PRODUCT MANUAL

for

IonPac® CS15

Now sold under the
Thermo Scientific brand

Thermo
SCIENTIFIC

DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

IONPAC® CG15 GUARD COLUMN

(2 x 50 mm, P/N 052256)

(4 x 50 mm, P/N 052200)

IONPAC® CS15 ANALYTICAL COLUMN

(2 x 250 mm, P/N 052252)

(4 x 250 mm, P/N 051795)

Document No. 031272
Rev. 06
9 April 2009

TABLE OF CONTENTS

SECTION 1 - INTRODUCTION

The IonPac® CS15 4-mm (P/N 051795) and 2-mm (P/N 052252) Analytical Columns are designed specifically for the analysis of alkali metals, alkaline earth metals, and ammonium at extreme concentration ratios. The CS15 and CG15 stationary phase is functionalized with a crown ether and relatively weak phosphonic and carboxylic acids having a high selectivity for hydronium ion. The crown ether functionality has a high selectivity for potassium and ammonium which retains potassium at the end of the run and greatly improves the resolution between sodium and ammonium. The weak carboxylate functional groups require low ionic strength eluents to isocratically elute both monovalent and divalent cations in a relatively short period of time. The CS15 is solvent-compatible with 100% acetonitrile, 20% tetrahydrofuran or 100% aqueous eluents 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.
Do NOT use the IonPac CS15 column with basic eluents

CAUTION

The IonPac CS15 should be used with a CSRS ULTRA P/N 053948 (4-mm), 053949 (2-mm) when operated with eluents containing organic solvents or when operating at elevated temperatures (40 °C)

Table 1
IonPac CS15/CG15 Packing Specifications

Column	Particle Diameter µm	Substrate[a] X-linking %	Column Capacity meq/column	Functional Group	Hydrophobicity
CS15 4 x 250 mm	8.5	55	2.8	Crown Ether/ Carboxylic/ Phosphonic acid	Medium to low
CG15 4 x 50 mm	8.5	55	0.56	Crown ether/ Carboxylic/ Phosphonic acid	Medium to low
CS15 2 x 250 mm	8.5	55	0.7	Crown ether/ Carboxylic/ Phosphonic acid	Medium to low
CG15 2 x 50 mm	8.5	55	0.14	Crown ether/ Carboxylic/ Phosphonic acid	Medium to low

[a] macroporous divinylbenzene/ethylvinylbenzene polymer

Table 2
CS15/CG15 Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS15 4-mm Analytical	≤ 1,350 (9.31)	1.2	3.0
CG15 4-mm Guard	≤ 450 (3.10)	1.2	3.0
CS15 + CG15 4-mm columns	**≤ 1,800 (12.41)**	**1.2**	**3.0**
CS15 2-mm Analytical	≤ 1,350 (9.31)	0.3	0.75
CG15 2-mm Guard	≤ 450 (3.10)	0.3	0.75
CS15 + CG15 2-mm columns	**≤ 1,800 (12.41)**	**0.3**	**0.75**

Read the system manuals. This manual assumes that you are familiar with the installation and operation of the Dionex Ion Chromatograph (IC). If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis.

You may need to make a liquid line fitting. The IonPac CS15 Analytical Column and the IonPac CG15 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 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

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

CONFIGURATION	2-mm	4-mm
Eluent Flow Rate	0.30 mL/min	1.2 mL/min
SRS Suppressor	CSRS-ULTRA (2-mm) (P/N 053949)	CSRS-ULTRA (4-mm) (P/N 053948)
MMS Suppressor	CMMS III (2-mm) (P/N 056753) Operation without solvents	CMMS III (4-mm) (P/N 056752) Operation without solvents
NOTE Do not run suppressors over 40 °C. If application requires a higher temperature, place suppressor outside of chromatographic oven.		
Regenerant Flow Rate	See suppressor manual.	See suppressor manual.
Injection Loop	2 - 15 µL Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.	10-50 µL
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (2-mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2 , GM-3 or recommended gradient mixers.
Pumps	Use the GS50/GP50/GP40/IP20 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. No External Gradient Mixer is required for GS50/GP50/GP40 Pump when performing gradient analysis The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.	Use the GP40/GP50/IP20/IP25 in Standard-Bore Configuration. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP40/GP50/IP20/IP25 and the DX-300 HPLC Pump.. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.

CONFIGURATION	2-mm	4-mm
Detectors	AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL, P/N 043120) CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield, P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. Ensure 30–40 psi back pressure after the cell.	AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) VDM-2 Cell (6-mm, 10 µL) P/N 043113 CD20, CD25, CD25A, ED40, ED50 or ED50A Conductivity Cell with DS3 P/N 044130 or with shield, P/N 044132 CDM-2/CDM-3 Cell P/N 042770 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 ED40/ED50/ED50A or the CD20/CD25/CD25A. Ensure 30–40 psi back pressure after the cell.

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/cm at 1 mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 2-mm Operation

The IonPac CS15 mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a gradient pump configured for narrow bore operation.

3.1.2 System Requirements for 4-mm Operation

The IonPac CS15 4-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a system having a gradient pump configured for standard bore operation.

3.1.3 System Void Volume

When using 2-mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4-mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" i.d. PEEK tubing (P/N 044221), 0.010" i.d. PEEK tubing (P/N 042260), or 0.012" Tefzel tubing (see, "Dionex Product Selection Guide") may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers. If you need assistance in properly configuring your system contact the Dionex Office nearest you (see, "Dionex Worldwide Offices").

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

For IonPac CS15 applications using solvents the EG40 or EG50 with EGC II MSA cartridge, should not be used. See the CR-TC Product Manual (Document No. 031910) for instructions. The CTC-1 Trap Column (P/N 040192) should be used for gradient work. The CTC-1 Trap Column will require off-line regeneration. To use the CTC Cation Trap Column, see Section 3.3.

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

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

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

A. Remove the Gradient Mixer. It is installed between the gradient pump pressure transducer and the injection valve.

B. Connect the gradient pump directly to the CTC. Connect a waste line to the CTC outlet and direct the line to a waste container.

C. Flush the CTC. Use 200 mL of a 10X eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min. Note that with the guard and analytical columns out of line, there is no need for 2-mm flow rate restrictions.

D. Rinse the CTC. Use the strongest eluent that will be used during the gradient analysis.

E. Reconnect the CTC. Connect the CTC to the eluent line that is connected to the injection valve.

The background conductivity of your system should be less than 3 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, the CTC should be cleaned using steps A–E above.

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

A. Disconnect the CTC. It should be installed between the injection valve

B. Direct the outlet of the CTC to a separate waste container.

C. Flush the CTC. Use 30 mL of a 10X eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min.

D. Flush the CTC prior to start-up. Prior to the use of the chromatographic system on the next day, flush the CTC with 30 mL of the strongest eluent used in the gradient program. Reconnect CTC to the eluent line.

3.4 The Injection Loop

Table 4
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
Rheodyne Microinjection Valve Model 9126 (0.8 µL Internal Volume) (10 cm Loop)	8.0	3.3	5.9	2.0

3.4.1 The 2-mm System Injection Loop, 2–15 µL

For most applications on a 2-mm analytical system, a 2–15 µL injection loop is sufficient. Dionex recommends that a 2.5 µL injection loop be used to avoid overloading the CS15 2-mm Analytical Column. Generally, you should not inject more than 2.5 nanomoles (100–200 ppm) of any one analyte onto a 2-mm analytical column. Depending on sample concentration, injecting larger volumes of samples may result in overloading the column which can affect the detection linearity. The CS15 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4-mm analytical system (Section 2, “Comparison of Ion Chromatography Systems”).

3.4.2 The 4-mm System Injection Loop, 10–50 µL

For most applications on a 4-mm 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 CS15 4-mm Analytical Column. Generally, do not inject more than 10 nanomoles (100–200 ppm) of any one analyte onto the 4-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.

3.5 Sample Concentration

The IonPac CG15 Guard Column or the Low-Pressure Trace Cation Concentrator, TCC-LP1, should be used for trace cation concentrator. Trace cation concentrators are used primarily in high purity water analysis. The function of 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 CG15) leading to a lowering of detection limits by 2–5 orders of magnitude.

The IonPac CG15 2-mm Guard Column (P/N 052256) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 0496027) must be used for sample concentration with the IonPac CS15 2-mm Analytical Column.

