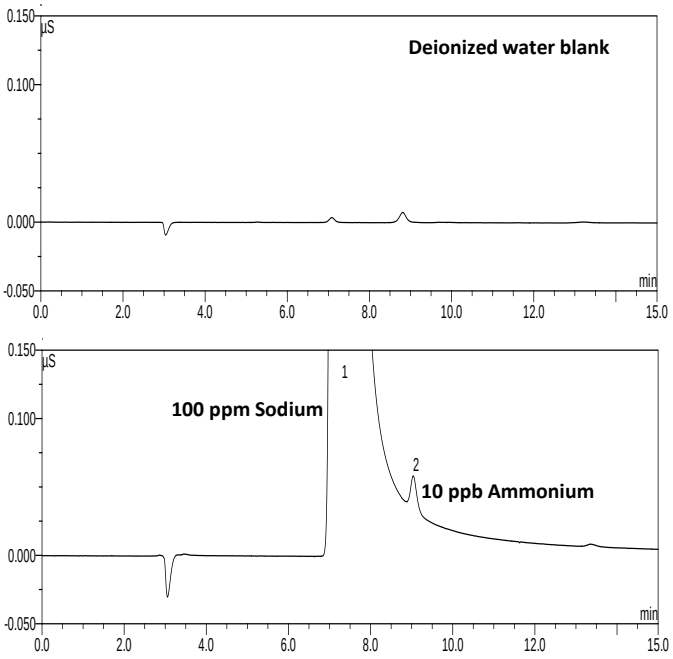
Peaks:

1.	Lithium	0.30 mg/L
2.	Sodium	1.20
3.	Ammonium	1.50
4.	Methylamine	1.00
5.	Dimethylamine	0.80
6.	Potassium	3.00
7.	Trimethylamine	3.00
8.	Magnesium	1.50
9.	Calcium	3.00

5.4 10,000:1 Sodium-to-Ammonium Concentration Ratio using the Dionex IonPac CS16-4µm Column with the IonPac CG16-4µm Guard Column

The following chromatograms show the isocratic eluent separation of a 10,000:1 concentration ratio of sodium to ammonium on the Dionex IonPac CS16-4µm in 250 mm length and 4-mm and 2-mm i.d. In the case of the 4-mm column, a smaller injection volume than shown can be used to minimize the amount of sodium injected on the column. This will depend on the expected level of ammonium in the sample, for it should be present above the method's minimum quantitation limit.

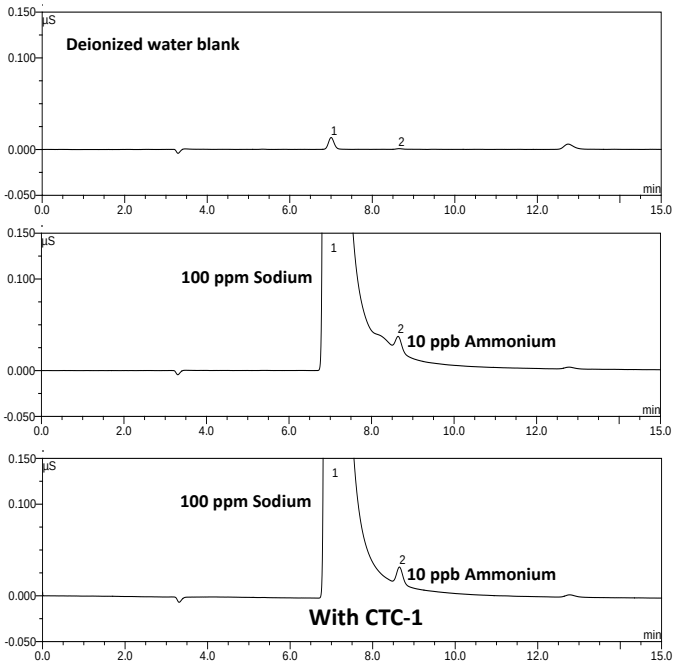
Figure 14 CG/CS16-4µm (4 mm): 10,000-to-1 ratio of Sodium-to-Ammonium



Columns:	Dionex IonPac CG16-4µm (4 × 50 mm) and CS16-4µm (4 × 250 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.64 mL/min
Inj. Volume:	15 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4-mm, AutoSuppression, recycle mode

In the case of the 2-mm i.d. column, as can be seen in the middle trace of the slide below, there was a "hump" eluting between the sodium and ammonium peaks, making quantitation of the trace level ammonium peak difficult. By installing a cation trap column CTC-1 between the Cell OUT and the suppressor's Regen IN ports, the large amount of sodium is trapped and thus prevented from going into the Regenerant chamber of the suppressor.
An alternative would be to use the suppressor in the External Water Mode.

