Errata

Product Manual for Dionex IonPac™ CS16 and CG16 Columns
031747-05

For new orders of the following parts discussed in this manual, please use the updated part numbers listed below.

Part	Old Part Number in this manual	Updated Part Number to use for new orders
PROD,COL,IP,CS16,5X250MM	*057573*	*079805*
PROD,COL,IP,CG16,3X50MM	*059595*	*079931*

PRODUCT MANUAL

for

IonPac® CS16
IonPac® CG16

Now sold under the
Thermo Scientific brand

Thermo
SCIENTIFIC

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DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

Product Manual

for

IONPAC® CG16 GUARD COLUMN

5 x 50 mm, P/N 057574
3 x 50 mm, P/N 059595
0.5 x 50 mm, P/N 075402

IONPAC® CS16 ANALYTICAL COLUMN

5 x 250 mm, P/N 057573
3 x 250 mm, P/N 059596
0.5 x 250 mm, P/N 075401

Document No. 031747
Revision 05
December 2010

TABLE OF CONTENTS

SECTION 1 – INTRODUCTION

The IonPac® CS16 5-mm and 3-mm Analytical Columns and the CS16 0.5 x 250 mm (P/N 075401) Capillary Column have been designed specifically for the analysis of alkali metals, alkaline earth metals, and ammonium at diverse concentration ratios. The CS16 stationary phase is a high-capacity weak cation exchanger functionalized with carboxylic acid groups having a high selectivity for hydronium ion. It has both cation exchange and reverse phase properties. The CS16 is solvent-compatible with 100% aqueous eluents, 100% acetonitrile, or 20% tetrahydrofuran without loss of performance.

CAUTION

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

Table 1
IonPac CS16/CG16 Packing Specifications

Column	Particle Diameter µm	Substrate[a] X-linking Capacity %	Column meq/column	Functional Group	Hydrophobicity
CS16 5 x 250 mm	5.5	55	8.4	Carboxylic acid	Medium
CG16 5 x 50 mm	5.5	55	1.7	Carboxylic acid	Medium
CS16 3 x 250 mm	5.5	55	3.0	Carboxylic acid	Medium
CG16 3 x 50 mm	5.5	55	0.6	Carboxylic acid	Medium
CS16 0.5 x 250 mm	5.5	55	0.084	Carboxylic acid	Medium
CG16 0.5 x 50 mm	5.5	55	0.017	Carboxylic acid	Medium

[a] macroporous (100 Å) divinylbenzene/ethylvinylbenzene polymer

Table 2
CS16/CG16 Operating Parameters

Column	Typical Back Pressure at 1.0 mL/min Psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS16 5-mm Analytical	≤ 1,400 (9.65)	1.0	2.0
CG16 5-mm Guard	≤ 450 (3.10)	1.0	3.0
CS16 + CG16 5-mm columns	≤ 1,850 (12.75)	1.0	2.0
CS16 3-mm Analytical	≤ 1,400 (9.65)	0.36	0.72
CG16 3-mm Guard	≤ 450 (3.10)	0.36	1.08
CS16 + CG16 3-mm columns	≤ 1,850 (12.75)	0.36	0.72
CS16 0.5-mm Capillary	≤ 1,400 (9.65)	0.01	0.02
CG16 0.5-mm Capillary Guard	≤ 450 (3.10)	0.01	0.02
CS16 + CG16 0.5-mm columns	≤ 1,850 (12.75)	0.01	0.02

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. All instrument manuals are available on the Dionex Literature and Manuals CD-ROM supplied with this column.

You may need to make a liquid line fitting. The IonPac CS16 Analytical Column and the IonPac CG16 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.

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 – THE ION CHROMATOGRAPHY SYSTEM

Condition	3-mm System Operation Summary	5-mm System Operation Summary	0.5-mm System Operation Summary
Eluent Flow Rate	Typically 0.36 mL/min	Typically 1.0 mL/min	Typically 0.01 mL/min
Cation Self-Regenerating Suppressor	CSRS 300 2-mm (P/N 053949)	CSRS 300 4-mm (P/N 053948)	CCES 300 (P/N 072053)
Cation Atlas Electrolytic Suppressor	CAES® (P/N 065118) (max. 25 mM MSA)	CAES® (P/N 065118) (max. 25 mM MSA)	N/A
Cation MicroMembrane Suppressor 300	CMMS 300 2-mm (P/N 056753)	CMMS 300 2-mm (P/N 056752)	N/A
Regenerant Flow Rate	Typically 50-100% of 5-mm System	Typically 10-15 mL/min	N/A
Injection Loop	5-25 µL	10-50 µL	N/A
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (s-mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplerscan be used. Use the GM-2, GM-3, GM-4 or recommended gradient mixers.	Use only on an IC System equipped for capillary analysis.
Pumps	Use the GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. 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 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 Configuration. The GPM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP50 and IP25. The GP40 has an active mixer. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.	Use only on a pump designed for capillary flow rates, such as the ICS-5000 Capillary Pump.
Detectors	AD25/AD20 Cell (6-mm, 7.5 µL) P/N 046423 CD25 or ED50/ED40 Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) ED40/ED50 Integrated Amperometry Either the TS-1 or 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 ED50/ED40 or the CD20. Ensure 30-40 psi back pressure after the cell.	AD25/AD20 Cell (10-mm, 9 µL) P/N 049393 CD25 or ED50/ED40 Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) ED40/ED50 Integrated Amperometry Either the TS-1 or 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 ED50/ED40 or the CD20. Ensure 30-40 psi back pressure after the cell.	Use only a conductivity detector designed for capillary flow rates such as the ICS-5000 Capillary CD.

Table 3
Tubing Back Pressures

Color	Dionex P/N	ID inches	ID cm	Volume mL/ft	Back Pressure Psi/ft at 1 mL/min	Back Pressure Psi/ft at 0.25 mL/min	Back Pressure Psi/ft 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 0.5 mm Operation

The IonPac CS16 0.5-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 5-mm Operation

The IonPac CS16 5-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 microbore operation.

3.1.3. System Requirements for 3-mm Operation

The IonPac CS16 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 a gradient pump configured for standard bore operation. For alternate detection than conductivity refer to Section 2.

3.1.4. Installation of the Capillary Column

1. Before installing the new separator column, tear off the column label and slide it into the holder on the front of the cartridge (see Figure 1).
2. For reference, Figure 1 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 2 shows the column cartridge after installation of only a capillary separator column.

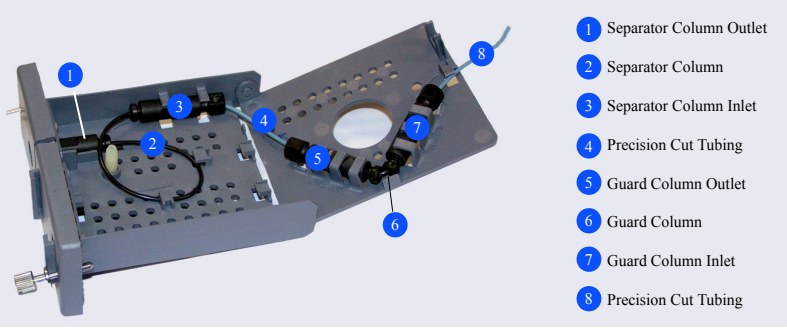


Figure 1
Separator and Guard Columns Installed in Column Cartridge

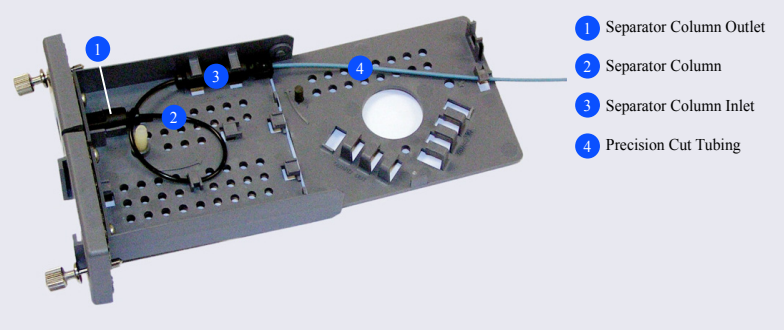


Figure 2
Separator Column Only Installed in Column Cartridge

3. Locate the 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

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

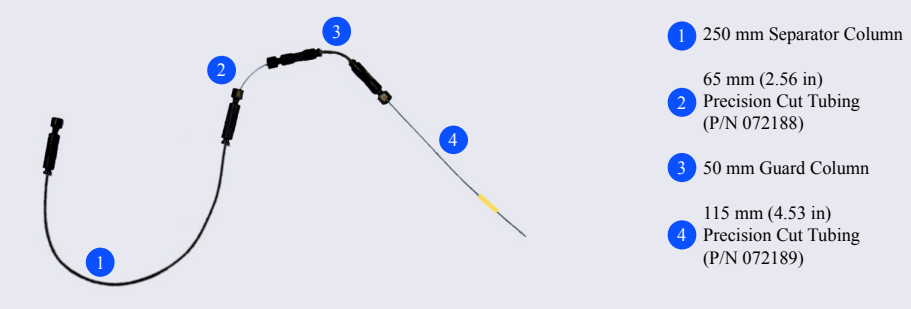


Figure 3
Tubing Connections for 250-mm Separator Column and 50-mm Guard Column

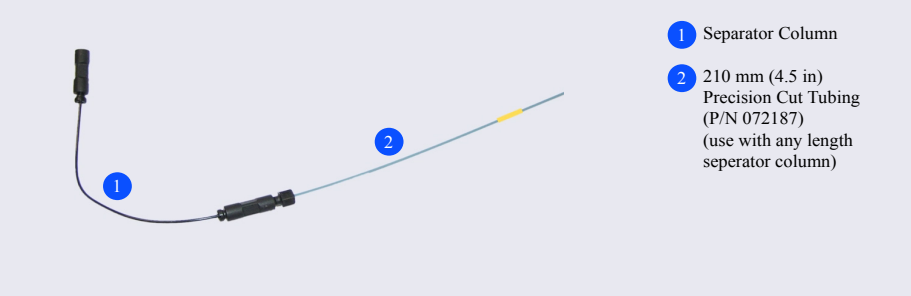


Figure 4
Tubing Connections for Separator Column Only

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

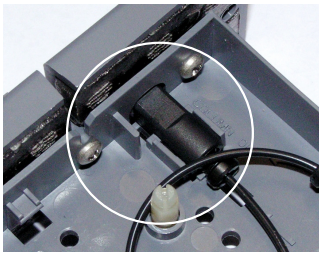


Figure 5
Column Outlet Fitting Installed in Column Cartridge

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

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

Figure 6
Column Cartridge Closed

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

For IonPac CS16 applications using the MSA cartridge, a CR-CTC Continuously Regenerated Trap Column (P/N 060478 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

For gradient operation, an IonPac Cation Trap Column (CTC-1, P/N 040192, for 5-mm CS16 or CTC (2-mm), P/N 043132 for 3-mm CS16) 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. To install the CTC, 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. For the CTC-1 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 flow rate restrictions. For the CTC (2-mm), use 50 mL of a 10x eluent concentrate of the strongest eluent required by the application at a flow rate of 0.5 mL/min
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 inlet.

The background conductivity of your system should be less than 3 µS when 10 mN H_2SO_4 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 µS during a gradient concentration ramp from 10 to 40 mM methanesulfonic acid (MSA). If the baseline shifts are greater than 5 µS after equilibration, the CTC should be cleaned using steps A - E above.

3.4. The Injection Loop

Table 5
Smallest Injectable Volumes (µL)

Valve Type	Using 0.012" ID Tefzel Tubing	Using 0.007" ID Tefzel Tubing	Using 0.010" ID PEEK Tubing	Using 0.005" ID PEEK Tubing
Dionex BF2 Valve (8 µL Internal Volume) (10 cm Loop)	15.2	10.5	13.1	9.2
Dionex MicroInject Valve (10.5 µL Internal Volume) (14 cm Loop)	20.5	14.0	17.6	12.2
Dionex Microinjection Valve Model 9126 (0.8 µL Internal Volume) (10 cm Loop)	8.0	3.3	5.9	2.0

For most applications on a standard bore analytical system, a 10–50 µL injection loop will be sufficient. Dionex recommends that a 10 µL injection loop be used to avoid overloading the CS16 5-mm Analytical Column. Generally, do not inject more than 10 nanomoles (100–200 ppm) of any one analyte onto the 5-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. This phenomenon will be more prevalent at higher concentrations of the analytes of interest. In the case of the CS16 3-mm, do not inject more than 4 nanomoles (35–70 ppm) of any one analyte onto the 3-mm analytical column.

3.5. Sample Concentration

The IonPac CG16 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 a 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 CG16) 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 CG16) for the analytical chemist in these applications is the capability of performing routine trace analyses of sample matrix ions at ng/L levels without extensive and laborious sample pretreatment.

The IonPac CG16 5-mm Guard Column (P/N 057574) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027) should be used for sample concentration with the IonPac CS16 5-mm Analytical Column. The IonPac CG16 3-mm Guard Column (P/N 059595) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027) should be used for sample concentration with the IonPac CS16 3-mm Analytical Column. For the Trace Cation Concentrator to work with the CS16 0.5 mm Column use the CG16 0.5 mm Column or the IonSwift MCC-100 (0.5 x 80 mm) or MCC-200 (0.75 x 80 mm) Concentrator Column.

CAUTION

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 CS16 eluents will not properly elute ions concentrated on this column.