The IonPac CG15 4-mm Guard Column (P/N 052200) or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 0496027) should be used for sample concentration with the IonPac CS15 4-mm Analytical Column.

CAUTION

The Trace Cation Concentrator (TCC-2, P/N 043103) should NOT be used for sample concentration with the IonPac CS15. The TCC-2 column packing is functionalized with a strong cation exchange resin and the recommended IonPac CS15 eluents will not properly elute ions concentrated on this column.

3.6 IonPac CG15 Guard Columns

An IonPac CG15 Guard Column is normally used with the IonPac CS15 Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. For maximum life of the analytical column, the guard column should be changed or replaced as part of a regular maintenance schedule or at the first sign of performance deterioration. Use the test chromatogram that is shipped with the analytical column or the initial application run for a performance benchmark.

3.7 Eluent Storage

IonPac CS15 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 Requirements

A Cation Self-Regenerating Suppressor (CSRS® ULTRA) should be used with the IonPac CS15 for applications that require suppressed conductivity detection. The CSRS ULTRA 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 ULTRA modes of operation. The CSRS ULTRA is compatible with operation at elevated temperatures up to 40 °C.

NOTE

For dependable operation, the CSRS ULTRA P/N 053948 (4-mm), 053949 (2-mm) must be used to suppress eluents containing solvents at elevated temperatures. Solvent containing eluents should be used with the AutoSuppression External Water Mode.

If you are installing an IonPac CS15 4-mm Analytical Column, use a CSRS ULTRA (4-mm, P/N 053948).
If you are installing an IonPac CS15 2-mm Analytical Column, use a CSRS ULTRA (2-mm, P/N 053949).

For detailed information on the operation of the Cation Self-Regenerating Suppressor ULTRA, see Document No. 031370, the "Product Manual for the Cation Self-Regenerating Suppressor ULTRA, the CSRS ULTRA."

3.9 Cation MicroMembrane Suppressor Requirements

A Cation MicroMembrane Suppressor, CMMS® III, may be substituted for the CSRS ULTRA for operation without solvents and at ambient temperature. For detailed information on the operation of the Cation MicroMembrane Suppressor III, see Document No. 031728, the "Product Manual for the Cation MicroMembrane Suppressor III, the CMMS III."

If you are installing an IonPac CS15 4-mm Analytical Column, use a CMMS III (4-mm, P/N 056752).
If you are installing an IonPac CS15 2-mm Analytical Column, use a CMMS III (2-mm, P/N 056753).

3.10 Suppressor Caution

Do not run suppressors over 40 °C. If an application requires a higher temperature, place suppressor outside of chromatographic oven.

3.11 Using AutoRegen

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

CAUTION

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

When using an AutoRegen System, the regenerant passes through 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.

3.12 Detector Requirements

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

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	2-mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume 4-mm: 25 µL Loop + 0.8 µL Injection valve dead volume
Column:	2-mm: CS15 2-mm Analytical Column (+ CG15 2-mm Guard Column) 4-mm: CS15 4-mm Analytical Column (+ CG15 4-mm Guard Column)
Eluent:	10 mN H_2SO_4/ 9% Acetonitrile
Eluent Flow Rate:	2-mm: 0.3 mL/min 4-mm: 1.2 mL/min
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (2-mm or 4-mm) External Water Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (2-mm or 4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

4.2 Operating Precautions

CAUTION

IonPac CS15 Operation Precautions

Operate below 4,000 psi (27.57 MPa)
Filter and Degas Eluents
Filter Samples

Do Not use this column with alcohols

If the column has not been in use for a few days, pump eluent through the column to waste (i.e. bypass the suppressor) for 20 minutes before connecting column to the suppressor.

4.3 Chemical Purity Requirements

Obtaining reliable, consistent and accurate results requires eluents that are free of ionic impurities. Chemicals, solvents and deionized water used to prepare eluents must be of the highest purity available. Low trace impurities and low particle levels in eluents minimize contamination of 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. Bottled HPLC-Grade Water (with the exception of Burdick & Jackson) should not be used as most bottled water contains an unacceptable level of ionic impurities.

4.3.2 Inorganic Chemicals

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

A. Use only concentrated H_2SO_4, ACS grade or BAKER INSTRA-ANALYZED® for trace metals.

B. Use Fluka or Aldrich Methanesulfonic Acid (MSA) (>99% pure).

C. Use Dionex Cation Regenerant Solution, tetrabutylammonium hydroxide (TBAOH), P/N 039602, to ensure maximum system performance when operating with a CMMS III or a CSRS ULTRA in the Chemical Suppression Mode.

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

4.4 Preparation of Eluent Stock Solution Concentrates

WARNING

Sulfuric acid (H_2SO_4) is very corrosive. Methanesulfonic acid (MSA) is also a corrosive and a strong irritant.
Avoid breathing the vapors.
Always use these reagents in a fume hood. Wear gloves and goggles.

4.4.1 1.0 N (0.5M) 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 (0.5 M) solution, weigh out:

1 liter = 98.08 g/1 mole x 1 mole/ 2 Eq x 1 mole/liter x 100 g/98 g

4.4.2 1.0 N (1.0 M) Methanesulfonic Acid (MSA) Stock Solution

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

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

4.4.3 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 (0.5 M) H_2SO_4 or 1.0 N (0.5 M) MSA eluent concentrate (see Section 4.4, "Preparation of Eluent Stock Solution Concentrates") into a 1-L volumetric flask. Dilute to 1 L using deionized water with a specific resistance of 18.2 megohm-cm. Degas the eluent.

Table 6
mN Eluents from Stock Solutions

mN H_2SO_4 or MSA Stock Solution	# mL H_2SO_4 or MSA Stock Solution
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

Eluent: 10 mN Sulfuric acid 9% Acetonitrile

To a 1-liter volumetric flask, add 10 mL of the 1.0 N Sulfuric acid eluent concentrate and 90 mL of (100%) acetonitrile. Dilute to 1 L using deionized water with a specific resistance of 18.2 megohm-cm.

4.5 Eluents with Solvents

Solvents can be added to the ionic eluents used with IonPac CS15 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 generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-acetonitrile mixture varies. The practical back pressure limit for the IonPac CS15 columns is 4,000 psi (27.57 MPa).

The IonPac CS15 is compatible with the HPLC solvents listed in Table 5, "HPLC Solvents for Use with the CS15 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 5
HPLC Solvents for Use with IonPac CS15 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 ULTRA) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

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

SECTION 5 - EXAMPLE APPLICATIONS

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

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

5.1 Production Test Chromatogram

Selectivity of the IonPac CS15 Analytical Column has been optimized utilizing a sulfuric acid/acetonitrile eluent at 40 °C. Using this eluent and temperature, the weak carboxylate functionalized packing isocratically separates mono- and divalent cations in a single injection. To guarantee that all IonPac CS15 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

As the analytical column ages it is normal to see some decrease in retention time or change in operating parameters. If the analytical column is older than one year it is recommended to run the column under test chromatogram conditions and confirm that the column is meeting the QAR specifications to ensure optimum performance of the column. It is recommended to repeat this performance test once a year.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm) No Guard Column
Column Temp:	40 °C
Eluent:	10 mN H_2SO_4/9% Acetonitrile
Eluent Flow Rate:	1.2 mL/min (4-mm)
Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm) External Water Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Lithium	1.0
2.	Sodium	4.0
3.	Ammonium	10.0
4.	Magnesium	5.0
5.	Calcium	10.0
6.	Potassium	10.0

