



PRODUCT MANUAL

for

IonPac[®] CS10
IonPac[®] CS11
IonPac[®] CG10
IonPac[®] CG11

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PRODUCT MANUAL

for the

**IONPAC® CS10 ANALYTICAL COLUMN
(4 x 250 mm, P/N 043015)**

**IONPAC CS11 2-mm ANALYTICAL COLUMN
(2 x 250 mm, P/N 043127)**

**IONPAC® CG10 GUARD COLUMN
(4 x 250 mm, P/N 043016)**

**IONPAC CG11 2-mm GUARD COLUMN
(2 x 50 mm, P/N 043128)**

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SECTION 1 - INTRODUCTION

The IonPac® CS10 Analytical Column (P/N 043015) and the IonPac® CS11 2-mm Analytical Column (P/N 043127) are designed specifically for the analysis of alkali metals and alkaline earth metals.

The 4 mm x 250 mm IonPac CS10 Analytical Column has an ion exchange capacity of approximately 80 µeq/column. This resin is composed of 8.5 micron polystyrene/divinylbenzene substrate agglomerated with 275 nanometer cation exchange latex and has a nominal efficiency of 24,000 plates/meter for magnesium using standard operating conditions.

The CS11 2-mm has an ion exchange capacity of approximately 35 µeq/column. This resin is composed of 7.5 micron ethylvinylbenzene/divinylbenzene substrate that is 55% crosslinked and agglomerated with 275 nanometer cation exchange latex that is 5% crosslinked. The high crosslink of the substrate makes the column compatible with all common HPLC solvents. The column has a nominal efficiency of 20,000 plates/meter for magnesium using standard operating conditions.

The IonPac CS10 Analytical Column can be operated at flow rates up to 3.0 mL/min. Optimally, the IonPac CS10 Analytical Column should generate a system back pressure of approximately 1200 psi when operated with an eluent flow rate of 1.0 mL/min. The CS11 2-mm can be operated at flow rates up to 0.75 mL/min. Optimally, the CS11 should generate a system backpressure of approximately 1,200 psi when operated with an eluent flow rate of 0.25 mL/min.

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.

Table 1
CS10/CS11 Packing Specifications

Column	Particle Diameter µm	Substrate X-Linking %	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
CS10 Analytical (4 x 250 mm)	8.5	55	275	5	80	Sulfonic acid	Medium
CS10 Guard (4 x 50 mm)	8.5	55	275	5	26	Sulfonic acid	Medium
CS11 Analytical (2 x 250 mm)	7.5	55	275	5	35	Sulfonic acid	Medium
CS11 Guard (2 x 50 mm)	7.5	55	275	5	7	Sulfonic acid	Medium

Table 2
CS10/CS11 Operating Parameters

Column	Typical Back Pressure psi (MPa) at 30°C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
CS10 Analytical (4 x 250 mm)	1,200 (8.27)	1.0	3.0
CS10 Guard (4 x 50 mm)	350 (2.41)	1.0	3.0
CS10 Analytical + Guard	1,550 (10.69)	1.0	3.0
CS11 Analytical (2 x 250 mm)	1,200 (8.27)	0.25	0.75
CS11 Guard (2 x 50 mm)	350 (2.41)	0.25	0.75
CS11 Analytical + Guard	1,550 (10.69)	0.25	0.75

SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

This section describes the performance comparison of 2-mm i.d. analytical columns and systems to 4-mm i.d. analytical columns and systems. Note the similarities and differences of the two column and system types below.

CONDITION	2-mm CS11	4-mm CS10
Eluent Flow Rate	0.25 mL/min	1.0 mL/min
SRS	CSRS®-ULTRA (2-mm) (P/N 053949)	CSRS®-ULTRA (4-mm) (P/N 053948)
MMS	CMMS III (2-mm) (P/N 056753)	CMMS III (4-mm) (P/N 056752)
Injection Loop	(1 - 15 µL) Use the Rheodyne Microinjection Valve, Model No. 9126 (DIONEX P/N 044697) for full loop injections <15 µL	10 µL
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. No Gradient Mixer is required for the DX-300 HPLC Pump	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	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.
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/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 or the CD20/CD25. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) 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/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 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 30-40 psi back pressure after the cell.

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" on the Dionex Reference Library CD-ROM.