3.6. IonPac CG16 Guard Columns

An IonPac CG16 Guard Column is normally used with the IonPac CS16 Analytical or Capillary Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical/capillary column. A guard is placed prior to the analytical/capillary column to prevent sample contaminants from eluting onto the analytical/capillary column. It is easier to clean or replace a guard column than it is an analytical/capillary column. For maximum life of the analytical/capillary column, the guard column should be changed or replaced as part of a regular maintenance schedule or at the first sign of performance deterioration. Use the test chromatogram that is shipped with the analytical/capillary column or the initial application run for a performance benchmark.

3.7. Eluent Storage

IonPac CS16 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 and Cation Capillary Electrolytic Suppressor Requirements

A Cation Self-Regenerating Suppressor should be used for applications that require suppressed conductivity detection. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all CSRS 300 modes of operation.

NOTE

Solvent containing eluents must be used in the Chemical Suppression Mode using either the CSRS 300 or CMMS 300 Suppressor. (If the CSRS 300 is used with solvents, be sure to use TBAOH as regenerant and with no current applied to the CSRS 300 suppressor)

When the oven is operated at > 40 °C, the suppressor should be placed outside the oven.

If you are installing an IonPac CS16 5-mm Analytical Column, use a CSRS 300 (4-mm, P/N 053948). If you are installing an IonPac CS16 3-mm Analytical Column, use a CSRS 300 (2-mm, P/N 053949). If you are installing an IonPac CS16 0.5-mm Capillary Column, use a CCES 300 (P/N 072053).

For detailed information on the operation of the Cation Self-Regenerating Suppressor, see Document No. 031139, the "Product Manual for the Cation Self-Regenerating Suppressor 300, the CSRS 300 (4-mm) and the CSRS 300 (2-mm)." For detailed information on the operation of the Cation Capillary Electrolytic Suppressor, see Document No. 065386.

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 µeq/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 Self-Regenerating Suppressor, CSRS 300, should be used for applications that require suppressed conductivity detection. It is compatible with all solvents in the AutoSuppression External Water Mode (see Section 3.7, "Cation Self-Regenerating 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. 034359, the "Product Manual for the Cation MicroMembrane Suppressor 300, the CMMS 300."

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

Dionex recommends using the Displacement Chemical Regeneration (DCR) Mode for chemical suppression using tetrabutylammonium hydroxide (TBAOH) and the Cation MicroMembrane Suppressor (CMMS 300). See the DCR kit manual, Document P/N 031664, for details.

3.12. Using AutoRegen® with the Chemical Suppression Mode

Dionex recommends using an AutoRegen Accessory (P/N 039594) with eluents that do not contain acetonitrile. It should be used with the CMMS 300. 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 H_2SO_4 or methanesulfonic acid step or linear gradient, a high regenerant flow rate (10–15 mL/min) is required.

3.13. Detector Requirements

See Section 2, “The 5-mm and 3-mm Ion Chromatography System,” system detector, cell and thermal stabilizer requirements.

SECTION 4 – OPERATION

4.1. General Operating Conditions

Column:	CS16 0.5-mm Capillary Column (+ CG16 0.5-mm Capillary Guard Column) CS16 5-mm Analytical Column (+ CG16 5-mm Guard Column); CS16 3-mm Analytical Column (+ CG16 3-mm Guard Column)
Sample Volume 0.5-mm CS16:	0.4 µL Loop
Sample Volume 5-mm CS16:	25 µL Loop + 0.8 µL Injection valve dead volume
Sample Volume 3-mm CS16:	10 µL Loop + 0.8 µL Injection valve dead volume
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate CS16 5-mm:	1.0 mL/min
Eluent Flow Rate CS16 3-mm	0.36 mL/min
Eluent Flow Rate CS16 0.5-mm	0.010 mL/min
Temperature:	40 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode for the CS16 5-mm; for CS16 3-mm use the CSRS 300 (2-mm)
or CES Suppressor:	Cation Capillary Electrolytic Suppressor, CCES 300 (0.4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm or 4-mm)
MMS Regenerant:	Tetrabutylammonium hydroxide (TBAOH)
MMS Mode:	Displacement Chemical Regeneration (DCR) Mode is recommended
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

4.2. Operating Precautions

IonPac CS16 Operation Precautions

CAUTION

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 µm. Filter water with a 0.2 µm filter. Bottled HPLC-Grade Water (with the exception of Burdick & Jackson) should not be used since most bottled water contains an unacceptable level of ionic impurities.

4.3.2. Inorganic Chemicals

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

A. Use Fluka or Aldrich Methanesulfonic Acid (MSA) (>99% pure) or Dionex Methanesulfonic Acid (0.4 M).

B. Use only concentrated sulfuric acid (H_2SO_4), ACS grade or BAKER INSTRA-ANALYZED® for trace metals.

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. For the DCR Mode, use Dionex TBAOH (P/N 057561).

D. Use deionized water with a specific resistance of at least 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 Methanesulfonic Acid (MSA) Stock Solution

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

A. Weigh out 96.10 g of methanesulfonic acid (MSA, ≥ 99%, P/N 033478).

B. Carefully add this amount to a 1-liter volumetric flask containing about 500 mL of deionized water.

C. Dilute to the mark and mix thoroughly.

4.4.2 0.4 N Methanesulfonic Acid (MSA) Eluent Concentrate

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

4.4.2. 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 18.2 megohm-cm. Dilute to the 1 liter mark and mix thoroughly.

In other words:

FW of H_2SO_4 = 98.08 g
H_2SO_4 concentration = 98%

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

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

4.4.3. Eluent Preparation

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

Using the table below, pipet x.0 mL of the 1.0 N H_2SO_4 or 1.0 N MSA eluent concentrate (see Section 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 6
mN Eluents from Stock Solutions

mN H_2SO_4 or MSA	# mL H_2SO_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

4.5. Eluents with Solvents

Solvents can be added to the ionic eluents used with IonPac CS16 columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column 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 CS16 columns is 4,000 psi (27.57 MPa).

The IonPac CS16 is compatible with the HPLC solvents listed in Table 7, "HPLC Solvents for Use with the CS16 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 7
HPLC Solvents for Use with IonPac CS16 Columns

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

4.5.1. 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 when using eluents containing solvents. Dionex recommends the Chemical Suppression Mode for long term trouble free operation.

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

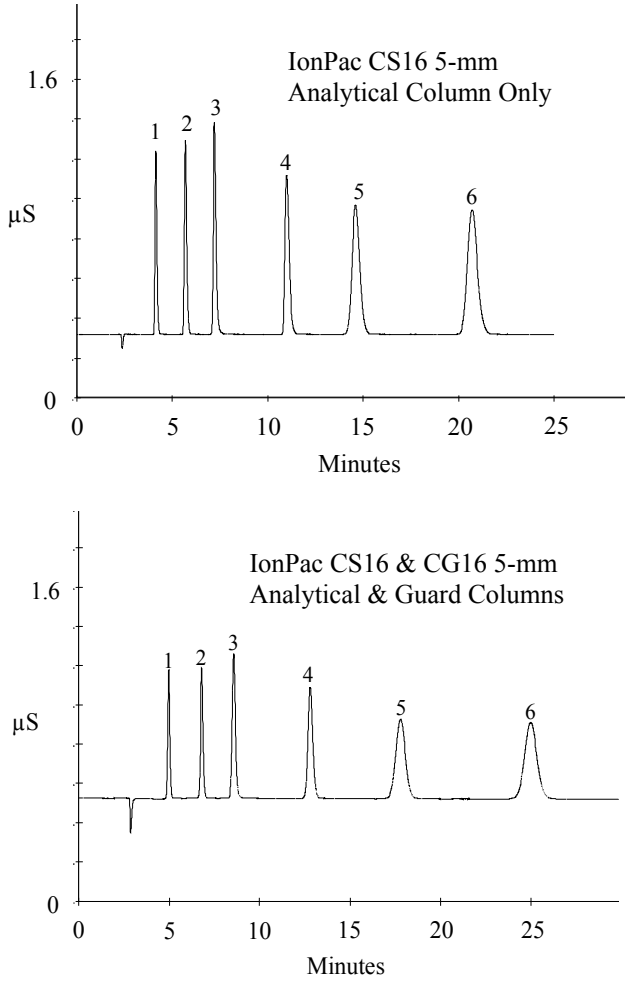
The chromatograms in this section were obtained using columns that reproduced the Production Test Chromatogram (see Section 5.3, "Production Test Chromatogram") on optimized Ion Chromatographs (see Section 3, "Installation"). Different systems will differ slightly in performance due to slight differences 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 at 40 °C

Isocratic elution of cations on the IonPac CS16 Analytical Column has been optimized utilizing a methanesulfonic acid eluent. Using this eluent, the weak carboxylate functionalized packing isocratically separates mono- and divalent cations in a single injection. To guarantee that all IonPac CS16 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Column:	CS16 5-mm
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode,
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

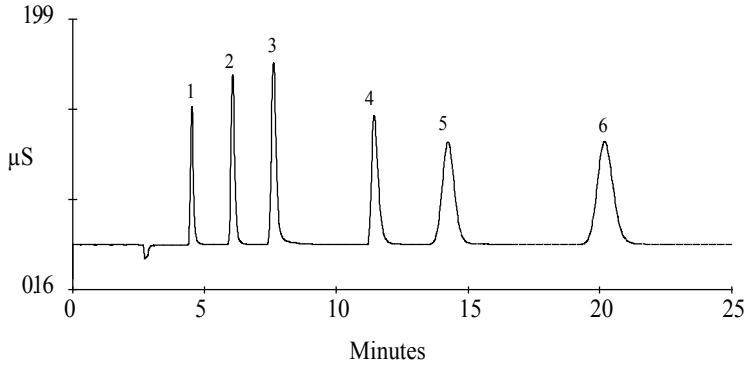


	Analyte	mg/L
1.	Lithium	0.1
2.	Sodium	0.4
3.	Ammonium	0.5
4.	Potassium	1.0
5.	Magnesium	0.5
6.	Calcium	1.0

where 1 mg/L = 1 ppm

Figure 7
IonPac CS16 5-mm Production Test Chromatogram

Column:	CS16 3-mm (no guard)
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.36 mL/min
Temperature:	40 °C
Suppressor*:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode,
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



	Analyte	mg/L
1.	Lithium	0.05
2.	Sodium	0.20
3.	Ammonium	0.25
4.	Potassium	0.50
5.	Magnesium	0.25
6.	Calcium	0.50

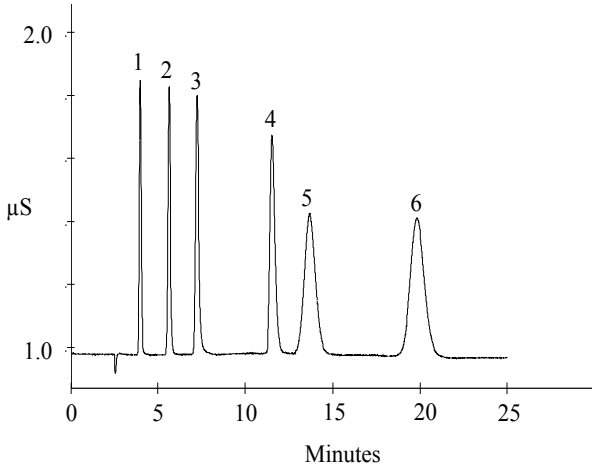
where 1 mg/L = 1 ppm

Figure 8
IonPac CS16 3-mm Production Test Chromatogram

5.2. Reproducing the Production Test Chromatogram Using a Sulfuric Acid Eluent at 40 °C

When substituting sulfuric acid for methanesulfonic acid, the eluent concentration must be increased to 34 mN sulfuric acid in order to resolve all of the peaks in the production test chromatogram with the runtime within 20 minutes.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	34 mN Sulfuric acid (H_2SO_4)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode,
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



	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

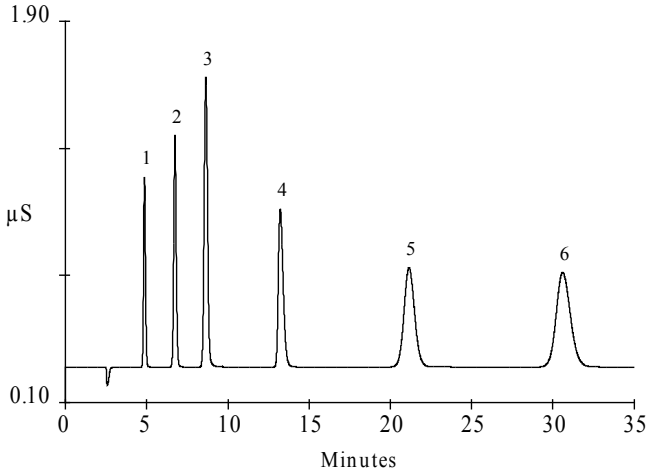
where 1 mg/L = 1 ppm

Figure 9
IonPac CS16 5-mm Production Test Chromatogram
Using Sulfuric Acid

5.3. 6-Cations (Li^+, Na^+, $NH4^+$, K^+, Mg^{2+}, Ca^{2+}) with CS16 3-mm and CAES

In order to use a CS16 3-mm column with an Atlas Suppressor, the maximum eluent concentration allowed is 25 mM MSA.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	25 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.36 mL/min
Temperature:	40 °C
AES Suppressor:	Cation Atlas Electrolytic Suppressor, CAES
or SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 (2-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 0.5 µS
Storage Solution:	Eluent



Analyte	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.25
4. Potassium	0.50
5. Magnesium	0.25
6. Calcium	0.50

where 1 mg/L = 1 ppm

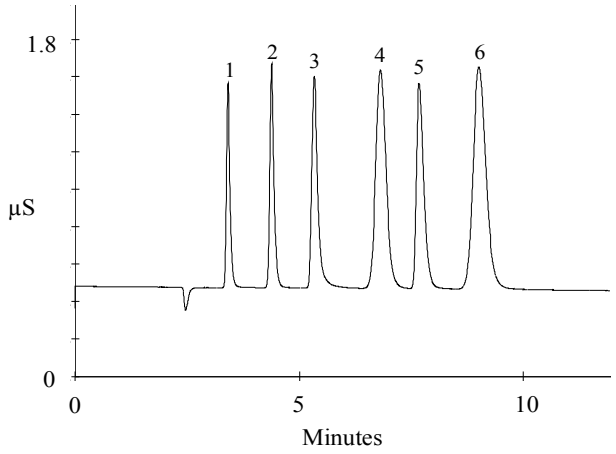
Figure 10
IonPac CS16 3-mm 6-Cation Run at 40 °C with an Atlas Suppressor

The Cation Atlas Electrolytic Suppressor (P/N 056118) can be used with up to 25 mM Methanesulfonic acid at 1.0 mL/min, as with a CS16 5-mm column.