where 1 mg/L = 1 ppm

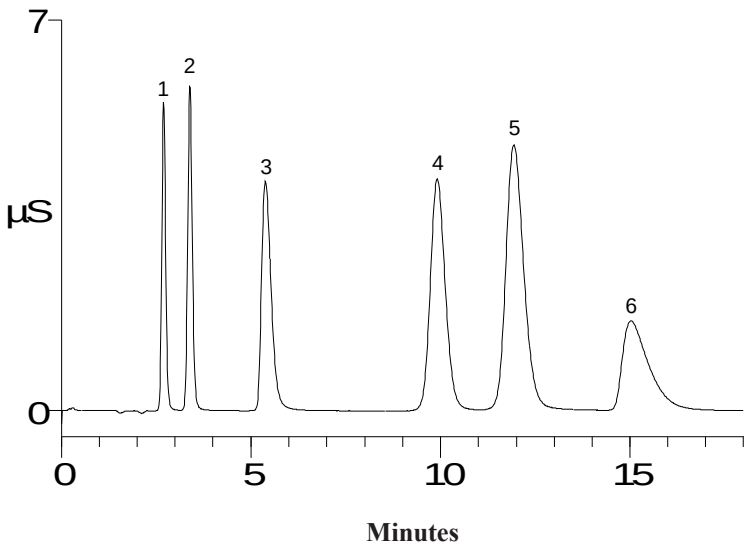


Figure 2
IonPac CS15 Production Test Chromatogram

5.1.1 Test Chromatogram Comparison

The following test chromatograms show the isocratic elution of ammonia, plus Group I and Group II cations (Li^+, Na^+, NH_4^+, Mg^{2+}, Ca^{2+}, and K^+) on the CS15 analytical column with CG15 guard column using different isocratic eluents with the column either at room temperature (21–25 °C) or at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	See Chromatogram
Eluent:	See Chromatogram
Eluent Flow Rate:	1.2 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Lithium	1.0
2.	Sodium	4.0
3.	Ammonium	10.0
4.	Magnesium	5.0
5.	Calcium	10.0
6.	Potassium	10.0

where 1 mg/L = 1 ppm

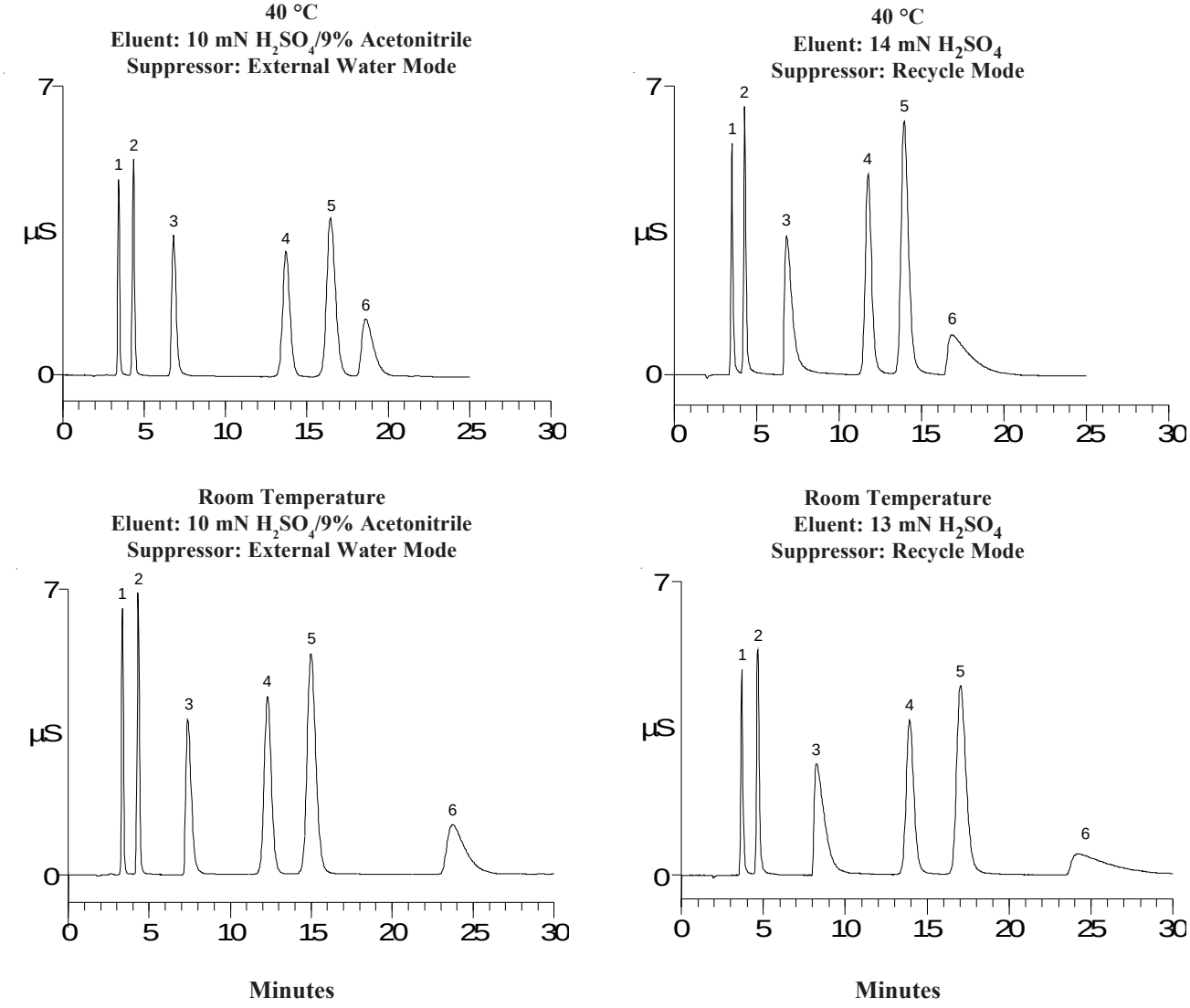


Figure 3
IonPac CS15 Production Test Chromatogram Comparison

5.2 Isocratic Elution of 6-Cations plus Ethanolamine

The following chromatograms show the isocratic elution of ammonia, ethanolamine, plus Group I and Group II cations (Li^+, Na^+, ethanolamine, NH_4^+, Mg^{2+}, Ca^{2+}, and K^+) on the CS15 analytical column with CG15 guard column using different isocratic eluents with the column either at room temperature (21–25 °C) or at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	See chromatogram
Eluent:	See chromatogram
Eluent Flow Rate:	1.2 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Lithium	1.0
2.	Sodium	4.0
3.	Ethanolamine	3.0
4.	Ammonium	5.0
5.	Magnesium	5.0
6.	Calcium	10.0
7.	Potassium	10.0