Table 3
Tubing Backpressures

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 4-mm Operation

The IonPac CS10 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.

3.1.2 System Requirements for 2-mm Operation

The IonPac CS11 2-mm Guard and Analytical Columns are designed to be run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient or isocratic methods should be performed on a system having either a standard or a narrow bore pumps (4-mm or 2-mm), as most applications are run at 0.5 mL/min.

3.1.3 System Void Volume

When using 2-mm and CS11, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4-mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" (P/N 044221 PEEK tubing). PEEK tubing with an i.d. of 0.010" (P/N 042260) may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers. Make sure all tubing is cut in a straight (not slanted) manner. If you need assistance in properly configuring your system contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices")

3.2 Installing the Cation Trap Column

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 with the strongest eluent that will be used during the gradient analysis.
- E. **Reconnect the CTC.** Connect the CTC to the eluent line that is connected to the injection valve.

The background conductivity of your system should be less than 3 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 from the injection valve.**
- B. Direct the outlet of the CTC to a separate waste container.**
- C. Flush the CTC.** Use 30 mL of a 10X eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min.
- D. Flush the CTC prior to start-up.** Prior to the use of the chromatographic system on the next day, flush the CTC with 30 mL of the strongest eluent used in the gradient program.

3.3 Sample Concentration

Detection limits can be enhanced through concentration of the sample onto a concentrator column and using this column in lieu 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.

The following columns can be used for concentration with the IonPac CS10 and CS11 Analytical Columns:

- A. The IonPac Trace Cation Concentrator (TCC-1, P/N 037032) provides the least baseline disturbance when the concentrator column is switched in line during analysis. This is due to its low internal volume which reduces back pressure fluctuation. However, its column capacity can be a limiting factor.
- B. The IonPac CG10 Guard Column (P/N 043128) and CG11 Guard Column (P/N 043016) have higher capacity than the IonPac Trace Cation Concentrator, but will create a larger baseline disturbance due to the back pressure fluctuation caused by its appreciably larger internal volume.

The practical backpressure limit for the IonPac CS10 and CS11 columns is 4,000 psi. Therefore, any combination of the above contributions to operating backpressure that totals up to 4,000 psi can be used.

CAUTION

In methods that include salts, always be careful to determine the aqueous salt solubility at the maximum solvent concentration before running the gradient to prevent precipitation of the salt in the system during the gradient run.

The salt's solubility under the desired eluent solvent concentration can be empirically determined by making small samples of gradually increasing levels of salt in the solvent containing eluent and observing when precipitation occurs.

3.4 The Injection Loop

The injection loop can vary from 2.5 μL to about 1000 μL , depending on the sample concentration and the cation exchange capacity of the column. Using very large sample loops result in a slight drop of peak efficiencies, but this could be offset by the gain in sensitivity. It is much easier to use a large sample loop injection than to do sample preconcentration, as besides requiring the extra hardware, the latter could be subject to contamination from external sources such as the preconcentration pump.

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

For most applications on a CS10 or CS11 analytical column, a 10 to 50 μL injection loop will be sufficient. Generally, do not inject more than 50 - 100 nanomoles of any one analyte onto the analytical column. Injecting larger amounts than this can result in overloading of the column which affects linearity as well as peak efficiency and asymmetry. This phenomenon will be more prevalent at higher concentrations of the analytes of interest, and especially so in the lower capacity analytical columns.

Table 4
Injection Loop

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

3.5 IonPac CG10/CG11 Guard Columns

An IonPac CG10 or CG11 Guard Column is normally used with the IonPac CS10 or CS11 Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column.

3.6 Cation Self-Regenerating Suppressor Requirements

A Cation Self-Regenerating Suppressor should be used for applications that require suppressed conductivity detection. Aqueous ionic eluents can be used in all CSRS ULTRA modes of operation. When using Aqueous Chloride Eluents use the CSRS in the Chemical Suppression Mode.

NOTE

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

If you are installing an IonPac CS10 4-mm Analytical Column, use a CSRS ULTRA 4-mm, P/N 053948.

If you are installing an IonPac CS11 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, see Document No. 031370, the "Product Manual for the Cation Self-Regenerating Suppressor ULTRA, the CSRS ULTRA (4-mm) and the CSRS ULTRA (2-mm)."

