



PRODUCT MANUAL

for

IonPac[®] CG12A
IonPac[®] CS12A

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 **DIONEX**

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

IONPAC® CG12A GUARD COLUMN

- (8.5 µm, 2 x 50 mm, P/N 046076)
- (5 µm, 3 x 30 mm, P/N 057184)
- (8.5 µm, 4 x 50 mm, P/N 046074)
- (5 µm, 0.4 x 35 mm, P/N 072069)
- (8.5 µm, 0.4 x 50 mm, P/N 072067)

IONPAC® CS12A ANALYTICAL COLUMN

- (8.5 µm, 2 x 250 mm, P/N 046075)
- (5 µm, 3 x 150 mm, P/N 057185)
- (8.5 µm, 4 x 250 mm, P/N 046073)
- (8.5 µm 2 x 100 mm, P/N 059960)
- (5 µm, 0.4 x 150 mm, P/N 072068)
- (8.5 µm, 0.4 x 250 mm, P/N 072066)

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SECTION 1 - INTRODUCTION

The IonPac® CS12A 4x250 mm (P/N046073), CS12A-5 μ m 3-mm 3x150 mm (P/N057185), CS12A 2x250 mm (P/N046075) Analytical Columns and CS12A 0.4x250 mm (P/N072066) Capillary Column are designed specifically for the analysis of alkali metals, alkaline earth metals, and ammonium. The CS12A, CS12A-MS and CG12A stationary phase is functionalized with relatively weak phosphonic and carboxylic acids having a high selectivity for hydronium ion. The weak carboxylate functional groups require low ionic strength eluents to isocratically elute both monovalent and divalent cations in a relatively short period of time. It has both cation exchange and reverse phase properties. The CS12A and CS12A-MS are solvent-compatible with 100% aqueous eluents, 100% acetonitrile, or 20% tetrahydrofuran without loss of performance.

The IonPac® CS12A-5 μ m 3x150-mm (P/N057185) Analytical Column and CS12A-5 μ m 0.4x150 mm (P/N072068) Capillary Column are designed specifically for the fast analysis of alkali metals, alkaline earth metals, and ammonium. They use the same functional groups (carboxylate and phosphonate) as the standard CS12A 4-mm. They differ in both the column format and the resin particle size. The smaller resin size of the CS12A-5 μ m, together with optimized packing conditions, results in high peak efficiencies in spite of the fact that the CS12A-5 μ m column is shorter than the standard CS12A column. The smaller column internal diameter requires half the flow rates of the standard 4-mm column, thus decreasing eluent consumption and eluent disposal. Minimum detection levels are improved by a factor of about 2.5. The shorter length together with the smaller id allows for faster analysis of the common cations.

Thus, the advantages of the CS12A-5 μ m column are:

- Fast analysis of the group I and the group II cations plus ammonium ion.
- Low eluent consumption.
- Improved minimum detection limits.

The disadvantages are that the column has about 1/3 the column cation exchange capacity as the CS12A 4-mm, and will therefore be not as tolerable as the CS12A with respect to sample pH. While the CS12A 4-mm can determine samples containing up to 50 mM H⁺ when a 25 μ L sample loop is used, the CS12A-5 μ m 3-mm column can only tolerate up to 20 mM H⁺. To overcome this limitation either a smaller sample loop or the OnGuard II A cartridge can be used to treat the sample making it possible for the CS12A-5 μ m 3-mm to analyze samples with up to 50 mM H⁺ concentration. Due to its lower capacity, the CS12A-5 μ m 3-mm will also overload sooner than the CS12A 4-mm when high concentration samples are injected. Thus, for a given sample loop and for samples containing diverse concentration ratios, the 5- μ m 3-mm column is inferior to the 4-mm.

The CS12A-5 μ m 3-mm column can be used with either a standard or narrow bore pump (2-mm or 4-mm eluent pump), but the system's plumbing should be optimized to minimize band dispersion. Plumbing should be done with 5/1000 ID PEEK tubing (red) minimizing the lengths of it and making sure the tubing is cut with a flat (and not slanted) edge. A CSRS 300 2-mm suppressor should be used instead of a 4-mm for the same reasons. Using a 4-mm CSRS 300 will decrease the peaks' efficiencies by at least 1000 plates for monovalent cations. In order to minimize noise, the CSRS 300 2-mm should be used in the lowest current setting required to suppress the eluent.

Most of the CS12A-5 μ m applications shown in this manual have been run at 30 °C. If you do not have an oven to do this, you can still run the same applications at room temperature. Peak efficiencies will be slightly lower. Selectivity can sometimes be different when the column is run at room temperature vs at 30 °C. For example, resolution of magnesium and manganese will actually be poor at 30 °C but baseline resolved at room temperature.

Chromatographic methods developed for CS12A applications, such as amine applications, should be applicable also to the CS12A-5 μ m columns, with just the one change in eluent flow rate; while the CS12A is usually run at 1.0 mL/min, the CS12A-5 μ m 3-mm should be run at 0.5 mL/min.

1.1 Important Considerations When Using the CS12A-MS (2 x 100 mm) Column

The IonPac CS12A-MS column is an IC-MS screening column packed with the same CS12A (8.5 μ m) resin in a 2-mm I.D. x 100-mm length format, ideally suited for fast elution and low flow rates required for interfacing with IC-MS detectors. Typically, the CS12A-MS column will be operated in an ion chromatography system containing a post column cation eluent suppressor for eluent suppression and removal of salt counter anions, followed by a conductivity detector and ESI-MS detector in series. When an eluent suppressor is used after the analytical column in electrospray IC-MS, standard IC nonvolatile ionic eluent components can be used. The electrolytic suppressor (CSRS 300) lowers the ionic strength of the effluent entering the ESI interface, producing

a more stable signal and improving sensitivity by eliminating ionization suppression of the analyte by eluent ions. The system can also be operated without a suppressor by using formic acid and ammonium acetate as the mobile phase. The advantage of operating without a suppressor is that an MS signal can be obtained for both anions and cations since the anions will not be removed by the suppressor.

A typical IC system for IC-MS consists of a 2-mm suppressed IC system such as a ICS-5000 with a conductivity detector. A ground union is installed between the conductivity cell and the chassis to eliminate voltage buildup between the two detectors. The use of microbore (2-mm) components is recommended because the sensitivity and ease-of-use are better at the lower flow rates with the electrospray interface.

The use of a suppressor in IC-MS provides several advantages. Methods developed for conductivity detection conditions can be used without modification. The counterions are removed, reducing MS system maintenance. The electrospray signal is considerably more stable when the ionic load entering the interface is lowered, even in comparison to volatile eluent systems. Detection limits are improved due to increased signal stability and the elimination of ionization suppression. The availability of a high sensitivity conductivity trace provides useful identification information.

One advantage to operating without a suppressor is that it is possible to obtain a MS signal for both anions and cations because neither is removed in the suppressor. However, the absence of the CSRS suppression leads to a more erratic signal and increased signal suppression. A useful mobile phase is formic acid and ammonium acetate for cation applications.

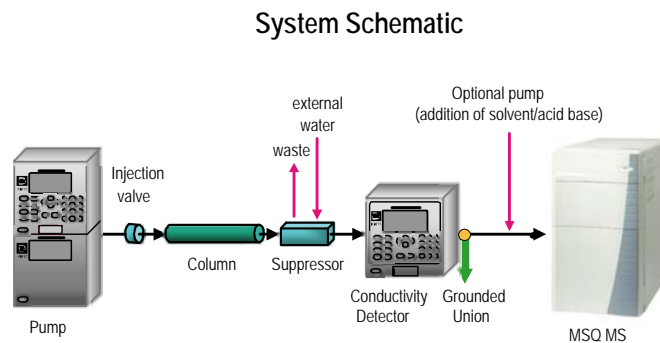
The schematic below shows the components in an IC/MS System.



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.



Slide 16159

Figure 1
IC/MS System Schematic

Table 1
IonPac CS12A/CG12A Packing Specifications

Column	Particle Diameter μm	Substrate^a X-linking %	Column Capacity meq/column	Functional Group	Hydrophobicity
CS12A 4 x 250 mm	8.5	55	2.8	Carboxylic/ Phosphonic acid	Med-low
CG12A 4 x 50 mm	8.5	55	0.56	Carboxylic/ Phosphonic acid	Med-low
CS12A-5μm 3 x 150 mm	5.5	55	0.94	Carboxylic/ Phosphonic acid	Med-low
CG12A-5μm 3 x 30 mm	5.5	55	0.19	Carboxylic/ Phosphonic acid	Med-low
CS12A 2 x 250 mm	8.5	55	0.7	Carboxylic/ Phosphonic acid	Med-low
CG12A 2 x 50 mm	8.5	55	0.14	Carboxylic/ Phosphonic acid	Med-low
CS12A-MS 2x100	8.5	55	0.28	Carboxylic/ Phosphonic acid	Med-low
CS12A 0.4x250mm	8.5	55	0.028	Carboxylic/ Phosphonic acid	Med-low
CG12A 0.4x50mm	8.5	55	0.0056	Carboxylic/ Phosphonic acid	Med-low
CS12A-5μm 0.4x150mm	5.5	55	0.0094	Carboxylic/ Phosphonic acid	Med-low
CS12A-5μm 0.4x35mm	5.5	55	0.0019	Carboxylic/ Phosphonic acid	Med-low

^a macroporous (100 Å) divinylbenzene/ethylvinylbenzene polymer

Table 2
CS12A/CG12A Operating Parameters

Column	At standard flow rate, Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS12A 4-mm Analytical	≤ 1,200 (8.27)	1.0	2.0
CG12A 4-mm Guard	≤ 450 (3.10)	1.0	3.0
CS12A + CG12A 4-mm columns	≤ 1,650 (11.37)	1.0	2.0
CS12A-5µm 3-mm Analytical	≤ 1,760 (12.13)*	0.5	1.0
CG12A-5µm 3-mm Guard	≤ 605 (4.17)*	0.5	1.0
CS12A + CG12A-5µm 3-mm columns	≤ 2,365 (17.30)*	0.5	1.0
CS12A 2-mm Analytical	≤ 1,200 (8.27)	0.25	0.5
CG12A 2-mm Guard	≤ 450 (3.10)	0.25	0.75
CS12A + CG12A 2-mm columns	≤ 1,650 (11.37)	0.25	0.5
CS12A 0.4 mm Capillary	≤ 1,320 (9.10)	0.01	0.020
CG12A 0.4 mm Capillary Guard	≤ 495 (3.41)	0.01	0.020
CS12A + CG12A 0.4 mm columns	≤ 1,815 (12.51)	0.01	0.020
CS12A-5µm 0.4 mm Capillary	≤ 1,760 (12.13)*	0.008	0.02
CG12A-5µm 0.4 mm Capillary Guard	≤ 605 (4.17)*	0.008	0.02
CG12A-5µm 0.4 mm columns	≤ 2,365 (17.30)*	0.008	0.02
CS12A-MS (2 x 100 mm) Analytical	≤ 800 (5.51)	0.25	0.75

* at 30 °C

Read the system manuals. This manual assumes that you are familiar with the installation and operation of the 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.

You may need to make a liquid line fitting. The IonPac CS12A Analytical Column and the IonPac CG12A Guard Column have 10-32 PEEK end fittings for use with ferrule/bolt liquid line fittings. If you have an Ion Chromatograph with Tefzel® liquid lines having 1/4-28 ThermoFlare fittings, it will be necessary to obtain one or more Tefzel liquid lines with a PEEK bolt and ferrule fitting on one end and a 1/4-28 ThermoFlare fitting on the other end. See, “Dionex Liquid Line Fittings,” for detailed instructions on purchasing or making these lines.



NOTE

Even though the end fittings for the CS12A 4-mm and 3-mm are the same, notice that the bed support is NOT.

If you need to replace the bed support in the CS12A-5µm 3-mm, make sure you use the correct one. The i.d. of the 3-mm frit is smaller than the one used in the 4-mm format.

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of Dionex instrumentation and columns through the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the Dionex Offices listed in, “Dionex Worldwide Offices.”

SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

The proper configuration of 2-mm system injection volume, mass loading, system void volume and flow rate is based on the ratio of the 2-mm to 4-mm column cross-sectional area which is a factor of 1:4. The ratio of the 3-mm to 4-mm column cross-sectional area is a factor of 1:1.8.

Condition	2-mm	3-mm	4-mm	0.4-mm
Eluent Flow Rate	0.25 mL/min	0.5 mL/min	1.0 mL/min	0.010 mL/min
Injection Loop	2 – 15 µL Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections < 15 µL.	5 – 25 µL	10 – 50 µL	0.4 µL (typical)
Suppressors	CSRS 300 (2-mm) (P/N 064557) CMMS 300 (2-mm) (P/N 064561) CAES ® (P/N 056118)	CSRS 300 (2-mm) (P/N 064557) CMMS 300 (2-mm) (P/N 064561) CAES ® (P/N 056118)	CSRS 300 (4-mm) (P/N 064556) CMMS 300 (4-mm) (P/N 064560) CAES ® (P/N 056118)	CCES 300 (P/N 072053)
System Void Volume	Eliminate switching valves, coupler and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (2-mm) Mixer (P/N 049135).	Eliminate switching valves, coupler and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (2-mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2, GM-3 or recommended gradient mixers.	Use only on an IC System equipped for Capillary Analysis.
Pumps	Use the GS50/GP50/GP40/IP20 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. No External Gradient Mixer is required for GS50/GP50/GP40 Pump when performing gradient analysis. The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.	Use the GS50/GP50/GP40/IP20 in Microbore or Standard Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. No External Gradient Mixer is required for GS50/GP50/Gp40 pump when performing gradient analysis. The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.	Use the GP40/GP50/IP20/IP25 in Standard-Bore Configurations. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP40/GP50/IP20/IP25 and the DX-300 HPLC Pump. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.	Use only a pump designed for Capillary flow rates such as the ICS-5000 Capillary pump.



Do not run suppressors over 40°C. If you have an application that requires higher temperatures, place the suppressor outside the oven.

Condition	2-mm	3-mm	4-mm	0.4-mm
Chromatographic Module	A thermally controlled column oven such as the LC25, LC30, ICS-10, 11, 15, 16, 20, 2100, 3000, 5000 DC	A thermally controlled column oven such as the LC25, LC30, ICS-10, 11, 15, 16, 20, 2100, 3000, 5000 DC	A thermally controlled column oven such as the LC25, LC30, ICS-10, 11, 15, 16, 20, 2100, 3000, 5000 DC	A thermally controlled column compartment such as the ICS-5000 DC or IC-Cube.
Detectors	<p>AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423)</p> <p>VDM-2 Cell (3-mm, 2.0 µL, P/N 043120)</p> <p>CD20, CD25, ED40 or ED50 Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132</p> <p>CDM-2/CDM-3 Cell P/N 042770</p> <p>Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25.</p> <p>Ensure 30 – 40 psi back pressure after the cell.</p>	<p>AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) or an AD20/AD25 cell (10-mm, 9 µL) P/N 049393</p> <p>VDM-2 Cell (3-mm, 2.0 µL, P/N 043120) or VDM-2 cell (6-mm, 10 µL) P/N 043113</p> <p>CD20, CD25, ED40 or ED50 Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132</p> <p>CDM-2/CDM-3 Cell P/N 042770</p> <p>Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25.</p> <p>Ensure 30 – 40 psi back pressure after the cell.</p>	<p>AD20/AD25 Cell (10-mm, 9 µL P/N 049393)</p> <p>VDM-2 Cell (6-mm, 10 µL) P/N 043113</p> <p>CD20, CD25, ED40 or ED50 Conductivity Cell with DS3 P/N 044130 or with shield P/N 044132</p> <p>CDM-2/CDM-3 Cell P/N 042770</p> <p>Either the TS-1 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50 or the CD20/CD25.</p> <p>Ensure 30 – 40 psi back pressure after the cell.</p>	<p>Use only a Conductivity detector designed for Capillary flow rates such as the ICS-5000 Capillary CD</p>

Table 3
Tubing Back Pressures

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

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 0.4 mm Operation

The IonPac CS12A and CS12A - 5 μ m 0.4 mm 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 ICS-5000 Capillary System for best performance.

3.1.2 System Requirements for 4-mm Operation

The IonPac CS12A 4-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a system having a gradient pump configured for standard bore operation.

3.1.3 System Requirements for CS12A-5 μ m 3-mm Operation

The IonPac CS12A-5 μ m 3-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a system having either a standard or a narrow bore pumps (4-mm or 2-mm), as most applications are run at 0.5 mL/min.

3.1.4 System Requirements for 2-mm Operation

The IonPac CS12A 2-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a gradient pump configured for narrow bore operation.

3.1.5 System Void Volume

When using 2-mm and CS12A-5 μ m 3-mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4-mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" (P/N 044221) ID PEEK tubing. 0.010" ID PEEK tubing (P/N 042260) or 0.012" Tefzel tubing (see "Dionex Product Selection Guide") 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 remove all unnecessary switching valves and couplers. Make sure all tubing is cut in a straight (not slanted) manner. If you need assistance in properly configuring your system contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices")

3.1.6 Installation of the Capillary Column

Before installing the new separator column, tear off the column label and slide it into the holder on the front of the cartridge (see Figure 2).

For reference, Figure 2 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 3 shows the column cartridge after installation of only a capillary separator column. Before installing the new separator column, tear off the column label and slide it into the holder on the front of the cartridge (see Figure 2).

For reference, Figure 2 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 3 shows the column cartridge after installation of only a capillary separator column.

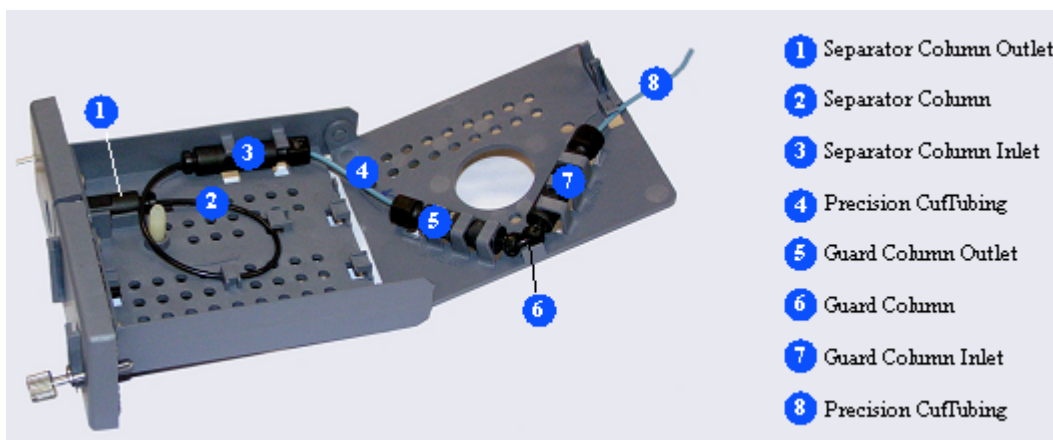


Figure 2
Separator and Guard Columns Installed in Column Cartridge

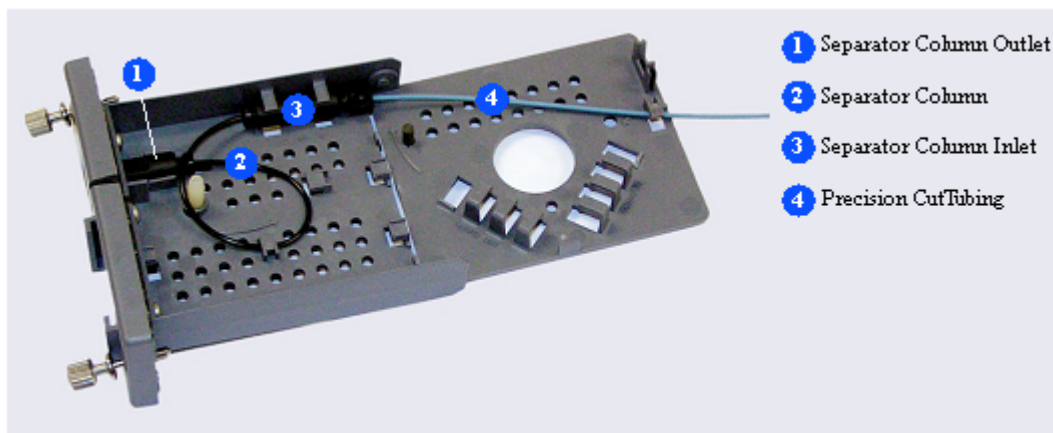


Figure 3
Separator Column Only Installed in Column Cartridge

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

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

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

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

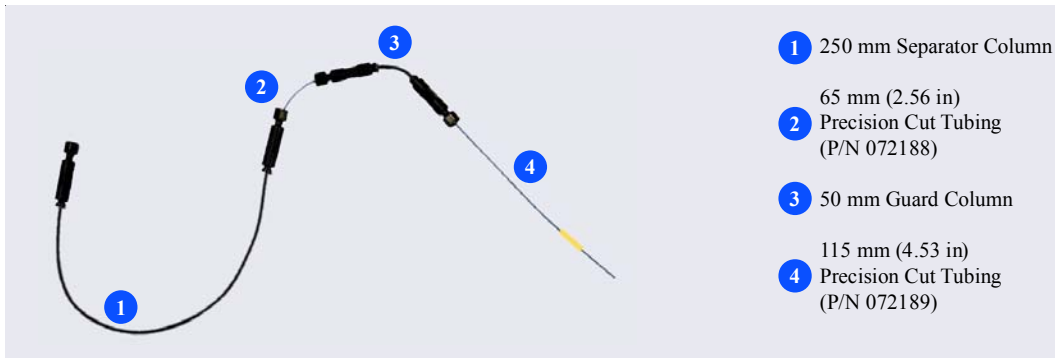


Figure 4
Tubing Connections for 250-mm Separator Column and 50-mm Guard Column

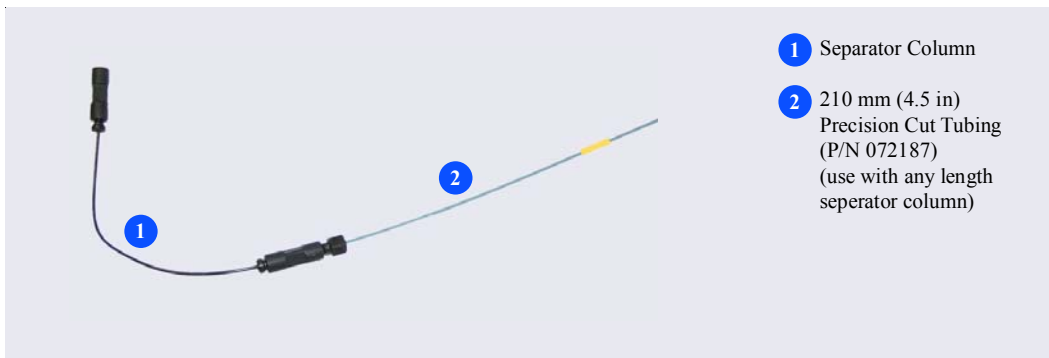


Figure 5
Tubing Connections for Separator Column Only

3. Lift up the lid of the column cartridge to open it.
4. Remove the fitting plug from the outlet fitting on the separator column. Orient the fitting with a flat side up (see Figure 6) and push the fitting into the opening at the front of the column cartridge until it stops.