where 1 mg/L = 1 ppm

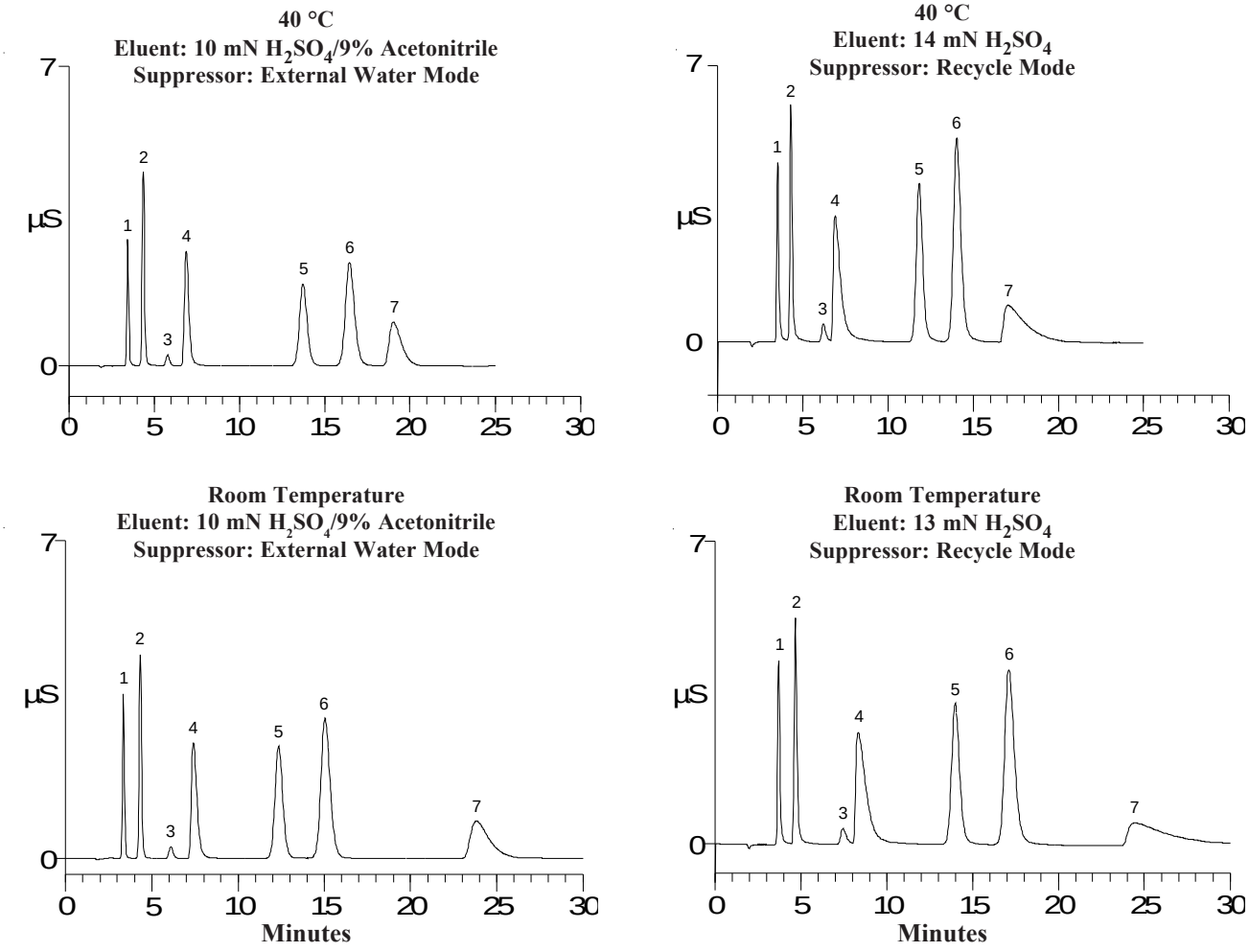


Figure 4
Isocratic Elution of 6-Cations Plus Ethanolamine (Li^+, Na^+, Ethanolamine, NH_4^+, Mg^{2+}, Ca^{2+}, and K^+)

5.3 Isocratic Elution of 6-Cations Plus Morpholine

The following chromatograms show the isocratic elution of ammonia, morpholine plus Group I and Group II cations (Li^+, Na^+, Morpholine, NH_4^+, Mg^{2+}, Ca^{2+}, and K^+) on the CS15 analytical column with CG15 guard column using different isocratic eluents with the column either at room temperature (21–25 °C) or at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	See chromatogram
Eluent:	See chromatogram
Eluent Flow Rate:	1.2 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Lithium	1.0
2.	Sodium	4.0
3.	Morpholine	50.0
4.	Ammonium	5.0
5.	Magnesium	5.0
6.	Calcium	16.0
7.	Potassium	10.0

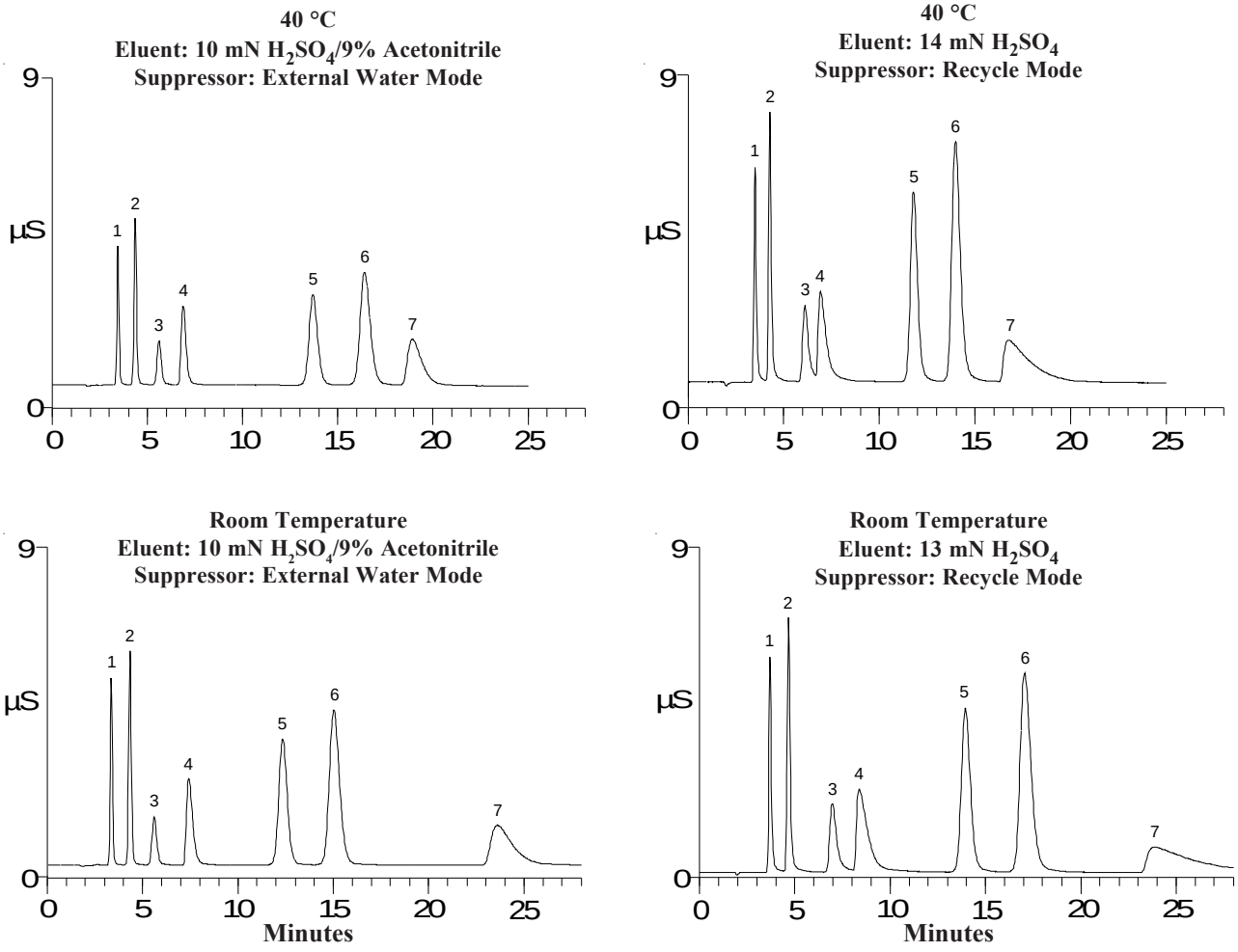


Figure 5
Isocratic Elution of 6-Cations Plus Morpholine (Li^+, Na^+, Morpholine, NH_4^+, Mg^{2+}, Ca^{2+}, and K^+)

5.4 4000:1 Ratio Sodium to Ammonium

The following shows a ratio of 4000:1 of sodium to ammonium on the CS15 analytical column with CG15 guard column using different isocratic eluents with the column either at room temperature (21–25 °C) or at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	See Chromatogram
Eluent:	See Chromatogram
Eluent Flow Rate:	1.2 mL/min (4-mm)
Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Sodium	100.0
2.	Ammonium	0.025