3.7 Cation MicroMembrane Suppressor Requirements

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

If you are installing an IonPac CS10 4-mm Analytical Column, use a CMMS III 4-mm, P/N 056752.

If you are installing an IonPac CS11 2-mm Analytical Column, use a CMMS III 2-mm, P/N 056753.

The recommended regenerant for the CSRS Cation Self-Regenerating Suppressor or the Cation MicroMembrane Suppressor (CMMS III) is 100 mM tetrabutylammonium hydroxide (TBAOH). The use of Dionex Cation Regenerant Solution (P/N 039602) to make the regenerant is highly recommended. There are several important operating precautions that must be observed in order to successfully use the Cation Self-Regenerating (CSRS) Suppressor or the Cation MicroMembrane Suppressor (CMMS III). Study the Installation Instructions and Troubleshooting Guide for the suppressor carefully before beginning an analysis.

3.8 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 III). See the DCR kit manual, Document P/N 031664, for details.

3.9 Detector

See Section 2, "Comparison of Ion Chromatography Systems," for CS10 4-mm and CS11 2-mm system detector, cell and thermal stabilizer requirements.

3.10 Eluent Flow Rate

Use the same eluent concentration used for a standard 4-mm system but reduce the flow rate to one-fourth of the flow rate (typically 0.25 mL/min) used on a 4-mm column system.

3.11 Eluent Storage

The storage solution for both the CS10 and CS11 is the eluent:

CS10: 40.0 mM HCl, 4.0 mM DL-2,3-diaminopropionic acid monohydrochloride

CS11: 50.0 mM HCl, 7.0 mM DL-2,3-diaminopropionic acid monohydrochloride

3.12 Using AutoRegen and Eluents Containing Solvent

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

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

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

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

SECTION 4 - OPERATION

4.1 Operating Precautions

CAUTION

Operate below 4,000 psi (4-mm)
Filter and Degas Eluents for 15 Minutes prior to use
Filter Samples
pH 0-14
0.75 mL/min Maximum Flow Rate (2-mm)
3.0 mL/min Maximum Flow Rate (4-mm)

Filtering and degassing eluents minimizes the chance of particles or air bubbles lodging in the pump system which can affect the pumps ability to provide consistent low flow rates.

4.1.1 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.

4.1.2 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.

4.1.3 Solvents

One of the benefits of the IonPac CS10 and CS11 Analytical Columns is the ability to use most common HPLC solvents in concentrations from 0% to 100% as mobile phase modifiers.

However when performing gradient analyses with solvent containing eluents, solvents and water should be premixed in concentrations that will allow rapid mixing by the gradient pump and will minimize outgassing. When using a solvent in an ionic eluent, column backpressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column backpressure varies with the composition of water-methanol and water-acetonitrile mixture.

4.2 Standard Operating Conditions

Sample loop:	10 μ L, CS10 4-mm 2.5 μ L, CS11 2-mm
Column:	CS10 4-mm CS11 2-mm
Eluent:	40 mM HCl/4 mM DAP·HCl
Eluent flow rate:	1 mL/min, CS10 4-mm; 0.25 mL/min, CS11 2-mm
SRS Suppressors:	Cation Self-Regenerating Suppressor, CSRS ULTRA (2-mm or 4-mm) AutoSuppression Chemical Suppression Mode
MMS Suppressors:	Cation MicroMembrane Suppressor, CMMS III (2-mm or 4-mm)
SRS or MMS Regenerant:	100mN tetrabutylammonium hydroxide (TBAOH) Use Dionex Cation Regenerant Solution (P/N 039602).
SRS or MMS Regenerant flow rate:	5-10 mL/min, CS10 4-mm 5.0 mL/min, CS11 2-mm
Expected Background Conductivity:	1-5 μ S
Storage Solution:	Eluent

4.3 Chemicals Required

It is very important for the eluent to be as free of impurities as possible. The chemicals and water required to prepare the eluent should be of the highest purity available. Use deionized water with a specific resistance of 18.2 megohm-cm.

- Use only concentrated HCl, ULTREX[®] grade or BAKER INSTRA-ANALYZED[®] for trace metals.
- Use Dionex DAP (DL-2,3-diaminopropionic acid, DAPHCl, P/N 039670) Reagent to prepare the Cation Eluents.
- Use Dionex Cation Regenerant Solution (tetrabutylammonium hydroxide (TBAOH), P/N 039602) as the regenerant solution to ensure maximum system performance.
- Use deionized water with a specific resistance of 18.2 megohm-cm to make all standards, eluents and regenerants.