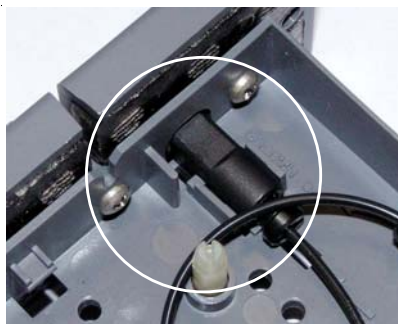


Figure 6
Column Outlet Fitting Installed in Column Cartridge

5. Coil the separator column tubing inside the cartridge as shown in Figure 2 or Figure 3. Secure the column tubing and the inlet fitting in the clips on the column cartridge.
6. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
7. Route the guard column inlet tubing (if used) or the separator column inlet tubing through the clip on the top edge of the column cartridge lid.
8. Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see Figure 7).



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 7
Column Cartridge Closed

3.2 Installing the CR-CTC Trap Column for Use with EGC MSA Cartridge

For IonPac CS12A applications using the EGC MSA cartridge, a CR-CTC Continuously Regenerated Trap Column (P/N 066262 or 072079) should be installed at the EGC eluent outlet to remove trace level cationic contaminants such as ammonium from the carrier deionized water. See the CR-TC Product Manual (Document No. 031910) for instructions. As an alternative, the CTC-1 Trap Column (P/N 040192) can be used. The CTC-1 Trap Column will require off-line regeneration. To use the CTC Cation Trap Column, see Section 3.3.

3.3 Installing the Cation Trap Column for Eluent Step Change or Gradient Operation

An IonPac Cation Trap Column (CTC (2-mm), P/N 043132 or CTC-1 (4-mm), P/N 040192) should be installed between the Gradient Pump and the injection valve. Remove the high pressure Gradient Mixer if present. The CTC is filled with high capacity cation exchange resin which helps to minimize the baseline shift caused by increasing cationic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis.

The CS12A-5 μ m 3-mm can be used with either CTC column.

To install the CTC (2-mm) or CTC-1 (4-mm), complete the following steps:

- A. **Remove the Gradient Mixer.** It is installed between the gradient pump pressure transducer and the injection valve.
- B. **Connect the gradient pump directly to the CTC.** Connect a waste line to the CTC outlet and direct the line to a waste container.
- C. **Flush the CTC.** Use 200 mL of a 10X eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min. Note that with the guard and analytical columns out of line, there is no need for 2-mm flow rate restrictions.
- D. Rinse the CTC with the strongest eluent that will be used during the gradient analysis.
- E. **Reconnect the CTC.** Connect the CTC to the eluent line that is connected to the injection valve.

The background conductivity of your system should be less than 3 μ S when 10 mM H₂SO₄ or methanesulfonic acid (MSA) is being pumped through the chromatographic system with the CSRS in-line and properly functioning. The baseline shift should be no greater than 1 μ S during a gradient concentration ramp from 10 to 40 mM methanesulfonic acid (MSA). If the baseline shifts are greater than 5 μ S, the CTC should be cleaned using steps A - E above.

Flush the CTC at the end of each operating day. This removes any impurities that may have accumulated on it. This will minimize periodic maintenance and lost data.

- A. **Disconnect the CTC from the injection valve.**
- B. **Direct the outlet of the CTC to a separate waste container.**
- C. **Flush the CTC.** Use 30 mL of a 10X eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min.
- D. **Flush the CTC prior to start-up.** Prior to the use of the chromatographic system on the next day, flush the CTC with 30 mL of the strongest eluent used in the gradient program.

3.4 The Injection Loop

The injection loop can vary from 0.4 μL to about 1000 μL , depending on the sample concentration and the cation exchange capacity of the column. Using very large sample loops result in a slight drop of peak efficiencies, but this could be offset by the gain in sensitivity. It is much easier to use a large sample loop injection than to do sample preconcentration, as besides requiring the extra hardware, the latter could be subject to contamination from external sources such as the preconcentration pump.

In cases where the samples' cation concentrations are high (or the column's capacity is low), it is better to use a smaller sample loop than to dilute the sample, as once again, the diluting water and vessels used could potentially contaminate the sample.

The CS12A-5 μm 3-mm column has about 1/3 the capacity of the CS12A 4-mm, so overloading of the column will occur at lower concentrations for the same injection loop volume.

For most applications on a CS12A analytical column, a 10 to 50 μL injection loop will be sufficient. Generally, do not inject more than 50 - 100 nanomoles of any one analyte onto the analytical column. Injecting larger amounts than this can result in overloading of the column which affects linearity as well as peak efficiency and asymmetry. This phenomenon will be more prevalent at higher concentrations of the analytes of interest, and especially so in the lower capacity analytical columns.

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 larger number of moles of a sample can result in overloading the column which can affect detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity.

3.5 Sample Concentration

The IonPac CG12A Guard Column or the Low-Pressure Trace Cation Concentrator, TCC-LP1, should be used for trace cation 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 concentrating large volumes of the sample onto a concentrator column and then using this column in place of the sample loop. 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 trace cation concentrator (the TCC-LP1 or the CG12A) leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the trace cation concentrator (TCC-LP1 or the CG12A) for the analytical chemist in these applications is the capability of performing routine trace analyses of sample matrix ions at $\mu\text{g/L}$ levels without extensive and laborious sample pretreatment.

The IonPac CG12A 4-mm Guard Column (P/N 046074) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027) should be used for sample concentration with the IonPac CS12A 4-mm Analytical Column.

The IonPac CG12A 2-mm Guard Column (P/N 046076) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027) must be used for sample concentration with the IonPac CS12A 2-mm, the CS12A-5 μm 3-mm, or the CS12A-MS Analytical Column.

In the case of the CS12A-5 μm 3-mm, either of the other two guards can also be used. Due to flow rate limitations, it is recommended that the CG12A-5 μm 3-mm column not be used as a preconcentrator column. The TCC-LP1, P/N 046027, should be used for trace cation preconcentration work on the CS12A-5 μm 3-mm.

For Trace Cation Concentration work with the CS12A and CS12A-5 μm 0.4 mm Column use the CG12A or CG12A-5 μm 0.4 mm Columns.



The Trace Cation Concentrator (TCC-2, P/N 043103) should not be used for sample concentration. The TCC-2 column packing is a strong cation exchange resin functionalized with sulfonic acid. The recommended IonPac CS12A eluents will not properly elute ions concentrated on this column.

3.6 IonPac CG12A Guard Columns

An IonPac CG12A Guard Column is normally used with the IonPac CS12A Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column. For maximum life of the analytical 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 column or the initial application run for a performance benchmark.

3.7 Eluent Storage

IonPac CS12A columns are designed to be used with sulfuric acid or methanesulfonic acid (MSA) eluents. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

3.8 Cation Self-Regenerating Suppressor & Cation Capillary Electrolytic Suppressor Requirements

A Cation Self-Regenerating Suppressor should be used for applications that require suppressed conductivity detection. Aqueous ionic eluents can be used in all CSRS 300 modes of operation.



NOTE

Solvent containing eluents must be used in the AutoSuppression External Water Mode or in the Chemical Suppression Mode.

Column	Suppressor	P/N
IonPac CS12A 4-mm Analytical Column	CSRS3004-mm	053948
IonPac CS12A-5 μ m 3-mm Analytical Column	CSRS3002-mm	053949
IonPac CS12A 2-mm Analytical Column	CSRS3002-mm	053949
IonPac CS12A 0.4-mm Capillary Column	CCES300	072053
IonPac CS12A-5 μ m 0.4 mm Capillary Column	CCES300	072053

For detailed information on the operation of the Cation Self-Regenerating Suppressor, see Document No. 031370, the “Product Manual for the Cation Self-Regenerating Suppressor 300.” For detailed information on the operation of the Cation Capillary Electrolytic Suppressor, see Document No. 065386.

3.8.1 IC-MS Current Settings for the CSRS 300

Most IC-MS applications use low current for the CSRS 300 suppressors. See section 3.3.4 of the CSRS 300 Manual (Document No. 031370).

3.9 Cation Atlas Electrolytic Suppressor Requirements

A Cation Atlas Electrolytic Suppressor, CAES, may be substituted for the CSRS 300 for applications up to 25 μ eq/min. For detailed information on the operation of the Cation Atlas Electrolytic Suppressor, see Document No. 031770, the “Product Manual for the Cation Atlas Electrolytic Suppressor.”

3.10 Cation MicroMembrane Suppressor Requirements

A Cation MicroMembrane Suppressor, CMMS, may be substituted for the CSRS 300. For detailed information on the operation of the Cation MicroMembrane Suppressor, see Document No. 031728, the Product Manual for the Cation MicroMembrane Suppressor 300.

3.11 Using AutoRegen

Dionex recommends using an AutoRegen® Accessory (P/N 039594) with eluents that do not contain acetonitrile. It should be used with the CSRS 300 in the Chemical Suppression mode or with the CMMS. The AutoRegen Accessory saves regenerant preparation time and reduces regenerant consumption and waste.



CAUTION

Acetonitrile is not compatible with the AutoRegen Cation Regenerant Cartridge. The acetonitrile diffuses into the TBAOH regenerant, concentrates during recirculation and eventually hydrolyzes to acetate and ammonia, depleting the capacity of the AutoRegen Cation Regenerant Cartridge. If acetonitrile is used with suppressed conductivity, a pressurized vessel rather than the AutoRegen must be used.

When using an AutoRegen System, the regenerant passes over the hydroxide form anion exchange resin in the AutoRegen Cation Regenerant Cartridge where specific anionic contaminants (such as chloride ions) are continuously removed from the regenerant (TBAOH) to restore the salt form of the regenerant to the base form. If solvents are used in the eluent, ionic contaminants from the solvent component of the eluent which are not removed by the AutoRegen Regenerant Cartridge slowly accumulate in the regenerant. This results in slowly increasing background conductivity. The rate at which the background conductivity increases versus the required analysis sensitivity will determine how often the regenerant must be changed. It is not necessary to change the AutoRegen Regenerant Cartridge until it is completely expended.

Use Dionex Cation Regenerant Solution (TBAOH, 0.1 M tetrabutylammonium hydroxide, P/N 039602). This ensures maximum system performance. If you are using the AutoRegen Accessory (P/N 039594) equipped with an AutoRegen Cation Regenerant Cartridge (P/N 039563), prepare 0.5 to 1.0 liter of the regenerant. If you plan to use a pressurized vessel, prepare several liters.

Equilibrate the AutoRegen Cation Regenerant Cartridge to new regenerant. When replacing the recycled regenerant, the first 200 mL of the regenerant should be pumped to waste before recycling of the regenerant is started. Utilizing AutoRegen in this manner will allow the use of high regenerant flow rates with the minimum of consumption and waste.

Increase the regenerant flow rate for gradient analysis. To minimize the baseline shift when performing an analysis that requires a H₂SO₄ or methanesulfonic acid step or linear gradient, a high regenerant flow rate (10 - 15 mL/min) is required.

3.12 Detector Requirements

See Section 2, "Comparison of 4-mm, CS12A-5µm 3-mm and 2-mm Ion Chromatography Systems," for 4-mm, CS12A-5µm 3-mm and 2-mm system detector, cell and thermal stabilizer requirements.

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	0.4-mm: 0.4 μ L Loop 4-mm: 25 μ L Loop + 0.8 μ L Injection valve dead volume 3-mm: 25 μ L Loop + 0.8 μ L Injection valve dead volume 2-mm: 2.5 μ L Loop + 0.8 μ L Injection valve dead volume
Column:	4-mm: CS12A 4-mm Analytical Column (+ CG12A 4-mm Guard Column) 3-mm: CS12A-5 μ m 3-mm Analytical Column (+ CG12A-5 μ m 3-mm Guard Column) 2-mm: CS12A 2-mm Analytical Column (+ CG12A 2-mm Guard Column) 0.4-mm: CA12A 0.4 mm Capillary Column (+ CG12A 0.4-mm Capillary Guard Column) 0.4-mm: CS12A-5 μ m 0.4 mm Capillary Column (+ CG12A-5 μ m 0.4 mm Capillary Guard Column)
Eluent:	22 mN H ₂ SO ₄ or 20 mM Methanesulfonic Acid
Eluent Flow Rate:	0.4 x 250 mm: 0.010 mL/min 0.4 x 150 mm: 0.008 mL/min 4-mm: 1.0 mL/min 3-mm: 0.5 mL/min 2-mm: 0.25 mL/min
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 4-mm in AutoSuppression Recycle Mode for the CS12A 4-mm Cation Self-Regenerating Suppressor 300 2-mm in AutoSuppression Recycle Mode for the CS12A-5 μ m 3-mm and 2-mm
or CES Suppressor:	Cation Capillary Electrolytic Suppressor, CCES 300 (0.4-mm)
or AES Suppressor:	Cation Atlas Electrolytic Suppressor (CAES) (if eluent suppression required is less than 25 μ eq/min)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm or 4-mm)
MMS Regenerant:	100 mN tetrabutylammonium hydroxide (TBAOH) Use Dionex Cation Regenerant Solution (P/N 039602)
Expected Background Conductivity:	< 3 μ S
Storage Solution:	Eluent

4.2 Operating Precautions



CAUTION

IonPac CS12A Operation Precautions
Operate below 4,000 psi (27.57 MPa)
Filter and Degas Eluents
Filter Samples

Do NOT use this column with alcohols

4.3 Chemical Purity Requirements

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

4.3.1 Deionized Water

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

4.3.2 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 only concentrated sulfuric acid (H_2SO_4), ACS grade or BAKER INSTRA-ANALYZED® for trace metals.
- B. Use Fluka or Aldrich Methanesulfonic Acid (MSA) (>99% pure).
- C. Use Dionex Cation Regenerant Solution, tetrabutylammonium hydroxide (TBAOH), P/N 039602, to ensure maximum system performance when operating with a CMMS 300 or a CSRS 300 in the Chemical Suppression Mode.
- D. Use deionized water with a specific resistance of 18.2 megohm-cm to make all standards, eluents and regenerants.

4.4 Preparation of Eluent Stock Solution Concentrates



WARNING

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

Avoid breathing the vapors.

Always use these reagents in a fume hood. Wear gloves and goggles.

4.4.1 1.0 N Sulfuric Acid Stock Solution

This solution will be used in the preparation of each of the eluents in Section 5, "Example Applications."

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 at least 17.8 megohm-cm. Dilute to the 1 liter mark and mix thoroughly.

In Other Words:

$$\begin{aligned} \text{FW of H}_2\text{SO}_4 &= 98.08 \text{ g} \\ \text{H}_2\text{SO}_4 \text{ concentration} &= 98\% \end{aligned}$$

Therefore, for a 1 N H_2SO_4 solution, weigh out:

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

4.4.2 1.0 N Methanesulfonic Acid (MSA) Stock Solution

A 1.0 N methanesulfonic acid stock solution can be prepared as follows:

Weigh out 96.10 g of methanesulfonic acid (MSA). Carefully add this amount to a 1-liter volumetric flask containing about 500 mL of deionized water. Dilute to the mark and mix thoroughly.

4.5 Eluent Preparation

Eluent: X mN Sulfuric Acid (H₂SO₄) or Methanesulfonic acid (MSA)

Using the table below, pipet X.0 mL of the 1.0 N H₂SO₄ or 1.0 N MSA eluent concentrate (see Section 5.1, “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

mN H ₂ SO ₄ or MSA	# mL H ₂ SO ₄ or MSA
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

4.6 Eluents with Solvents

Solvents can be added to the ionic eluents used with IonPac CS12A columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. At Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-acetonitrile mixture varies. The practical back pressure limit for the IonPac CS12A columns is 4,000 psi (27.57 MPa).

The IonPac CS12A and the CS12A-MS are compatible with the HPLC solvents listed in Table 5, “HPLC Solvents for Use with the CS12A Columns.” Alcohols, however, should be avoided, since the column capacity for cation exchange may be reduced due to the reversible formation of esters in the column packing. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 6
HPLC Solvents for Use with IonPac CS12A and CS12A-MS Columns

Solvent	Maximum Operating Concentration
Acetonitrile	100%
Methanol	0%
2-Propanol	0%
Tetrahydrofuran	20%

4.6.1 Making and Using Eluents that Contain Solvents

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

**NOTE**

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

Always degas and store all eluents in glass or plastic eluent bottles pressurized with helium. Only helium can be used to purge and degas ionic eluents containing solvents, since nitrogen is soluble in solvent containing eluents.

Avoid creating high viscosity pressure fronts that may disrupt the column packing when the eluent solvent component is changed. To do this, equilibrate the column 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.

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

**CAUTION**

The Cation Self-Regenerating Cation Suppressor (CSRS 300) must be operated in the AutoSuppression External Water Mode or the Chemical Suppression Mode when using eluents containing solvents.

Acetonitrile is not compatible with the Cation Regenerant Cartridge when using an AutoRegen Accessory Unit. The acetonitrile diffuses into the TBAOH regenerant, concentrates during recirculation and eventually hydrolyzes to acetate and ammonia, depleting the capacity of the AutoRegen Cation Regenerant Cartridge. If acetonitrile is used with suppressed conductivity, a pressurized vessel rather than the AutoRegen must be used.

SECTION 5 - EXAMPLE APPLICATIONS

The chromatograms in this section were obtained using columns that reproduced the Production test Chromatogram (see Section 5.3, “Production Test Chromatogram”) on optimized Ion Chromatographs (see Section 3, “Installation”). Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components and laboratory temperatures.

Before attempting any of the following example applications, take the time to ensure that your system is properly configured. Ensure that all of the eluents have been made from high purity reagents and deionized water. All water used in the preparation of eluents should be degassed, deionized water. For chemical purity requirements, see Section 4.3, “Chemical Purity Requirements.” After running synthetic standards to calibrate your system, you may find that real sample matrices foul your columns. For this reason it is always advisable to use a guard column to protect the analytical column. If column performance deteriorates and it is determined that the guard or the analytical column has been fouled, refer to the column cleanup protocols in, “Column Care.” If your sample matrices are relatively low in ionic concentration, you may be able to increase the sensitivity of your system by using sample concentration techniques (see Section 4.5, “Sample Concentration”).

5.1 Production Test Chromatogram

Isocratic elution of cations on the IonPac CS12A Analytical Column has been optimized utilizing a sulfuric acid or methanesulfonic acid eluent. Using the appropriate eluent, the weak carboxylate functionalized packing isocratically separates mono- and divalent cations in a single injection. To guarantee that all IonPac CS12A Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Volume: 25 µL Loop (4-mm or 3-mm)
 Column: CS12A Analytical Column (4-mm or 3-mm)
 Eluent: 20 mN Methanesulfonic Acid (4-mm or 3-mm)
 Eluent Flow Rate: 1.0 mL/min (4-mm) at room temperature
 0.5 mL/min (3-mm) at 30 °C
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode (used with CS12A 4-mm)
 Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode (used with CS12A-5µm 3-mm)
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (2-mm or 4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 µeq/min
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent



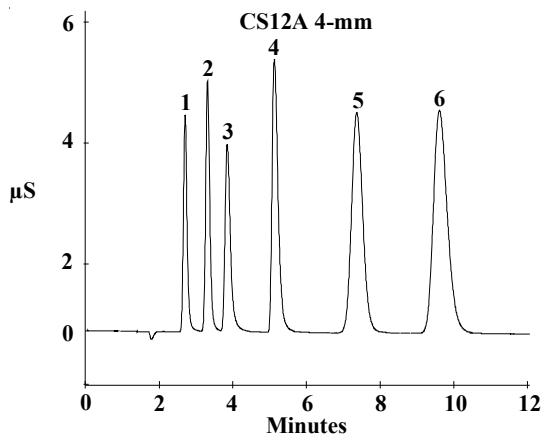
NOTE

2-mm application chromatograms are the same as the 4-mm using a 6.25 µL sample loop, 0.25 mL/min eluent flow rate, and a CSRS 300 2-mm suppressor.