where 1 mg/L = 1 ppm

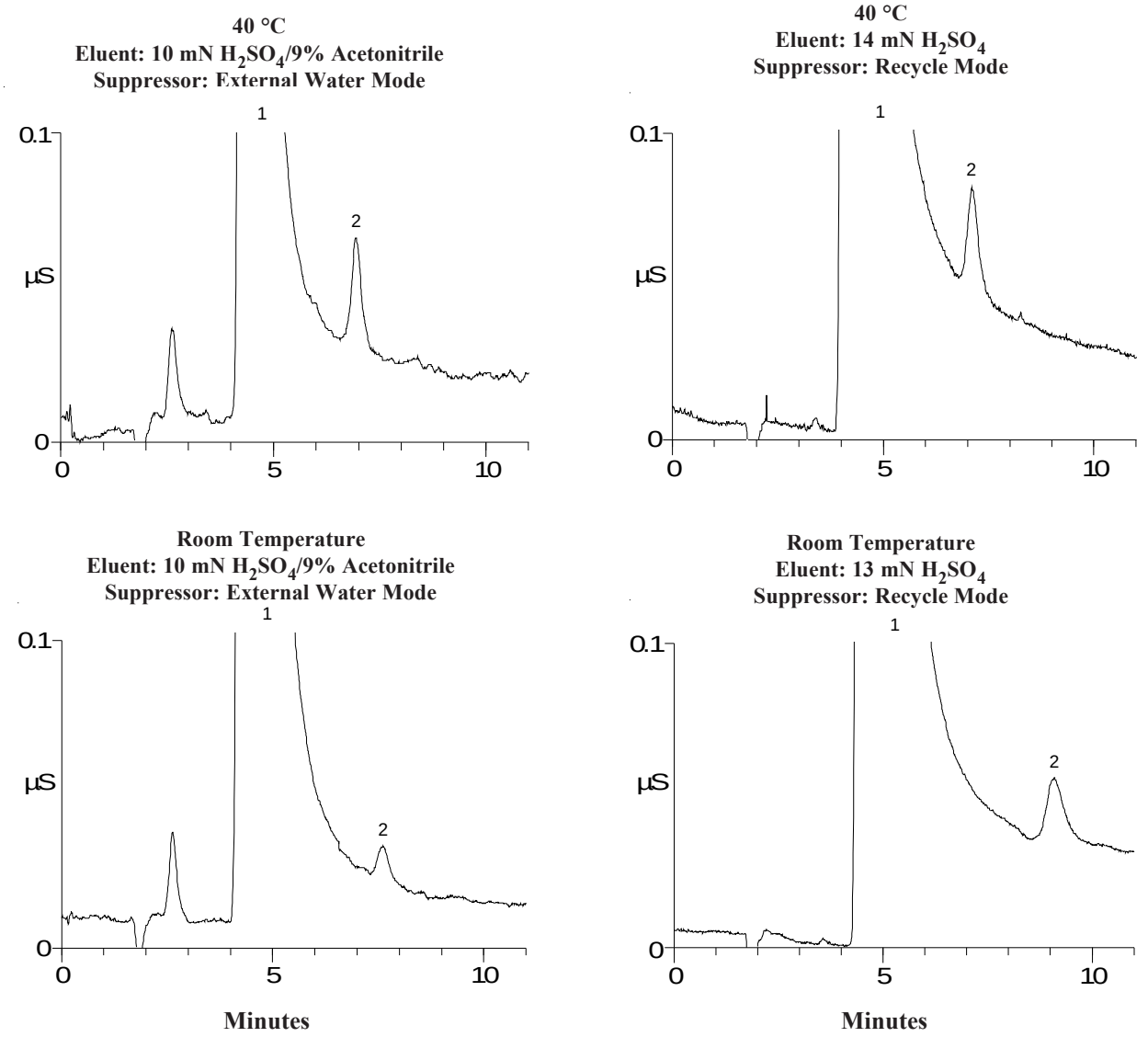


Figure 6
High-to-low concentration ratio of sodium to ammonium

5.5 10,000:1 Ratio Ammonium to Sodium

The following shows a ratio of 10,000:1 of ammonium to sodium on the CS15 analytical column with CG15 guard column at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	40 °C
Eluent:	10 mN H_2SO_4/9% Acetonitrile
Eluent Flow Rate:	1.2 mL/min (4-mm)
Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm), External water mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Sodium	0.01
2.	Ammonium	100.0

where 1 mg/L = 1 ppm

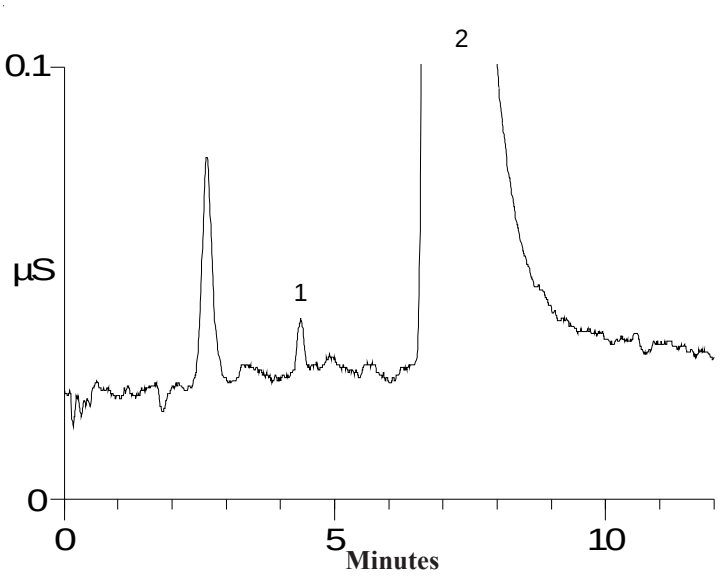


Figure 7
High-to-low concentration ratio of ammonium to sodium

5.6 10,000:1 Ratio Potassium to Ammonium

The following shows a ratio of 10,000:1 of potassium to ammonium on the CS15 analytical column with CG15 guard column at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	40 °C
Eluent:	10 mN H_2SO_4/9% Acetonitrile
Eluent Flow Rate:	1.2 mL/min (4-mm)
Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm) External water mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Ammonium	0.01
2.	Potassium	100.0

where 1 mg/L = 1 ppm

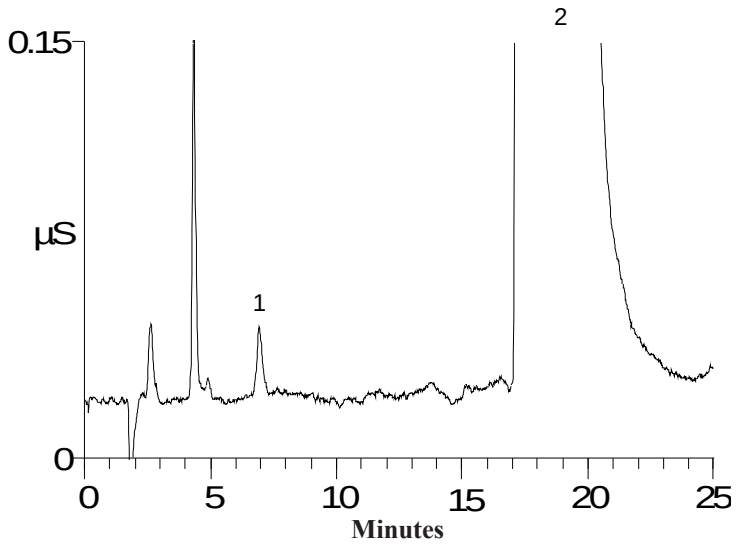


Figure 8
High-to-low concentration ratio of potassium to ammonium

5.7 Fast Run Analysis using CS15 and CG15

The following shows a fast run analysis using the CS15 analytical column with CG15 guard column at 40 °C.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), CG15 Guard Column (4-mm)
Column Temp:	40 °C
Eluent:	18 mN H_2SO_4/13% Acetonitrile
Eluent Flow Rate:	1.2 mL/min (4-mm)
Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm) External water mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

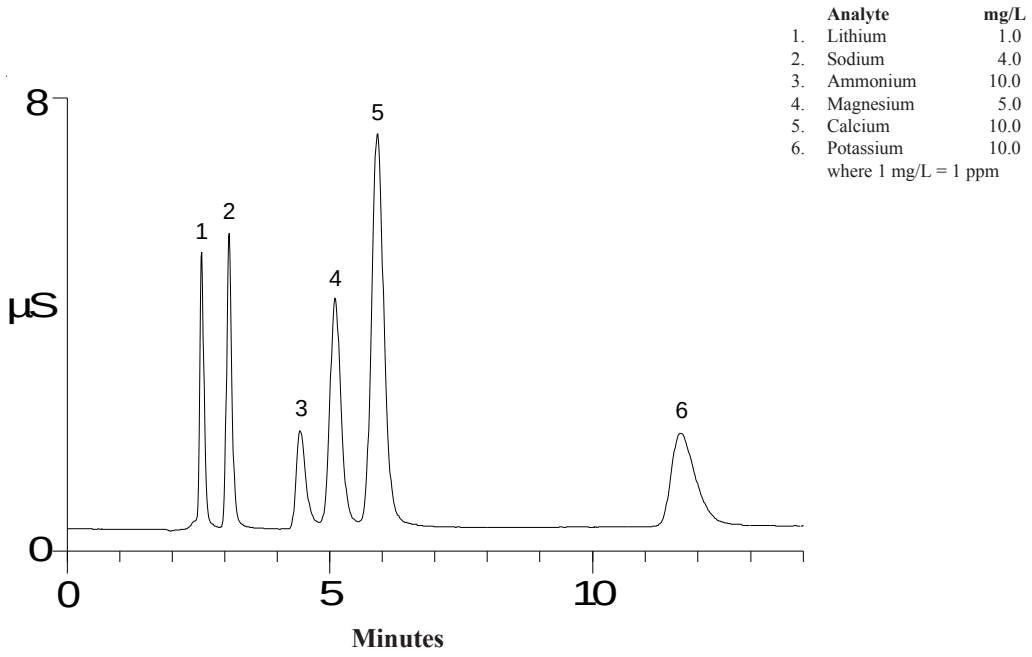
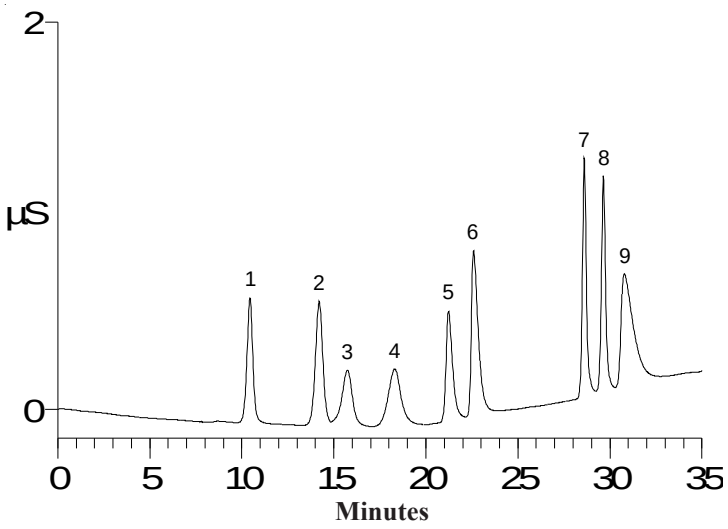