Obtaining reliable, consistent and accurate results requires eluents that are free of ionic and spectrophotometric 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 expensive 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.

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.

Since solvents used with the IonPac CS10 and CS11 columns are added to ionic eluents to modify the ion exchange process, the solvents used must be free of ionic impurities. 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 your solvent. At Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima Solvents by Fisher Scientific.

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

4.4 Solutions Required

WARNING

Hydrochloric acid (HCl) vapors are very corrosive. Avoid breathing the vapors. Dilutions of HCl from the concentrated acid (38%) should be made in a fume hood.

4.4.1 1.0 M HCl Stock Solution

This solution will be used in the preparation of each of the eluents below.

- A. Calculate the amount (in grams) of concentrated hydrochloric acid (HCl) that you need to add to a 1 liter volumetric flask by using the % HCl composition stated on the label of the particular bottle of HCl you are using. For example, if the HCl concentration is 38%, you need to weigh out 95.95 grams of concentrated HCl.
- B. Carefully add this amount of HCl to a 1 liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm.
- C. Then dilute to the 1 liter mark and mix thoroughly.

4.4.2 Preparation of Eluent Stock Solution Concentrates

To prepare Eluents from stock solutions, use the following formula and table as a guide:

- A. Weigh out Y g of DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl) into a 1000 mL volumetric flask.
- B. Add X mL of 1 M HCl stock solution to the flask.
- C. Use deionized water having a specific resistance of 18.2 megohm-cm to dilute the eluent to 1 liter.
- D. Mix thoroughly to dissolve.

Table 5
mM Eluents from Stock Solutions

Eluent Concentration			Components Diluted to 1 L		Use
X mM HCl	/ Y mM DAP·HCl		# mL 1 M HCl	# g DAP·HCl	
40	/	2	40	0.28	Alkali metals and high dynamic ranges
40	/	4	40	0.56	Standard Mixed monovalent/divalent eluent
40	/	12	40	1.69	Separation of Strontium and Barium
50	/	7	50	0.98	
60	/	6	60	0.84	
60	/	12	60	1.69	

Where: DAP·HCl FW =140.6 g/mole

4.5 Eluents Containing Solvents

Applications utilizing eluents that contain solvents such as methanol and acetonitrile are straightforward as long as the solvents and water are premixed in concentrations that allow proper mixing by the gradient pump proportioning valve. Premixing water with solvent will minimize the possibility of outgassing and refractive index problems associated with using the gradient pump proportioning valve to mix neat solvents with water.

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 to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a true volume/volume eluent is obtained.

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

4.6 Regenerant Preparation

The regenerant is 100 mN tetrabutylammonium hydroxide. Use Dionex Cation Regenerant Solution (P/N 039602) without further dilution. 2-mm column applications on systems with the AutoRegen Accessory use the same regenerant flow rate as used for 4-mm column applications. Regenerant flow rate can be reduced to conserve regenerant if a pressurized vessel rather than the AutoRegen is being used.

SECTION 5 - EXAMPLE APPLICATIONS

The following example applications demonstrate the optimization of the separation of different combinations of alkali and alkaline earth metals. Before attempting any of the following example applications, take the time to ensure that your system is properly configured. It is very important that applications run on the proper pump configuration (see Section 2 - Comparison of Ion Chromatography Systems) and have all system void volumes minimized (see Section 3.1.3).

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. See Section 4.3 - Chemicals Required, for chemical purity requirements and Section 4.4 - Solution Required, for the preparation of stock solutions and regenerant.

In order to guarantee reproducible retention times of analytes when doing gradient chromatography, it is important to install a Cation Trap Column in the system (see Section 3.2 - Installing the Cation Trap Column).

NOTE

Eluents should be degassed, filtered and maintained under an inert helium atmosphere to avoid contamination and to ensure proper gradient pump performance.

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 and analytical columns 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 3.3 - Sample Concentration).