5.4. 6-Cation Fast Run (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) at 40 °C

Isocratic elution of ammonia, selected alkali metals, and selected alkaline earth cations. (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) can be completed within 10 minutes. Notice that with this higher eluent concentration, magnesium, a divalent, elutes before potassium, a monovalent. This is an ideal operation mode when analytes are present in similar concentrations.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	48 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



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

where 1 mg/L = 1 ppm

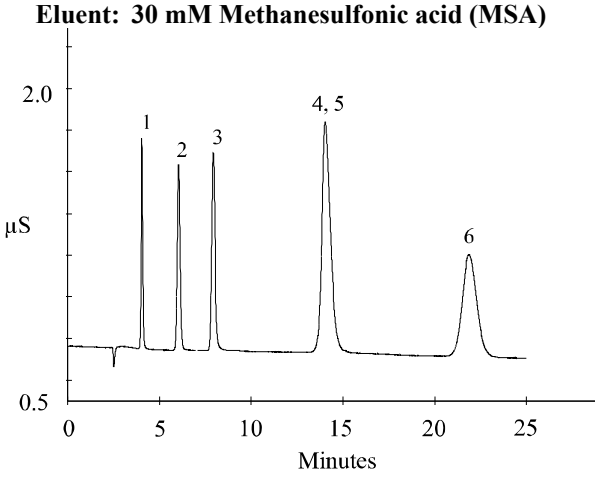
Figure 11
IonPac CS16 5-mm 6-Cation Fast Run at 40 °C

5.5. 6-Cation Run (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) at Room Temperature Operation

If the isocratic elution of ammonia, selected alkali metals, and selected alkaline earth cations (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca2+) is run at room temperature using 30 mM Methanesulfonic acid (MSA), as shown below, potassium and magnesium co-elute. By adjustment of the eluent concentration to 36 mM Methanesulfonic acid (MSA) separation of potassium and magnesium is obtained. If better resolution between potassium and magnesium is required, the eluent should be increased by 2 mM MSA to 38 mM MSA. This higher eluent concentration will actually be more forgiving to room temperature fluctuations, as shown in the following section.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	See Chromatogram
Eluent Flow Rate:	1.0 mL/min
Temperature:	Room Temperature (approximately 22 °C)
Suppressor:	Cation Self-Regenerating Suppressor 300 (4-mm) in AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

Eluent: 30 mM Methanesulfonic acid (MSA)



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

where 1 mg/L = 1 ppm

Eluent: 36 mM Methanesulfonic acid (MSA)

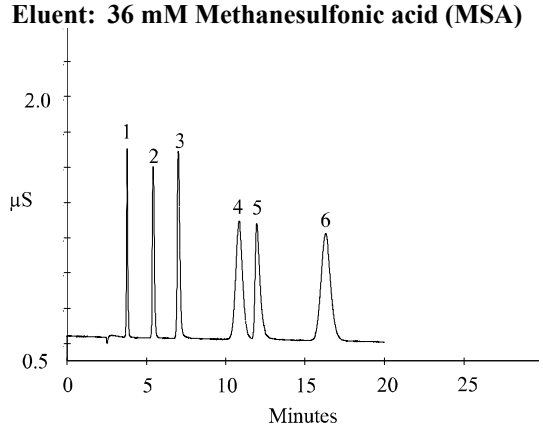


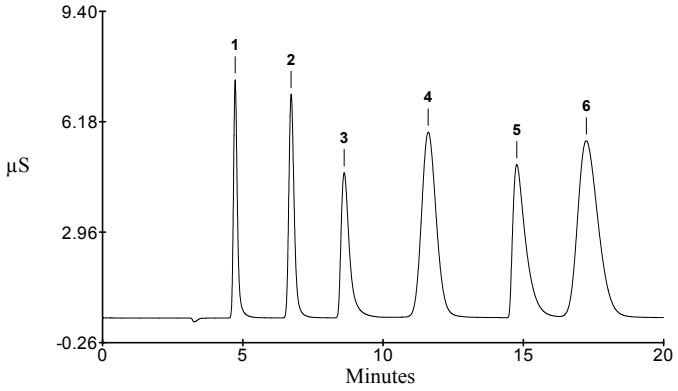
Figure 12
IonPac CS16 5-mm 6-Cation Run at Room Temperature

5.5.1. 6-Cation Run (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) at Room Temperature Operation Continuation

To minimize potential peak overlap between magnesium and potassium with changes in lab temperature, it is better to use a 38 mM methanesulfonic acid eluent, as shown below at 22 °C and 29 °C lab temperature.

Column:	IonPac CS16 3-mm Analytical Column with guard
Sample Volume:	25 µL Loop
Eluent:	38 mM Methanesulfonic acid
Eluent Flow Rate:	0.36 mL/min
Temperature:	See chromatogram
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

Eluent: 38 mM Methanesulfonic acid (MSA), 22 °C



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

where 1 mg/L = 1 ppm

Eluent: 38 mM Methanesulfonic acid (MSA), 29 °C

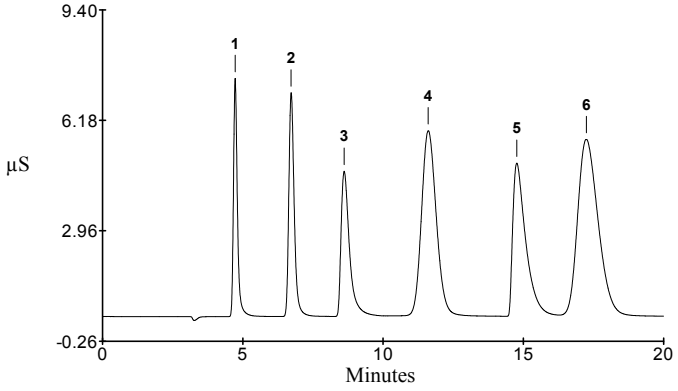
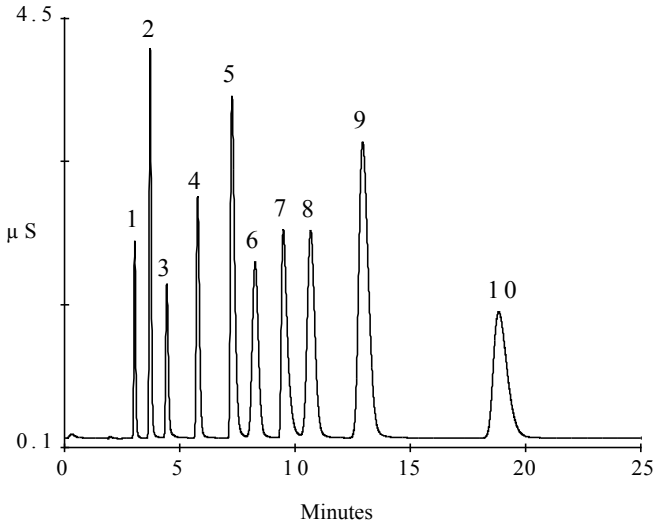


Figure 13
IonPac CS16 3-mm 6-Cation Run Optimized for Room Temperature Operation

5.6. 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+}) at 60 °C

The isocratic elution of ammonia plus Group I and Group II cations (Li^+, Na^+, NH_4^+, K^+, Rb^+, Cs^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}) can be completed within 20 minutes using a methanesulfonic acid eluent (MSA) at 60 °C. A sulfuric acid eluent should not be used for the determination of barium. The advantage of the higher capacity of the CS16 for this application is improved resolution between sodium and ammonium, enabling these two analytes to be present at disparate concentration ratios. Note that with the higher capacity CS16 column, cesium, a monovalent, elutes after magnesium, a divalent.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	36 mM Methanesulfonic acid (MSA)
Eluent Source:	EG40
Eluent Flow Rate:	0.5 mL/min
Temperature:	60 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode, (suppressor placed outside the oven)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



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

where 1 mg/L = 1 ppm

Figure 14
IonPac CS16 Isocratic Elution of
(Li^+, Na^+, NH_4^+, K^+, Rb^+, Mg^{2+}, Cs^+, Ca^{2+}, Sr^{2+}, Ba^{2+})

5.7. Isocratic Elution of 6-Cations Plus Morpholine

(Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Morpholine and Ca^{2+}) at 40 °C

Isocratic elution of ammonia, morpholine plus Group I and Group II cations (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Morpholine and Ca^{2+}) can be completed within 20 minutes. Morpholine is well resolved from magnesium.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	34 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

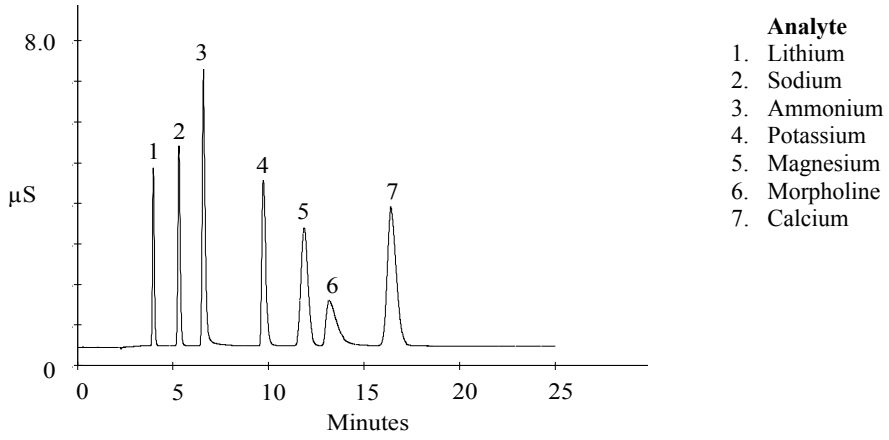


Figure 15
IonPac CS16 Isocratic Elution of
Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Morpholine and Ca^{2+}

5.8. 10,000:1 Ratio Sodium to Ammonium at 40 °C

A. CS16 5-mm

The following chromatograms show a ratio of 10,000:1 of sodium to ammonium on the CS16 5-mm using an isocratic eluent. In cases where the sodium peak does not return to the baseline (see chromatogram A), it is recommended that a CTC-1 trap column (P/N 040192) be placed between the CELL OUT and the suppressor REGEN IN ports (see chromatogram B). Alternatively, the suppressor can be used in the External Water mode.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

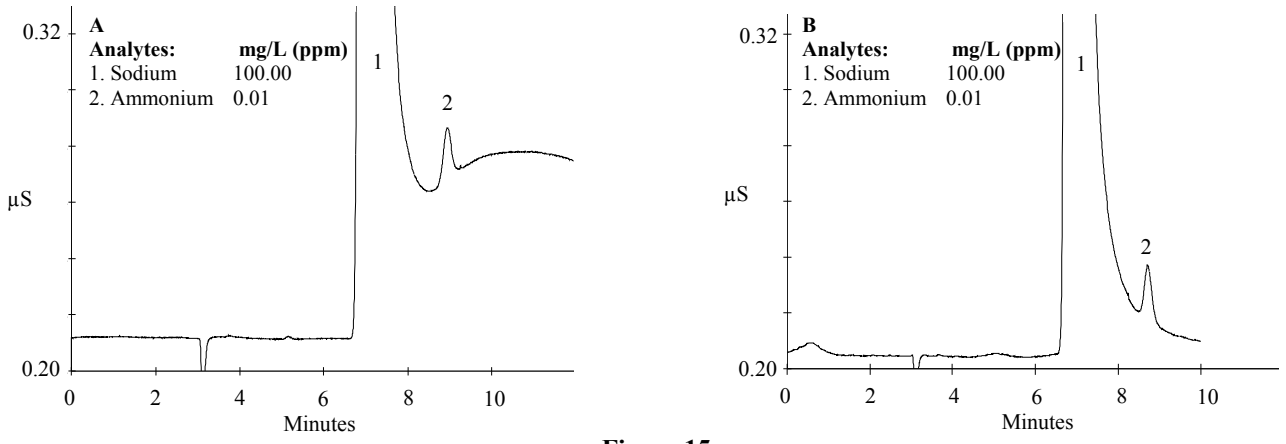


Figure 15a
Trace-level Quantification of Ammonium in Brine (10,000:1 ratio)

The conditions for the 6-Cation Fast Run, see Section 5.6, "6-Cation Fast Run (Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}) at 40 °C," can also be used for this application, although the separation between sodium and ammonium is not as good.