Figure 9
Fast run analysis using CS15 and CG15

5.8 Gradient Separation of Alkanolamines

The following shows a gradient separation of alkanolamines using the CS15 analytical column with CG15 guard.

Sample Volume:	2.5 µL Loop (2-mm)
Column:	CS15 Analytical Column (2-mm), CG15 Guard Column (2-mm)
Trap Column:	CTC (2-mm)
Eluent:	2 mM Methanesulfonic acid for 14 min., to 27 mM at 30 min.
Eluent Flow Rate:	0.3 mL/min
Column Temp:	40 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA External water mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)



	Analyte	mg/L
1.	Lithium	0.5
2.	Sodium	2.0
3.	Diethanolamine	10.0
4.	Triethanolamine	100.0
5.	Ethanolamine	5.0
6.	Ammonium	2.5
7.	Magnesium	1.0
8.	Calcium	1.5
9.	Potassium	5.0

where 1 mg/L = 1 ppm

Figure 10
Gradient Separation of Alkanolamines using CS15 and CG15

5.9 Gradient Elution of Ethylamines using CS15 and CG15

The following shows a gradient elution of ethylamines using the CS15 analytical column with CG15 guard.

Sample Volume:	2.5 µL Loop (2-mm)
Column:	CS15 Analytical Column (2-mm), CG15 Guard Column (2-mm)
Eluent:	2 mM Methanesulfonic acid / 6% acetonitrile for 10 min., to 14 mM at 15 min.
Eluent Flow Rate:	0.3 mL/min.
Column Temp:	40 °C
Detection:	Suppressed Conductivity
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA External water mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)

	Analyte	mg/L
1.	Lithium	0.5
2.	Sodium	2.0
3.	Diethylamine	2.5
4.	Triethylamine	10.0
5.	Ammonium	5.0
6.	Ethylamine	2.5
7.	Magnesium	0.5
8.	Calcium	1.0
9.	Potassium	5.0

where 1 mg/L = 1 ppm

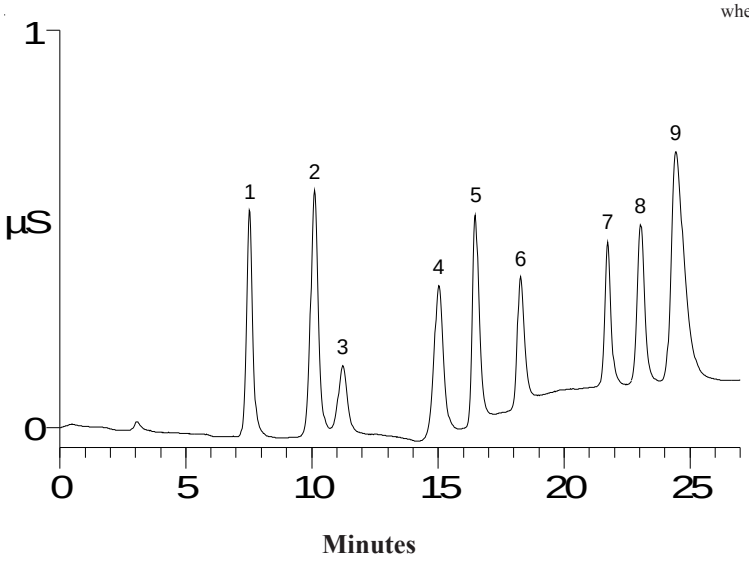


Figure 11
Gradient separation of ethylamines using CS15 and CG15

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac CS15 columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the nearest Dionex Office (see, "Dionex Worldwide Offices").

Table 7
CS15/CG15 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown Component	Isolate Blockage	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2
	Plugged System Hardware	Unplug, Replace	Component Manual
High Background Conductivity			
Improper Suppressor Operation	CSRS Not Suppressing	Check Current	6.5 A, Component Manual
		Check **REGEN OUT** Flow	6.5 A, 6.5 E Component Manual
		Check for leaks	6.5 B, Component Manual
	CMMS Not Suppressing	Check Regenerant	6.5.C, Component Manual
		Check AutoRegen Cartridge	6.5.C, Component Manual
Contamination	Contaminated Eluents	Remake Eluents	6.2, 6.4
	Contaminated Column	Clean Column	6.3.1, Column Care
	Contaminated CSRS or CMMS	Clean Suppressor	6.5, Component Manual
Hardware Operation			
	Proportioning Valve	Service Valve	Component Manual
Poor Peak Resolution			
Poor Efficiency	Large System Void Volumes	Replumb System	6.6.1 B, Component Manual
	Sluggish Injection Valve	Service Valve	6.6.4 A, Component Manual
	Column Headspace	Replace Column	6.6.1.A
	Column Overloading	Reduce Sample Size	6.6.3.B
Fronting Peaks	Column Overloading	Reduce Sample Size	6.6.3.B
Tailing Peaks	Contaminated CSRS or CMMS	Clean Suppressor	6.5, Component Manual
Short Retention Times	Flow Rate Too Fast	Recalibrate Pump	6.6.2.A
	First Peaks Elute Too Fast	Equilibrate to First Eluent	6.6.3.A
	Bad Eluent Concentrations	Remake Eluents	6.6.2.B
	Column Contamination	Clean Column	6.6.2.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
Coelution of Ca^{+2}/K^{+}	Eluent Concentration Too Low	Increase Eluent Concentration	6.6.2 B
	Column Temp. Too High	Increase Column Temp.	6.6.2.3
Drift retention times	Column Temp Has Not Yet Reached Equilibration	Let Column Temp. Stabilize	
Poor Quantification 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 CG15 (4-mm) Guard Column plus the CS15 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 1,800 psi (12.41 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.

A. Make sure that the pump is set to the correct eluent flow rate. Higher than recommended eluent flow rates will cause higher pressure. Measure the pump flow rate if necessary with a stop watch and graduated cylinder.

B. Determine which part of the system is causing the high pressure. High pressure could be due to a plugged tubing or tubing with collapsed walls, an injection valve with a clogged port, a column with particulates clogging the bed support, a clogged High-Pressure In-Line Filter, the suppressor or the detector cell.

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi (0.34 MPa). Continue adding system components (injection valve, column(s), suppressor and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 8, "Typical CS15/CG15 Operating Back Pressures").

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

Table 8
Typical CS15/CG15 Operating Back Pressures

Column psi (MPa)	Typical Back Pressure mL/min	Flow Rate
CS15 4-mm Analytical	≤ 1,350 (9.31)	1.2
CG15 4-mm Guard	≤ 450 (3.10)	1.2
CS15 + CG15 4-mm columns	**≤ 1,800 (12.41)**	**1.2**
CS15 2-mm Analytical	≤ 1,350 (9.31)	0.3
CG15 2-mm Guard	≤ 450 (3.10)	0.3
CS15 + CG15 2-mm columns	**≤ 1,800 (12.41)**	**0.3**

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

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 CS15 4-mm Columns (P/N)	IonPac CS15 2-mm Columns (P/N)
Analytical Column	051795	052252
Guard Column	052200	052256
Bed Support Assembly	042955	044689
End Fitting	052809	043278

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 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 CG15 Guard and CS15 Analytical 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 CG15 at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. A regular program to clean or replace the guard column will improve results and prolong the life of the analytical column. 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 (GM-2, GM-3, or GM-4) in the Gradient Pump Module should be flushed thoroughly to remove eluents containing DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl) prior to use with the IonPac CS15. Chloride containing eluents should not be pumped through the CSRS ULTRA.