5.1 Production Test Chromatogram, CS10 (4-mm)

Isocratic Elution of Ammonia, Alkali Metals and AlkalineEarth Metals (Na^+ , NH_4^+ , K^+ , Mg^{2+} , Ca^{2+})

Separation and elution of cations on the IonPac CS10 4-mm Analytical Column has been optimized utilizing a hydrochloric acid/DL-2,3-diaminopropionic acid (DAP) eluent. By using this eluent, monovalent and divalent cations can be isocratically separated and quantitated in a single injection. To guarantee that all IonPac CS10 4-mm Analytical Columns meet high quality and reproducible performance specifications standards, all analytical columns undergo the following production control test.

Sample loop:	10 μL
Column:	IonPac CS10 (4-mm)
Eluent:	40 mM HCl/4 mM DAP·HCl
Eluent flow rate:	1.0 mL/min
Regenerant flow rate:	5-10 mL/min
Detector range:	30 μS full scale
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
or MMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (4-mm)
SRS or MMS Regenerant:	100 mM Tetrabutylammonium hydroxide (TBAOH)
Expected Background Conductivity:	$\leq 5 \mu\text{S}$

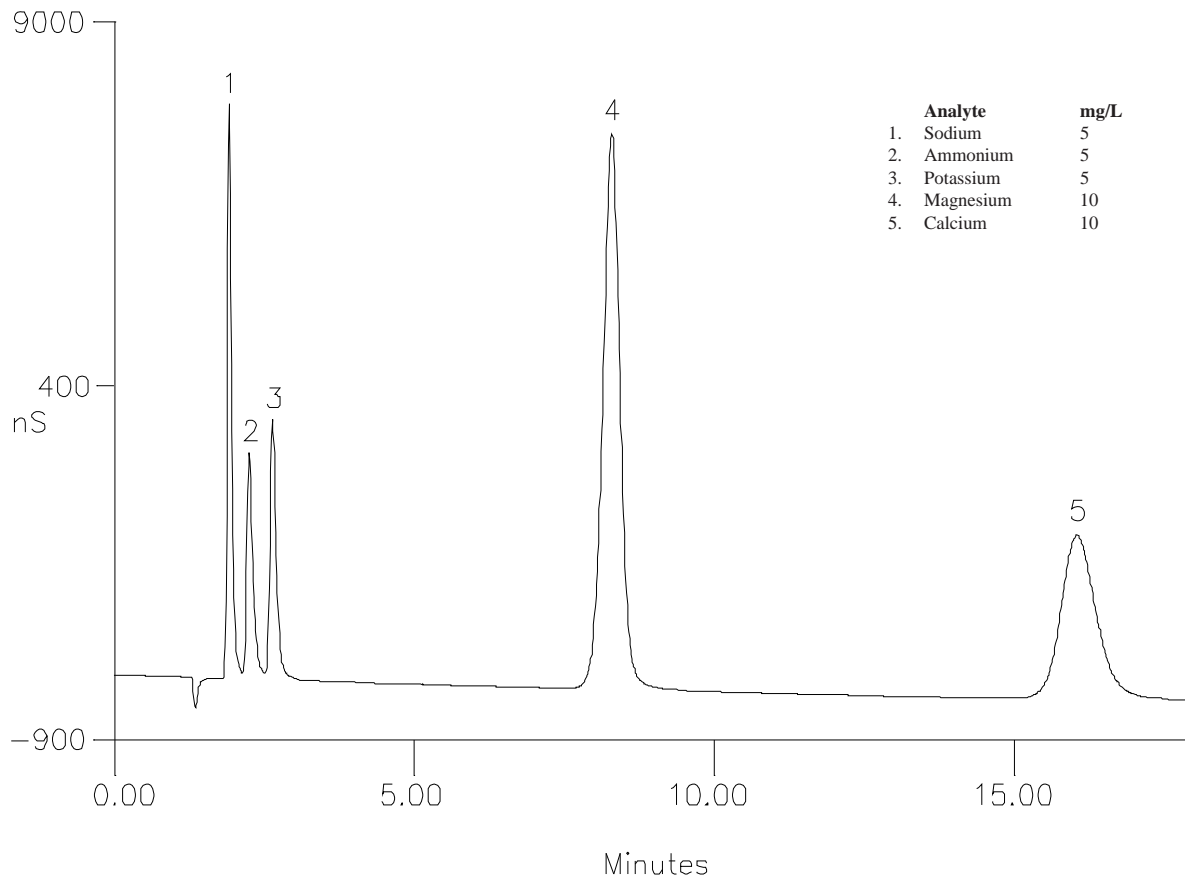


Figure 1
Production Test Chromatogram CS10 (4-mm)
Isocratic Elution of Ammonia, Alkali Metals and Alkaline Earth Metals (Na^+ , NH_4^+ , K^+ , Mg^{2+} , Ca^{2+})