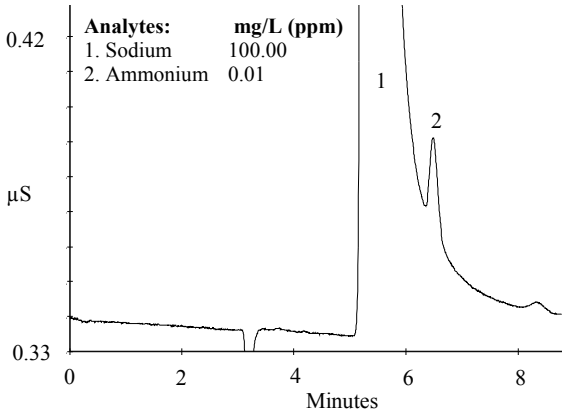
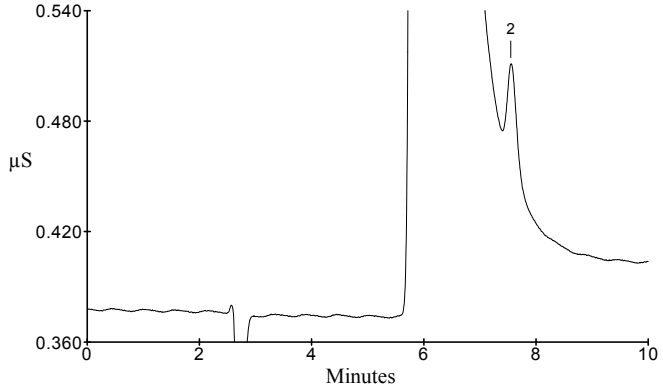


Figure 16
Trace-level Quantification of Ammonium in Brine (10,000:1 ratio) Using the 6-Cation Fast Run

B. CS16 3-mm

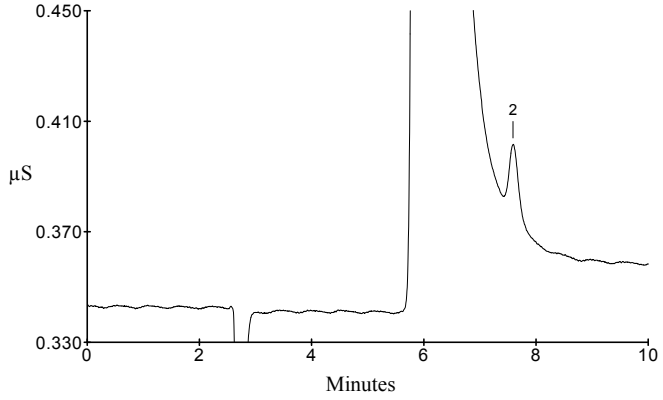
The following shows a ratio of 10,000:1 of sodium to ammonium on the CS16 3-mm using an isocratic eluent. Either a smaller sample loop size than with a CS16 5-mm should be used or the sample should be diluted not to overload the column.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.36 mL/min
Temperature:	40 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode, with CTC-1 between **CELL OUT** and *REGEN IN* ports
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



A

Analytes:	**mg/L (ppm)**
1. Sodium	100.00
2. Ammonium	0.01



B

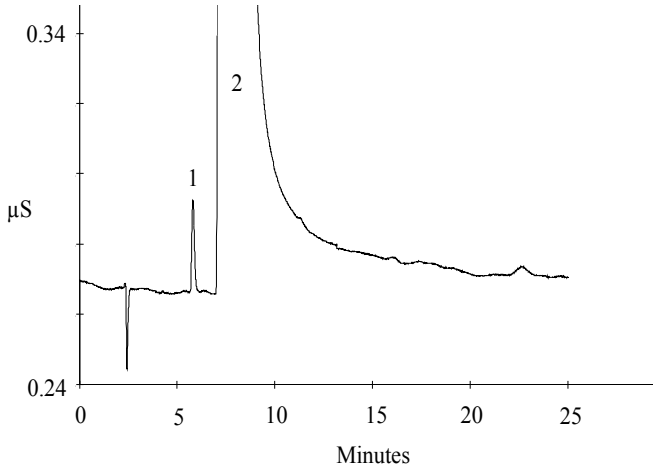
Analytes:	**mg/L (ppm)**
1. Sodium	33.3
2. Ammonium	0.0033

Figure 17
Trace-level Quantification of Ammonium in Brine (10,000:1 ratio)

5.9. 1:10,000 Ratio Sodium to Ammonium at 40 °C

The following chromatogram shows a separation of a ratio of 1:10,000 of sodium to ammonium on the CS16 5-mm using an isocratic eluent at 40 °C. It is recommended that a CTC-1 trap column (P/N 040192) be placed between the **CELL OUT** and the suppressor REGEN IN ports when using the CSRS 300 in the AutoSuppression Recycle Mode. Alternatively, the suppressor can be used in the External Water Mode.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Trap Column:	CTC-1 is placed between the CELL OUT and the REGEN IN ports
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



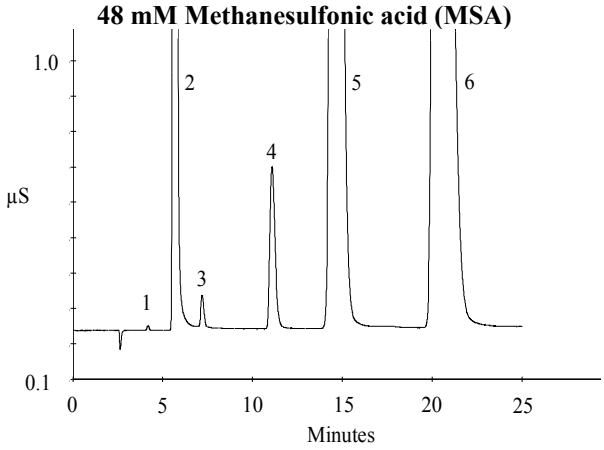
Analytes:	**mg/L (ppm)**
1. Sodium	100.00
2. Ammonium	0.01

Figure 18
1:10,000 Ratio Sodium to Ammonium

5.10. Analysis of Tap Water at 40 °C

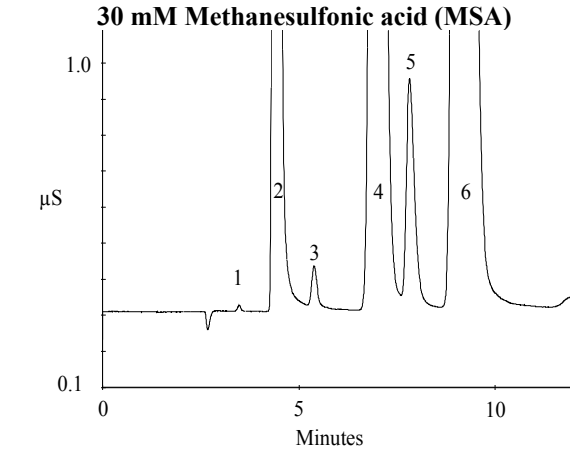
Tap water samples will typically contain high levels of sodium, magnesium and calcium and relatively low levels of other cations. In the top chromatogram, a weaker 30 mN methanesulfonic acid eluent was used, while the bottom chromatogram was obtained with the stronger 48 mN methanesulfonic acid eluent. The approximate calculated 300:1 ratio of sodium-to-ammonium is easy to determine in either case (see peaks 2 and 3).

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	See Chromatogram
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



Analyte	mg/L
1. Lithium	0.002
2. Sodium	19.73
3. Ammonium	0.065
4. Potassium	0.987
5. Magnesium	7.210
6. Calcium	18.54

where 1 mg/L = 1 ppm



Analyte	mg/L
1. Lithium	0.002
2. Sodium	19.73
3. Ammonium	0.065
4. Magnesium	7.210
5. Potassium	0.987
6. Calcium	18.54

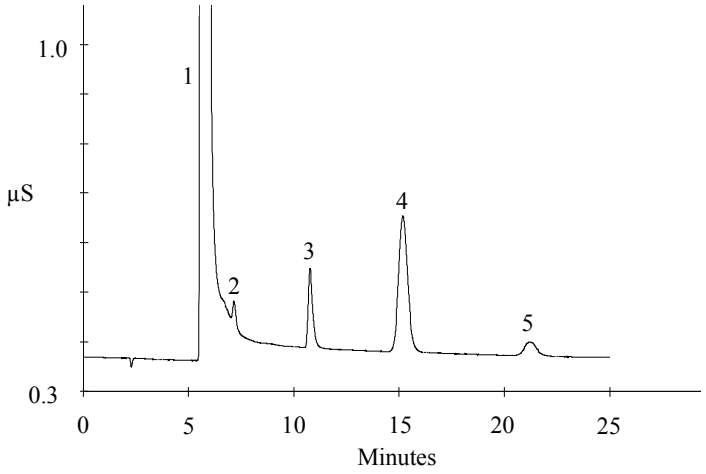
where 1 mg/L = 1 ppm

Figure 19
IonPac CS16 5-mm Analysis of Tap Water

5.11. Analysis of Waste Water at 40 °C

Wastewater samples generally contain more diverse concentration ratios of sodium and ammonium, as shown in the example below. The calculated sodium-to-ammonium ratio is 6800:1. Furthermore, wastewater samples may contain amines and transition metals which can potentially interfere with the analysis of the common cations. The high capacity CS16 column minimizes the chance to overload the column and offers a greater chance of resolving the sample's component peaks.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Sample Dilution:	0.5 : 100
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



Analyte	mg/L *
1. Sodium	27.85
2. Ammonium	4.1
3. Potassium	44.8
4. Magnesium	49.8
5. Calcium	10.6

where 1 mg/L = 1 ppm
* (Calculated in Original Sample)

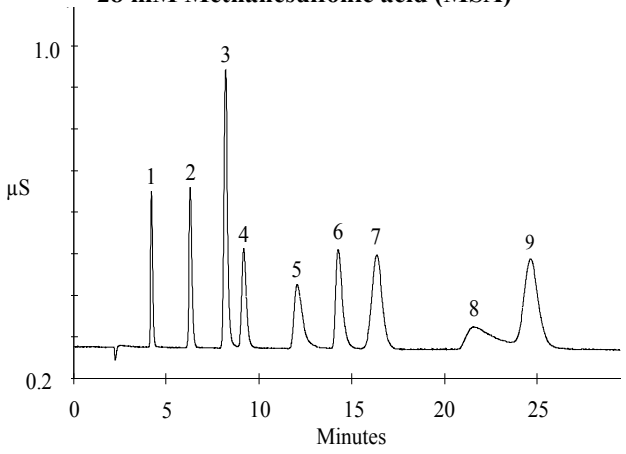
Figure 20
IonPac CS16 5-mm Analysis of Waste Water

5.12. Separation of Methylamines, Group I and Group II Cations and Ammonium at Room Temperature

The chromatograms below were obtained at room temperature, and the eluent concentration adjusted for optimized separations. The analytes consisted of a mixture of the common inorganic cations, ammonium plus methyl-, dimethyl- and trimethylamine. Here, ammonium and methylamine, peaks number 3 and 4, are well resolved from each other. By increasing the eluent concentration (bottom chromatogram) the run time is decreased to about 15 minutes with improved amine peak shapes. See Section 5.15, "Amine Selectivity with Common Cations at 65 °C," for improved chromatography of trimethylamine.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	See Chromatogram
Eluent Flow Rate:	1.0 mL/min
Temperature:	Room Temperature (approximately 22 °C)
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

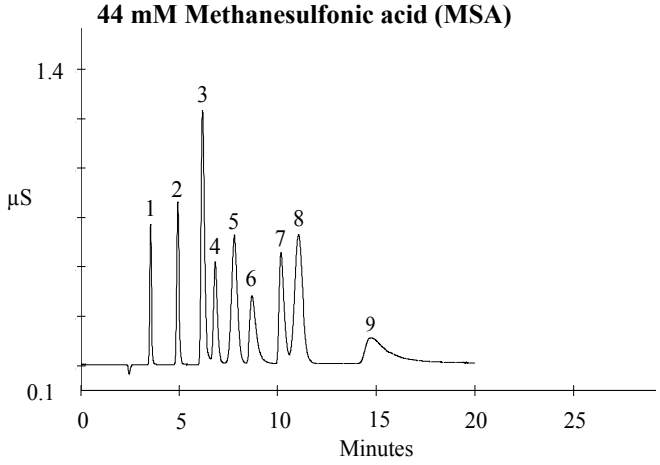
28 mM Methanesulfonic acid (MSA)



Analyte	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.30
4. Methylamine	0.50
5. Dimethylamine	1.00
6. Potassium	0.50
7. Magnesium	0.25
8. Trimethylamine	1.50
9. Calcium	0.50

where 1 mg/L = 1 ppm)

44 mM Methanesulfonic acid (MSA)



Analyte	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.30
4. Methylamine	0.50
5. Magnesium	0.25
6. Dimethylamine	1.00
7. Potassium	0.50
8. Calcium	0.50
9. Trimethylamine	1.50

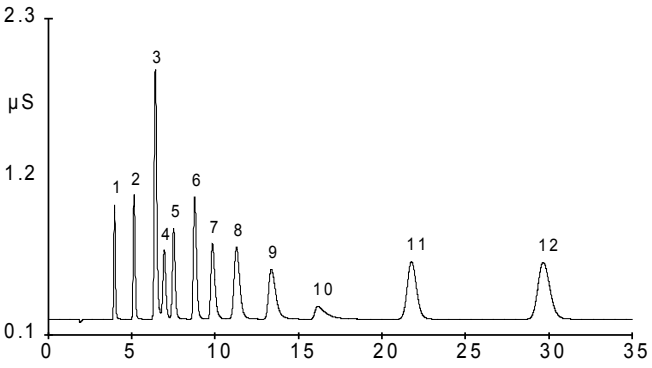
where 1 mg/L = 1 ppm)