CS12A 4-mm

Analyte	mg/L
1. Lithium	0.5
2. Sodium	2.0
3. Ammonium	2.5
4. Potassium	5.0
5. Magnesium	2.5
6. Calcium	5.0

where 1 mg/L = 1 ppm



CS12A-5µm 3-mm

Analyte	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Potassium	1.0
5. Magnesium	0.5
6. Calcium	1.0

where 1 mg/L = 1 ppm

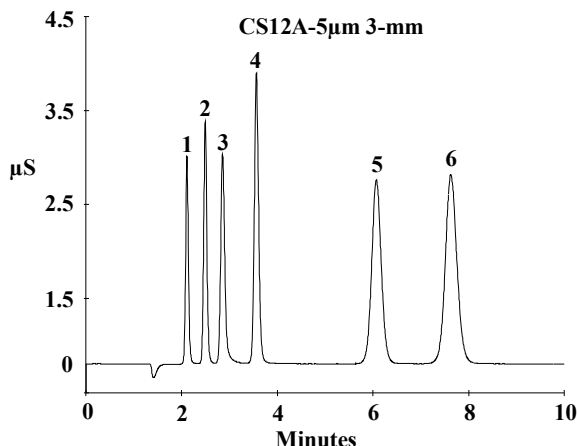


Figure 8a
IonPac CS12A Production Test Chromatogram

5.1.1 IonPac CS12A and CS12A-5µm Capillary Column Production Test Chromatogram

Eluent: 20 mM Methanesulfonic acid
Eluent Flow Rate: 0.01 mL/min
Temperature: Ambient Temperature
Detection: Suppressed Conductivity
Suppressor: Cation Capillary Electrolytic Suppressor (CCES-300)
 AutoSuppression® Recycle Mode
Applied Current: 7 mA
Injection Volume: 400 nL
Storage Solution: Eluent

No.	Peak Name	Ret.Time (min)	Concentration (mg/L)
1	Lithium	2.84	0.5
2	Sodium	3.36	0.5
3	Ammonium	3.83	0.5
4	Potassium	4.82	0.5
5	Magnesium	7.16	0.5
6	Calcium	9.00	5.0

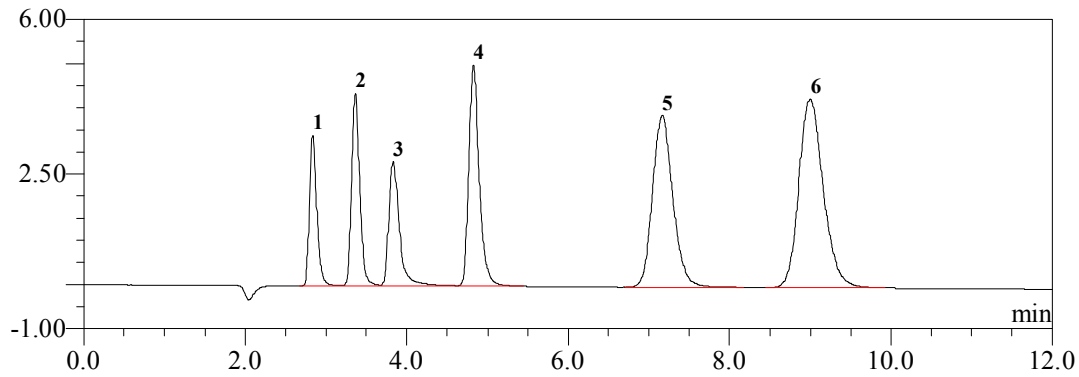


Figure 8b
IonPac CS12A Capillary Column Production Test Chromatogram

Eluent: 20 mM Methanesulfonic acid
Eluent Source: EGC-MSA Capillary Cartridge
Eluent Flow Rate: 0.008 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Cation Capillary Electrolytic Suppressor (CCES-300)
 AutoSuppression® Recycle Mode
Applied Current: 7 mA
Injection Volume: 400 nL
Storage Solution: Eluent

No.	Peak Name	Ret. Time (min)	Concentration (mg/L)
1	Lithium	2.84	0.1
2	Sodium	3.36	0.4
3	Ammonium	3.83	0.5
4	Potassium	4.82	1.0
5	Magnesium	7.16	0.5
6	Calcium	9.00	1.0

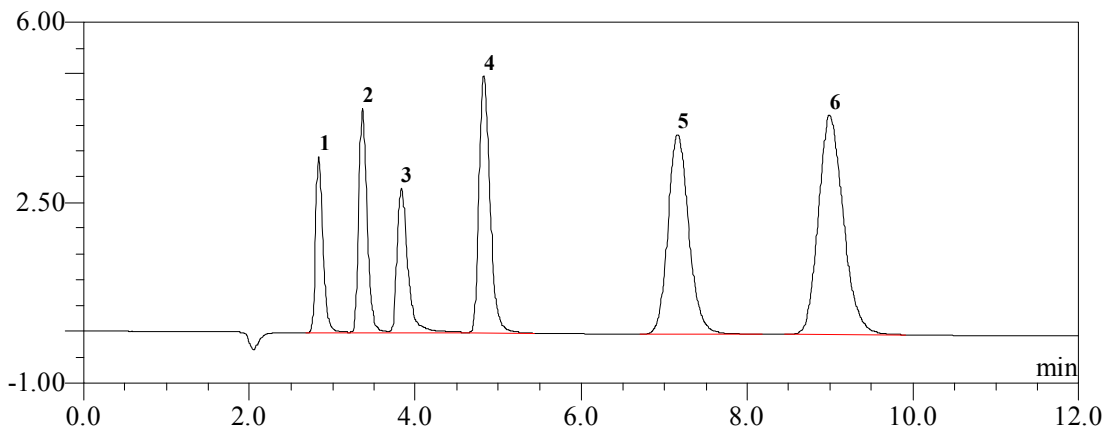


Figure 8c
IonPac CS12A-5µm Capillary Column Production Test Chromatogram

5.1.2 IonPac CS12A-MS Production Test Chromatogram

Sample Volume: 2.5 µL Loop
 Column: CS12A-MS (2 x 100 mm)
 Eluent: 20 mM Methane sulfonic acid
 Eluent Flow Rate: 0.25 mL/min
 AES Suppressor: CAES, Atlas Cation Electrolytic Suppressor
 or SRS Suppressor CSRS 300 (2-mm)
 Temperature: Room Temperature

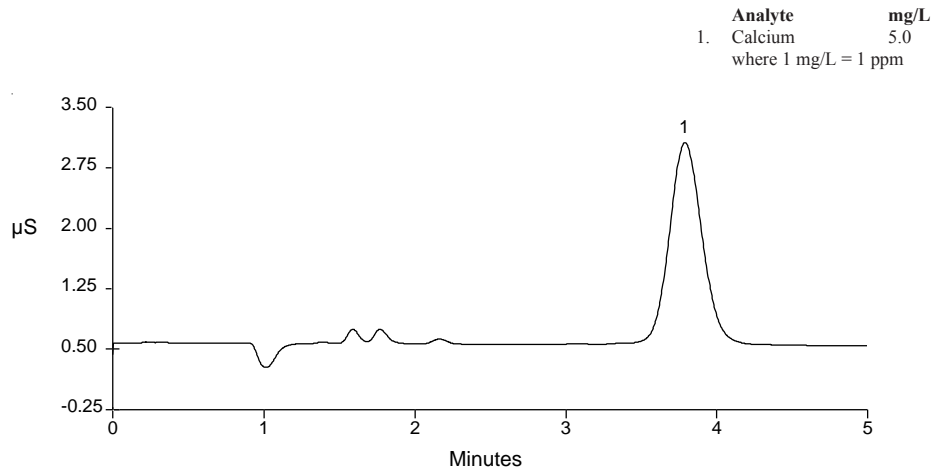


Figure 8d
IonPac CS12A-MS Production Test Chromatogram

5.2 Fast Separation of the Six Common Cations Using the IonPac CS12A-5 μ m (0.4 x 150 mm) Capillary Column

The CS12A-5 μ m Capillary Column provides faster analysis time for inorganic cations and ammonium, reduced eluent consumption and increased sensitivity.

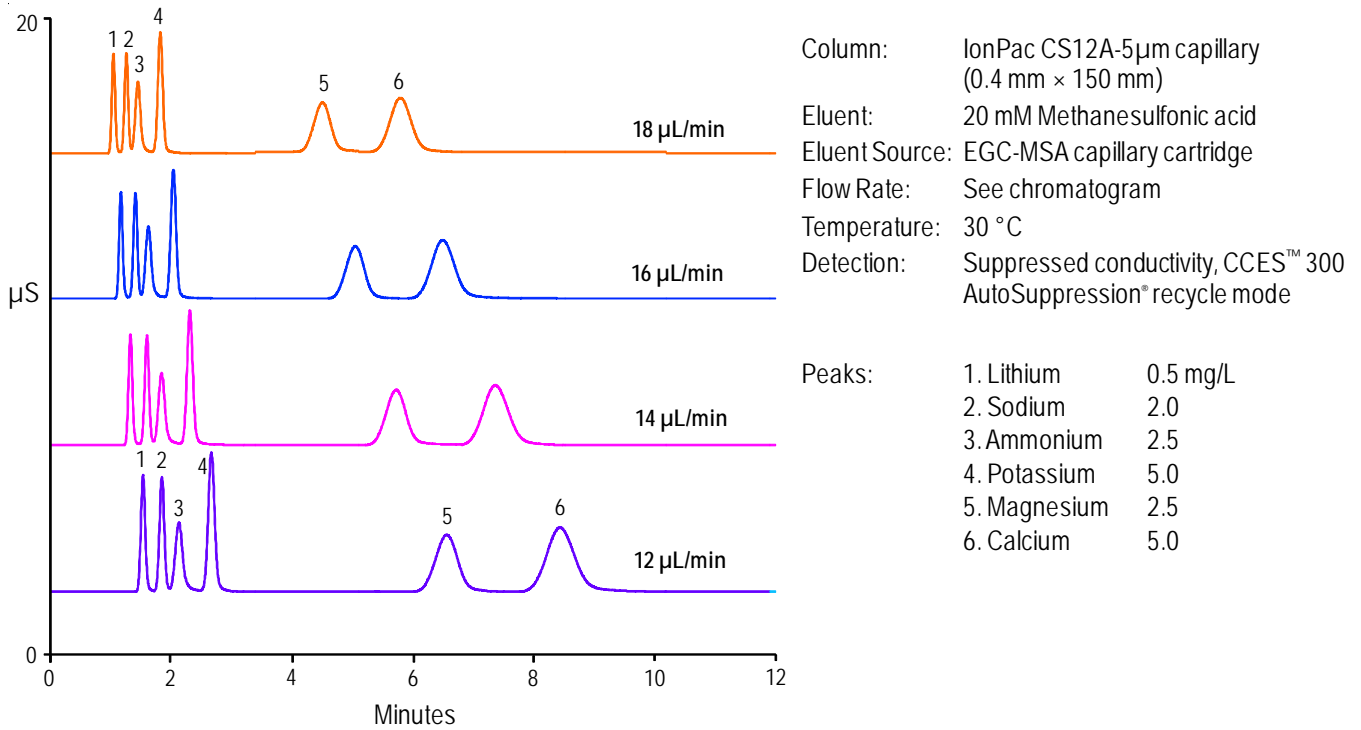


Figure 9
Fast Separation of the Six Common Cations Using the IonPac CS12A-5 μ m Capillary Column

5.3 Isocratic Separation of the Six Common Cations using an IonPac CS12A (0.4 x 250 mm) Capillary Column

The CS12A Capillary Column separates the Mono and divalent Cations in less than 15 minutes using an isocratic MSA eluent delivered by and Eluent Generator.

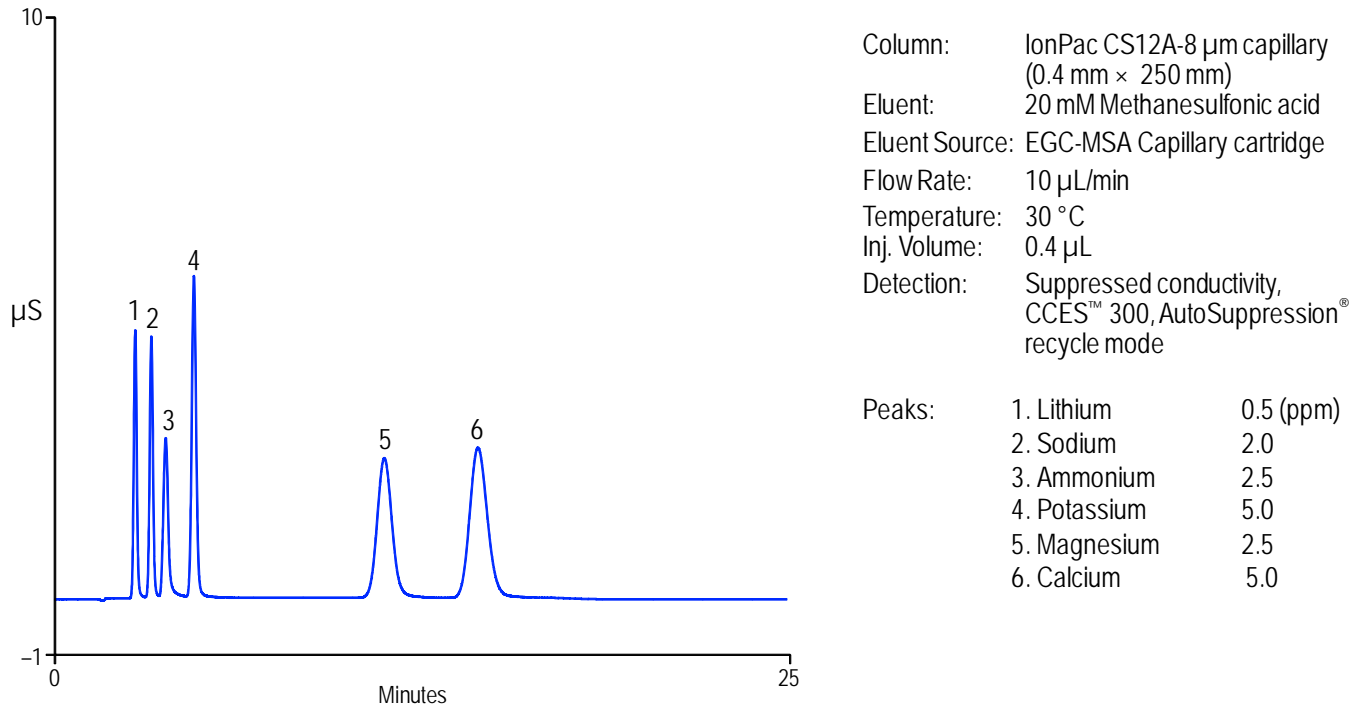


Figure 10
Isocratic Separation of the Six Common Cations using an IonPac CS12A Capillary Column

5.4 6-Cation Fast Run (Li⁺, Na⁺, NH₄⁺, K⁺, Mg²⁺, Ca²⁺)

Isocratic elution of ammonia, selected alkali metals, and selected alkaline earth cations (Li⁺, Na⁺, NH₄⁺, K⁺, Mg²⁺, Ca²⁺) can be completed in 7 minutes with a CS12A 4-mm column, or within 3 minutes with a CS12A-5µm 3-mm column at optimized conditions.

Sample Volume:	25 µL Loop (4-mm or 3-mm)
Column:	CS12A Analytical Column (4-mm)
Eluent:	30 mN H ₂ SO ₄ (4-mm)
	33 mN Methanesulfonic Acid (3-mm)
Eluent Flow Rate:	1.0 mL/min (4-mm) at room temperature
	0.5 mL/min (3-mm) 30 °C
	0.8 mL/min (3-mm) 30 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode (used with CS12A 4-mm)
	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode (used with CS12A-5µm 3-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm or 4-mm)
MMS Regenerant:	100 mN tetrabutylammonium hydroxide TBAOH
or AES:	Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 µeq/min
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



2-mm application chromatograms are the same as the 4-mm using a 6.25 µL sample loop, 0.25 mL/min eluent flow rate, and a CSRS 300 2-mm suppressor.

NOTE

CS12A 4-mm

Analyte	mg/L
1. Lithium	0.5
2. Sodium	2.0
3. Ammonium	2.5
4. Potassium	5.0
5. Magnesium	2.5
6. Calcium	5.0

where 1 mg/L = 1 ppm

CS12A-5µm 3-mm

Analyte	mg/L
1. Lithium	0.12
2. Sodium	0.5
3. Ammonium	0.62
4. Potassium	1.25
5. Magnesium	0.62
6. Calcium	1.25

where 1 mg/L = 1 ppm

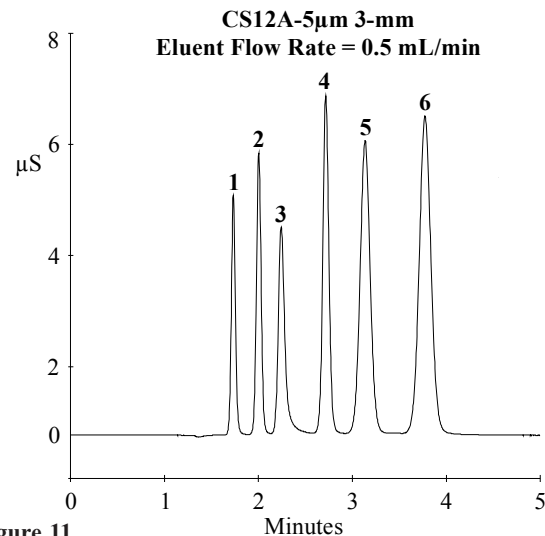
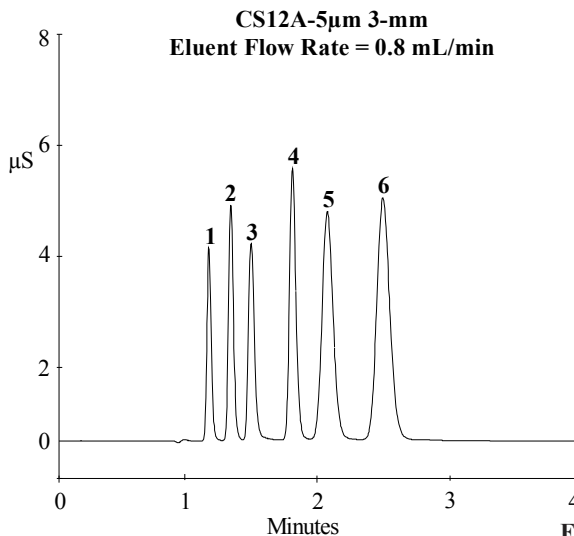
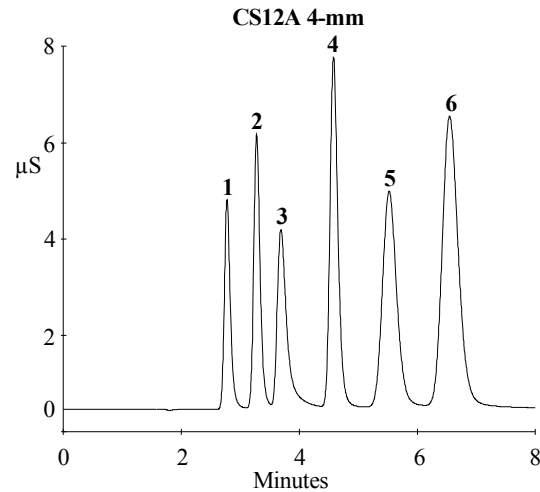


Figure 11
6-Cation Fast Run (Li⁺, Na⁺, NH₄⁺, K⁺, Mg²⁺, Ca²⁺)

5.5 Trace Cations by Direct Injection

Using a 500 µL injection loop on a CS12A 2-mm column or 25 µL on a CS12A-5µm 3-mm, low ppb levels of Group I and II cations and ammonium can be determined. Injection loop volume can be increased up to 1000 µL to provide quantitation of low ppt levels of cations with the CS12A-5µm 3-mm column.

Sample Volume: 500 µL loop (2-mm)
 25 µL loop (3-mm)

Column: CS12A, 2-mm
 CS12A-5µm 3-mm plus Guard

Eluent: 22 mN H₂SO₄ (2-mm)
 20 mM Methanesulfonic acid (3-mm)

Eluent Flow Rate: 0.25 mL/min (2-mm) at room temperature
 0.5 mL/min (3-mm) at room temperature

SRS Suppressor: Cation Self-Regenerating Suppressor 300 (2-mm) in
 AutoSuppression Recycle Mode (used with CS12A 2-mm)
 Cation Self-Regenerating Suppressor 300 (2-mm) in
 AutoSuppression Recycle Mode, (used with CS12A-5µm 3-mm)

or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (2-mm)

MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH

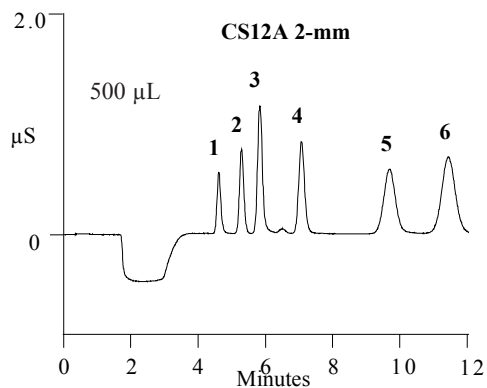
or AES: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 µeq/min

Expected Background Conductivity: < 2 µS

Storage Solution: Eluent

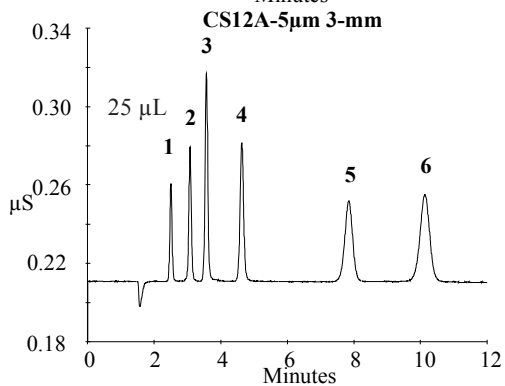
CS12A 2-mm	
Analyte	µg/L
1. Lithium	1.4
2. Sodium	3.8
3. Ammonium	7.6
4. Potassium	12.8
5. Magnesium	6.9
6. Calcium	14.8

where 1 mg/L = 1 ppm



CS12A-5µm 3-mm	
Analyte	µg/L
1. Lithium	2.5
2. Sodium	10
3. Ammonium	12.5
4. Potassium	25
5. Magnesium	12.5
6. Calcium	25

where 1 mg/L = 1 ppm



CS12A-5µm 3-mm	
Analyte	µg/L
1. Lithium	0.25
2. Sodium	1.0
3. Ammonium	1.25
4. Potassium	2.5
5. Magnesium	1.25
6. Calcium	2.5

where 1 mg/L = 1 ppm

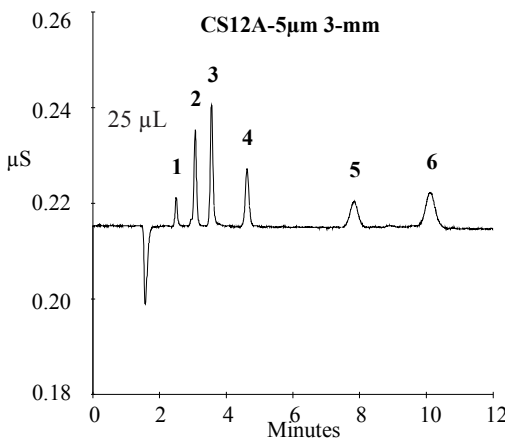


Figure 12
Trace Cations by Direct Injection

5.6 Isocratic Elution of Ammonium, Alkali Metals and Alkaline Earth Metals

With a CS12A 4-mm column, isocratic elution of ammonia, plus Group I and Group II cations (Li^+ , Na^+ , NH_4^+ , K^+ , Rb^+ , Cs^+ , Mg^{2+} , Ca^{2+} , Sr^{2+} and Ba^{2+}) can be completed in 25 minutes using a methanesulfonic acid eluent (MSA). With a CS12A-5 μm 3-mm column and guard, the analysis can be done within 20 minutes. A sulfuric acid eluent should not be used for the determination of barium.

Sample Volume:	25 μL Loop (4-mm or 3-mm)
Column:	CS12A Analytical Column (4-mm) CG12A Guard + CS12A Analytical Column (3-mm)
Eluent:	18 mN MSA (4-mm) 20 mN MSA (3-mm)
Eluent Flow Rate:	1.0 mL/min (4-mm) at room temperature 0.5 mL/min (3-mm) at room temperature
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode (used with CS12A 4-mm) Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode, (used with CS12A-5 μm 3-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm or 4-mm)
MMS Regenerant:	100 mN tetrabutylammonium hydroxide TBAOH
or AES Suppressor:	Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 $\mu\text{eq}/\text{min}$
Expected Background Conductivity:	< 2 μS
Storage Solution:	Eluent

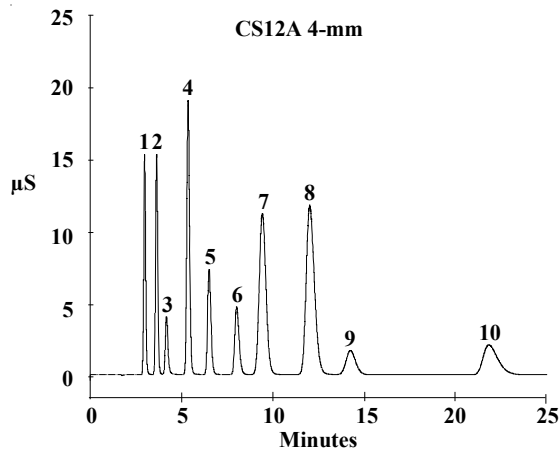
NOTE

2-mm application chromatograms are the same as the 4-mm using a 6.25 μL sample loop, 0.25 mL/min eluent flow rate, and a CSRS 300 2-mm suppressor.