5.2 Production Test Chromatogram, CS11 (2-mm)

Separation and elution of cations on the IonPac CS11 2-mm Analytical Column has been optimized utilizing a hydrochloric acid/DL-2,3-diaminopropionic acid (DAP) eluent. By using this eluent, monovalent and divalent cations can be isocratically separated and quantitated in a single injection. To guarantee that all IonPac CS11 2-mm Analytical Columns meet high quality and reproducible performance specifications standards, all analytical columns undergo the following production control test.

Sample Loop Volume: 2.5 μ L (2-mm)
 Analytical Column: IonPac CS11 2-mm Analytical Column
 Eluent: 40 mM HCl/4 mM DAP·HCl
 Eluent Flow Rate: 0.25 mL/min
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA (2-mm)
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III (2-mm)
 SRS or MMS Regenerant: 100 mM TBAOH
 Expected Background Conductivity: $\leq 5 \mu$ S
 Column Storage Solution: Eluent (40 mM HCl/4 mM DAP)

	Analyte	mg/L
1.	Sodium	5
2.	Ammonium	5
3.	Potassium	5
4.	Magnesium	10
5.	Calcium	10

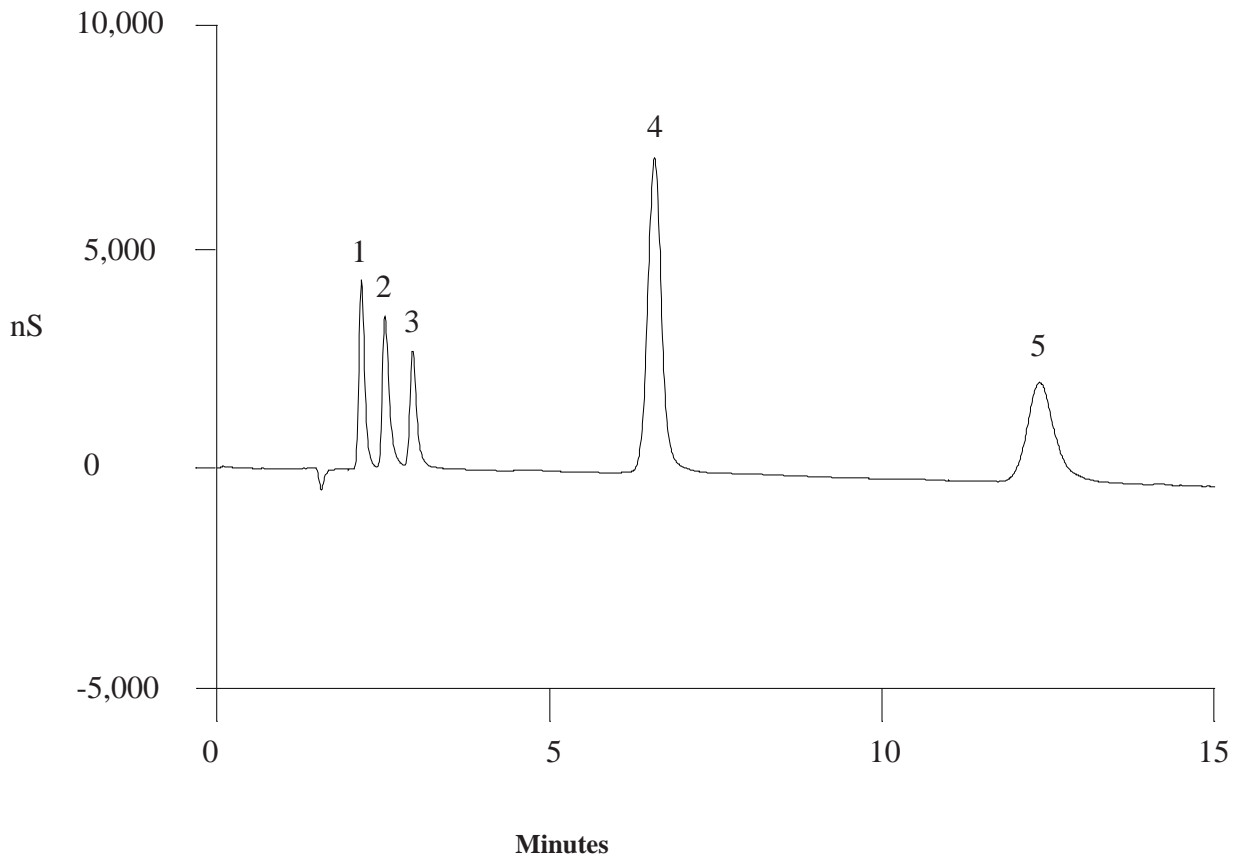


Figure 2
Production Test Chromatogram CS11 (2-mm)

5.3 Isocratic Elution of Ammonia, Alkali Metals and Alkaline Earth Metals

Column: IonPac CS10 (4-mm) Analytical Column
 Eluent flow rate: 1.0 mL/min
 Eluent: 40 mM HCl/2 mM DAPHCl
 Regenerant flow rate: 5-10 mL/min
 Detector range: 30 μ S full scale
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III
 SRS or MMS Regenerant: 100 mM TBAOH
 Sample loop: 10 μ L
 Expected Background Conductivity: $\leq 5 \mu$ S

Analyte	mg/L
1. Sodium	5
2. Ammonium	5
3. Potassium	5
4. Magnesium	10
5. Calcium	10

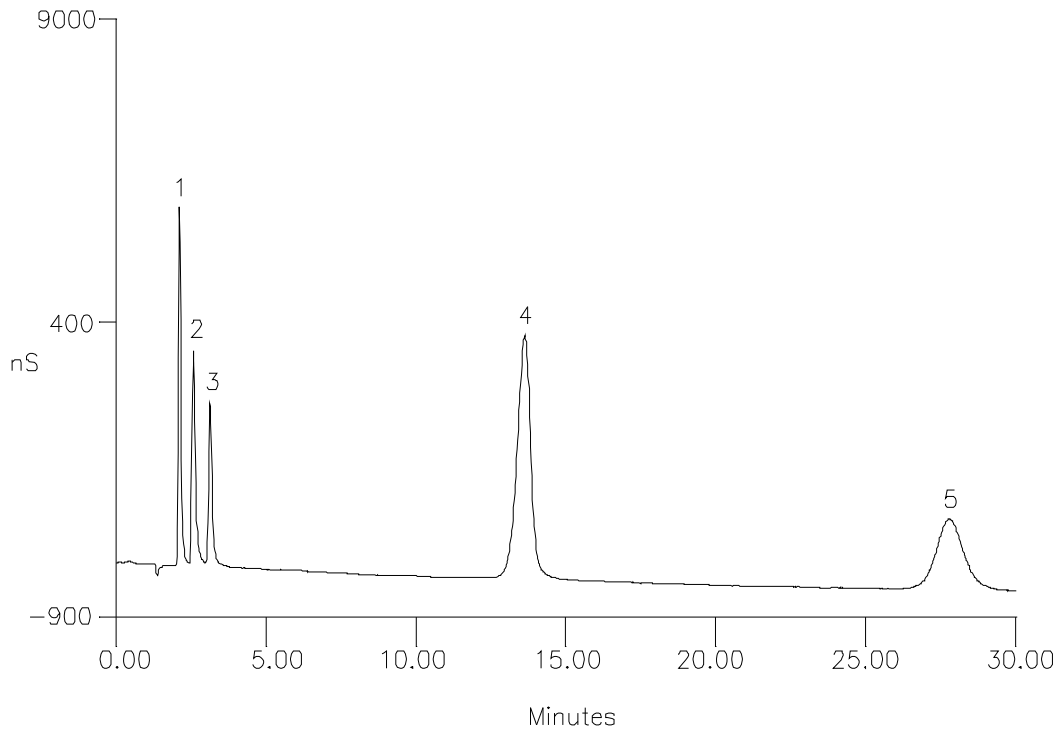


Figure 3
Isocratic Elution of Ammonia, Alkali Metals and Alkaline Earth Metals
 (Na^+ , NH_4^+ , K^+ , Mg^{2+} , Ca^{2+})