B. Use chemicals and deionized water of the proper purity. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

C. The system should be as metal-free as possible. Gripper tubing fittings in the system are a potential source for metal contamination of the column. The new Dionex ThermoFlare or PEEK ferrule fittings are preferred. Inspect the eluent pumps periodically for any signs of leakage.

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

E. For EG50 or EG40 operation, use a CR-CTC Trap Column. Install a CR-CTC Cation Trap Column (P/N 060478) if using an Eluent Generator with EGC II MSA cartirdge.

F. Install an IonPac Cation Trap Column (CTC-1, P/N 040192). It should be positioned between the pump and the injection valve. It is highly recommended for all cation gradient analyses. The CTC-1 strips the eluent of cation contaminants that will bind strongly to the analytical column resulting in the loss of column capacity and potentially interfering with the desired cation analyses. The CTC-1 minimizes baseline changes when performing gradient analysis. The **CTC (2-mm), P/N 043132**, should be used in 2-mm systems.

6.3.2 Sample Loop and/or Tubing Contamination

A. General Discussion

Eluents made with deionized water that is contaminated with bacteria as well as certain samples such as humic acids can potentially contaminate eluent lines and sample loops. Weak cation exchange sites are created on (or attached to) the tubing. This can happen to either Tefzel or PEEK tubing. Thus, the sample loop itself can act as a concentrator and depending on the sample or pH of the standard and the method of sample injection, inaccurate quantitation for divalent analytes on weak cation exchange resins such as the IonPac CS15 may be observed.

B. Weak Cation Exchangers

Carboxylated resins (used in the IonPac CS12, CS12A, CS14, CS15, CS16, and CS17) 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 retained. This can result in a quantification problem for divalent cations when the sample pH is >4.

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

A simple test can be performed (when using a column such as the IonPac CS15 which contains a carboxylated resin) with methanesulfonic acid or sulfuric acid to determine if the sample loop has been contaminated:

1. Prepare a standard containing 0.5 ppm of calcium and add a small amount of 0.2 mM sodium hydroxide so that the final pH of the standard is between 6.5 and 7.5.

2. With the sample loop in the load position, flush the loop with just enough standard to rinse and fill the loop (e.g. if the loop is 25 µL, flush it with no more than 100 µL).

3. Run the standard and record the peak area.

4. Repeat steps 2 and 3, but this time flush the loop with about 5 mL of standard.

5. If after repeating steps 2 through 4, the peak area for calcium recorded in 4 is significantly larger than that in 3, then the sample loop is contaminated and acting as a concentrator.

6. Replace the sample loop with new tubing and repeat this test.

7. If there is still a quantification problem, check other components of the system (tubing, injection valve, detector cell) or call your Dionex 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 a small percentage.

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 functioning system, the background conductivity using the operating conditions described in the test chromatogram should be < 3 µS with a CSRS ULTRA.

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 ULTRA, or Cation MicroMembrane Suppressor, CMMS ULTRA, that Does Not Suppress Properly."

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

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
10 mN H_2SO_4/9% Acetonitrile	< 3 µS
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 or the Cation MicroMembrane Suppressor is causing the problem, refer to the Cation Self-Regenerating Suppressor ULTRA Product Manual (Document No. 031370) for detailed troubleshooting assistance or to the Cation MicroMembrane Suppressor III Product Manual (Document No. 031728).

A. Check that the CSRS ULTRA is not in an alarm state.

B. Check for CSRS ULTRA leaks.

C. Make sure that the back pressure tubing is properly installed on the outlet of the CSRS ULTRA. Backpressure is necessary to minimize noise

D. Check the regenerant flow rate at the REGEN OUT port of the CSRS. Turn the power to the CSRS off. Measure the regenerant flow rate. If it is being used in the recycle mode, it should be the same flow rate as the eluent (typically 1 mL/min for 4-mm operation). If it is used in the AutoSuppression External Water Mode, it should be at least 5 mL/min for non-solvent containing eluents. When solvents are used in the eluent, the regenerant flow rate should be at least 10 mL/min.

E. Check the eluent flow rate. See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. Refer to the Cation Self-Regenerating Product Manual (Document No. 031139) 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), 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.**

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

NOTE

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

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. 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.

B. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an i.d. of no greater than 0.010" for 4-mm systems or no greater than 0.005" for 2-mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks.

C. Check the sample for column overloading. Overloading the column and/or injecting samples in very acidic matrices (>20 mM H^+) can cause poor efficiencies.

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. Gradient pumps may also have 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 nearest Dionex Office (see, "Dionex Worldwide Offices").

E. Increasing the acid concentration has a larger effect on divalent cations than on monovalent cations. In the CS15 column, if potassium is partially coeluting with calcium, increasing the acid concentration in the eluent will separate these by decreasing the retention of the divalent cations more with respect to the monovalent cations. Total run time will also be shorter in this case. See chromatograms below.

6.6.3 Increasing Peak Resolution by Increasing Acid Concentration

The following two chromatograms show the resolution of calcium and potassium when the sulfuric acid concentration is increased.

Sample Volume:	25 µL Loop (4-mm)
Column:	CS15 Analytical Column (4-mm), (No Guard Column)
Column Temp:	40 °C
Eluent:	See chromatogram
Eluent Flow Rate:	1.2 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor-II (4-mm) in External Water Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
MMS Regenerant:	TBAOH
MMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 3 µS
Storage Solution:	Eluent

	Analyte	mg/L
1.	Lithium	1.0
2.	Sodium	4.0
3.	Ammonium	5.0
4.	Magnesium	5.0
5.	Calcium	10.0
6.	Potassium	10.0

where 1 mg/L = 1 ppm

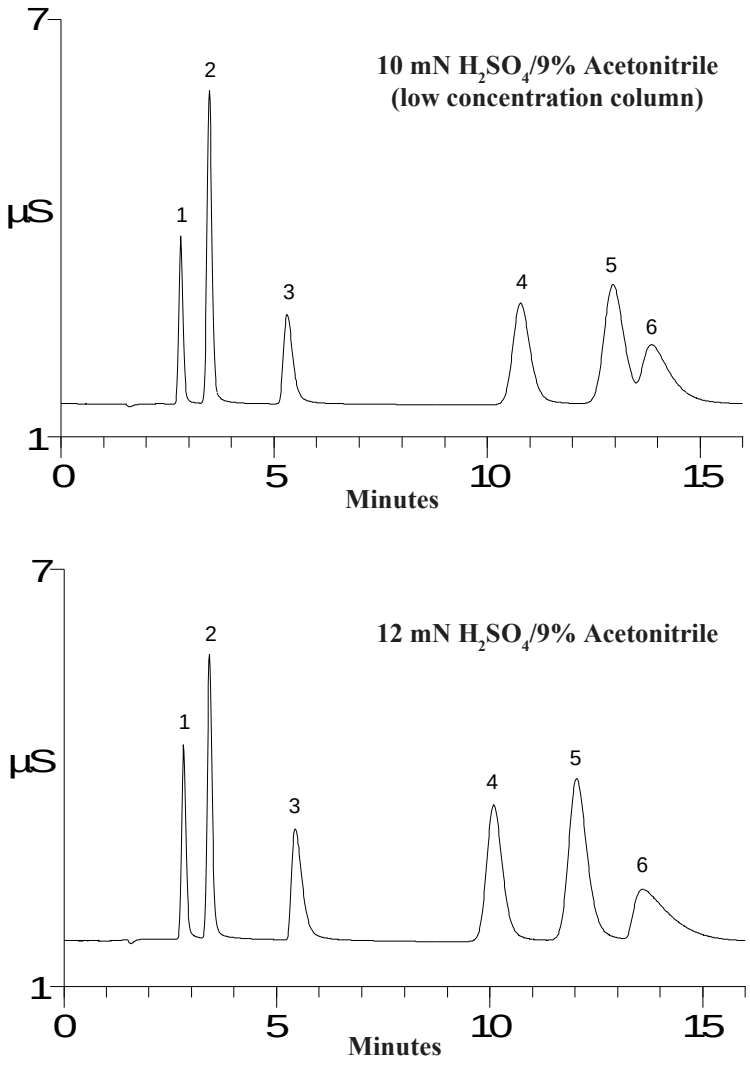


Figure 12
Increasing resolution by increasing eluent acid concentration

6.6.4 Loss of Early Eluting Peak Resolution

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

A. Be sure that the column is equilibrated 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 concentration 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) and often look like shark fins. Typically this will occur when the injection 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.