CS12A 4-mm

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

where 1 mg/L = 1 ppm



CS12A-5 μm 3-mm

Analyte	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Potassium	1.0
5. Rubidium	5.0
6. Cesium	5.0
7. Magnesium	0.5
8. Calcium	1.0
9. Strontium	5.0
10. Barium	5.0

where 1 mg/L = 1 ppm

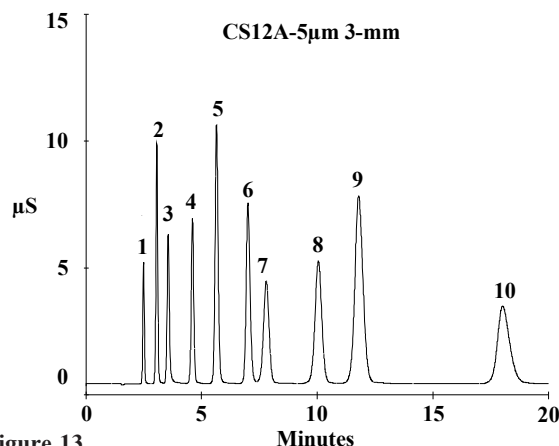


Figure 13
Isocratic Elution of Ammonium, Alkali Metals and Alkaline Earth Metals
(Li^+ , Na^+ , NH_4^+ , K^+ , Rb^+ , Cs^+ , Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+})

5.7 Fast Elution of Ammonium, Alkali Metals and Alkaline Earth Metals with the CS12A-5µm 3-mm

Isocratic elution of ammonia, plus Group I and Group II cations (Li⁺, Na⁺, NH₄⁺, K⁺, Rb⁺, Cs⁺, Mg²⁺, Ca²⁺, Sr²⁺ and Ba²⁺) can be completed in 10 minutes using a methanesulfonic acid eluent (MSA). A sulfuric acid eluent should not be used for the determination of barium.

Sample Volume: 25 µL Loop
 Column: CS12A-5µm 3-mm Analytical and Guard Columns
 20 mN MSA at 30 °C
 Eluent Flow Rate: 1.0 mL/min
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 µeq/min
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent

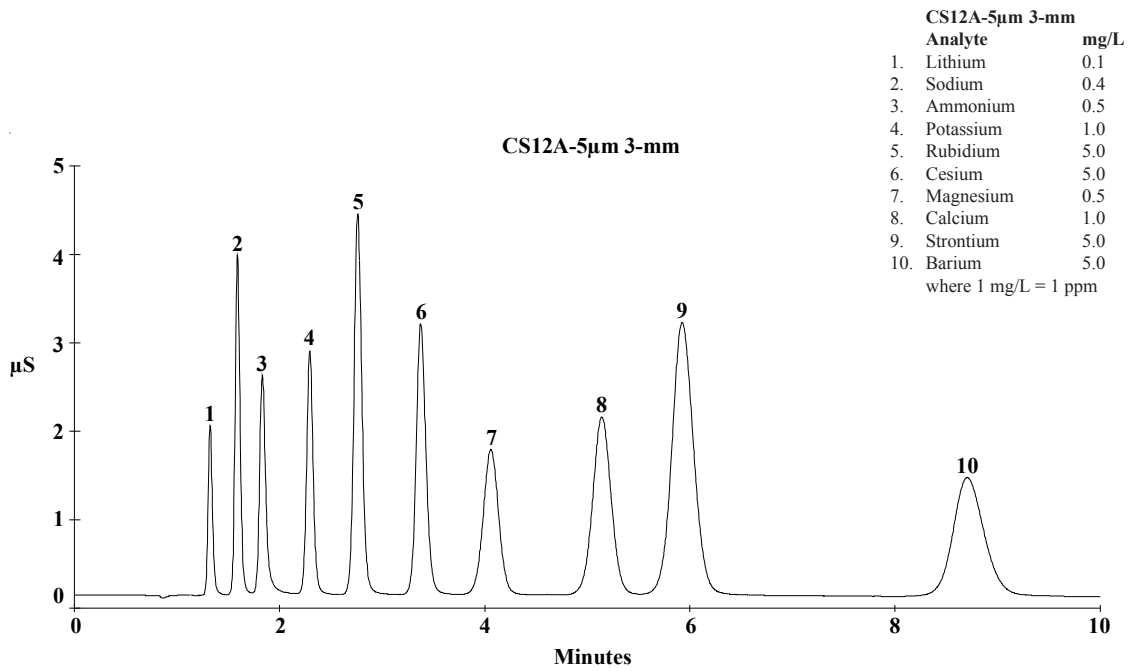


Figure 14
Isocratic Elution of Ammonium, Alkali Metals and Alkaline Earth Metals
(Li⁺, Na⁺, NH₄⁺, K⁺, Rb⁺, Cs⁺, Mg²⁺, Ca²⁺, Sr²⁺, Ba²⁺)
at higher flow rate and 30 °C

5.8 Isocratic Elution of 6 Cations plus Morpholine

Sample Volume: 25 µL Loop (4-mm or 3-mm)
 Column: CG12A (4-mm) Guard + CS12A Analytical Column (4-mm)
 CG12A-5µm 3-mm Guard + CS12A-5µm 3-mm Analytical Column
 Eluent: 20 mN H₂SO₄ (4-mm)
 20 mN Methanesulfonic Acid (3-mm)
 Eluent Flow Rate: 1.0 mL/min (4-mm) at room temperature
 0.5 mL/min at room temperature (3-mm)
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in
 AutoSuppression Recycle Mode (used with CS12A 4-mm)
 Cation Self-Regenerating Suppressor 300 (2-mm) in
 AutoSuppression Recycle Mode (used with CS12A-5µm 3-mm)
 or MMS Suppressor: Cation MicroMembrane Suppressor , CMMS 300 (2-mm or 4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES Suppressor: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 µeq/min
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent



2-mm application chromatograms are the same as the 4-mm using a 6.25 µL sample loop, 0.25 mL/min eluent flow rate, and a CSRS 300 2-mm suppressor.

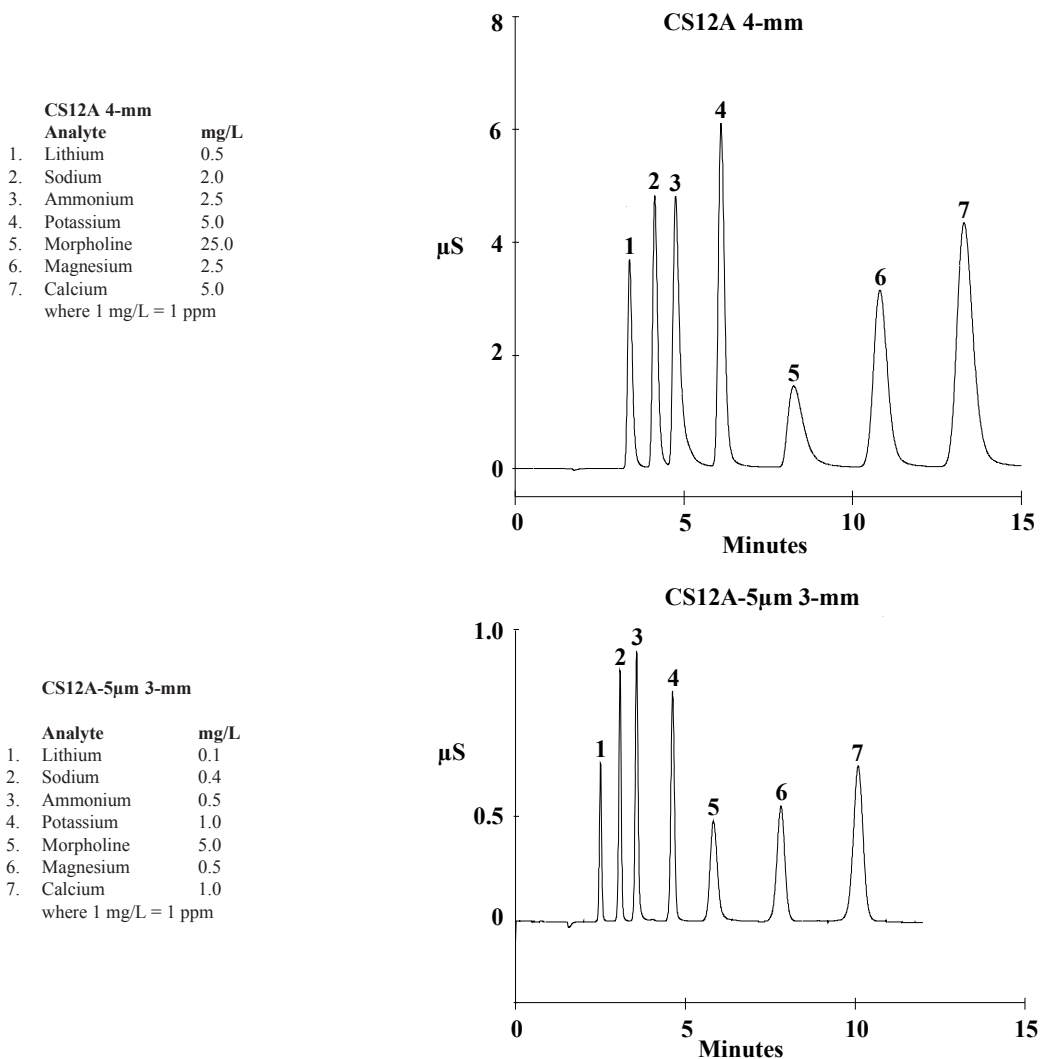


Figure 15
Isocratic Elution of 6 Cations plus Morpholine
(Li⁺, Na⁺, NH₄⁺, K⁺, Morpholine, Mg²⁺ and Ca²⁺)

5.9 Isocratic Elution of 6 Cations plus Manganese with the CS12A-5 μ m 3-mm

Isocratic elution of ammonia, manganese plus Group I and Group II cations (Li^+ , Na^+ , NH_4^+ , K^+ , Mg^{2+} , Mn^{2+} and Ca^{2+}) can be completed in 12 minutes. At higher temperatures (e.g. 30 °C) manganese is not well resolved from calcium.

Sample Volume: 25 μL Loop
 Column: CG12A-5 μm 3-mm Guard + CS12A-5 μm 3-mm Analytical Column
 Eluent: 20 mN Methanesulfonic Acid
 Eluent Flow Rate: 0.5 mL/min at room temperature
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 $\mu\text{eq}/\text{min}$
 Expected Background Conductivity: < 2 μS
 Storage Solution: Eluent

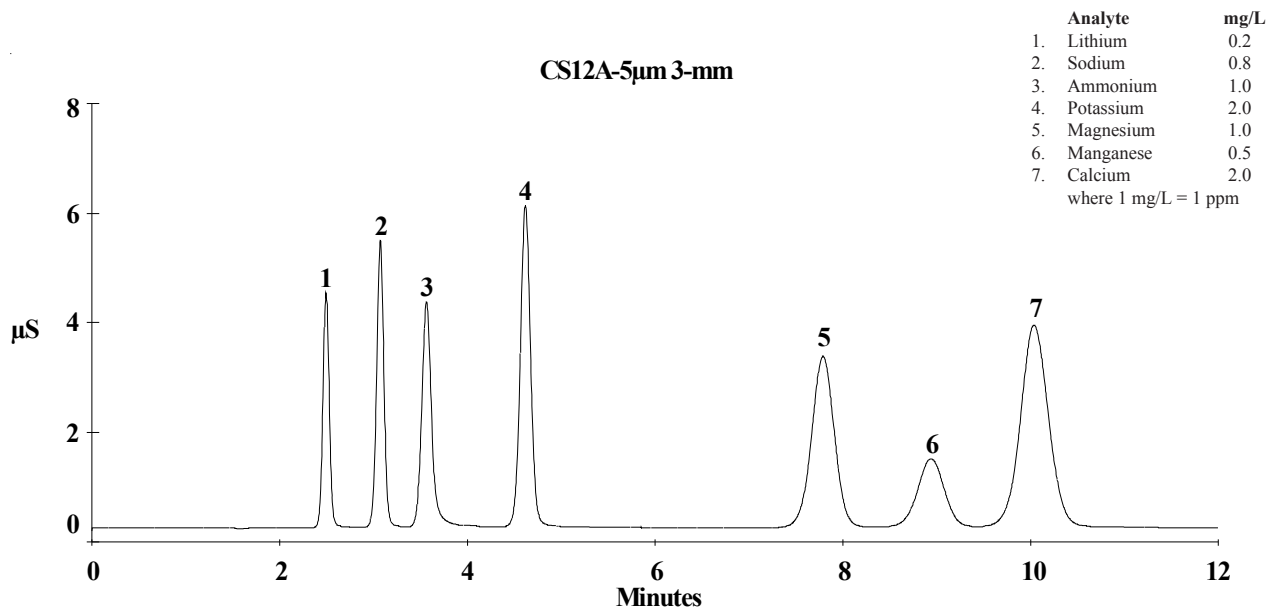


Figure 16
Isocratic Elution of 6 Cations plus Manganese
(Li^+ , Na^+ , NH_4^+ , K^+ , Mg^{2+} , Mn^{2+} and Ca^{2+}) with the CS12A-5 μ m 3-mm

5.10 Comparison of Sulfuric Acid and Methanesulfonic Acid Eluents

The following runs compare the sulfuric acid selectivity to methanesulfonic acid selectivity. To isocratically resolve Mn^{2+} from Mg^{2+} and Ca^{2+} , use a methanesulfonic acid eluent.

Sample Volume: 25 μ L Loop
 Column: CS12A Analytical Column (4-mm)
 Eluent: See Chromatogram
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES Suppressor: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 μ eq/min
 Expected Background Conductivity: < 2 μ S
 Storage Solution: Eluent



2-mm application chromatograms are the same as the 4-mm using a 6.25 μ L sample loop, 0.25 mL/min eluent flow rate, and a CSRS 300 2-mm suppressor.

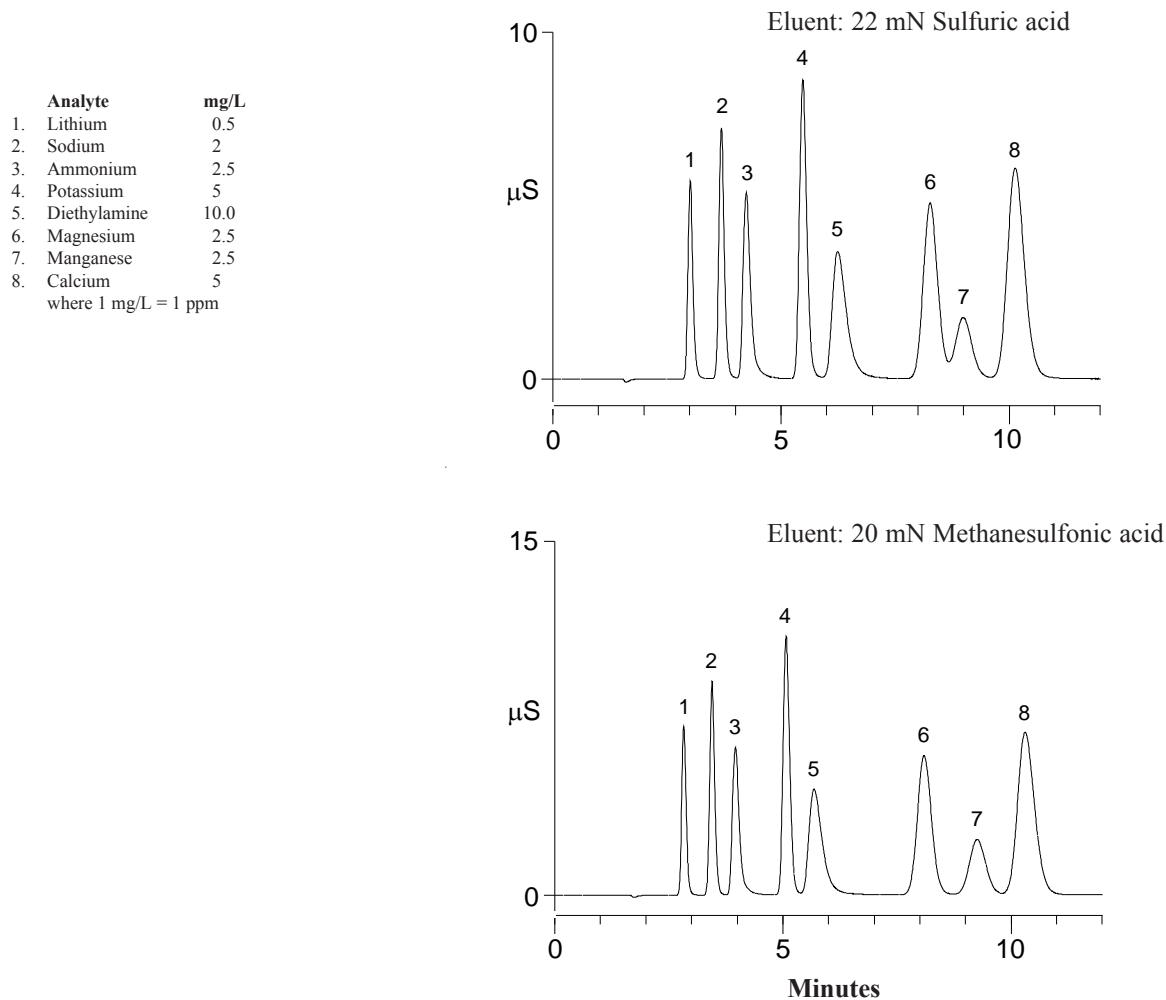


Figure 17
IonPac CS12A Eluent Selectivity Comparison Using 8 Cations

5.11 1000:1 Ratio Sodium to Ammonium

The following example shows a ratio of 1000:1 of sodium to ammonium on the CS12A using an eluent step change. For samples with greater than 1000:1 of sodium to ammonium or ammonium to sodium, the IonPac CS16 column is recommended. Please contact your local Dionex office for more details on this column.

Sample Volume: 25 μ L Loop
 Column: CS12A Analytical Column (4-mm)
 Eluent: Step change at 12 min from 3 mN to 20 mN Methanesulfonic acid
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 or AES: Cation Atlas Electrolytic Suppressor, CAES, if eluent suppression required is less than 25 μ eq/min
 Expected Background Conductivity: < 2 μ S
 Storage Solution: Eluent

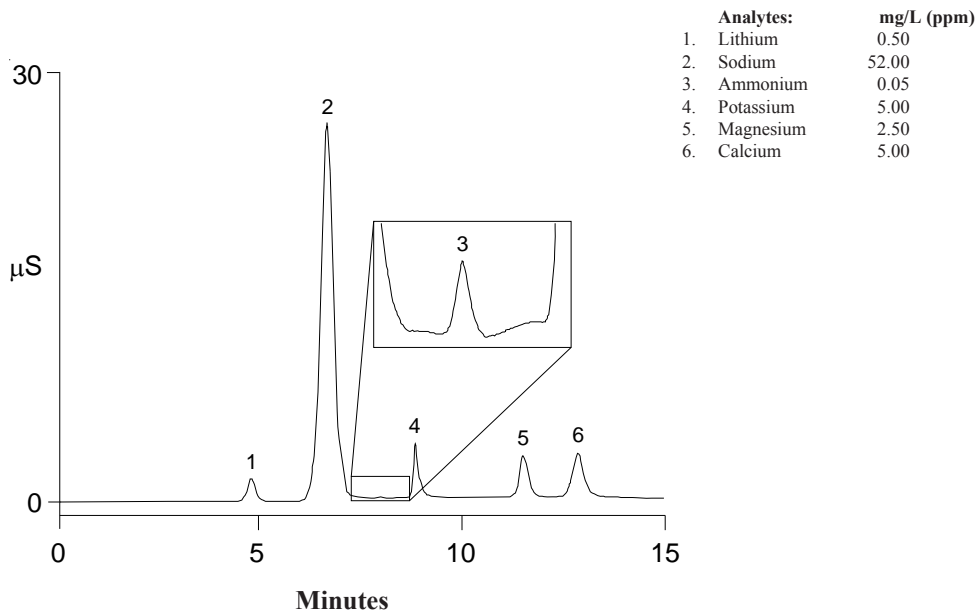


Figure 18
Trace-level Quantification of Ammonium in Brine (1000:1 ratio)

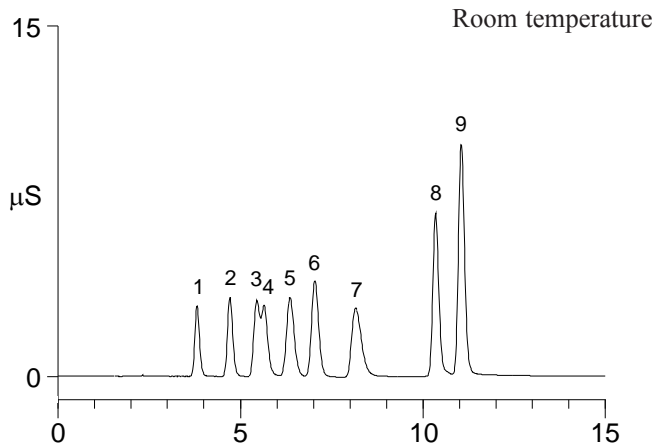
5.12 Comparison of Amine Selectivity at Different Temperatures Using Gradient Elution

The following runs compare the methylamine selectivity of the CS12A at room temperature and 60 °C.

Sample Volume: 25 µL Loop
 Column: CS12A Analytical Column (4-mm)
 Eluent: E1: Degassed Type I Reagent Grade Water
 E2: 100 mN H₂SO₄
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode at Room Temperature
 AutoSuppression External Water Mode at 60 °C, (The CSRS 300 **must** be placed outside of the column oven using the shortest connecting tubing possible.)
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm); place outside of chromatographic oven
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent

Analyte	mg/L
1. Lithium	0.5
2. Sodium	2
3. Ammonium	2.5
4. Methylamine	5
5. Potassium	5
6. Dimethylamine	10
7. Trimethylamine	15
8. Magnesium	2.5
9. Calcium	5

where 1 mg/L = 1 ppm



Gradient Conditions

Time	%E1	%E2	Comments
Init	84	16	Initial Eluent
0.0	84	16	Load Position
0.1	84	16	Inject
0.3	84	16	Load Position
4.0	84	16	End of Isocratic, Begin Gradient
8.0	60	40	End Gradient, Begin Isocratic
12.0	60	40	End Isocratic
12.1	84	16	Initial Conditions

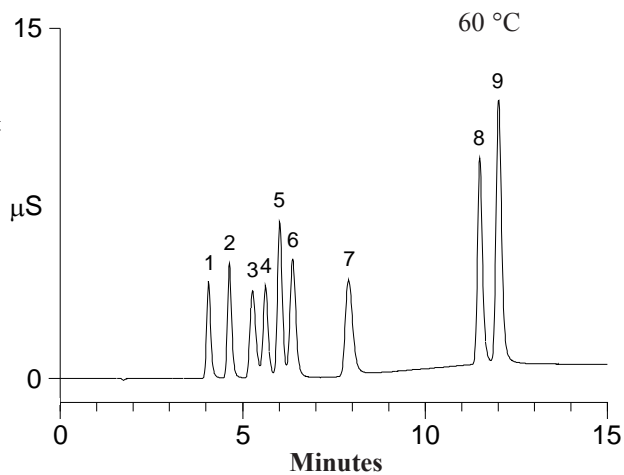


Figure 19
IonPac CS12A Amine Selectivity Comparison at Different Temperatures

5.13 Separation of Alkyl Amines at Elevated Temperature Using Gradient Elution

The following run demonstrates the separation of alkyl amines at 60 °C using gradient elution. The elevated temperature allows the use of a solvent-free eluent.