Figure 15 CG/CS16-4µm (2 mm): Isocratic Separation of 10,000-to-1 ratio of Sodium-to-Ammonium

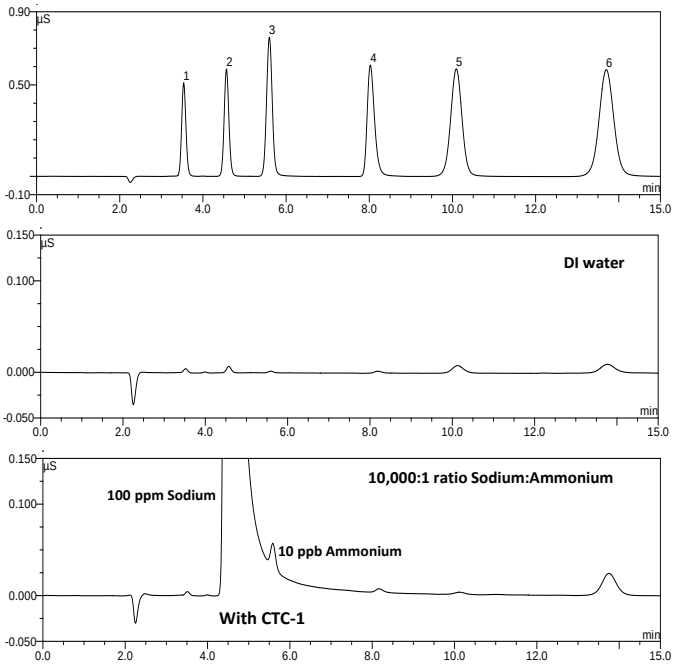


Columns:	Dionex IonPac CG16-4µm (2 x 50 mm) & Dionex IonPac CS16-4µm (2 x 250 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

5.5 10,000:1 Sodium-to-Ammonium Concentration Ratio using the Dionex IonPac CS16-Fast-4µm Column with the IonPac CG16-Fast-4µm Guard Column

In the chromatograms below, the separation was done under the same conditions but on an IonPac CS16-Fast-4µm 2x150 mm column with its 2x30 mm guard column. This shorter column set is meant for faster analysis, but due to its reduced column length, peak efficiency is lower and therefore the resolution is not as good as with the 250 mm length.

Figure 16 IonPac CG/CS16-Fast-4µm (2x30 mm & 2x150 mm): Isocratic Separation of 10,000-to-1 Ratio of Sodium-to-Ammonium



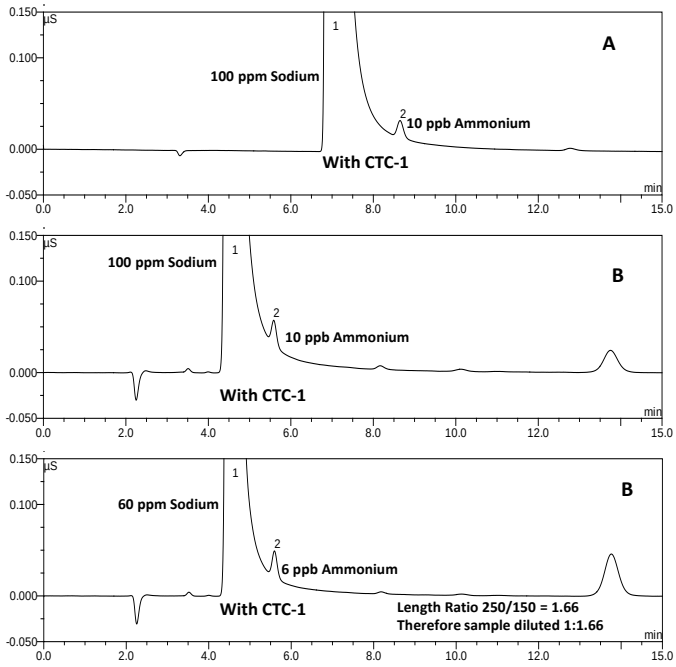
Columns:	Dionex IonPac CG16-Fast-4µm (2 x 30 mm) & Dionex IonPac CS16-Fast-4µm (2 x 150 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