Figure 21
IonPac CS16 5-mm Methylamine Selectivity with Common Cations Using Two Different Eluents

5.13. Isocratic vs. Gradient Elution of Common Cations and Amines at 60 °C

This chromatogram shows the isocratic separation of the common inorganic cations, ammonium, three alkylamines, and three commonly used additives in the Power Industry: ethanolamine, 5-amino-1-pentanol, and morpholine. The CS16 high capacity carboxylated analytical column can accomplish this separation using an isocratic, solvent-free eluent consisting of 20 mM methanesulfonic acid. To resolve ammonium from ethanolamine, the column is used at 60 °C. The high temperature has the added benefit of improving the peak efficiencies and symmetries of the analyte peaks, especially for the alkylamines (such as peak # 10, trimethylamine). The CSRS 300 is placed outside of the column oven.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	20 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.5 mL/min
Temperature:	60 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode (Suppressor placed outside oven)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



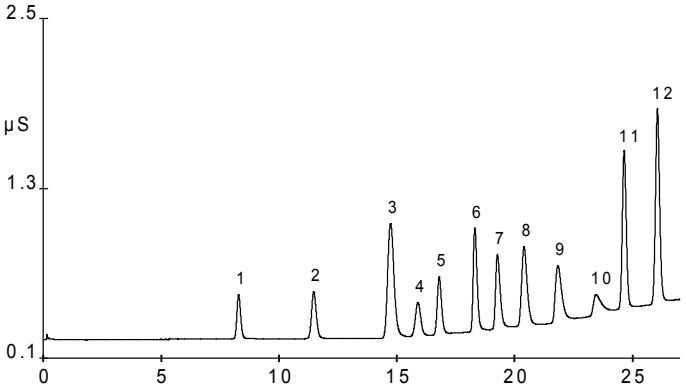
Analyte	mg/L
1.Lithium	0.05
2. Sodium	0.20
3.Ammonium	0.42
4.Ethanolamine	0.50
5.Methylamine	0.50
6. Potassium	0.50
7.Dimethylamine	1.00
8.5-amino-1-pentanol	2.00
9.Morpholine	2.00
10.Trimethylamine	1.50
11.Magnesium	0.25
12.Calcium	0.50

where 1 mg/L = 1 ppm

Figure 22
Isocratic Elution of Common Cations and Amines at 60 °C

Under gradient conditions, from 6 mM up to 55 mM methanesulfonic acid, using an Eluent Generator, peaks # 3 and 4, ammonium and ethanolamine, are well separated, and the total run time is even a few minutes shorter than before. Starting the elution with a low concentration acidic eluent and using the column at high temperature, 60 °C, aids the separation of these two components. The acid concentration in the eluent is then increased in a gradient to speed the analysis, having the added benefit of sharpening the later-eluting peaks and improving their minimum detection levels. The eluent contained no organic solvent, so the suppressor was used in the eluent recycle mode. Note that the suppressor is placed outside the chromatography oven when temperatures exceed 40 °C as they do here.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Eluent:	6 mM MSA, gradient from 0 to 13 min to 8 mM MSA, gradient from 13 to 25 min to 55 mM MSA
Eluent Source:	EG40
Flow Rate:	0.5 mL/min
Inj. Volume:	25 µL
Detection:	Suppressed conductivity CSRS 300 2-mm outside oven AutoSuppression® Recycle Mode



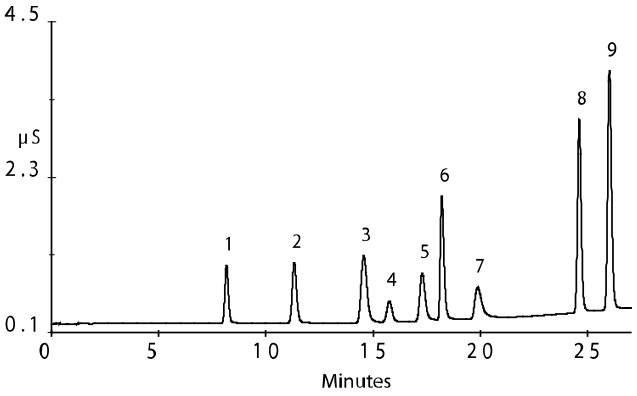
Analyte	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.42
4. Ethanolamine	0.50
5. Methylamine	0.50
6. Potassium	0.50
7. Dimethylamine	1.00
8. 5-Amino-1-pentanol	2.00
9. Morpholine	2.00
10. Trimethylamine	1.50
11. Magnesium	0.25
12. Calcium	0.50

Figure 22 Continued
Gradient Elution of Common Cations and Amines at 60 °C

5.14. Gradient Elution of the Six Common Cations plus Mono-, Di-, and Triethanolamines at 60 °C

Six common inorganic cations, ammonium plus three ethanolamines can be separated by gradient elution. To minimize the baseline change, the Eluent Generator (EG40) was used to generate the methanesulfonic acid gradient. An elevated temperature of 60 °C is used to improve the resolution of ammonium and ethanolamine. The CSRS 300 is placed outside of the column oven.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent Source:	EG40
Gradient:	6 mM MSA, gradient from 0 to 13 minutes to 8 mM MSA, gradient from 13 to 25 minutes to 55 mM MSA
Eluent Flow Rate:	0.5 mL/min
Temperature:	60 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode (Suppressor placed outside oven)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



Analyte	mg/L
1. Lithium	0.1
2. Sodium	0.4
3.Ammonium	0.5
4. Ethanolamine	0.5
5. Diethanolamine	1.0
6. Potassium	1.0
7. Triethanolamine	18.0
8. Magnesium	0.5
9. Calcium	1.0

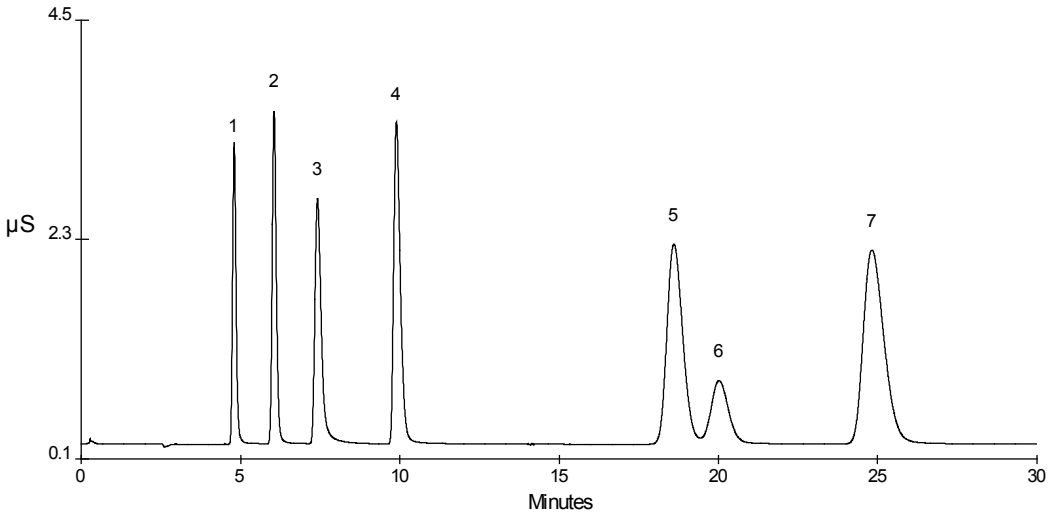
where 1 mg/L = 1 ppm

Figure 23
Gradient Elution of the Six Common Cations plus Mono-, Di-, and Triethanolamines at 60 °C

5.15. Isocratic Elution of the Six Common Cations Plus Manganese at 60 °C

Six common inorganic cations, ammonium plus manganese can be separated by isocratic elution at 60 °C. The CSRS 300 is placed outside of the column oven. This separation can be achieved on a CS12A at room temperature using isocratic conditions. The CS16 should be used where disparate concentration ratios of sodium and ammonium besides calcium, magnesium and manganese are to be determined.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	26 mM Methanesulfonic acid (MSA)
Eluent Source:	EG40
Eluent Flow Rate:	0.36 mL/min
Temperature:	60 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode (placed outside oven)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



Analyte	µg/L
1. Lithium	67
2. Sodium	267
3. Ammonium	330
4. Potassium	667
5. Magnesium	330
6. Manganese	167
7. Calcium	667

where 1 µg/L = 1 ppb

Figure 24
Isocratic Elution of the Six Common Cations plus Manganese at 60 °C

5.16. Sample pH at 40 °C

The IonPac CS16 resin contains relatively weak carboxylic acid cation exchange sites. The ionization of the sites are dependent on the eluent and on the sample pH. Furthermore, because these cation exchange sites are hydronium-selective, the sample pH will have an impact on the elution of the analyte cations from such sites. As the sample pH decreases, so do peak efficiencies and symmetries. Due to higher cation exchange capacity (approximately 8.4 meq/column), the IonPac CS16 5-mm column can tolerate samples of low pH. Quantitation can be performed for samples with pH 1.0 (or 100 mM acid). At pH 0.82 (150 mM acid), significant peak fronting and loss of efficiency is observed especially for lithium and magnesium. Samples of lower pH can be pre-treated before injection with an OnGuard II A cartridge in the bicarbonate form. Anions in the sample will be exchanged for the bicarbonate in the OnGuard resin. The bicarbonate ions neutralize the hydronium ion in the sample.

Column:	IonPac CS16 5-mm Analytical Column (no guard)
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

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

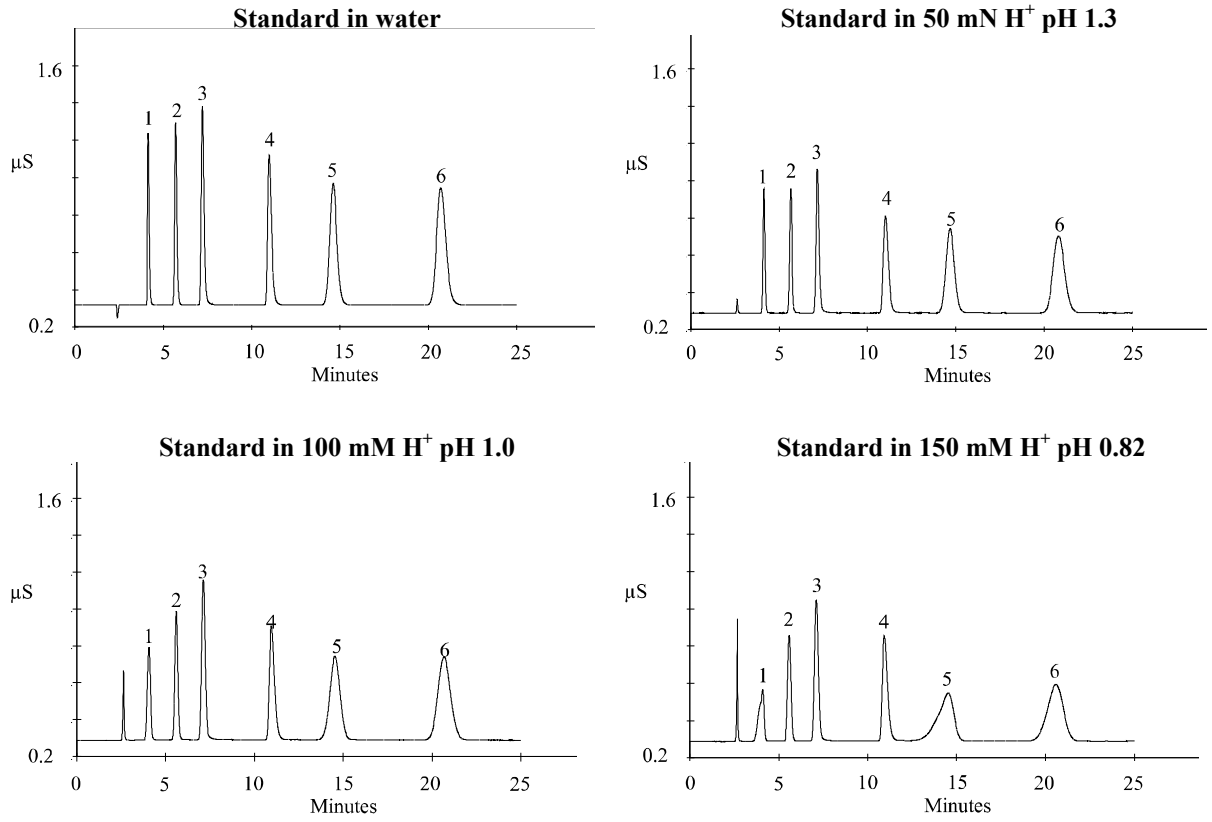


Figure 25
Effect of Sample pH

5.17. Linear Working Range at 40 °C

The higher cation exchange capacity of the CS16 5-mm stationary phase reduces overload with sample concentration. The chromatogram shown in this slide was obtained under standard conditions for this stationary phase (30 mM Methanesulfonic acid, 1 mL/min, 40 °C). The highest level standard injected with a 25 µL sample loop contained 50 ppm lithium, 200 ppm sodium, 250 ppm ammonium, 500 ppm potassium, 250 ppm magnesium and 500 ppm calcium. At these concentration levels, tailing of the late eluting peaks, potassium, magnesium and calcium is observed, indicating column overloading. Plots of peak areas versus concentration give very good correlation coefficients (> 0.9999) as shown here for calcium and potassium even when including the highest calibration point. The large resolution among all peaks permits quantitation even when efficiency/symmetry are poor due to overloading. Quantification using peak areas is possible from low ppb to 500 ppm concentration.