APPENDIX A - QUALITY ASSURANCE REPORT

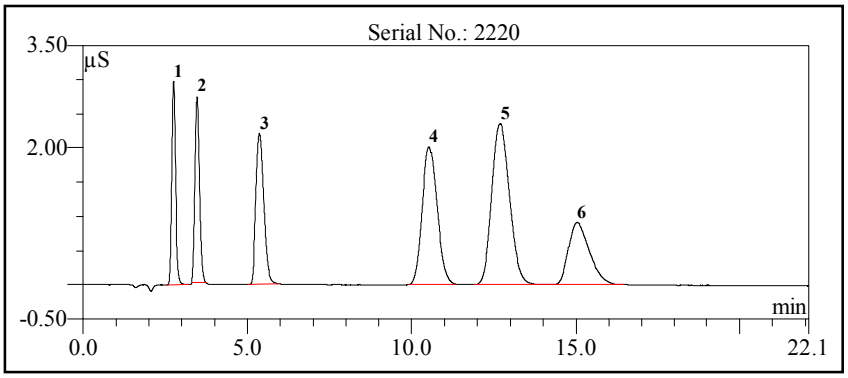
Quality Assurance Report - IonPac CS15 Analytical Column - 2 x 250 mm

Quality Assurance Report - IonPac CS15 Analytical Column - 4 x 250 mm

IonPac® CS15
Analytical (2 x 250 mm)
Product No. 052252

Date: 04-Mar-09 13:13
Serial No. : 002220
Lot No. : 008-20-019

Eluent: 10 mN H2SO4 + 9%(by volume) acetonitrile
Regenerant DI water
Regenerant Flow Rate: 10 psi
Eluent Flow Rate: 0.3 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Cation Self-Regeneratin g Suppressor (CSRS® ULTRA II, 2mm)
Applied Current: 9 mA
Injection Volume: 2.5 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	2.74	1.3	3.14	2635	1.0
2	Sodium	3.47	1.3	5.41	3013	4.0
3	Ammonium	5.36	1.4	8.12	2268	10.0
4	Magnesium	10.53	1.2	2.40	2594	5.0
5	Calcium	12.70	1.2	2.17	2701	10.0
6	Potassium	15.03	1.8	n.a.	2604	10.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=2070	Passed
Calcium	Asymmetry	1.1-1.9	Passed
Sodium	Efficiency	>=2700	Passed
Potassium	Retention Time	-20.90	Passed
	Pressure	<=1485	797

Production Reference:
Datasource: Column
Directory: CPE\CPE_4
Sequence: 638250_CS15_2x250
Sample No.: 48

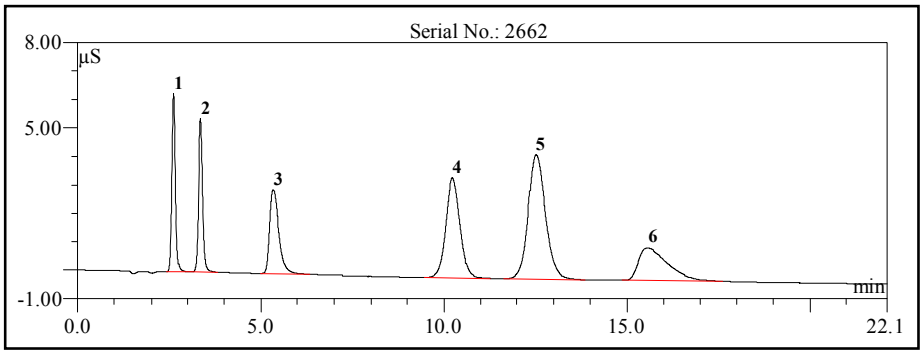
6.80 Build 2212 (Demo-Installation)

066891-02 (QAR)

IonPac® CS15
Analytical (4 x 250 mm)
Product No. 051795

Date: 03-Feb-09 11:32
Serial No. : 002662
Lot No. : 008-05-035

Eluent: 10 mN H2SO4 + 9%(by volume) acetonitrile
Regenerant DI water
Regenerant Flow Rate: 30 psi
Eluent Flow Rate: 1.2 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Suppressor: Cation Self-Regenerating Suppressor (CSRS® ULTRA Ⅱ, 4mm)
Applied Current: 36 mA
Injection Volume: 25 µL
Storage Solution: Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Lithium	2.63	1.1	3.98	4062	1.0
2	Sodium	3.36	1.1	6.28	4307	4.0
3	Ammonium	5.34	1.9	8.75	2498	10.0
4	Magnesium	10.24	1.1	2.97	3473	5.0
5	Calcium	12.51	1.3	2.72	3527	10.0
6	Potassium	15.57	2.7	n.a.	1931	10.0

QA Results:

Analyte	Parameter	Specification	Results
Calcium	Efficiency	>=2070	Passed
Calcium	Asymmetry	1.1-1.9	Passed
Sodium	Efficiency	>=2700	Passed
Potassium	Retention Time	<=20.90	Passed
	Pressure	<=1485	1056

Production Reference:
Datasource: Column
Directory : CPE\CPE_4
Sequence : 634295_CS15_4x25 0
Sample No. : 14

6.80 Build 2212 (Demo-Installation)

066897-02 (QAR)

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 CS15 Analytical or Guard Column is 4,000 psi (27.57 MPa).

CAUTION

Do not use eluents containing alcohols in this column
Formation of esters will occur in the column packing.
This can significantly reduce the column capacity for cation exchange.
Do not use the ionpac CS15 column with basic eluents

Column Start-Up

The column is shipped with eluent as the storage solution. This eluent is shown in the test chromatogram. Before using a column which is new and/or has not been in use for some time (> 1 week), pump eluent through the column to waste for at least 15 minutes before connecting it to the suppressor. If you plan to use an eluent other than the test eluent, first equilibrate the column to waste with the desired eluent for 30 to 60 minutes. The column is equilibrated when two consecutive injections of 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.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to < 5% levels and the ionic strength of the eluent to < 50 mM levels. This intermediate low concentration step will prevent precipitation or high viscosity zones. Avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

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

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

NOTE

Nitric acid should not be used as a substitute for 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 CS15 Analytical Column. If your system is configured with both a guard column and an analytical column, move the guard after the analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.

CAUTION

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

C. Set the pump flow rate to 1.2 mL/min for a CS15 4-mm Analytical or Guard Column or set the pump flow rate to 0.3 mL/min for a CS15 2-mm Analytical or Guard Column.

D. Rinse the column for 15 minutes with 10 mM HCl before pumping the chosen cleanup solution over the column.

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

F. Rinse the column for 15 minutes with 10 mM HCl before pumping eluent over the column.

G. Equilibrate the column(s) with eluent before resuming normal operation for at least 5–10 minutes (send effluent to waste).

H. Reconnect the suppressor to the CS15 Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

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

Hydrophobic Cations and Organic Contaminants

A. Disconnect the analytical column from the injection valve and the suppressor. Disconnect the Gradient Mixer or the Cation Trap Column (CTC-1) from the gradient pump. Connect the IonPac CS15 2-mm or 4-mm Analytical Column directly to the gradient pump. Direct the effluent from the analytical column directly to a waste container.

B. Set the flow rate to 1.2 mL/min on 4-mm systems or 0.3 mL/min on 2-mm systems.

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

Eluent 1: 1 M HCl
Eluent 2: 90% Acetonitrile in deionized water

Time (min)	%E1	%E2
0.0	100	0
20.0	20	80
25.0	20	80
45.0	100	0
55.0	100	0

D. Rinse the column for 15 minutes with 10 mN HCl before pumping eluent over the column.

E. Equilibrate the column(s) with eluent before resuming normal operation for at least 5–10 minutes (send effluent to waste).

F. Reconnect the suppressor to the CS15 Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

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