Peaks:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

Diluting the sample by the ratio of the lengths improves the resolution of sodium and ammonium. Alternatively, the injection volume can be reduced by the same ratio, less sodium being injected on the column, thus improving the separation. Still, the longer column will provide better sodium-to-ammonium concentration ratios. Shown below is the comparison on a 2x250 mm IonPac CS16-4µm versus a 2x150 mm CS16-Fast-4µm column. The last peak shown in chromatogram "B" is calcium; it has not eluted yet in the 250 mm long column (chromatogram "A"):

Figure 17 Comparison of 250 mm vs. 150 mm long IonPac CG/CS16-4µm (2 mm i.d.): Isocratic Separation of 10,000-to-1 Ratio of Sodium-to-Ammonium

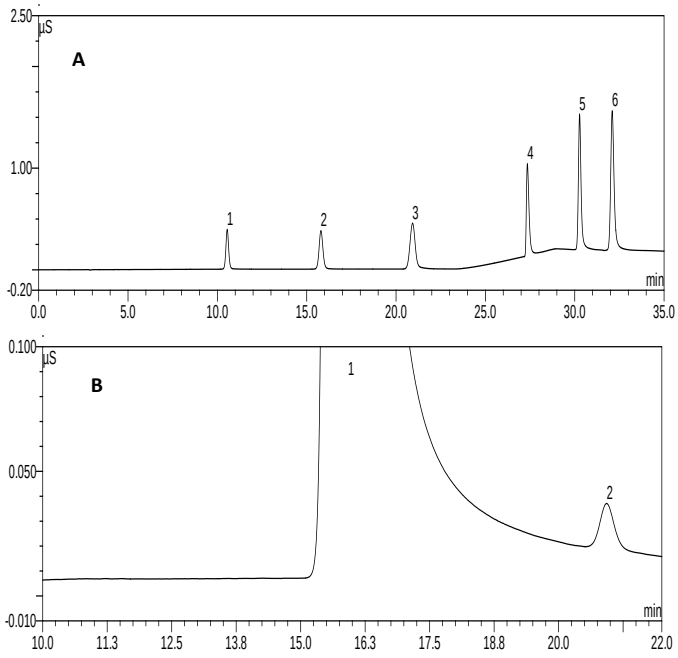


Columns A:	Dionex IonPac CS16-4µm (2 x 250 mm) & Dionex IonPac CG16-4µm (2 x 50 mm)
Columns B:	Dionex IonPac CS16-Fast-4µm (2 x 150 mm) & Dionex IonPac CG16-Fast-4µm (2 x 30 mm)
Eluent:	30 mM MSA
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.16 mL/min
Inj. Volume:	2.5 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 2-mm, AutoSuppression, recycle mode

5.6 Gradient Elution of a 10,000:1 Sodium-to-Ammonium Concentration Ratio Sample using the Dionex IonPac CS16- 4µm Column with the IonPac CG16-4µm Guard Column

Using gradient elution can further improve the sodium-to-ammonium peak resolution as shown below for a 4x250 mm IonPac CS16-4µm with its 50 mm guard. Using a more dilute acid eluent at the beginning improves the resolution between sodium and ammonium. The eluent concentration is then increased to expedite the elution of the later eluting cations in the sample.

Figure 18 CG/CS16-4µm (4 mm): Gradient Separation of 10,000-to-1 ratio of Sodium-to-Ammonium



Columns:	Dionex IonPac CG16-4µm (4 × 50 mm) and CS16-4µm (4 × 250 mm)
Eluent:	Isocratic 10.5 mM MSA to 20 min, gradient to 56 mM MSA at 25.0 min, isocratic to 33 min, back to 10.5 mM MSA at 33.1 min
Eluent Source:	Dionex EGC 500 MSA cartridge
Flow Rate:	0.64 mL/min
Inj. Volume:	15 µL
Temperature:	40 °C
Detection:	Suppressed Conductivity, Dionex CERS 500 4-mm, AutoSuppression, recycle mode

Peaks in A:

1.	Lithium	0.10 mg/L
2.	Sodium	0.40
3.	Ammonium	0.50
4.	Potassium	1.00
5.	Magnesium	0.50
6.	Calcium	1.00

Peaks in B:

1.	Sodium	100.0 mg/L
2.	Ammonium	0.010

6. Troubleshooting

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac CS16-4µm and Dionex IonPac CS16-Fast-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 CS16-4µm/CG16-4µm 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	Dionex CERS 500 or Dionex CCES 300 not suppressing	Check current	6.5, Component manual
Improper Suppressor Operation		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
	Column dehydrated	Hydrate column	Appendix D
	Contaminated or deformed bed support	Replace bed support	6.1.2
	Column headspace	Replace column	6.6.1 B
	Column overloading	Reduce sample size	34
	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	3.4, 1.0, OnGuard II A manual
Fronting Peaks	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	3.4, 1.0, OnGuard II A manual
	Column overload	Reduce sample size	3.4
	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	3.4
	Sluggish injection valve	Service valve	6.6.3 B, Component Manual
	Contaminated sample loop	Replace loop	6.3.3
	Capillary fittings/connections	Re-install	6.8
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 Quantification 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 CG16-4µm Guard/Capillary Guard Column plus the Dionex IonPac CS16-4µm Analytical/Capillary Column when using the test chromatogram conditions should be as indicated in Table 2. Table 3 shows the total system pressure for the Dionex IonPac CG16-Fast-4µm Guard/Capillary Guard Column plus the Dionex IonPac CS16-Fast-4µm Analytical/Capillary Column. 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 (0.64 mL/min, 0.16 mL/min, or 6 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 and Table 3 for the Typical 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 CS16-4µm and CS16-Fast-4µm 4 mm Columns (P/N)	Dionex IonPac CS16-4µm and CS16-Fast-4µm 2 mm Columns (P/N)	Dionex IonPac CS16-4µm and CS16-Fast-4µm 0.4 mm Columns (P/N)
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. If this has to be replaced, a Zitex membrane (P/N 060528 for the 4-mm and P/N 063167 for the 2-mm) should be placed between the resin bed and the outlet bed support.

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 CS16-4µm or CS16-Fast-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 deionized (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 CG16-4µm or CG16-Fast-4µm Guard and Dionex IonPac CS16-4µm or CS16-Fast-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 guard column 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 Capillary (P/N 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, CS19-4µm, CS16-4µm, and CS16-Fast-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 CS16-4µm or CS16-Fast-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 (for the CS16-4µm or the CS16-Fast-4µm, 0.64 mL/min for 4 mm operation or 0.16 mL/min for 2 mm operation). If the Dionex CERS 500 suppressor is used in the AutoSuppression External Water Mode, the regenerant flow rate should be a minimum of twice the eluent flow rate and a maximum of 5 mL/min (4 mm) or 2 mL/min (2 mm) for a 4-mm and a 2-mm suppressor respectively.

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, high flow rates or abrupt changes in eluent composition (such as high ionic strength or solvents). 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 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 (if injection valve is operated by air) 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").

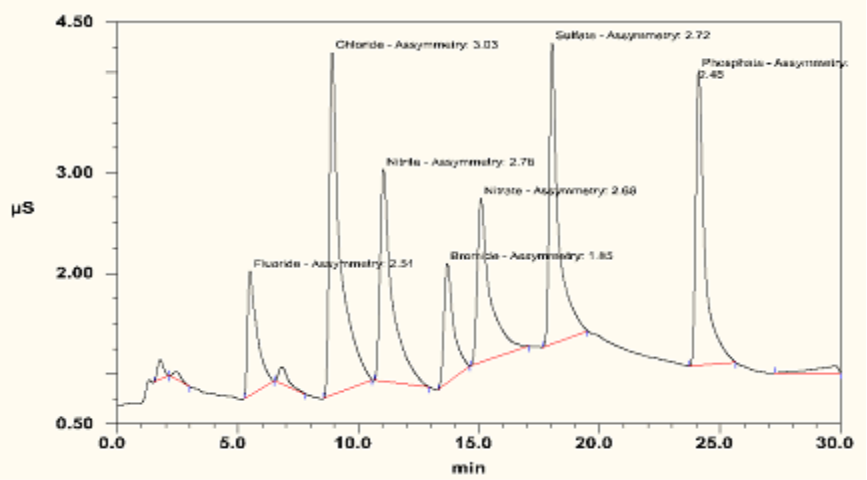
B. **Spurious peaks may be due to column contamination**. If the samples contain an appreciable level of polyvalent cations, polyvalent cations may contaminate the column. As a result, the retention times for the analytes will decrease, and spurious, inefficient peaks can show up at unexpected times. 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 19 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 20 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.