Column:	IonPac CG16 5-mm Guard Column + CS16 5-mm Analytical Column
Sample Volume:	25 µL Loop
Eluent:	30 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min
Temperature:	40 °C
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:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

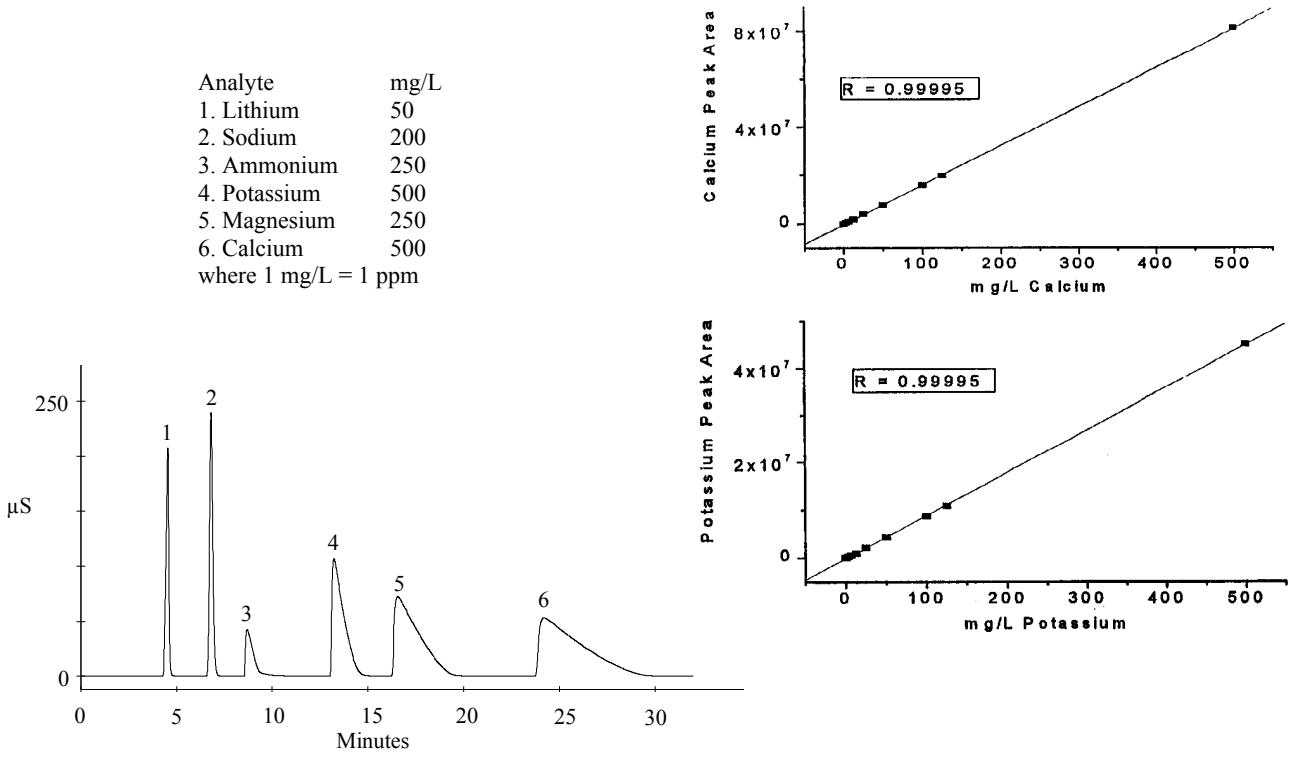
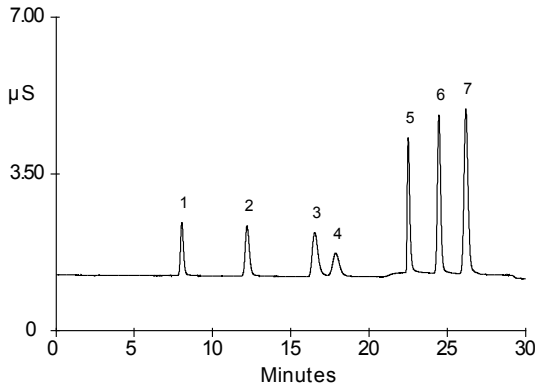


Figure 26
Potassium and Calcium Peak Area Plots and Overload Chromatogram

5.18. Step Change Elution of Ethanolamine and the Common Cations on the IonPac CS16 3-mm

Ethanolamine is a commonly used corrosion inhibitor in the Power Industry. By using a weaker acidic eluent resolution is improved between ammonium and ethanolamine (peaks #3, 4) so that diverse concentration ratios of these two can be determined using the CS16 column.

Column:	IonPac CS16 3-mm Analytical Column
Trap Column:	IonPac CTC (2-mm) (P/N 043132) between pump and injection valve
Sample Volume:	25 µL Loop
Eluent:	10.5 mM Methanesulfonic acid (MSA) step to 56 mM MSA at 17 min
Eluent Flow Rate:	0.36 mL/min
Temperature:	40 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode CTC-1 (P/N 040192) between CELL OUT and REGEN IN ports
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



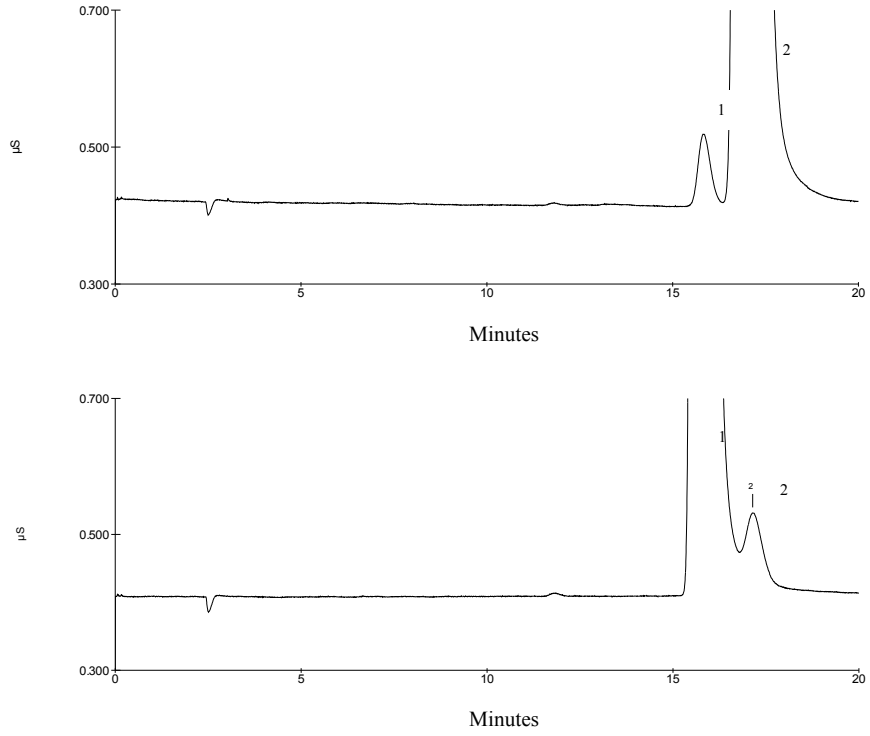
Analyte	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Ethanolamine	1.0
5. Potassium	1.0
6. Magnesium	0.5
7. Calcium	1.0

where 1 mg/L = 1 ppm

Figure 27
Step Change Elution of Ethanolamine

5.19. Diverse Concentration Ratios of Ammonium to Ethanolamine on the IonPac CS16 3-mm at 40 °C

Column:	IonPac CS16 3-mm Analytical Column
Trap Column:	IonPac CTC (2-mm) (P/N 043132) located between pump and injection valve
Sample Volume:	25 µL Loop
Eluent:	10.5 mM Methanesulfonic acid (MSA) step to 56 mM MSA at 17 min
Eluent Flow Rate:	0.36 mL/min
Temperature:	40 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode CTC-1 (P/N 040192) between CELL OUT and REGEN IN ports
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent



A

Analyte	µg/L
1. Ammonium	40
2. Ethanolamine	5000

B

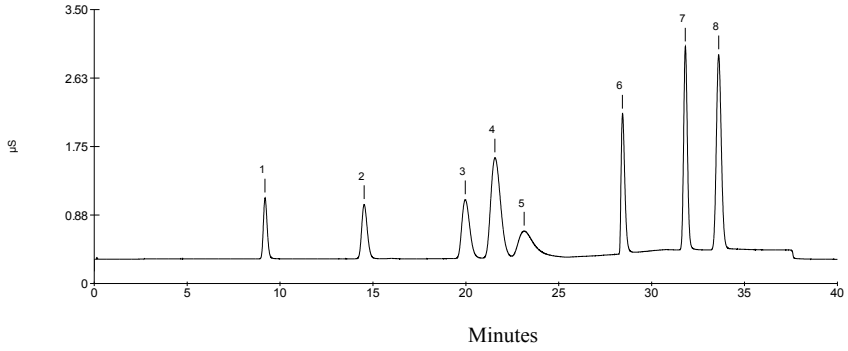
Analyte	µg/L
1. Ammonium	5000
2. Ethanolamine	50

Figure 28
Diverse Concentration Ratios of Ammonium and Ethanolamine

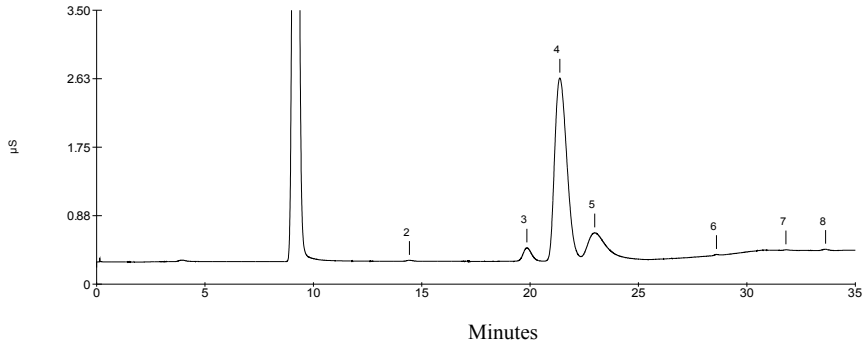
5.20. Gradient Elution of Ethanolamine, Hydrazine, and the Common Cations

Ethanolamine and hydrazine are common additives in Power Plant waters and these together with ammonium, sodium and the other cations need to be carefully monitored. The CS16 3-mm uses lower eluent flow rate than the CS16 5-mm and therefore is better suited for process applications. Ratios of 20,000:1 of sodium to ammonium can be separated on the CS16 3-mm using a MSA gradient.

Column:	IonPac CS16 3-mm Analytical Column
Sample Volume:	25 µL Loop
Eluent:	7 mM MSA, gradient to 52 mM MSA from 23 to 28 min, step to 7 mM MSA at 35 min.
Eluent Source:	EG40
Eluent Flow Rate:	0.43 mL/min
Temperature:	40 °C
SRS Suppressor:	Cation Self-Regenerating Suppressor 300 (2-mm) in AutoSuppression Recycle Mode CTC-1 (P/N 043132) between CELL OUT and REGEN IN ports
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS 300 (2-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 2 µS
Storage Solution:	Eluent

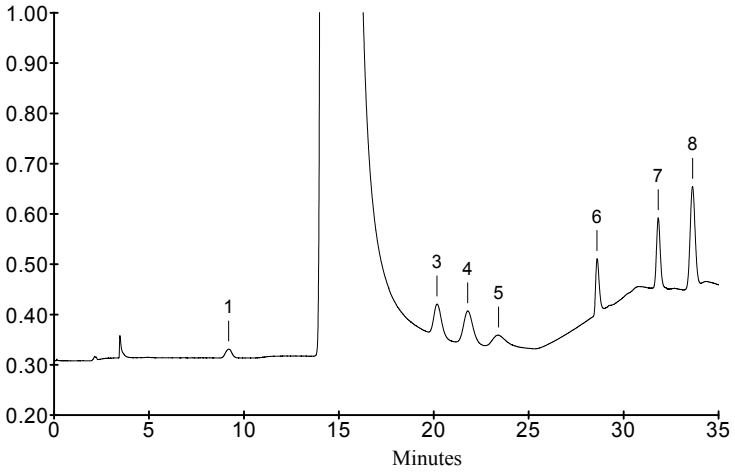


Analyte	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Ethanolamine	1.0
5. Hydrazine	4.0
6. Potassium	1.0
7. Magnesium	0.5
8. Calcium	1.0



Analyte	µg/L
1. Lithium	2500
2. Sodium	6
3. Ammonium	100
4. Ethanolamine	2000
5. Hydrazine	4000
6. Potassium	4
7. Magnesium	1
8. Calcium	6

Figure 29
Gradient Elution of Ethanolamine, Hydrazine, and the Common Cations



Analyte	µg/L
1. Lithium	5
2. Sodium	500,000
3. Ammonium	25
4. Ethanolamine	50
5. Hydrazine	200
6. Potassium	50
7. Magnesium	25
8. Calcium	50

* With this same gradient condition, 20,000:1 concentration ratio of Na^+ to NH_4^+ (ammonium) can be determined

Figure 29 Continued
Gradient Elution of Ethanolamine, Hydrazine, and the Common Cations

5.21. Gradient Elution of Common Cations and Amines at 60 °C

Here a gradient of methanesulfonic acid from 6 mM to 60 mM was done with an Eluent Generator, EG40, on the 3x250 mm high capacity weak carboxylic acid column at 60 °C. The analytes eluted are the common cations, lithium, sodium, ammonium, potassium, magnesium and calcium, and are all well separated from the amines. Among the amines separated are methylamine, dimethylamine and trimethylamine, and the more hydrophilic hydroxylamines ethanolamine, diethanolamine, and triethanolamine. Besides ethanolamine, other power waters corrosion-prevention additives shown here are 5-amino-1-pentanol (shown under these conditions partially coeluting with triethanolamine), morpholine and 2-diethylaminoethanol (last eluting peak). The eluent contained no organic solvent, so the suppressor was used in the eluent recycle mode. The suppressor was placed outside the chromatographic oven as the temperature exceeded 40 °C.