Sample Volume: 25 µL Loop
 Column: CS12A Analytical Column (4-mm)
 Eluent: E1: Degassed Type I Reagent Grade Water
 E2: 100 mN H₂SO₄
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 Temperature: 60 °C
 (The CSRS 300 **must** be placed outside of the column oven using the shortest connecting tubing possible.)
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression External Water Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent

Time	Gradient Conditions		Comments
	%E1	%E2	
Init	69	31	Initial Eluent
0.0	69	31	Load Position
0.1	69	31	Inject, Begin Gradient
(0.3			Return to Load Position)
10.0	50	50	End Gradient, Begin Isocratic
19.9	50	50	End Isocratic
20.0	69	31	Initial Conditions

Analyte	mg/L
1. Ethylamine	5
2. Propylamine	7.5
3. tert-Butylamine	12.5
4. sec-Butylamine	12.5
5. iso-Butylamine	12.5
6. n-Butylamine	37.5
7. 1,2-Propanediamine	20
8. 1,2-Dimethylpropylamine	20
9. Di-n-propylamine	40

where 1 mg/L = 1 ppm

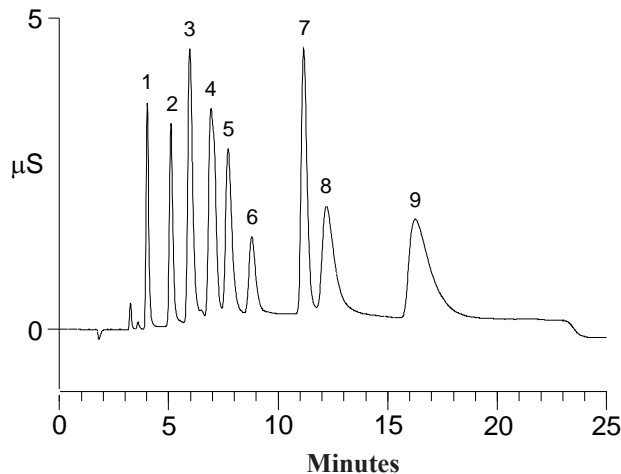


Figure 20
IonPac CS12A Elution of Alkyl Amines at Elevated Temperature Using Gradient Elution

5.14 Separation of Aliphatic Quaternary Amines Using Gradient Elution

The following run demonstrates the separation of aliphatic quaternary amines at 30 °C using gradient elution.

Sample Volume: 25 µL Loop
 Column: CS12A Analytical Column (4-mm)
 Cation Trap Column (CTC-1)
 Eluent: E1: 200 mN H₂SO₄
 E2: 100% Acetonitrile
 E3: Degassed deionized water
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 Temperature: 30 °C
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in Chemical Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 Expected Background Conductivity: 1.8 µS
 Storage Solution: Eluent

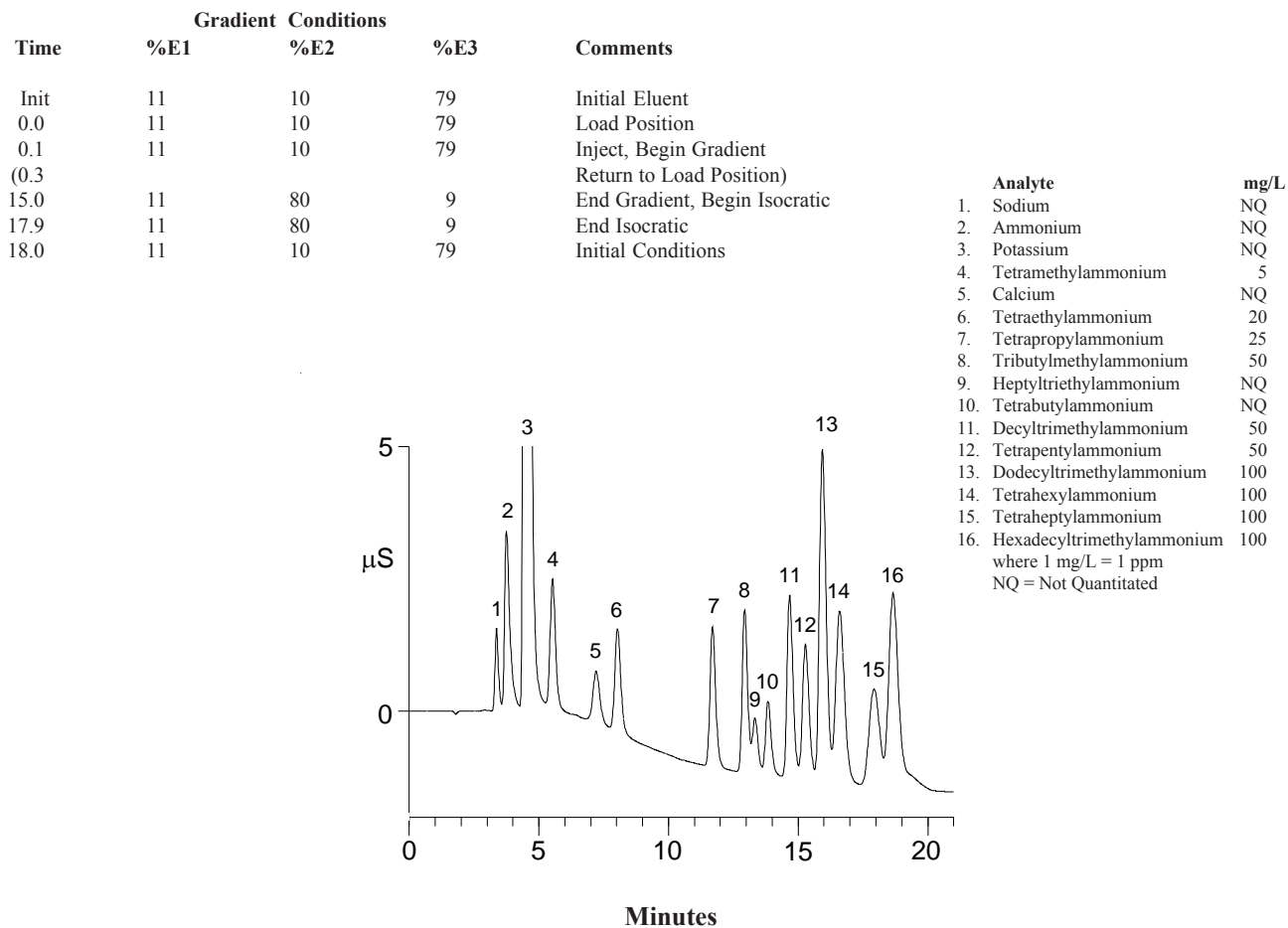


Figure 21
 IonPac CS12A Elution of Aliphatic Quaternary Amines at Elevated Temperature Using Gradient Elution

5.15 Separation of Alkyl Diamines at Elevated Temperature Using Gradient Elution

The following run demonstrates the separation of alkyl diamines at 40 °C using gradient elution.

Sample Volume: 25 µL Loop
 Column: CS12A Analytical Column (4-mm)
 Cation Trap Column (CTC-1)
 Eluent: E1: 200 mN H₂SO₄
 E2: 10% Acetonitrile
 E3: 100% Acetonitrile
 E4: Degassed deionized water
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 Temperature: 40 °C
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in Chemical Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 Expected Background Conductivity: 1.9 µS
 Storage Solution: Eluent

Time	Gradient Conditions				Comments
	%E1	%E2	%E3	%E4	
Init	11	20	0	69	Initial Eluent
0.0	11	20	0	69	Load Position
0.1	11	20	0	69	Inject, Begin 1st Gradient
(0.3					Return to Load Position)
10.0	22	0	16	62	End 1st Gradient, Begin 2nd Gradient
14.1	25	0	30	45	End 2nd Gradient, Begin Isocratic
19.9	25	0	30	45	End Isocratic
20.0	11	20	0	69	Initial Conditions

Analyte	mg/L
1. Lithium	0.2
2. Sodium	0.8
3. Ammonium	1
4. Potassium	2
5. Magnesium	1
6. Calcium	2
7. 1,2-Propanediamine	8
8. 1,6-Hexanediamine	8
9. 1,7-Heptanediamine	8
10. 1,8-Octanediamine	8
11. 1,9-Nonanediamine	8
12. 1,10-Decanediamine	8
13. 1,12-Dodecanediamine	8

where 1 mg/L = 1 ppm

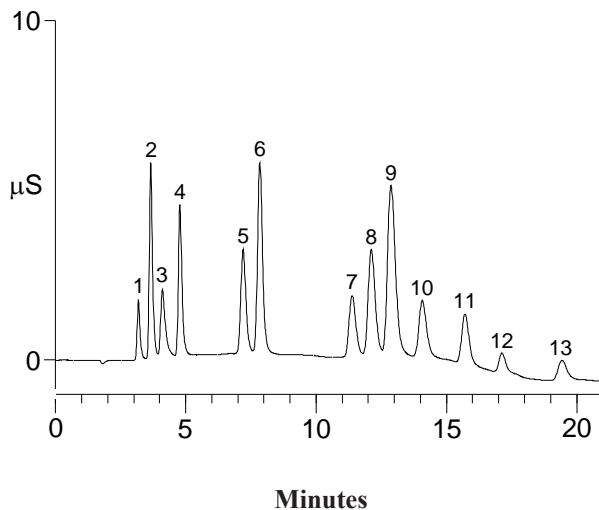


Figure 22
IonPac CS12A Elution of Alkyl Diamines
at Elevated Temperature Using Gradient Elution

5.16 Sample pH

The following run demonstrates the effect of sample pH on peak symmetry and efficiency and how treating the sample with the OnGuard II A can help. The CS12A 4-mm column having higher total cation exchange capacity than the CS12A-5µm 3-mm because of its size, can tolerate more acidic matrices than the CS12A-5µm 3-mm, but both can be helped by treating the acidic samples with the OnGuard II A cartridge.

Sample Volume: 25 µL Loop (4-mm or 3-mm)
 Column: CS12A Analytical Column (4-mm or 3-mm)
 Eluent: 22 mN H₂SO₄ (4-mm)
 20 mN MSA (3-mm)
 Eluent Flow Rate: 1.0 mL/min (4-mm) at room temperature
 0.5 mL/min (3-mm) at 30 °C
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in
 AutoSuppression Recycle Mode (used with CS12A 4-mm)
 Cation Self-Regenerating Suppressor 300 (2-mm) in
 AutoSuppression Recycle Mode (used with CS12A-5µm 3-mm)
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent

CS12A 4-mm		
Analyte		mg/L
1. Lithium		0.5
2. Sodium		2
3. Ammonium		2.5
4. Potassium		5
5. Magnesium		2.5
6. Calcium		5

where 1 mg/L = 1 ppm

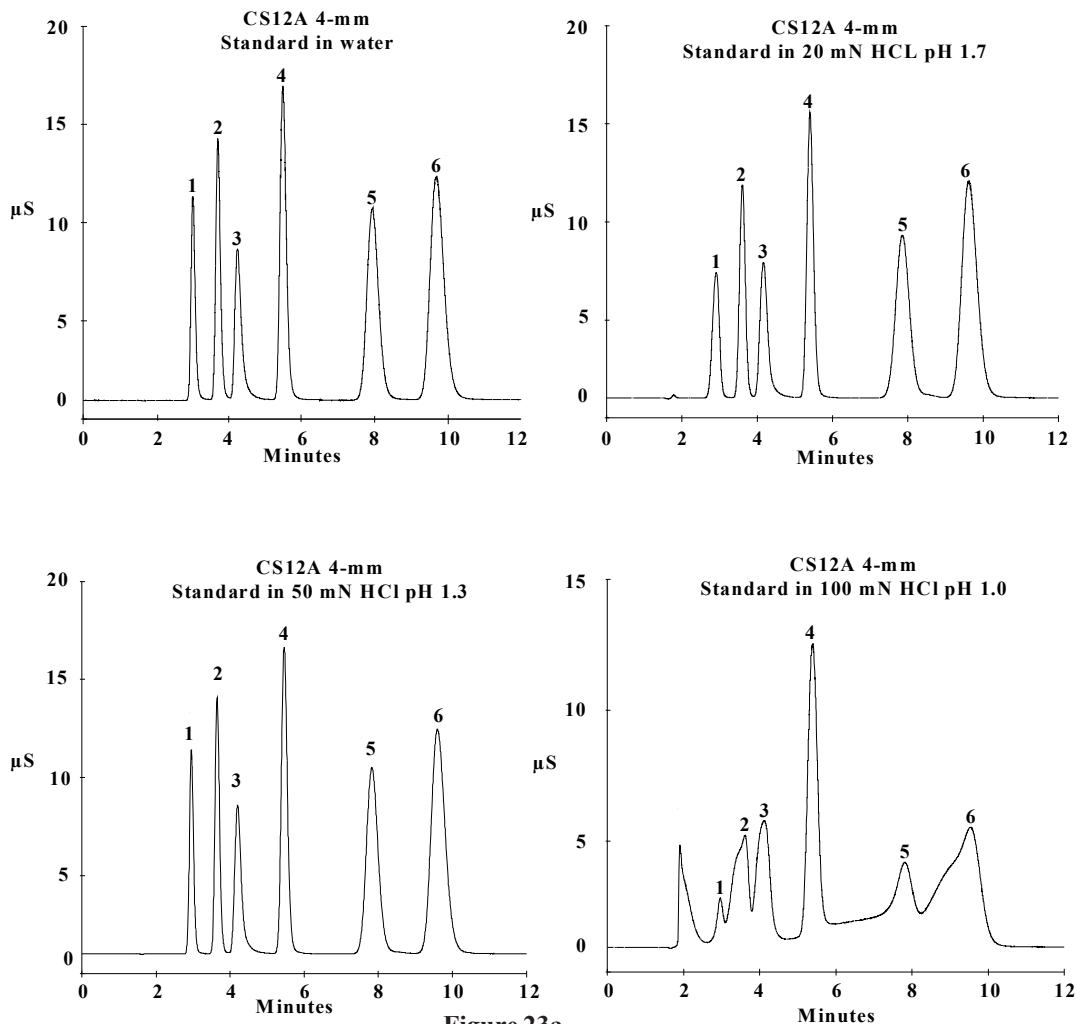


Figure 23a
 Effect of Sample pH

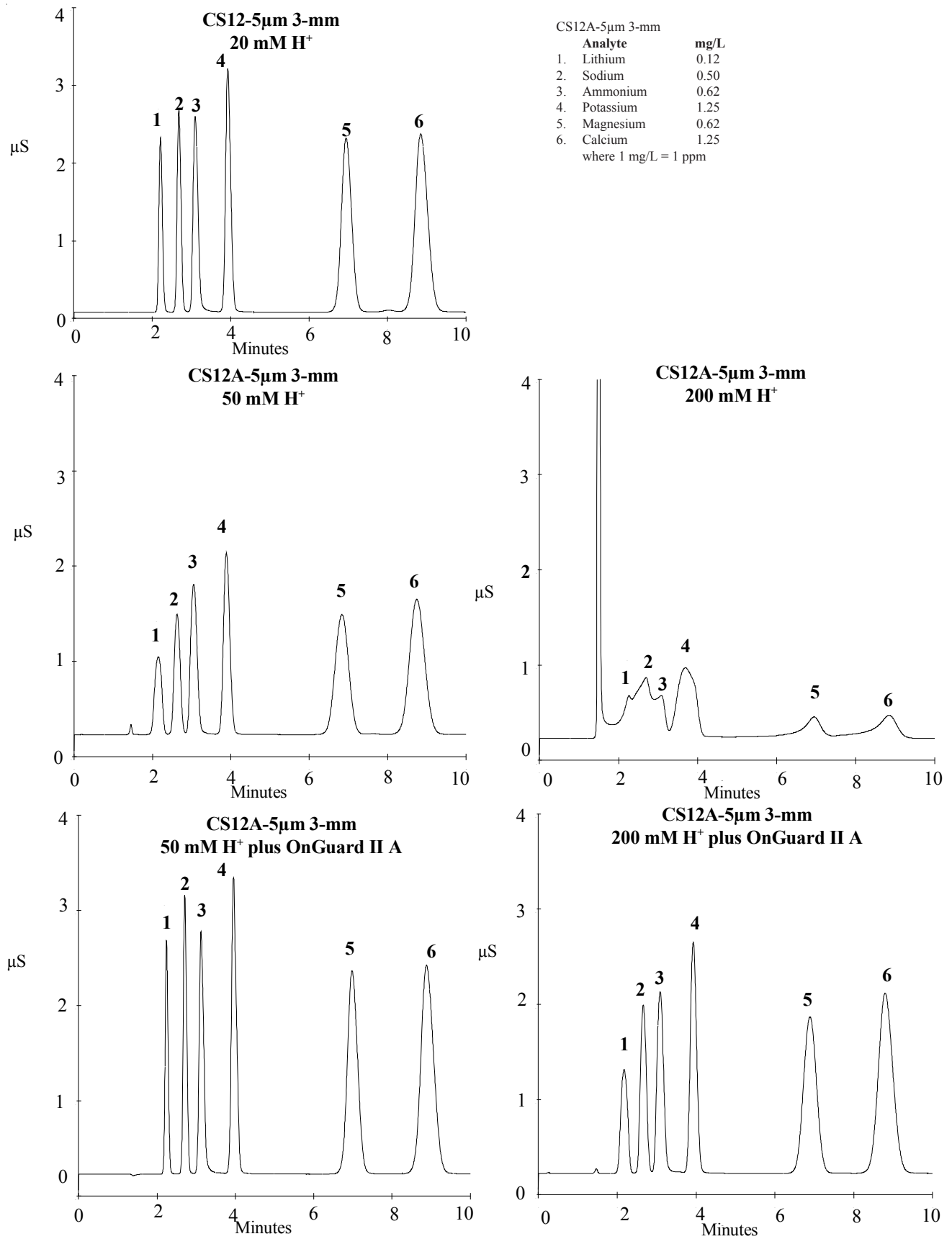


Figure 23b
Effect of Sample pH

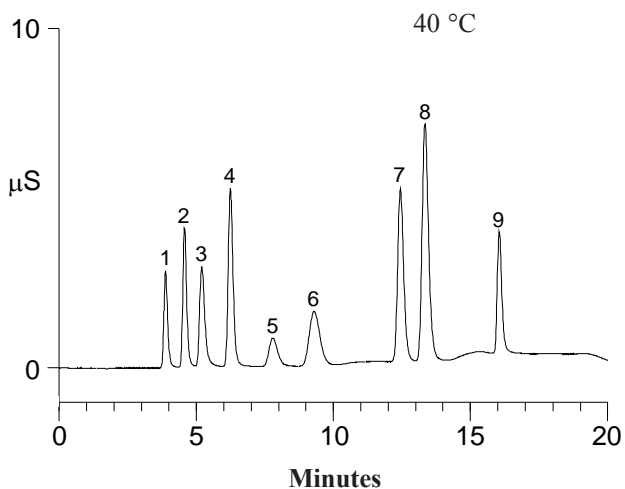
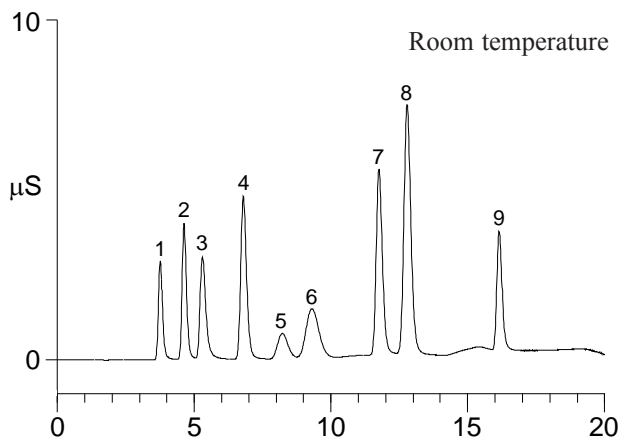
5.17 Separation of Cyclohexylamine, Morpholine and Diethylaminoethanol at Different Temperatures Using Step Elution

The following run demonstrates the separation of cyclohexylamine, morpholine and diethylaminoethanol at room temperature and 40 °C using gradient elution.

Sample Volume: 25 µL Loop
 Column: CS12A Analytical Column (4-mm)
 Cation Trap Column (CTC-1)
 Eluent: E1: 200 mN H₂SO₄
 E2: 20% Acetonitrile
 E3: Degassed deionized water
 Eluent Flow Rate: 1.0 mL/min (4-mm)
 Temperature: 40 °C
 SRS Suppressor: Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression External Water Mode
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
 MMS Regenerant: 100 mN tetrabutylammonium hydroxide TBAOH
 Expected Background Conductivity: < 2 µS
 Storage Solution: Eluent

Analyte	mg/L
1. Lithium	0.5
2. Sodium	2
3. Ammonium	2.5
4. Potassium	5
5. Morpholine	10
6. 2-Diethylaminoethanol	10
7. Magnesium	2.5
8. Calcium	5
9. Cyclohexylamine	15

where 1 mg/L = 1 ppm



Time	Gradient Conditions			Comments
	%E1	%E2	%E3	
Init	8	10	82	Initial Eluent
0.0	8	10	82	Load Position
0.1	8	10	82	Inject, Begin 1st Isocratic Region
0.3	8	10	82	Return to Load Position
7.0	8	10	82	End 1st Isocratic Region
7.1	14	20	66	Begin 2nd Isocratic Region
11.0	14	20	66	End 2nd Isocratic Region
11.1	25	75	0	Begin 3rd Isocratic Region
16.0	25	75	0	End 3rd Isocratic Region
16.1	8	10	82	Initial Conditions

Figure 24
IonPac CS12A Elution of Cyclohexylamine, Morpholine and Diethylaminoethanol at Different Temperatures Using Gradient Elution

5.18 Separation of Arenes at Two Different Temperatures Using Gradient Elution

The following run demonstrates the separation of Arenes at 28 °C and 40 °C using gradient elution.