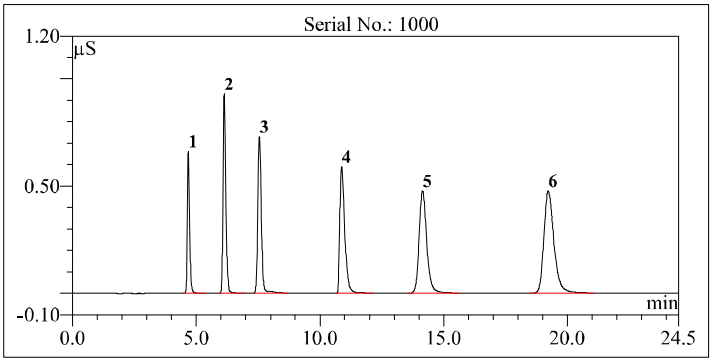
Figure 20 Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

Appendix A – Quality Assurance Reports

Dionex IonPac™ CS16-4µm
Analytical (4 x 250 mm)
Product No. 088584

Date: 10-Jul-15 15:42
Serial No. : 001000
Lot No. : 2011-34-89

Eluent: 30 mM Methanesulfonic acid
Eluent Flow Rate: 0.64 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Electrolytically Regenerated Suppressor (Dionex CERS™ 500 4 mm) AutoSuppression™ Recycle Mode
Applied Current: 57 mA
Injection Volume: 10 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	4.68	1.4	7.87	12117	0.1
2	Sodium	6.13	1.4	6.12	14883	0.4
3	Ammonium	7.55	1.1	10.70	13044	0.5
4	Potassium	10.87	2.1	7.30	14782	1.0
5	Magnesium	14.15	1.3	8.15	10918	0.5
6	Calcium	19.23	1.5	n.a.	11753	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=8100	Passed
Calcium	Retention Time	17.80-22.20	Passed
Potassium	Efficiency	>=11250	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=1.17	Passed
	Pressure	<=2970	1933

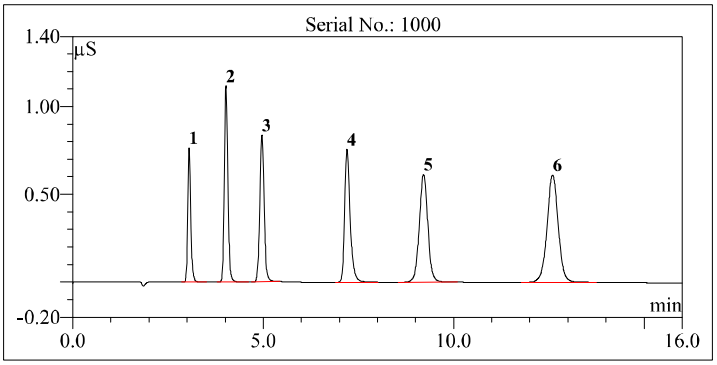
Production Reference:
Datasource: QAR
Directory: Cation\CS16_4µm
Sequence: CS16_4X250-4µm
Sample No.: 1

6.80 SR14 Build 4527 (238909) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS16-Fast-4µm
Analytical (4 x 150 mm)
Product No. 088599

Date: 23-Jul-15 11:25
Serial No. : 001000
Lot No. : 2011-34-96

Eluent: 30 mM Methanesulfonic acid
Eluent Flow Rate: 0.64 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Electrolytically Regenerated Suppressor (Dionex CERS™ 500 4 mm) AutoSuppression™ Recycle Mode
Applied Current: 57 mA
Injection Volume: 10 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.05	1.4	6.48	7633	0.1
2	Sodium	4.02	1.3	5.11	10061	0.4
3	Ammonium	4.97	1.0	9.49	8840	0.5
4	Potassium	7.20	1.6	6.10	12284	1.0
5	Magnesium	9.21	1.0	7.35	8431	0.5
6	Calcium	12.60	1.0	n.a.	9237	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=4860	Passed
Calcium	Retention Time	10.68-13.32	Passed
Potassium	Efficiency	>=6750	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=0.72	Passed
	Pressure	<=1980	1597