Column	IonPac CS16 3-mm Analytical Column (no guard)
Eluent	6 mM MSA, gradient from 0 to 13 min to 8 mM MSA, gradient from 13 to 25 min to 55 mM MSA, gradient from 25 to 30 min to 60 mM MSA
Eluent Source:	EG40
Flow Rate:	0.5 mL/min
Inj. Volume:	25 µL
Detection:	Suppressed conductivity CSRS® 300 2-mm outside oven AutoSuppression® Recycle Mode

Analyte	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.42
4. Ethanolamine	0.50
5. Methylamine	0.50
6. Diethanolamine	1.00
7. Potassium	0.50
8. Dimethylamine	1.00
9. Triethanolamine	18.00
10. 5-Amino-1-pentanol	2.00
11. Morpholine	2.00
12. Trimethylamine	1.50
13. Magnesium	0.25
14. Calcium	0.50
15. 2-Diethylaminoethanol	2.00

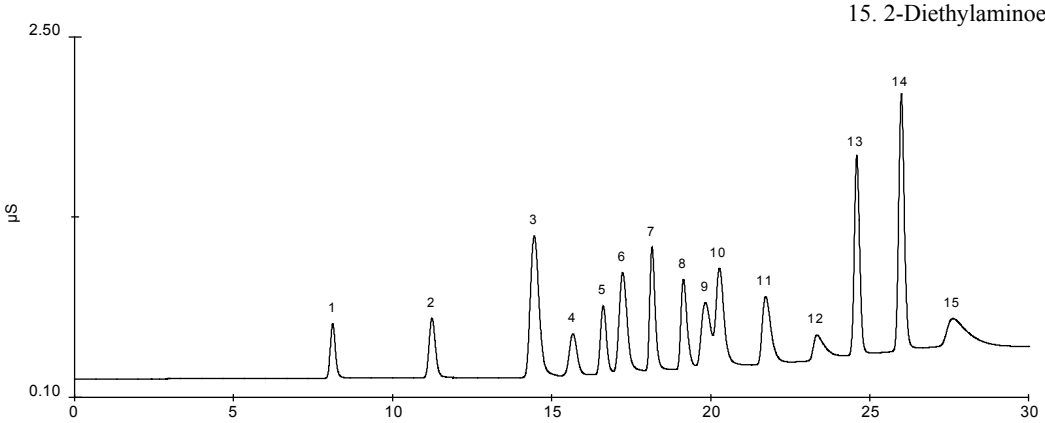


Figure 30
Gradient Elution of Common Cations and Amines at 60 °C

5.22. Gradient Elution of Common Cations and Alkanolamines at 60 °C

This is a methanesulfonic acid gradient from 6 mM to 70 mM, at 0.5 mL/min flow rate, done with an Eluent Generator, EG40, on the high capacity CS16 cation exchanger at 60 °C. The analytes are the common cations, sodium, ammonium, magnesium and calcium as well as different monoamines containing alkanol groups, which makes them more hydrophilic. Under these conditions, sodium partially coelutes with peak #2, 2-Amino-2-(hydroxymethyl)-1,3-propanediol, and potassium coelutes with peak # 7, 2-methylaminoethanol. No organic solvent was used in the eluent, and temperature and the acid concentration gradient is used to improve the elution of the larger alkanolamines. The suppressor can be used in the eluent recycle mode as there is no organic solvent used, however, the suppressor must be placed outside the chromatographic oven when temperatures exceed 40 °C as they do here.

Column:	IonPac CS16 3-mm Analytical Column (no guard)
Eluent:	6 mM MSA, gradient from 0 to 10 min to 9 mM MSA, gradient from 10 to 30 min to 70 mM MSA
Eluent Source:	EG40
Flow Rate:	0.5 mL/min
Inj. Volume:	25 µL
Detection:	Suppressed conductivity CSRS 300 2-mm outside oven AutoSuppression Recycle Mode

Analyte	**mg/L**
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.42
4. Ethanolamine	0.50
5. Methylamine	0.50
6. Diethanolamine	1.00
7. Potassium	0.50
8. Dimethylamine	1.00
9. Triethanolamine	18.00
10. 5-Amino-1-pentanol	2.00
11. Morpholine	2.00
12. Trimethylamine	1.50
13. Magnesium	0.25
14. Calcium	0.50
15. 2-Diethylaminoethanol	2.00

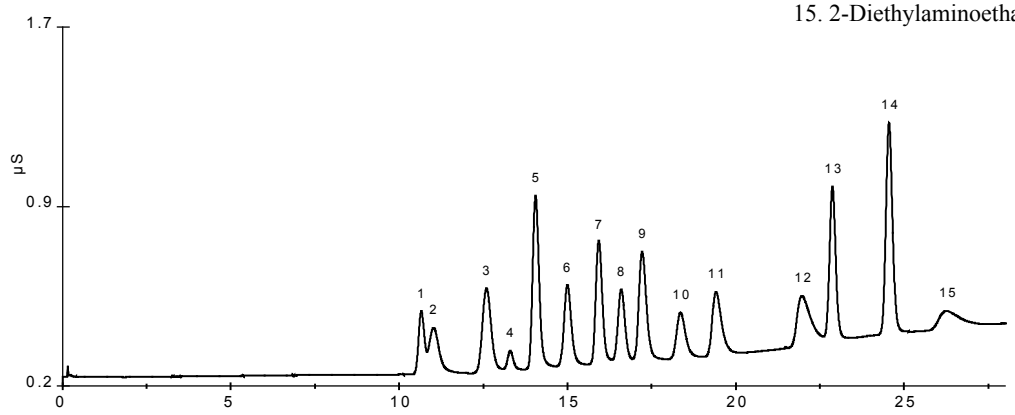


Figure 31
Gradient Elution of Common Cations and Amines at 60 °C

5.23. Separation of Six Cations on a Capillary CS16 column

Column:	IonPac® CS16 (0.5 mm x 250 mm)
Eluent Source:	Capillary EGC-MSA cartridge
Eluent:	30 mM MSA
Flow Rate:	10 µL/min
Temperature:	40 °C
Suppressor:	CCES 300 Cation Capillary Electrolytic Suppressor
Detection:	Suppressed conductivity
Injection Volume:	0.4 µL

Peak
1. Lithium
2. Sodium
3. Ammonium
4. Potassium
5. Magnesium
6. Calcium

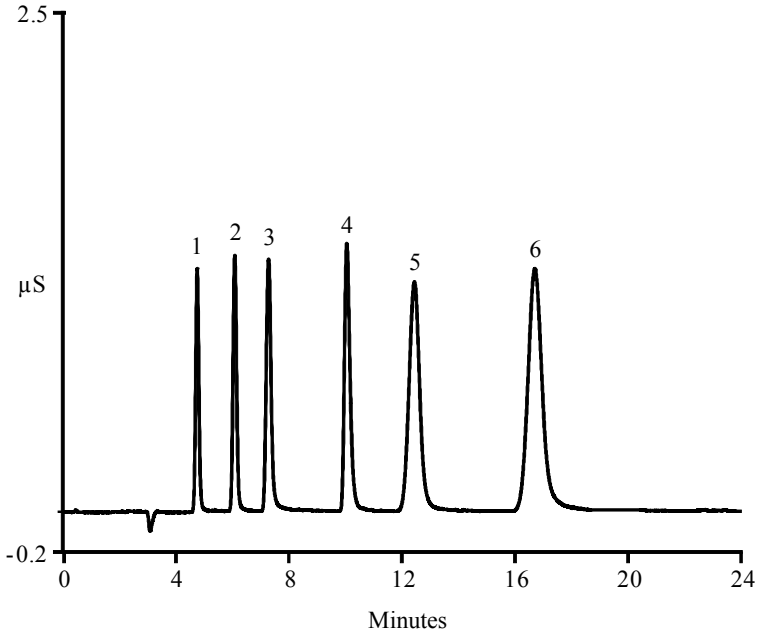


Figure 32
Separation of Six Cations on a Capillary CS16 column

SECTION 6 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac CS16 columns. For more information on problems that originate with the Ion Chromatograph (IC) or suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact 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 8
CS16/CG16 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown component Plugged column bed supports Plugged system hardware	Isolate blockage Replace bed supports Unplug, Replace	6.1.1 6.1.2 Component manual
High Background Conductivity and/or High Noise			
Improper Suppressor Operation	CSRS, CCES 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
	Air bubble trapped in CSRS or CAES	Remove bubble by loosening fittings	6.4
Contamination	Bad eluents	Remake eluents	6.2, 6.4
	Contaminated column	Clean column	6.3.1, 7.4
	Contaminated suppressor	Clean suppressor	6.3.1 A, 6.5, Component manual
Hardware Operation	Proportioning valve	Service valve	Component manual
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
	Contaminated or deformed bed support	Replace bed support	6.1.2
	Column headspace	Replace column	6.6.1 A
	Column overloading	Reduce sample size	5.18
	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	5.17
Fronting Peaks	Low sample pH	Reduce sample size, Dilute Sample, Use OnGuard II A	5.17
	Column overload	Reduce sample size	5.18
	Contaminated or deformed bed support	Replace bed support	6.1.2
	Column headspace	Replace column	6.6.1 A
Tailing Peaks	Contaminated suppressor	Clean suppressor	6.3.1 A, 6.5, Component Manual
	Column overloading	Reduce sample size	5.18
Short Retention	Flow rate too fast	Recalibrate pump	6.6.2 A
Times	First peaks elute too fast	Equilibrate to first eluent	6.6.3 A
	Bad eluents	Remake eluents	6.6.2 B
	Column contamination	Clean column	6.62 C
Spurious Peaks	Column contamination	Pretreat samples	6.3.1, 6.7 A, 6.7 B
	Sluggish injection valve	Service valve	6.7 C, Component Manual
Poor Qualifications of Divalents	Sample loop contamination	Flush, replace	6.3.2

6.1. High Back Pressure

6.1.1. Finding the Source of High System Pressure

Total system pressure for the IonPac CG16 Guard Column plus the CS16 Analytical Column when using the test chromatogram conditions should be equal or less than 1,850 psi (12.75 MPa). If the system pressure is higher than 1,950 psi (13.44 MPa), 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. The CTC-1, if required, should be installed after the High Pressure In-Line Filter.

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, 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 9, "Typical CS16/CG16 Operating Back Pressures").

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

Table 9
Typical CS16/CG16 Operating Back Pressures

Column	Typical Back Pressure Psi (MPa)	Flow Rate mL/min
CS16 5-mm Analytical	≤ 1,400 (9.65)	1.0
CG16 5-mm Guard	≤ 450 (3.10)	1.0
CS16 + CG16 5-mm columns	≤ 1,850 (12.75)	1.0
CS16 3-mm Analytical	≤ 1,400 (9.65)	0.36
CG16 3-mm Guard	≤ 450 (3.10)	0.36
CS16 + CG16 3-mm columns	≤ 1,850 (12.75)	0.36
CS16 0.5-mm Capillary	≤ 1,400 (9.65)	0.01
CG16 0.5-mm Capillary Guard	≤ 450 (3.10)	0.01
CS16 + CG16 0.5-mm columns	≤ 1,850 (12.75)	0.01

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

A. Disconnect the column from the system.

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

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

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

Part	IonPac CS16 5-mm Columns (P/N)	IonPac CS16 3-mm Columns (P/N)	IonPac CS16 0.5-mm Columns (P/N)
Analytical or Capillary Column	057573	059596	075401
Guard Column	057574	059595	075402
Bed Support Assembly	042955	056823	N/A
End Fitting	052809	052809	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.***

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/Capillary Column

Determine if the column is contaminated. Column contamination can lead to a loss of column capacity since all of the cation exchange sites will no longer be available for the sample ions. Polyvalent cations may be concentrating on the column over a series of runs. Remove the IonPac CG16 Guard and CS16 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 CG16 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 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

A. **Check for a contaminated Gradient Mixer.** Gradient Mixers in the Gradient Pump Module should be flushed thoroughly to remove 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 used in older systems are a potential source for metal contamination of the column. The new Dionex ThermoFlare or PEEK ferrule fittings are preferred. Inspect the eluent pumps periodically for any signs of leakage.

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

E. **For EG operation, use a CR-CTC Trap Column.** Install a CR-CTC Cation Trap Column (P/N 060478) 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 applications. 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 and 3-mm systems.

6.3.2. Sample Loop and/or Tubing Contamination

Eluents made with deionized water that is contaminated with bacteria and samples such as humic acids and soil extracts can potentially contaminate eluent lines and sample loops. Weak cation exchange sites are created on (or attached to) the tubing. This can happen to either Tefzel or PEEK tubing. Thus, the sample loop itself can act as a concentrator and depending on the pH of the sample or the standard and the way 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, CS14, CS15, 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 (pH 5), the weak cation exchange sites on the contaminated tubing are ionized and divalent cations are preferentially retained. When the sample pH is low (< pH 4), these sites are protonated by the sample and rendered inactive, so that the divalent quantification is not affected.