Sample Volume: 25 μ L
Column: IonPac CS12A (4-mm)
Eluent: 20 mN Sulfuric acid/30% acetonitrile to 20 mN sulfuric acid/90% acetonitrile in 30 minutes
Eluent Flow Rate: 1.0 mL/min
Detection: UV, 210 nm

Analyte

1. Phenylphosphoric acid
2. Phenol
3. 2,2'-Biphenol
4. Benzene
5. Styrene
6. Benzophenone
7. Cumene
8. Naphthalene

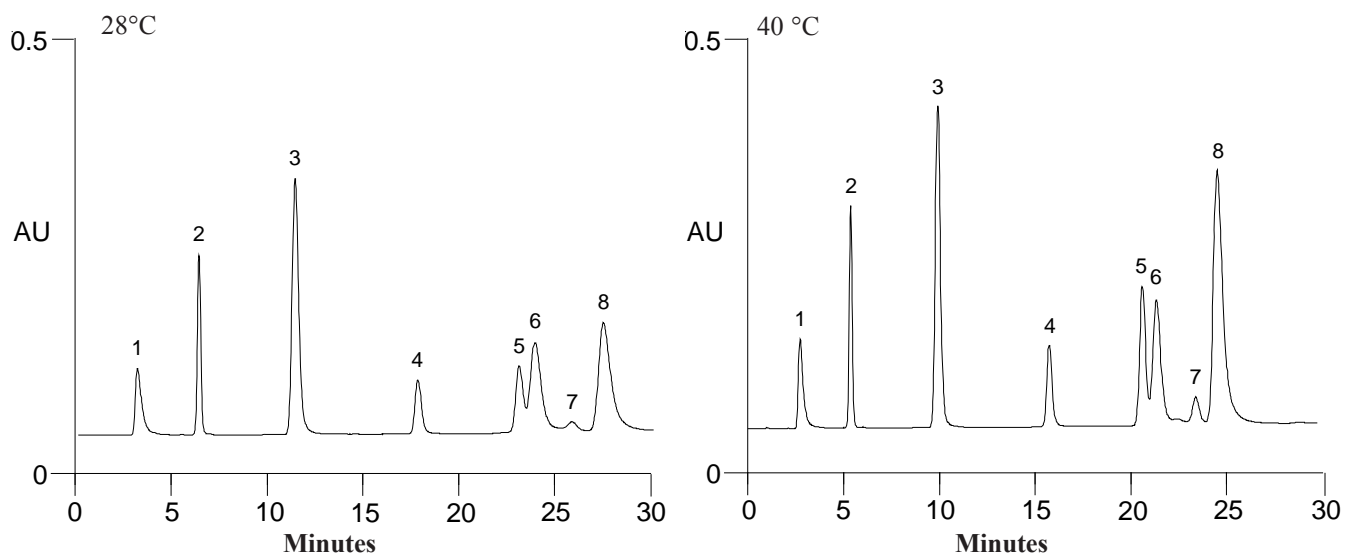


Figure 25
Separation of Arenes at Two Different Temperatures using Gradient Elution

5.19 Separation of Anilines at Two Different Temperatures Using Gradient Elution

The following run demonstrates the separation of anilines at 28 °C and 40 °C using gradient elution.

Sample Volume: 25 μ L
Column: IonPac CS12A (4-mm)
Eluent: 40 mN Sulfuric acid/5% acetonitrile to 40 mN sulfuric acid/25% acetonitrile in 10 minutes,
to 40 mN sulfuric acid/60% acetonitrile in 17 min.
Eluent Flow Rate: 1.0 mL/min
Detection: UV, 210 nm

- Analyte**
1. Aniline
 2. N-Methylaniline
 3. 3-Toluidine
 4. N,N'-Dimethylaniline
 5. N,N'-Diethylaniline
 6. 4,4'-Methylenedianiline
 7. Unknown
 8. Unknown
 9. Unknown
 10. 4-Nitroaniline
 11. 2-Nitroaniline
 12. N-Methyl-N-nitrosoaniline
 13. 2,6-Dichloro-4-nitroaniline

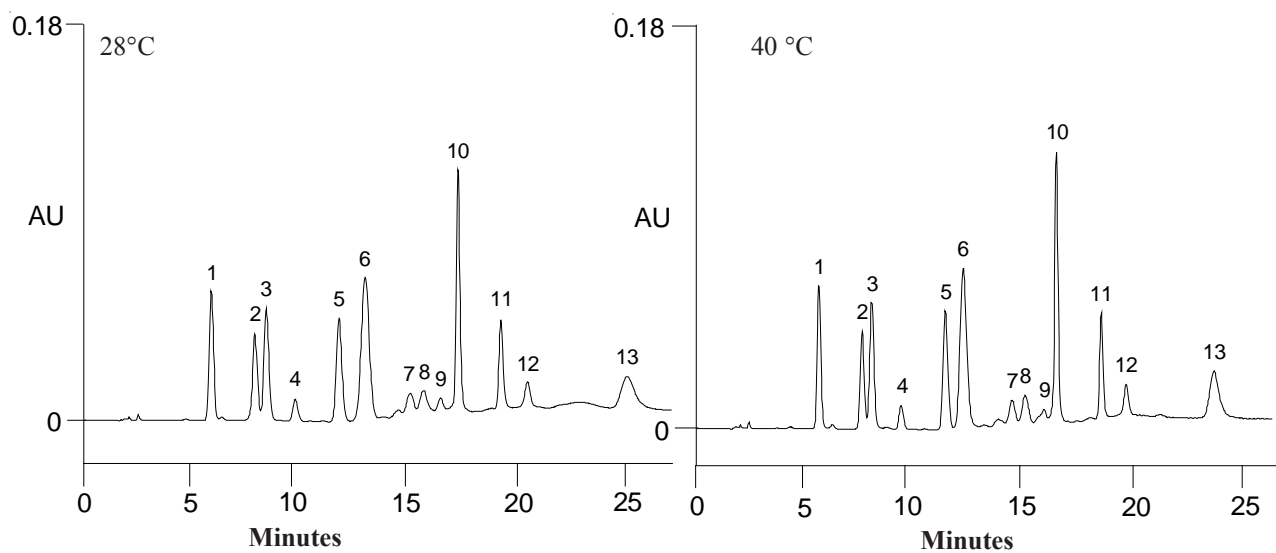


Figure 26
Separation of Anilines at Two Different Temperatures using Gradient Elution

5.20 Separation of Pyridines at Two Different Temperatures Using Gradient Elution

The following run demonstrates the separation of pyridines at 26 °C and 60 °C using gradient elution.

Sample Volume: 25 μ L
Column: IonPac CS12A (4-mm)
Eluent: 10 mN Sulfuric acid/10% acetonitrile
to 18 mN sulfuric acid/25% acetonitrile in 10 minutes,
to 40 mN sulfuric acid/50% acetonitrile in 13 min
Eluent Flow Rate: 1.0 mL/min
Detection: UV, 254 nm

- Analytes (Approx. 20 mg/L)**
1. Pyridine
 2. 2-Aminopyridine
 3. 4-Picoline
 4. 2-Dimethylaminopyridine
 5. 2,2'-bipyridine
 6. 4-Benzylpyridine
 7. 2-(2-Aminoethyl)pyridine

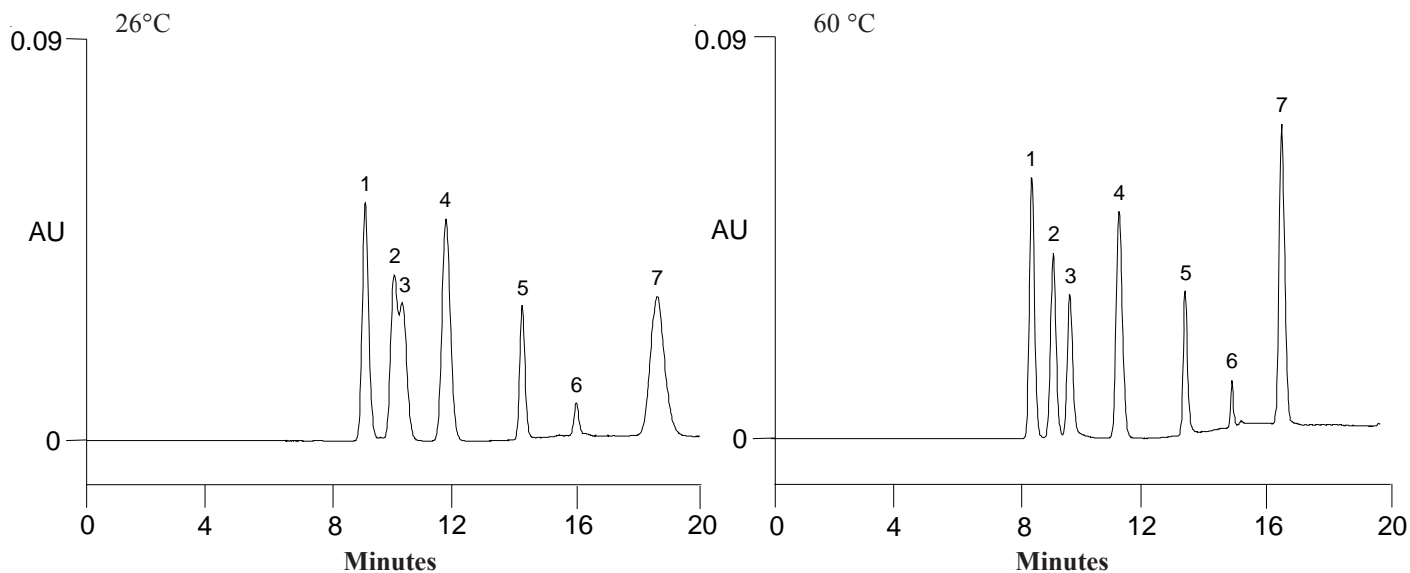


Figure 27
Separation of Pyridines at Two Different Temperatures using Gradient Elution

5.21 Separation of Benzylamines and Amides at Two Different Temperatures Using Gradient Elution

The following run demonstrates the separation of benzylamines and amides at 26 °C and 40° C using gradient elution.

Sample Volume: 25 µL
 Column: IonPac CS12A (4-mm)
 Eluent: 10 mN Sulfuric acid/5% acetonitrile
 to 10 mN sulfuric acid/25%acetonitrile in 10 minutes,
 to 10 mN sulfuric acid/60% acetonitrile in 17 min
 Eluent Flow Rate: 1.0 mL/min
 Detection: UV, 210 nm

- Analytes**
1. 4-Hydroxybenzamide
 2. Unknown
 3. Unknown
 4. Benzamine
 5. Benzamide
 6. N,N'-Dimethylbenzylamine
 7. Dibenzylamine
 8. Tribenzylamine

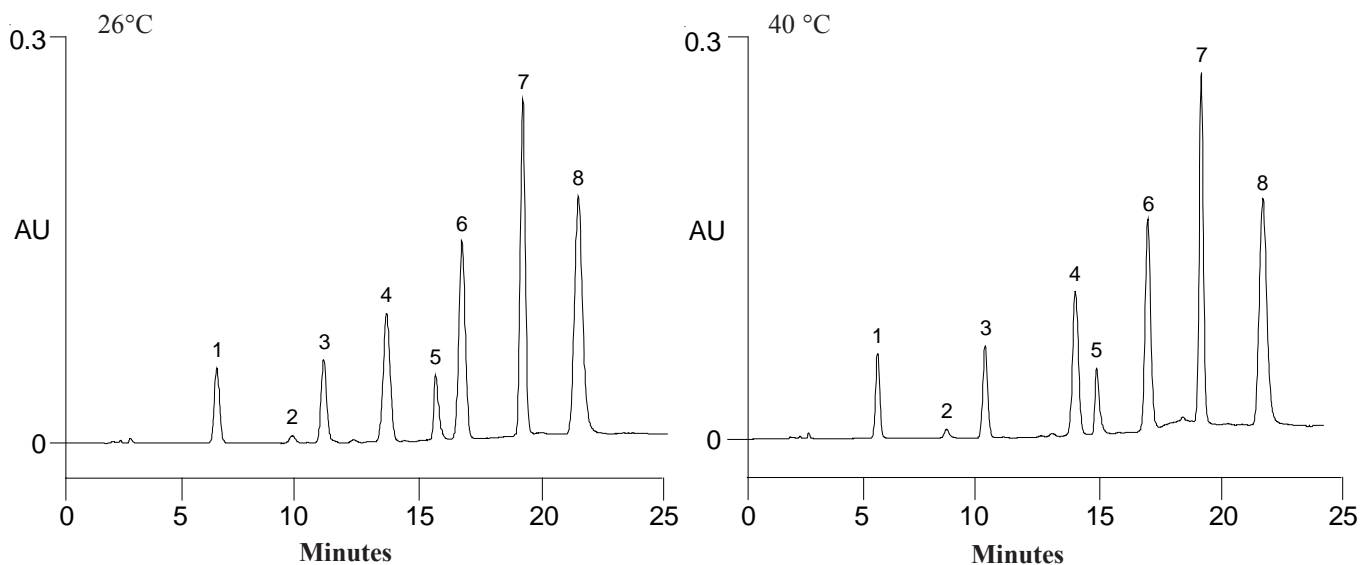


Figure 28
 Separation of Benzylamines and Amides at Two Different Temperatures using Gradient Elution

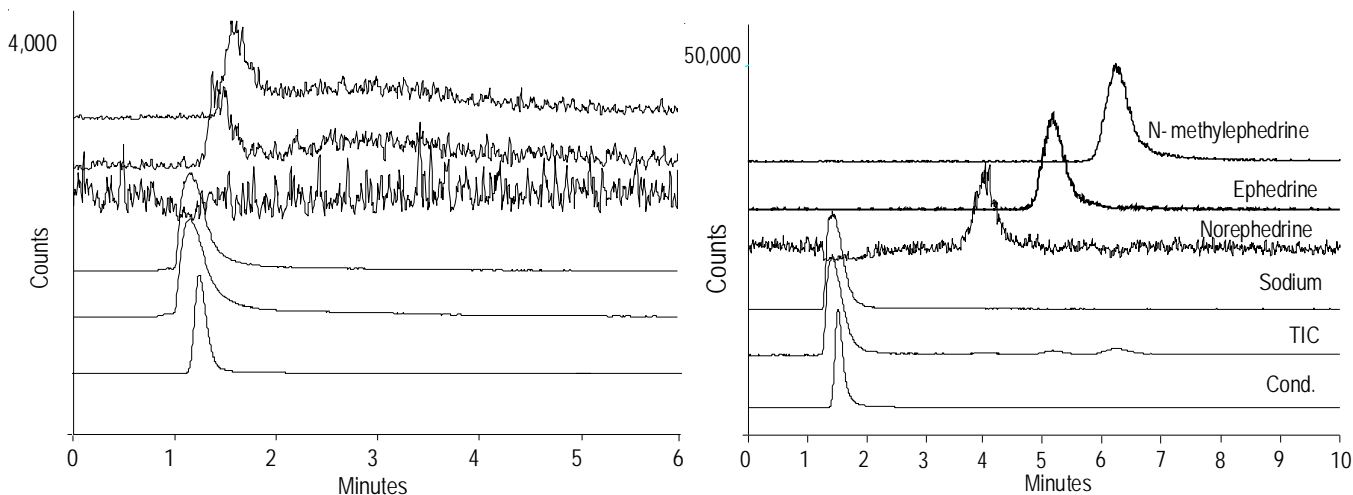
5.22 Separation of Matrix Ions from Analytes in IC-MS: 900 ppb Ephedrines in 130 ppm NaCl

Optimized sensitivity in IC-MS is achieved when analytes are separated from other matrix cations such as sodium. The following example shows two sets of overlays for the various signals generated for ephedrines in a salt matrix. On the left, the conditions were selected for minimal separation of the three ephedrines from the sodium peak. The identification of the traces is shown on the right with conductivity trace on the bottom, the total ion current, followed by the 105 amu extracted ion trace for sodium (a sodium acetonitrile cluster). Above the sodium trace are the extracted ion traces for the molecular ion plus a proton for norephedrine at 152 amu, ephedrine at 166 amu and N-methylephedrine at 180 amu. Without separation the norephedrine is not detected due to suppression of its ionization by the sodium. Detection limits of close-eluting ephedrine and N-methylephedrine are also diminished as seen by the full scale setting for the top 3 traces. All of the MS data generated is not smoothed.

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, external water, 10 mA*
 Eluent: 36% ACN, 25 mM MSA
 Flow rate: 0.25 mL/min
 Injection loop: 5 µL
 Detection: AQA-MS, +ESI, 20V, 200 °C, full scan

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, external water, 10 mA*
 Gradient: 5-18% ACN, 10-25 mM MSA in 6 min, hold 2 min.
 Flow rate: 0.25 mL/min
 Injection loop: 5 µL
 Detection: AQA-MS, +ESI, 20V, 200 °C, full scan

* See CSRS 300 manual, Section 3.3.4



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Figure 29
Separation of Matrix Ions from Analytes in IC-MS: 900 ppb Ephedrines in 130 ppm NaCl

5.23 Detection of Small Amines Using IC-MS

The following example shows an overlay of single ion traces of the M+H signals for 6 small amines. There is a large sensitivity difference among these amines. The triethanolamine hydrogen bonds easily and does not behave well in the electrospray process, leading to poor sensitivity. Trimethylamine is very volatile. The other amines have good sensitivity. This work was done with minimal separation of the amines using the CS12A-MS (2 x 100 mm) column.

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, external water, 10 mA*
 Gradient: 2-18% ACN, 10-25 mM MSA
 in 6 min, hold 2 min.
 Flow rate: 0.25 mL/min
 Injection loop: 5 µL
 Detection: AQA MS, +ESI, 20V, 200 °C, SIM as indicated

Peaks:	µg/L (ppb)
1. Ethanolamine	50
2. Diethanolamine	50
3. Triethanolamine	25,000
4. Trimethylamine	12,500
5. Ethyldimethylamine	500
6. Triethylamine	50

* See CSRS 300 manual, Section 3.3.4

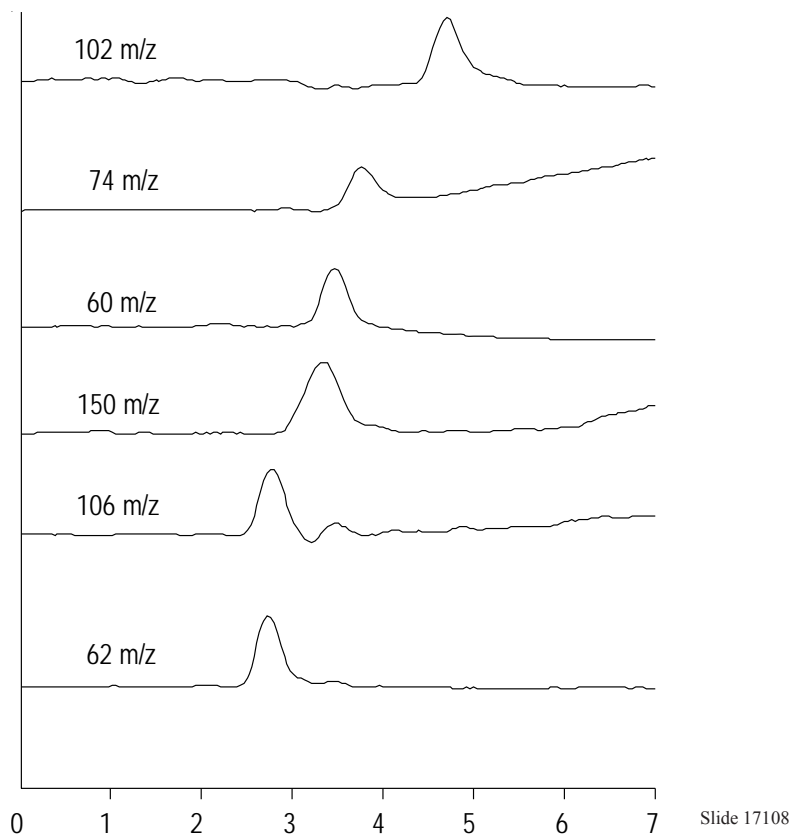


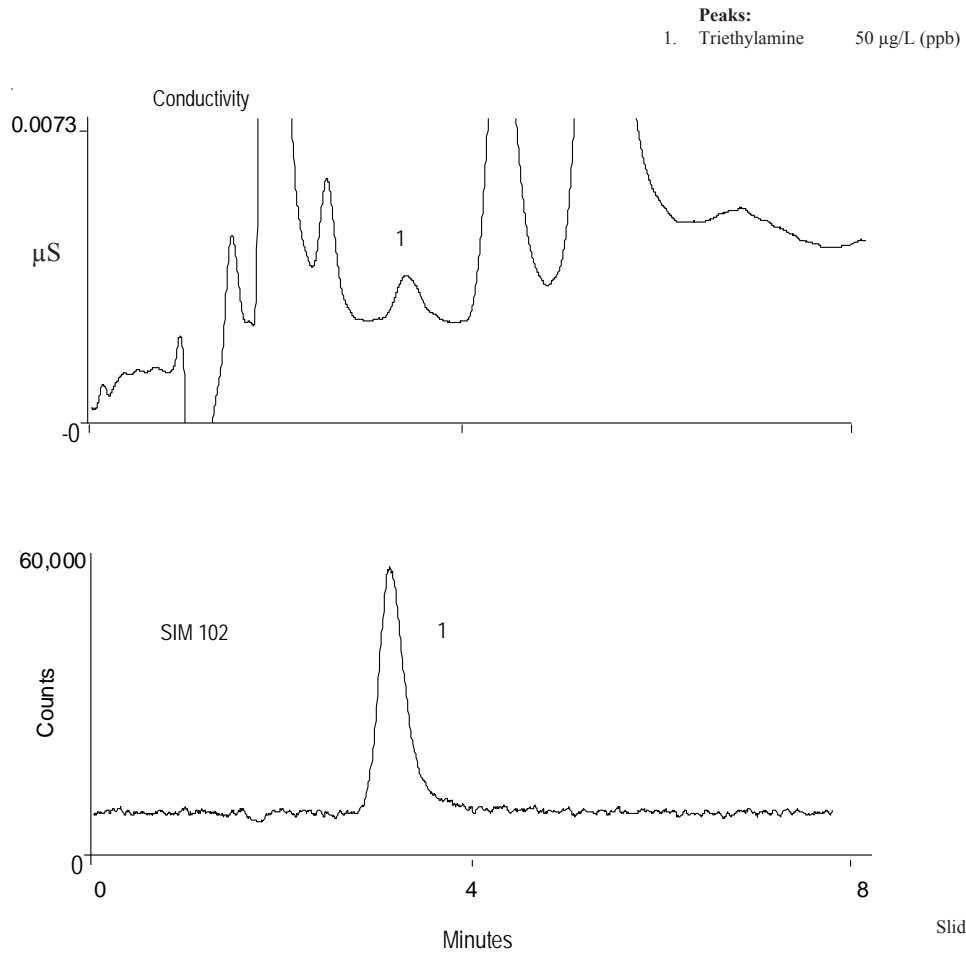
Figure 30
Detection of Small Amines Using IC-MS

5.24 Comparison of Conductivity and Electrospray-MS Detection of 50 ppb Triethylamine

The following example demonstrates the selective detection of triethylamine among other cations at the 50 ppb level. Both the conductivity detector and the MS provide useful information. The MS chromatogram is a Selected Ion Monitoring Trace at m/z 102 so that only the triethylamine is detected. The MS data generated is not smoothed.