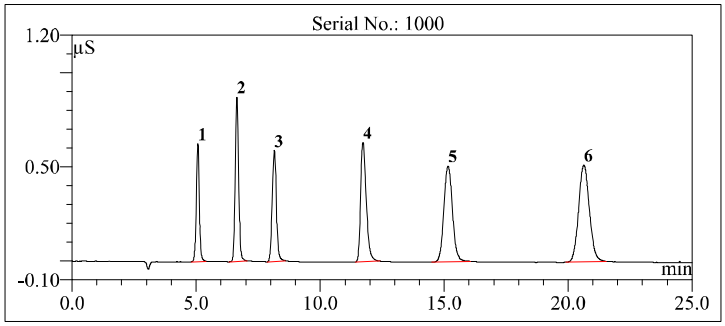
Production Reference:
Datasource: QAR
Directory: Cation\CS16_4µm
Sequence: CS16_4X150-4µm
Sample No.: 1

6.80 SR14 Build 4527 (238909) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS16-4µm
Analytical (2 x 250 mm)
Product No. 088582

Date: 30-Jun-15 14:27
Serial No. : 001000
Lot No. : 2011-34-93

Eluent: 30 mM Methanesulfonic acid
Eluent Flow Rate: 0.16 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Electrolytically Regenerated Suppressor (Dionex CERS™ 500 2 mm) AutoSuppression™ Recycle Mode
Applied Current: 15 mA
Injection Volume: 2.5 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	5.06	1.1	6.65	8393	0.1
2	Sodium	6.64	1.1	5.38	10747	0.4
3	Ammonium	8.15	1.1	10.24	11475	0.5
4	Potassium	11.73	1.6	6.73	13973	1.0
5	Magnesium	15.16	1.1	7.70	9406	0.5
6	Calcium	20.63	1.1	n.a.	10643	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=8100	Passed
Calcium	Retention Time	17.80-22.20	Passed
Potassium	Efficiency	>=11250	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=1.17	Passed
	Pressure	<=2970	1900

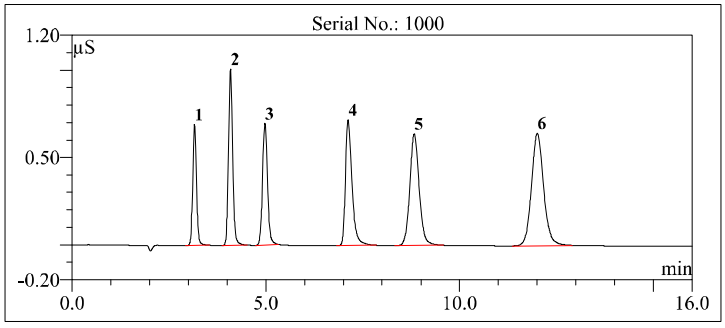
Production Reference:
Datasource: QAR
Directory: Cation\CS16_4µm
Sequence: CS16_2X250-4µM
Sample No.: 1

6.80 SR14 Build 4527 (238909) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS16-Fast-4µm
Analytical (2 x 150 mm)
Product No. 088601

Date: 22-Jul-15 17:03
Serial No. : 001000
Lot No. : 2011-34-96

Eluent: 30 mM Methanesulfonic acid
Eluent Flow Rate: 0.16 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Electrolytically Regenerated Suppressor (Dionex CERS™ 500 2 mm) AutoSuppression™ Recycle Mode
Applied Current: 15 mA
Injection Volume: 2.5 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.16	1.1	5.14	5447	0.1
2	Sodium	4.09	1.1	4.21	7267	0.4
3	Ammonium	4.98	1.1	8.14	7328	0.5
4	Potassium	7.12	1.6	4.71	9249	1.0
5	Magnesium	8.83	1.2	6.45	6761	0.5
6	Calcium	12.00	1.2	n.a.	7415	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=4860	Passed
Calcium	Retention Time	10.68-13.32	Passed
Potassium	Efficiency	>=6750	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Ret. Time diff	>=0.72	Passed
	Pressure	<=1980	1614