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

A simple test can be performed (when using a column such as the IonPac CS16 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 µS with a CSRS 300 (4-mm). If the background is low but the system is noisy, an air bubble may be trapped in the system. With the system running, disconnect the **ELUENT IN** line from the suppressor and apply pressure to the open port with your gloved finger to dislodge a suspected bubble. Reconnect the line. Do not take too long to do this, as the current is still being applied to the CSRS 300 and the eluent flow is needed to produce regenerant.

Check the conductivity flow cell for bubbles. See the conductivity detector manual for details.

A system with a high background (> 3 µS) 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 Cation Self-Regenerating Suppressor, CSRS 300, Cation Capillary Electrolytic Suppressor, CCES 300, or Cation MicroMembrane Suppressor, CMMS 300, that Does Not Suppress Properly."

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

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
22 mN H_2SO_4 or 20 mN Methanesulfonic acid	< 1 µS
50 mN H_2SO_4 or Methanesulfonic acid	< 2 µS

6.5. Suppressor Not Suppressing Properly

If the Cation Self-Regenerating Suppressor, Cation Capillary Electrolytic Suppressor, Cation Atlas Electrolytic Suppressor, or the Cation MicroMembrane Suppressor is causing the problem, refer to the Cation Self-Regenerating Suppressor Product Manual (Document No. 031139), the Cation Atlas Electrolytic Suppressor Product Manual (Document No. 031770), the Cation MicroMembrane Suppressor Product Manual (Document No. 034359), or 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 correct back pressure tubing is properly installed after 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 5-mm operation or 0.36 mL/min for 3-mm operation or 0.10 mL/min for 0.5-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 approximately 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. 031139) or to the Cation MicroMembrane Suppressor Product Manual (Document No. 034359), or the Cation Capillary Electrolytic Suppressor Product Manual (Document No. 065386) 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.

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

NOTE

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, causing loss of peak efficiencies.** Make sure you are using PEEK tubing with an i.d. of no greater than 0.010" for 5-mm systems or 0.005" for 3-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 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 or improperly swept out of void volumes are usually the cause of poor resolution or efficiency of peaks eluting near the system void volume compared to the later eluting peaks.

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

B. **Sluggish operation of the injection valve may be the problem.** Check the air pressure and make sure there are no gas leaks or partially plugged port faces. Refer to the valve manual for instructions.

C. **Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem.** Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change.

6.7. Spurious Peaks

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

6.7.1. Poor Efficiency Using Capillary Columns

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

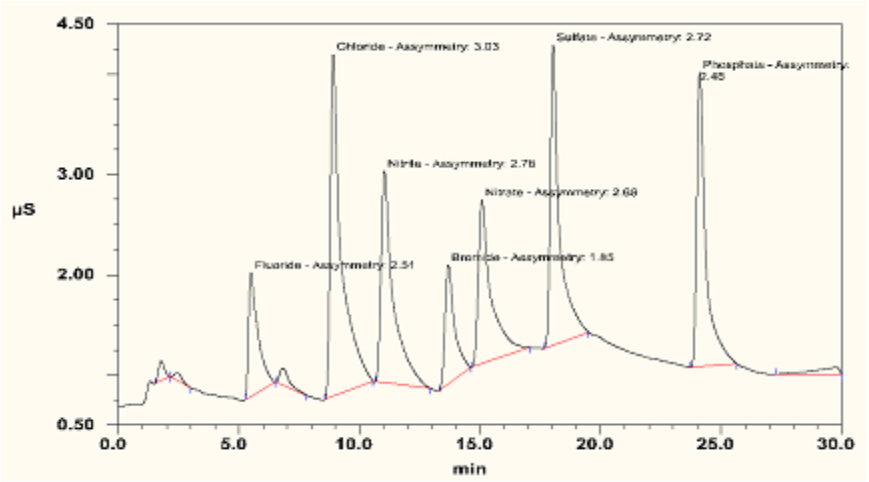


Figure 33
Tailing Peaks Caused by Incorrectly Installed Capillary Tubing Fittings

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 33 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

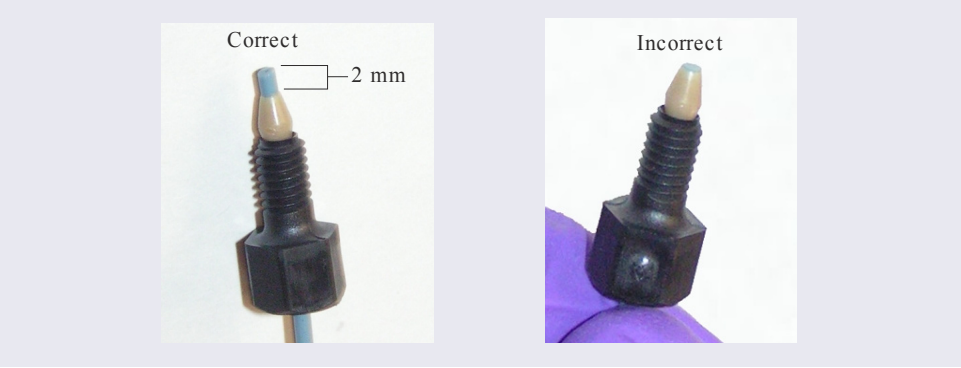


Figure 34
Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

APPENDIX A - QUALITY ASSURANCE REPORT

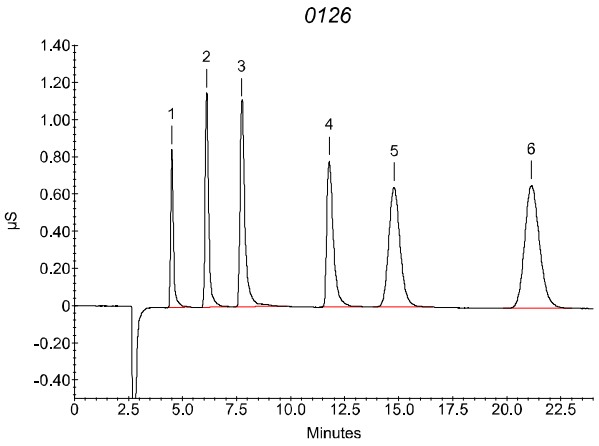
Quality Assurance Report - IonPac CS16 Analytical Column - 3 x 250 mm

Quality Assurance Report - IonPac CS16 Analytical Column - 5 x 250 mm

Quality Assurance Report - IonPac CS16 Capillary Column – 0.5 x 250 mm

IonPac® CS16
Analytical (3 x 250 mm)
Product No. 059596

Serial No. : 0126 Pressure (PSI) : 820 Date : 5/13/02 11:45:16 AM



Eluent: 30 mM MSA (Methanesulfonic Acid)

Eluent Flow Rate: 0.36 mL/min

Operating Temperature: 40°C

Detection: Suppressed Conductivity CSRS® 300 2 mm, AutoSuppression® Recycle Mode, 100 mA

Background Conductivity: < 2 µS

Injection Volume: 25 µL

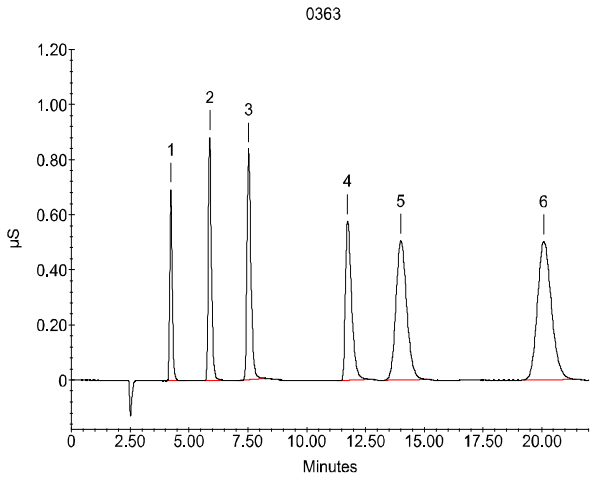
Storage Solution: Eluent (30 mM MSA) Methanesulfonic

Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	4.50	Lithium	0.05	6812	1.8	6.47
2	6.11	Sodium	0.20	7593	1.7	4.94
3	7.73	Ammonium	0.25	6643	2.3	8.99
4	11.78	Potassium	0.50	8091	2.1	4.07
5	14.78	Magnesium	0.25	3838	1.3	5.75
6	21.13	Calcium	0.50	4498	1.3	n/a

IonPac® CS16
Analytical (5 x 250 mm)
Product No. 057573

Serial No. : 0363 Pressure (PSI) : 912 Date : 7/16/01 3:42:08 PM



Eluent: 30 mM MSA
(Methanesulfonic Acid)

Eluent Flow Rate: 1.0 mL/min

Operating Temperature: 40°C

Detection: Suppressed Conductivity
CSRS® 300 4 mm,
AutoSuppression® Recycle Mode,
100 mA

Background Conductivity: < 2 µS

Injection Volume: 25 µL

Storage Solution: Eluent (30 mM MSA)
Methanesulfonic

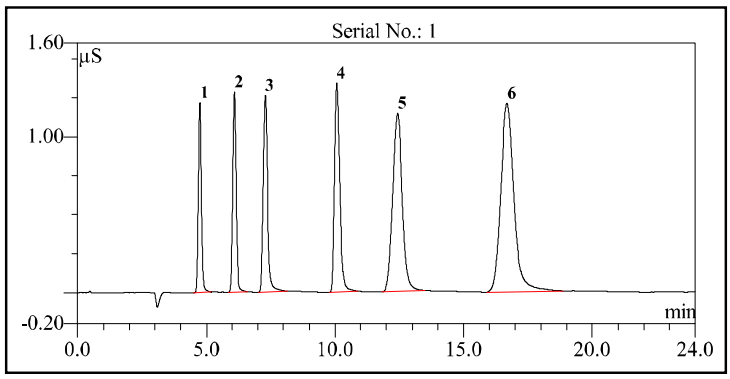
Peak Information : Found Components

Peak No.	Retention Time	Name	(mg/L)	Efficiency	Asymmetry (10%)	Resolution
1	4.23	Lithium	0.1	8205	1.5	7.79
2	5.88	Sodium	0.4	9784	1.5	6.08
3	7.53	Ammonium	0.5	9704	1.7	10.94
4	11.75	Potassium	1.0	10114	2.1	3.48
5	14.00	Magnesium	0.5	4553	1.4	6.39
6	20.08	Calcium	1.0	5542	1.3	n/a

IonPac® CS16
Capillary (0.5 x 250 mm)
Product No. 075401

Date: 22-Oct-10 09:59
Serial No. : 000001
Lot No. :

Eluent: 30 mM Methanesulfonic acid
Eluent Flow Rate: 0.010 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Cation Capillary Electrolytic Suppressor (CCES 300)
AutoSuppression® Recycle Mode
Applied Current: 8 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	4.75	1.1	6.11	8693	0.1
2	Sodium	6.08	1.1	4.43	10714	0.4
3	Ammonium	7.28	1.2	8.31	8954	0.5
4	Potassium	10.06	1.6	4.58	12358	1.0
5	Magnesium	12.43	1.1	5.47	5433	0.5
6	Calcium	16.68	1.2	n.a.	5681	1.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=3600	Passed
Calcium	Retention Time	18.35-21.65	*****
Potassium	Efficiency	>=6300	Passed
Potassium	Asymmetry	1.1-3.0	Passed
Sodium	Asymmetry	1.1-1.9	Passed
Ammonium - Sodium	Retention Time difference	>=1.08	Passed
	Pressure	<=1540	525

Production Reference:
Datasource: Column
Directory: Capillary\Cap_Cation
Sequence: CS16_p5x250mm_09-08-10
Sample No.: 13

6.80 SR9a Build 2680 (163077) (Demo-Installation)

Chromeleon® Dionex Corp. 1994-2010

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 CS16 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 CS16 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. Disconnet 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 with 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 steps to reduce the solvent content of the eluent to < 5% levels and the ionic strength of the eluent to < 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing. This intermediate low concentration step will prevent precipitation or high viscosity zones. Avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

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

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

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

B. **Disconnect the Suppressor from the IonPac CS16 Analytical or Capillary Column.** If your system is configured with both a guard column and an analytical 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.

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

DO NOT pump hydrochloric acid through the CSRS 300

C. Set **the pump flow rate** to 1.0 mL/min for a CS16 5-mm Analytical or Guard Column and to 0.36 mL/min for a CS16 3-mm Analytical or Guard Column. Set the pump flow rate to 0.010 mL/min for a CS16 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** for at least 5–10 minutes before resuming normal operation (send effluent to waste).

H. **Reconnect the Suppressor to the CS16 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.

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

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

II. Hydrophobic Cations and Organic Contaminants

A. Disconnect the analytical/capillary column from the injection valve and the suppressor. Disconnect the Gradient Mixer or the Cation Trap Column (CTC-1) from the pump. Connect the IonPac CS16 Analytical/Capillary Column directly to the pump. Direct the effluent from the analytical column directly to a waste container.

B. Set the flow rate to 1 mL/min for a CS16 5-mm Analytical or Guard Column and to 0.36 mL/min for a CS16 3-mm Analytical or Guard Column. Set the pump flow rate to 0.010 mL/min for a CS16 Capillary or Capillary Guard Column.

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

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

Time (min)	**% E1**	**% E2**
0.0	100	0
20.0	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** for at least 30 minutes before resuming normal operation.

F. **Reconnect the IonPac CS16.** Connect the Analytical/Capillary Column outlet to the Suppressor and the inlet to either the IonPac CG16 Guard Column or the Pump Module.

G. Equilibrate the column with eluent before resuming normal operation.