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, external water, 10 mA*
 Eluent: 4.5% ACN/15 mM MSA
 Flow rate: 0.25 mL/min
 Injection loop: 5 µL
 Detection: AQA MS, +ESI, 20V, 200 °C, SIM as indicated

* See CSRS 300 manual, Section 3.3.4



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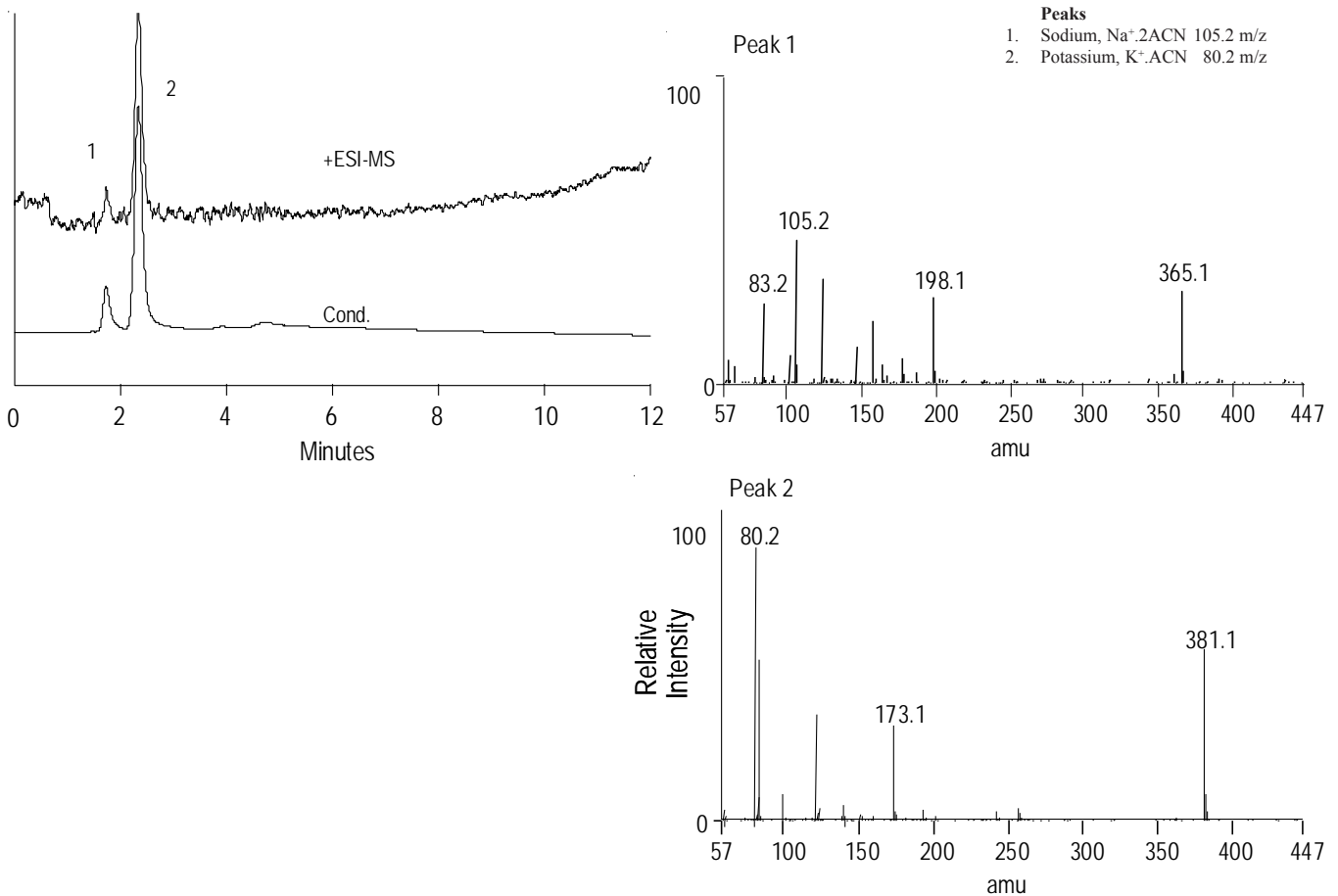
Figure 31
Comparison of Conductivity and Electrospray-MS Detection of 50 ppb Triethylamine

5.25 Identification of Cations in Biaxin Extract by IC-MS

The following example demonstrates the ability to identify inorganic cations in a Biaxin Extract. A MSA/ACN gradient was used to elute inorganic as well as organic amines for screening purposes. Sodium and potassium are easily detected under these conditions as clusters with acetonitrile and/or water. The highest masses for example are clusters with 19 water molecules while the lower masses contain acetonitrile.

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, 2-mm external water, 10 mA*
 Gradient Conditions: 2% ACN/17 mM MSA to 36% ACN/25 mM MSA in 10 mins, hold for 5 min.
 Flow rate: 0.25 mL/min
 Detection: Conductivity, +ESI-MS
 MS Conditions: +ESI, Full Scan 57-447 amu, 200 °C, 20V
 Sample: 40 mg Biaxin/10 mLs 50% ACN, sonicated and filtered

* See CSRS 300 manual, Section 3.3.4



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Figure 32
Identification of Cations in Biaxin Extract by IC-MS

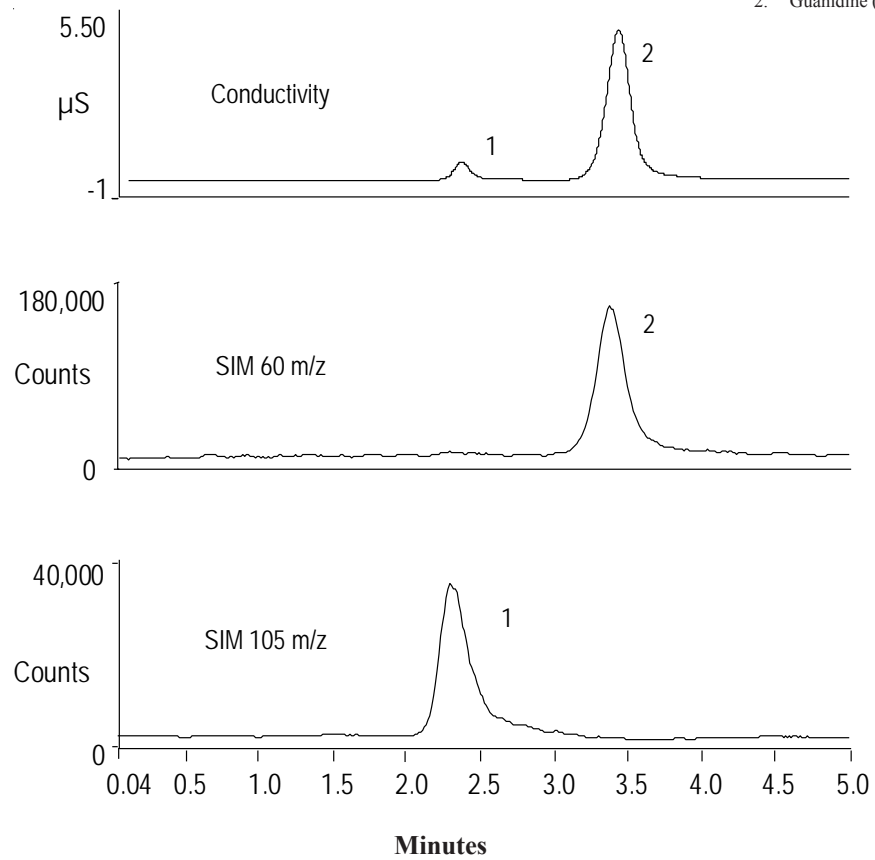
5.26 Guanidine by IC-MS

The following example shows the analysis of a guanidine stock with separation and detection of a sodium impurity in guanidine with good sensitivity under fast run, low solvent conditions.

Column: IonPac CS12A-MS, 2 x 100 mm
 Eluent: 1.8% ACN/12 mM MSA
 Flow Rate: 0.25 mL/min
 Suppressor: CSRS 300, external water, 9 mA*
 Detection: Conductivity/AQA MS
 MS Conditions: +ESI, 250 °C, 20V,
 SIM as indicated
 Sample: Guanidine 1 mg/mL
 Loop: 5 µL

* See CSRS 300 manual, Section 3.3.4

Peaks:	m/z
1. Sodium cluster	105
2. Guanidine (NH ₂) ₂ C=NH	60



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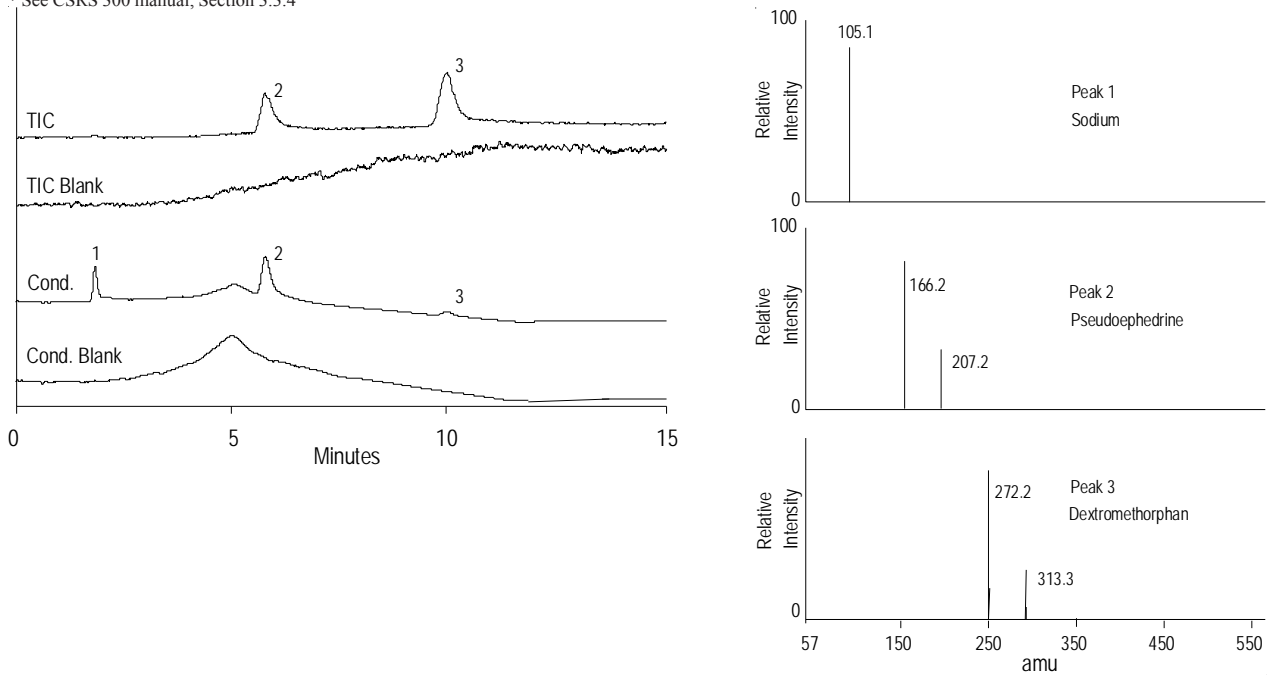
Figure 33
Guanidine by IC-MS

5.27 General Cation Gradient Screening of Histenol-Forte

The following example demonstrates a general cation screen applied to a generic cold medication. The sample was dissolved in water and filtered prior to injection. The four overlaid chromatograms show the conductivity and the electrospray MS traces along with the blank runs. The MS was operated in full scan mode and the masses shown here were extracted. Masses for the M+H and the M+H+ACN are shown.

Column: IonPac CS12A-MS, 2 x 100 mm
 Gradient: 1.8-45% ACN/10-50 mM MSA in 10 min, hold 5 min
 Flow Rate: 0.37 mL/min
 Detection: Conductivity/AQA MS
 MS Conditions: +ESI, 275 °C, 20V, full scan 57-550 amu
 Suppressor: CSRS 300, external water, 30 mA*

* See CSRS 300 manual, Section 3.3.4



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Figure 34
General Cation Gradient Screening of Histenol-Forte

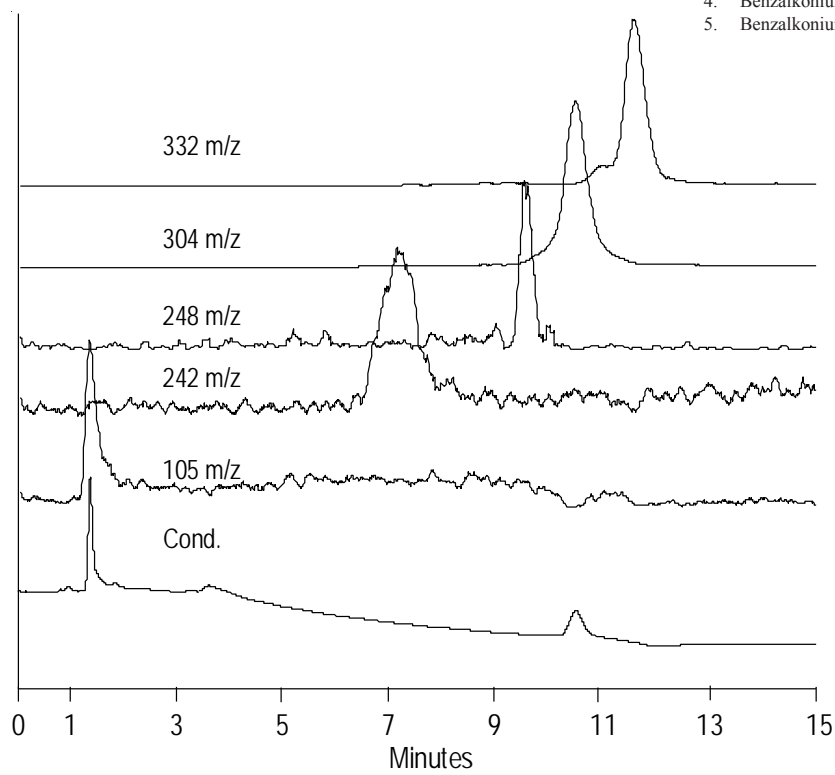
5.28 IC-MS of an Antiseptic Swab

The following example shows the use of a dual gradient applied to an antiseptic swab sample. Masses of the various chain lengths were extracted from the full scan run. Four of these masses are shown here. The conductivity trace did not indicate multiple peaks.

Column: IonPac CS12A-MS, 2 x 100 mm
 Suppressor: CSRS 300, external water, 30 mA*
 Gradient: 18-63% ACN, 10-50 mM MSA in 10 min, hold 2 min.
 Flow rate: 0.37 mL/min
 Injection loop: 5 µL
 Detection: AQA-MS, +ESI, 20V, 275 °C, full scan
 Sample: Antiseptic swab, 0.133% Benzalkonium chloride
 Sample preparation: 1 swab/20 mL water

* See CSRS 300 manual, Section 3.3.4

Peaks:	m/z
1. Sodium,	105
2. Benzalkonium (unsaturated)	242
3. Benzalkonium (C8)	248
4. Benzalkonium (C12)	304
5. Benzalkonium (C14)	332



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Figure 35
IC-MS of an Antiseptic Swab

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac CS12A columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

Table 7
CS12A/CG12A Troubleshooting Summary

Observation	Cause	Action	Reference Section	
Column Related Problems				
High Back Pressure	Unknown Component	Isolate Blockage	6.1.1	
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2 Component Manual	
	Plugged System Hardware	Unplug, Replace		
Poor Peak Resolution	Poor Efficiency	Large System Void Volumes	Replumb System	6.6.3.B, Component Manual
		Sluggish Injection Valve	Service Valve	6.6.3.A, Component Manual
		Column Headspace	Replace Column	6.6.1.A
		Column Overloading	Reduce Sample Size	6.6.3.B
	Fronting Peaks	Column Overloading	Reduce Sample Size	6.6.3.B
	Tailing Peaks	Contaminated CSRS, CAES or CMMS	Clean Suppressor	6.3.1.A, 6.5, Component Manual
	Short Retention Times	Flow Rate Too Fast	Recalibrate Pump	6.6.2.A
		First Peaks Elute Too Fast	Equilibrate to First Eluent	6.6.3.A
		Bad Eluents	Remake Eluents	6.6.2.B
	Spurious Peaks	Column Contamination	Clean Column	6.6.2.C
		Column Contamination	Pretreat Samples	6.3.1, 6.7.A, 6.7.B,
		Sluggish Injection Valve	Service Valve	6.7.C, Component Manual
	Poor Quantification of Divalents	Sample Loop Contamination	Flush	6.3.2
		Replace	6.3.2	
Suppressor Related Problems				
High Background Conductivity IC and IC-MS	Improper Suppressor Operation	CSRS or CAES Not Suppressing	Check Current	6.5.A, Component Manual
			Check REGEN OUT Flow	6.5.A, Component Manual
			Check for leaks	6.5.A, Component Manual
		CMMS Not Suppressing	Check Regenerant	6.5.C, Component Manual
			Check AutoRegen Cartridge	6.5.C, Component Manual
	Contamination	Bad Eluents	Remake Eluents	6.2, 6.4
		Contaminated Column	Clean Column	6.3.1
		Contaminated CSRS, CAES or CMMS	Clean Suppressor	6.3.1 A, 6.5, Component Manual
	Hardware Operation	Proportioning Valve	Service Valve	Component Manual

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac CG12A Guard Column plus the CS12A Analytical Column are listed in the table below using the test chromatogram conditions. If the system pressure is higher, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated.

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

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi (0.34 MPa). Continue adding system components (injection valve, column(s), suppressor and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 8, "Typical CS12A/CG12A Operating Back Pressures").

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

Table 8
Typical CS12A/CG12A Operating Back Pressures

Column	At standard flow rate, Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS12A 4-mm Analytical	≤ 1,200 (8.27)	1.0	2.0
CG12A 4-mm Guard	≤ 450 (3.10)	1.0	3.0
CS12A + CG12A 4-mm columns	≤ 1,650 (11.37)	1.0	2.0
CS12A-5µm 3-mm Analytical	≤ 1,760 (12.13)*	0.5	1.0
CG12A-5µm 3-mm Guard	≤ 605 (4.17)*	0.5	1.0
CS12A + CG12A-5µm 3-mm columns	≤ 2,365 (17.30)*	0.5	1.0
CS12A 2-mm Analytical	≤ 1,200 (8.27)	0.25	0.5
CG12A 2-mm Guard	≤ 450 (3.10)	0.25	0.75
CS12A + CG12A 2-mm columns	≤ 1,650 (11.37)	0.25	0.5
CS12A 0.4 mm Capillary	≤ 1,320 (9.10)	0.01	0.020
CG12A 0.4 mm Capillary Guard	≤ 495 (3.41)	0.01	0.020
CS12A + CG12A 0.4 mm columns	≤ 1,815 (12.51)	0.01	0.020
<small>* at 30 °C</small>			
CS12A-5µm 0.4 mm Capillary	≤ 1,760 (12.13)*	0.008	0.02
CG12A-5µm 0.4 mm Capillary Guard	≤ 605 (4.17)*	0.008	0.02
CG12A-5µm 0.4 mm columns	≤ 2,365 (17.30)*	0.008	0.02
CS12A-MS (2 x 100 mm) Analytical	≤ 800 (5.51)	0.25	0.75

* at 30 °C

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. 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.



NOTE

The CS12A-5µm 3-mm and CG12A-5µm 3-mm use a different bed support assembly than the CS12A 4-mm. It has a smaller frit than the 4-mm column. **DO NOT** substitute the 4-mm type bed support in the CS12A-5µm 3-mm columns. Efficiency will degrade.

- 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.

Table 9
IonPac CS12A Column Bed Supports

Part	IonPac CS12A Column Bed Supports				CS12A	CS12A-5µm
	4-mm (P/N)	3-mm (P/N)	2-mm (P/N)	2-mm MS (P/N)	0.4-mm (P/N)	0.4-mm (P/N)
Analytical Column	046034	057185	046055	059960	072066	072068
Guard Column	046035	057184	046056	N/A	072067	072069
Bed Support Assembly	042955	056823	044689	044689	N/A	N/A
End Fitting	052809	052809	043278	043278	N/A	N/A



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.

- E. **Screw the end fitting back onto the column.** Tighten it fingertight, then an additional 1/4 turn (25 in x 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. Wash the column with eluent to waste for about 15 minutes before connecting it back to the suppressor.

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 A Contaminated Guard or Analytical 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 IonPac CG12A Guard and CS12A 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 CG12A at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in, "Column Cleanup" (See, "Column Care"). 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 μ S. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

- A. **Check for a contaminated Gradient Mixer.** Gradient Mixers (GM-2) in the Gradient Pump Module should be flushed thoroughly to remove eluents containing DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl). Chloride containing eluents should not be pumped through the CSRS 300.
- 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 in the system 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 CR-CTC Trap Column.** Install a CR-TC Cation Trap Column (P/N 066262 or 072079) if using an Eluent Generator with EGC MSA cartridge.
- F. **Install an IonPac Cation Trap Column (CTC-1, P/N 040192).** It should be positioned between the pump and the injection valve. It is highly recommended for all cation gradient analyses. The CTC-1 strips the eluent of cation contaminants that will bind strongly to the analytical column resulting in the loss of column capacity and potentially interfering with the desired cation analyses. The CTC-1 minimizes baseline changes when performing gradient analyses. The CTC (2-mm), P/N 043132, should be used in 2-mm or CS12A-5 μ m 3-mm systems.

6.3.2 Sample Loop and/or Tubing Contamination

Eluents made with deionized water that is contaminated with bacteria as well as certain samples such as humic acids 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 sample or pH of the standard and the way these are introduced, inaccurate readings for divalent analytes on weak cation exchange resins may be observed.

A. Weak Cation Exchangers

Carboxylated resins (used in the IonPac CS12, CS12A, CS12A-5 μ m, CS12A-MS, CS14, CS16, CS17 and CS18) 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, the weak cation exchange sites on the contaminated tubing are ionized and divalent cations are retained. This can result in problems, such as poor linearity, for divalent cations when the sample pH is >4 .

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

A simple test can be performed (when using a column such as the IonPac CS12A 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 mL, flush it with no more than 100 mL).
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 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 < 1 μ S with a CSRS 300(4-mm) or CSRS 300(2-mm).

A system with a high background (> 3 μ S) will probably also have high noise, resulting in increased detection limits.