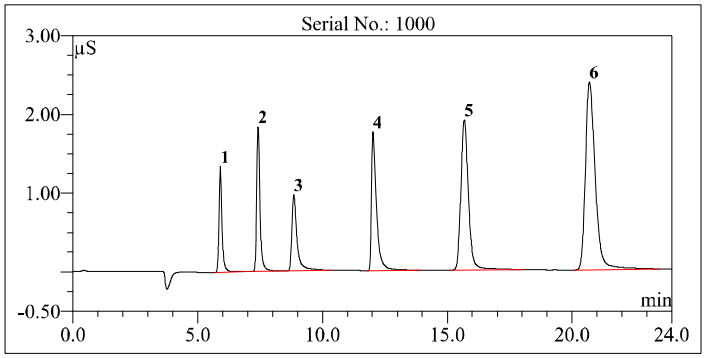
Production Reference:
Datasource: QAR
Directory: Cation\CS16_4µm
Sequence: CS16_2X150-4µM
Sample No.: 1

6.80 SR14 Build 4527 (238909) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS16-4µm Capillary (0.4 x 250 mm) Product No. 088615

Date: 14-Aug-15 07:11
Serial No. : 001000
Lot No. : 2011-34-83

Eluent: 30 mM Methanesulfonic acid (MSA)
Eluent Source: Dionex EGC-MSA (Capillary)
Eluent Flow Rate: 0.006 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Capillary Electrolytic Suppressor (Dionex CCES 300) AutoSuppression™ Recycle Mode
Applied Current: 7 mA
Injection Volume: 0.4µL
Storage Solution: Eluent (30 mM MSA)



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	5.90	1.5	7.09	12761	0.1
2	Sodium	7.42	1.4	5.17	18235	0.4
3	Ammonium	8.85	2.0	9.54	11143	0.5
4	Potassium	12.02	2.6	8.58	21062	1.0
5	Magnesium	15.68	1.3	8.33	14343	0.5
6	Calcium	20.69	1.6	n.a.	14668	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=8100	Passed
Calcium	Retention Time	17.80-22.20	Passed
Potassium	Efficiency	>=11250	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=1.17	Passed
	Pressure	<=2970	1584

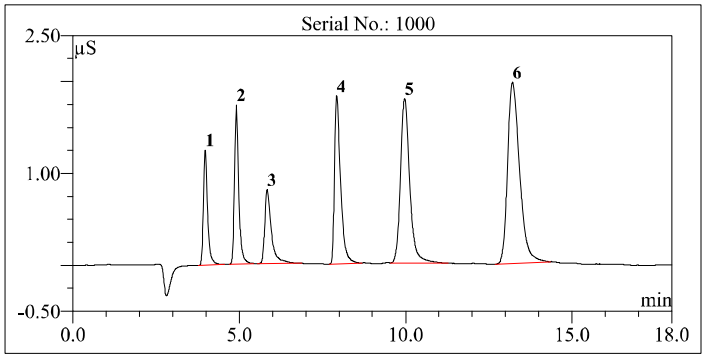
Production Reference:
Datasource: QAR
Directory: Cap\CS16-4µm
Sequence: CS16_p4x250-4µm
Sample No.: 1

6.80 SR14 Build 4527 (238909) (Demo-Installation)
Chromeleon™ Thermo Fisher Scientific

Dionex IonPac™ CS16-Fast-4µm Capillary (0.4 x 150 mm) Product No. 088641

Date: 18-Aug-15 10:09
Serial No. : 001000
Lot No. : 2011-34-96

Eluent: 30 mM Methanesulfonic acid (MSA)
Eluent Source: Dionex EGC-MSA (Capillary)
Eluent Flow Rate: 0.006 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Dionex Cation Capillary Electrolytic Suppressor (Dionex CCES 300) AutoSuppression™ Recycle Mode
Applied Current: 7 mA
Injection Volume: 0.4µL
Storage Solution: Eluent (30 mM MSA)



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	3.97	1.6	4.43	5765	0.1
2	Sodium	4.91	1.5	3.43	8368	0.4
3	Ammonium	5.84	2.0	6.50	5038	0.5
4	Potassium	7.93	2.1	5.12	10171	1.0
5	Magnesium	9.97	1.4	5.81	6783	0.5
6	Calcium	13.21	1.6	n.a.	6890	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=4860	Passed
Calcium	Retention Time	11.47-14.33	Passed
Potassium	Efficiency	>=6750	Passed
Potassium	Asymmetry	1.1-3.2	Passed
Sodium	Asymmetry	1.0-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=0.72	Passed
	Pressure	<=1980	798

Production Reference:
Datasource: QAR
Directory: Cap\CS16-4µm
Sequence: CS16_p4x150-4µm
Sample No.: 1

6.80 SR14 Build 4527 (238909) (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 CS16-4µm and CS16-Fast-4µm Analytical, Capillary or Guard Column is 5,000 psi (34.46 MPa). ***Maximum Operating Flow Rates for the column (see Table 2) should never be exceeded***.

CAUTION

Do not use alcohols in the eluent.
Do not use the Dionex IonPac CS16-4µm and CS16-Fast-4µm column with basic eluents. This can significantly increase column back pressures and disrupt the packing.

B.2 Column Start-Up

The column is shipped using 30 mM Methanesulfonic acid as the storage solution. Prepare the eluent shown on the Quick Start procedure (see Appendix D) and follow the Quick Start instructions to hydrate the columns prior to running the QAR. After the column has gone through the 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 30 mM Methanesulfonic acid for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends ***tightly***, 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 CS16-4µm or CS16-Fast-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 0.64 mL/min** for a Dionex IonPac CS16-4µm or CS16-Fast-4µm 4 mm Analytical or Guard Column, to 0.16 mL/min for a Dionex IonPac CS16-4µm or CS16-Fast-4µm 2 mm Analytical or Guard Column, or to 0.006 mL/min for a capillary Dionex IonPac CS16-4µm or CS16-Fast-4µm 0.4 mm Analytical or Guard Column.

D. **Rinse the column for 15 minutes with eluent** (30 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 30 mM MSA eluent for at least 60 minutes before resuming normal operation (send effluent to waste).

G. **Reconnect the Suppressor** to the Dionex IonPac CS16-4µm or CS16-Fast-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 CS16-4µm or CS16-Fast-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 0.3 mL/min** for a Dionex IonPac CS16-4µm or CS16-Fast-4µm 4 mm Analytical or Guard Column, to 0.08 mL/min for a Dionex IonPac CS16-4µm or CS16-Fast-4µm 2 mm Analytical or Guard Column, or to 0.003 mL/min for a capillary Dionex IonPac CS16-4µm or CS16-Fast-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
10.0	90	10
15.0	90	10
30.0	30	70
60.0	30	70
75.0	90	10
80.0	90	10
90.0	100	0

D. **Rinse and equilibrate the column(s)** with 30 mM MSA eluent for at least 60 minutes before resuming normal operation.

E. **Reconnect the Dionex IonPac CS16-4µm column**. Connect the Analytical/Capillary Column outlet to the Suppressor and the inlet to either the Dionex IonPac CG16-4µm or CG16-Fast-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 for Dionex IonPac CS16-4µm and CS16-Fast-4µm Columns

Condition	2 mm System Operation Summary	4 mm System Operation Summary	0.4 mm System Operation Summary
Eluent Flow Rate	Typically 0.16 mL/min	Typically 0.64 mL/min	Typically 6 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 064560)	N/A
Regenerant Flow Rate	Typically 1 – 2 mL/min	Typically 3-5 mL/min	N/A
Injection Loop	2.5-10 µL	10- 40 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 capillary systems.
Note **For Dionex IonPac CS16-4µm and CS16-Fast-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 (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	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 (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™ CS16-4µm and CS16-Fast-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 guard and separator column bed by following this Quick Start procedure, is **recommended 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.

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 CS16-4µm and CS16-Fast-4µm column is 5000 psi (34.5 MPa).

D.2 Gradient Program for Dionex IonPac CS16-4µm/ CG16-4µm and Dionex IonPac CS16-Fast-4µm/ CG16-Fast-4µm 2-mm and 4-mm i.d. formats

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 0.32 mL/min.
Conditioning flow rate for 2 mm columns is 0.08 mL/min.

Time	%E1 DI Water	%E2 300 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 CS16-4µm/ CG16-4µm and Dionex IonPac CS16-Fast-4µm/ CG16-Fast-4µm Capillary 0.4-mm i.d. formats

a. Prepare 30 mM MSA with 10% 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 3µL/min.
e. Disconnect the column and change the 30 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 30 mM MSA through the column directly to waste for one hour at 6 µL/min.
j. Proceed to section 4 to test the column under Quality Assurance Report conditions.

D.4 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 30 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.5 Storage

Store the column in 30 mM Methanesulfonic acid and ***tightly*** seal both ends immediately with column plugs to avoid drying of the column.

Tip ***For additional information, please refer to the Dionex IonPac CS16 Product Manual (Document No. 031725).***