- A. Make sure that the eluents and regenerant are prepared correctly (see Section 5.2, “Eluent Preparation”).**
- B. Determine if the columns or system are contaminated (see Section 6.3, “A Contaminated Guard or Analytical Column”).**
- C. 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, “A Suppressor That Does Not Suppress Properly.”

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

<u>ELUENT</u>	<u>EXPECTED BACKGROUND CONDUCTIVITY</u>
22 mN H ₂ SO ₄ or 20 mN Methanesulfonic acid	< 1 μS
50 mN H ₂ SO ₄ or Methanesulfonic acid	< 2 μS

6.5 Suppressor Not Suppressing Properly

If the Cation Self-Regenerating Suppressor, the Cation Capillary Electrolytic Suppressor, the Cation Atlas Electrolytic Suppressor, or the Cation MicroMembrane Suppressor is causing the problem, refer to the Cation Self-Regenerating Suppressor Product Manual (Document No. 031370), to the Cation Atlas Electrolytic Suppressor Product Manual (Document No. 031770), to the Cation MicroMembrane Suppressor Product Manual (Document No. 031728) or to the Cation Capillary Electrolytic Suppressor Product Manual (Document No. 065386) for detailed troubleshooting assistance.

- A. Check that the CSRS 300 is not in an alarm state.
- B. Check for CSRS 300 leaks.**
- C. Make sure that the back pressure tubing is properly installed in the CSRS 300.
- D. Check the regenerant flow rate at the REGEN OUT port of the CSRS.** Turn the power to the CSRS off. Measure the regenerant flow rate. If it is being used in the recycle mode, it should be the same flow rate as the eluent (typically 1 mL/min for 4-mm operation). If it is used in the AutoSuppression External Water Mode, it should be at least 5 mL/min for non-solvent containing eluents. When solvents are used in the eluent, the regenerant flow rate should be at least 10 mL/min.
- 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 Cation Self-Regenerating Suppressor Product Manual (Document No. 031370) or to the Cation MicroMembrane Suppressor Product Manual (Document No. 031728) for assistance in determining if the eluent is within suppressible limits.
- F. If you are using an AutoRegen Accessory with the CSRS (in the Chemical Suppression Mode) or the CMMS, prepare fresh regenerant solution.** Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.
 - 1. If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge, you probably need to clean or replace your CSRS or CMMS.**
 - 2. If the background conductivity is low when freshly prepared regenerant is run through the CSRS or CMMS without an AutoRegen Accessory in-line, test the AutoRegen Regenerant Cartridge to see if it is expended.** Connect the freshly prepared regenerant to the AutoRegen Regenerant Cartridge. Pump approximately 200 mL of regenerant through the AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir. If the background conductivity is high after placing the AutoRegen Accessory in-line, you probably need to replace the AutoRegen Regenerant Cartridge. Refer to the "AutoRegen Regenerant Cartridge Refill Product Manual" (Document No. 032852) for assistance.



NOTE

acetonitrile.

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

G. 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 CSRS 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, making the peaks' elution less efficient.** Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4-mm systems or no greater than 0.005" for CS12A-5 μ m 3-mm and 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.
- 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. 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 that is too concentrated 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, 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 at least 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, "Column Cleanup" in "Column Care").

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

6.6.3 Loss of Early Eluting Peak Resolution

Lack of equilibration to the initial eluent, improper system operation 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 to equilibrate to the initial eluent. The minimum equilibration time can be determined by making successive runs with increasing equilibration times. The column is equilibrated to the initial eluent when additional equilibration time does not increase the runtime of the first eluting peaks.
- B. Sluggish operation of the injection valve may be the problem.** Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.
- C. Improperly swept out volumes anywhere in the system prior to the guard and Analytical/Capillary 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, 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 Column

Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks (see Figure 36).

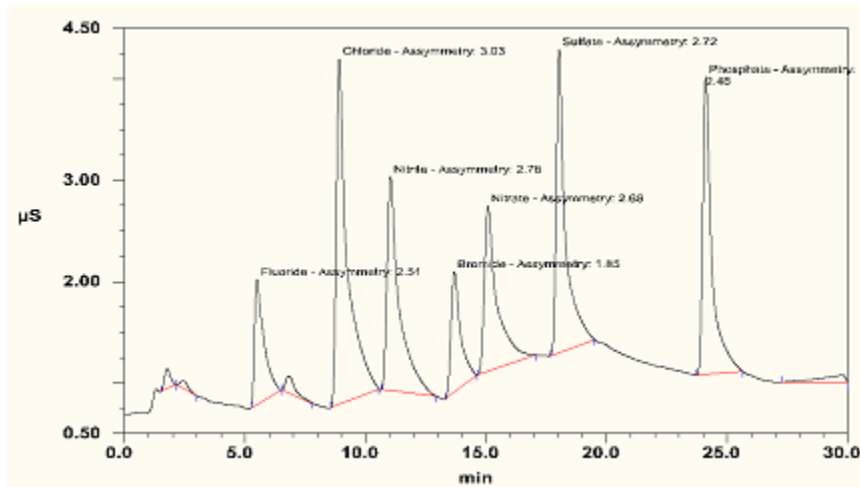


Figure 36
Tailing Peaks Caused by Incorrectly Installed
Capillary Tubing Fittings

When connecting a capillary tube fitting, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing. Figure 37 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

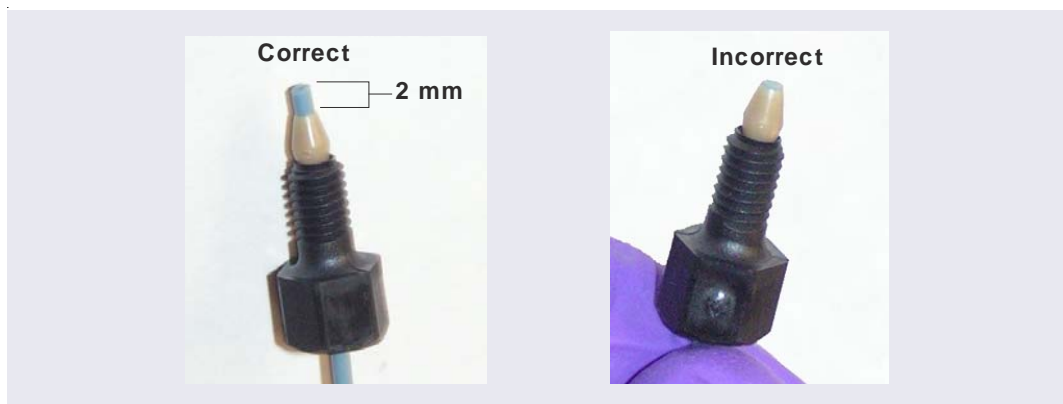


Figure 37
Correct and Incorrect Ferrule and
Fitting Bolt Placement for Capillary Tubing Connections

APPENDIX A - QUALITY ASSURANCE REPORT

Quality Assurance Report - IonPac CS12A Analytical Column - 2 x 250 mm

Quality Assurance Report - IonPac CS12A Analytical Column - 2 x 100 mm

Quality Assurance Report - IonPac CS12A Analytical Column - 3 x 150 mm

Quality Assurance Report - IonPac CS12A Analytical Column - 4 x 250 mm

Quality Assurance Report - Ion Pac CS12A-5 μ m Capillary Column - 0.4 x 150 mm

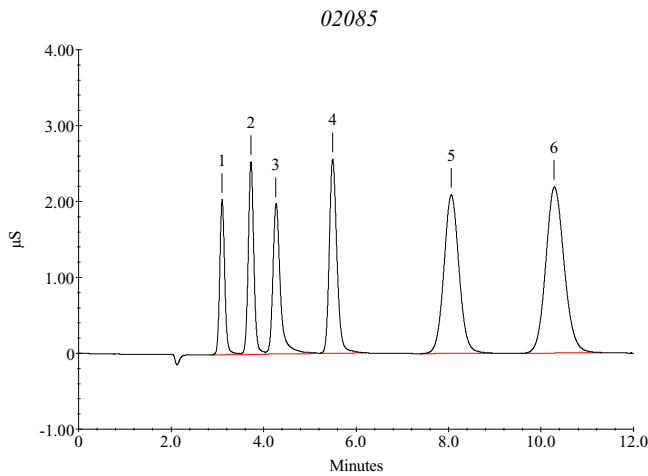
Quality Assurance Report - IonPac CS12A Capillary Column - 0.4 x 250 mm

**IonPac® CS12A
Analytical (2 x 250 mm)
Product No. 46075**

Serial No. : 02085

Pressure (PSI) : 720

Date : 1/25/01 11:47:02 AM



Eluent: 20 mM MSA*
(Methanesulfonic Acid)

Eluent Flow Rate: 0.25 mL/min

Detection: Suppressed Conductivity
CSRS®-ULTRA, 2-mm
AutoSuppression® Recycle Mode

Background Conductivity: < 2 µS

Injection Volume: 2.5 µL

Storage Solution: Eluent (20 mM
Methanesulfonic Acid)

* The IonPac CS12A test protocol has been changed to use 20 mM Methanesulfonic Acid, (rather than 22 mN Sulfuric Acid) as the test eluent.

Peak Information : Found Components

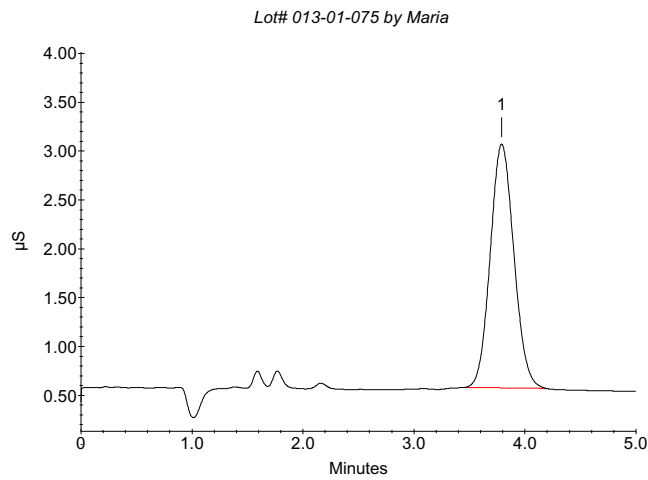
Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	3.10	Lithium	0.5	4270	1.2	3.09
2	3.73	Sodium	2.0	4791	1.2	2.24
3	4.27	Ammonium	2.5	4023	1.6	4.36
4	5.49	Potassium	5.0	5561	1.4	5.81
5	8.06	Magnesium	2.5	2965	1.1	3.40
6	10.28	Calcium	5.0	3243	1.2	n/a

**IonPac® CS12A-MS
Analytical (2 x 100 mm)
Product No. 59960**

Serial No. : Lot# 013-01-075 by Maria

Pressure (PSI) :

Date : 11/15/01 4:00:24 PM



Eluent: 20 mM MSA*
(Methanesulfonic Acid)

Eluent Flow Rate: 0.25 mL/min

Detection: Suppressed Conductivity
CSRS®-ULTRA 2-mm, 50 mA
AutoSuppression® Recycle Mode

Background Conductivity: < 2 µS

Injection Volume: 2.5 µL

Storage Solution: Eluent (20 mM
Methanesulfonic Acid)

Peak Information : Found Components

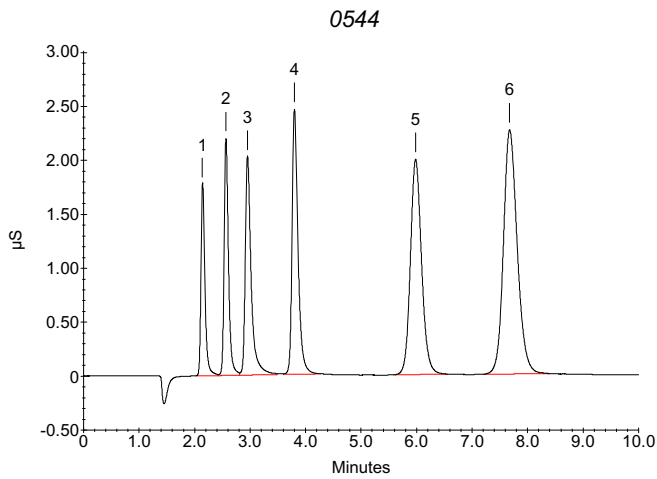
Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	3.79	Calcium	5.0	1462	1.1	n/a

**IonPac® CS12A-5µm 3-mm
Analytical (3 x 150 mm)
Product No. 057185**

Serial No. : 0544

Pressure (PSI) : 1174

Date : 9/6/02 1:28:31 PM



Eluent: 20 mM MSA
(Methanesulfonic Acid)

Eluent Flow Rate: 0.5 mL/min

Operating Temperature: 30°C

Detection: Suppressed Conductivity
CSRS®-ULTRA, 2-mm
AutoSuppression® Recycle Mode,
50 mA

Background Conductivity: < 2 µS

Injection Volume: 25 µL

Storage Solution: Eluent (20 mM MSA)
Methanesulfonic

Peak Information : Found Components

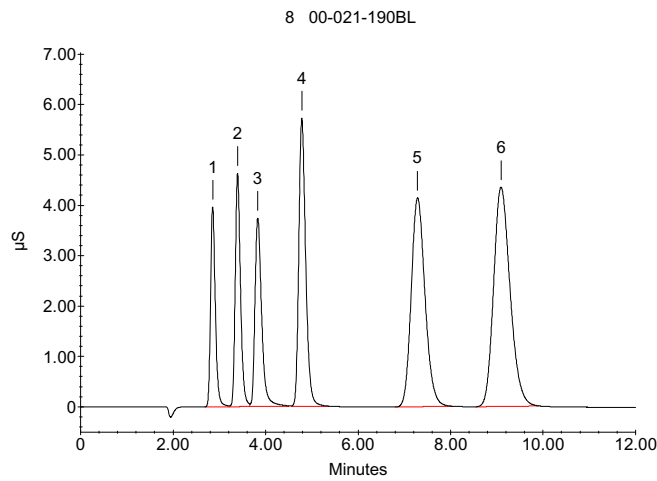
Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	2.14	Lithium	0.1	4725	1.8	3.24
2	2.57	Sodium	0.4	5472	1.5	2.45
3	2.95	Ammonium	0.5	4515	2.1	4.70
4	3.80	Potassium	1.0	6611	1.7	8.01
5	5.98	Magnesium	0.5	4427	1.2	4.22
6	7.67	Calcium	1.0	4766	1.4	n/a

IonPac® CS12A
Analytical (4 x 250 mm)
Product No. 46073

Serial No. : 8 00-021-190BL

Pressure (PSI) : 870

Date : 7/23/01 10:18:10 AM



Eluent: 20 mM MSA*
(Methanesulfonic Acid)

Eluent Flow Rate: 1.0 mL/min

Detection: Suppressed Conductivity
CSRS®-ULTRA
AutoSuppression® Recycle Mode

Background Conductivity: 1 µS

Injection Volume: 25 µL

Storage Solution: Eluent (20 mM
Methanesulfonic Acid)

* The IonPac CS12A test protocol has been changed to use 20 mM Methanesulfonic Acid, (rather than 22 mN Sulfuric Acid) as the test eluent.

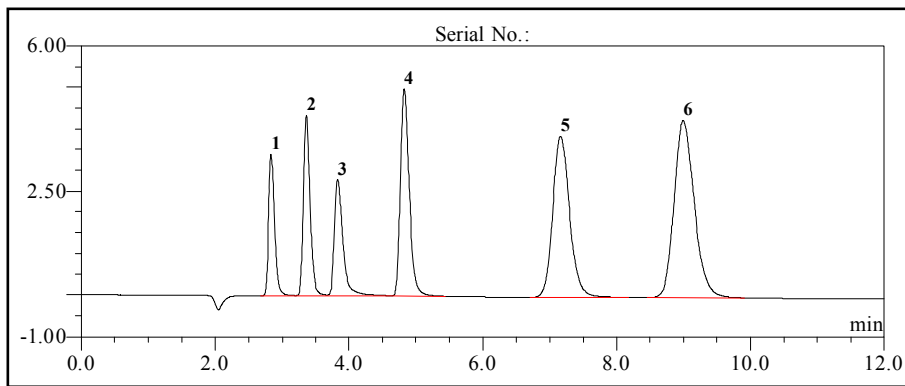
Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	2.86	Lithium	0.5	3736	1.5	2.75
2	3.39	Sodium	2.0	4430	1.4	1.89
3	3.83	Ammonium	2.5	3456	1.6	3.63
4	4.78	Potassium	5.0	5164	1.5	6.20
5	7.29	Magnesium	2.5	2873	1.3	3.03
6	9.09	Calcium	5.0	3115	1.4	n/a

**IonPac® CS12A-5µm
Capillary (0.4 x 150 mm)
Product No. 072068**

Date: 25-Mar-03 10:21
Serial No. :
Lot No. :

Eluent: 20 mM Methanesulfonic acid
Eluent Source: EGC-MSA Capillary Cartridge
Eluent Flow Rate: 0.008 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Cation Capillary Electrolytic Suppressor (CCES-300)
AutoSuppression® Recycle Mode
Applied Current: 7 mA
Injection Volume: 400 nL
Storage Solution: Eluent



No.	Peak Name	Ret. Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	2.84	1.4	3.17	5166	0.1
2	Sodium	3.36	1.4	2.43	6043	0.4
3	Ammonium	3.83	1.7	4.46	5101	0.5
4	Potassium	4.82	1.6	6.89	7037	1.0
5	Magnesium	7.16	1.2	3.70	4038	0.5
6	Calcium	9.00	1.3	n.a.	4393	1.0

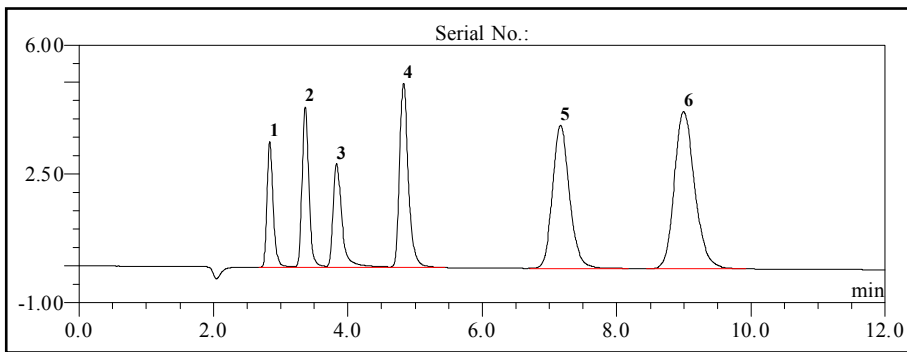
IonPac® CS12A
Capillary (0.4 x 250 mm)
Product No. 072066

Date: 25-Mar-03 10:21

Serial No. :

Lot No. :

Eluent: 20 mM Methanesulfonic acid
Eluent Flow Rate: 0.01 mL/min
Temperature: Ambient Temperature
Detection: Suppressed Conductivity
Suppressor: Cation Capillary Electrolytic Suppressor (CCES-300)
 AutoSuppression® Recycle Mode
Applied Current: 7 mA
Injection Volume: 400 nL
Storage Solution: Eluent



No.	Peak Name	Ret. Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	2.84	1.4	3.17	5166	0.5
2	Sodium	3.36	1.4	2.43	6043	0.5
3	Ammonium	3.83	1.7	4.46	5101	0.5
4	Potassium	4.82	1.6	6.89	7037	0.5
5	Magnesium	7.16	1.2	3.70	4038	0.5
6	Calcium	9.00	1.3	n.a.	4393	5.0

APPENDIX B - COLUMN CARE

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 IonPac CS12A Analytical/Capillary or Guard Column is 4,000 psi (27.57 MPa).



CAUTION

Do not use alcohols.

Formation of esters will occur in the column packing.

This can significantly reduce the column capacity for cation exchange.

Do not use the CS12A column with basic eluents.

Column Start-Up

The column is shipped with eluent as the storage solution. This eluent is the same one shown in the test chromatogram. If you plan to use an eluent other than the test eluent, first equilibrate the column with the desired eluent for 30 to 60 minutes. The column is equilibrated when two consecutive injections of the standard produce the same retention times.

Column Storage

The column's storage solution should be the eluent used for the particular application. If the column will not be used for one week or more, prepare it for long term storage by flushing the column for a few minutes with the eluent. Cap both ends securely, using the plugs supplied with the column.

Column Conditioning

For sample matrices that contain organic solvent content, it is recommended to condition the column with the following procedure:

- A. Disconnect the column and direct the column effluent to a waste container.
- B. Rinse the column for 90 minutes with 0.5 mN sulfuric acid and 10% acetonitrile.
- C. Rinse the column for 30 minutes with eluent.
- D. Reconnect the column to the suppressor.

Column Cleanup

The following column cleanup protocols have been divided into two general isocratic protocols:

- A. Acid soluble contaminants
- 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 include short column rinse steps to reduce the solvent content of the eluent to < 5% levels and the ionic strength of the eluent to < 50 mM levels. 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.

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.



NOTE

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 IonPac CS12A Analytical or Capillary Column. If your system is configured with both a guard column and an analytical or capillary column, place the guard behind the analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.



CAUTION

When cleaning an analytical or capillary column and a guard column in series, ensure that the guard column is placed after the analytical or 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.

- C. Set the pump flow rate to 1.0 mL/min for a CS12 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for a CS12 2-mm Analytical or Guard Column. Set the pump flow rate to 0.010 mL/min for a CS12A or CS12A-5 μ m Capillary or Capillary Guard Column.
- D. Rinse the column for 15 minutes with 10 mM HCl 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 the column for 15 minutes with 10 mM HCl before pumping eluent over the column.
- G. Equilibrate the column(s) with eluent before resuming normal operation for at least 30 minutes.
- H. Reconnect the suppressor to the CS12 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.



CAUTION

Do not pump HCl through the CSRS 300 when used in the electrolytic mode.

Hydrophobic Cations and Organic Contaminants

- A. Disconnect the analytical or capillary column from the injection valve and the suppressor. Disconnect the Gradient Mixer or the Cation Trap Column (CTC-1) from the gradient pump. Connect the IonPac CS12A 2-mm or 4-mm Analytical Column directly to the gradient pump. Direct the effluent from the analytical column directly to a waste container.
- B. Set the flow rate to 1 mL/min on 4-mm systems or 0.25 mL/min on 2-mm systems or 0.010 mL/min for Capillary Systems.

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

Eluent 1: 100mMHCl
Eluent 2: 90% Acetonitrile in deionized water

Time(min)	% E1	% E2
0.0	100	0
20.0	0	100
25.0	0	100
45.0	100	0
55.0	100	0

- D. Rinse the column for 15 minutes with 10 mM HCl before pumping eluent over the column.
- E. Equilibrate the column(s) with eluent before resuming normal operation for at least 30 minutes.
- F. Reconnect the IonPac CS12A Analytical or Capillary Column outlet to the suppressor, and the inlet to either the IonPac CG12A Guard Column, or the gradient pump.
- G. Equilibrate the system with eluent before resuming normal operation.
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