5.3.1 Isocratic Elution with 60 mM HCl/6 mM DAP·HCl

Column: IonPac CS10 (4-mm) Analytical Column
 Eluent flow rate: 1.0 mL/min
 Eluent: 60 mM HCl/6 mM DAP·HCl
 Regenerant flow rate: 5-10 mL/min
 Detector range: 30 μ S full scale
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III
 SRS or MMS Regenerant: 100 mM TBAOH
 Sample loop: 10 μ L
 Expected Background Conductivity: $\leq 5 \mu$ S

Analyte	mg/L
1. Sodium	5
2. Ammonium	5
3. Potassium	5
4. Magnesium	10
5. Calcium	10

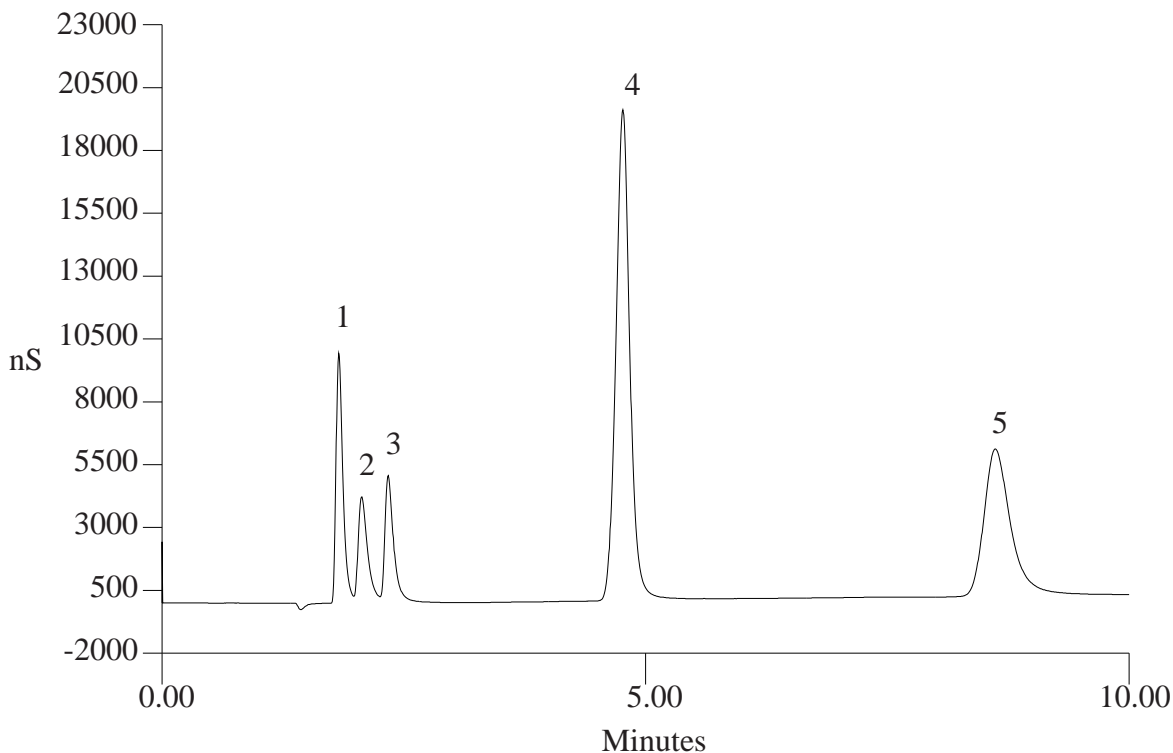


Figure 4
Isocratic Elution of Ammonia, Alkali Metals and Alkaline Earth Metals
 (Na^+ , NH_4^+ , K^+ , Mg^{2+} , Ca^{2+})

5.3.2 Isocratic Elution with 40 mM HCl/12 mM DAP·HCl

This chromatogram is obtained using the standard operating conditions:

Column: IonPac CS10 (4-mm) Analytical Column
 Eluent flow rate: 1.0 mL/min
 Eluent: 40 mM HCl/12 mM DAP·HCl
 Regenerant flow rate: 5-10 mL/min
 Detector range: 30 µS full scale
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III
 SRS or MMS Regenerant: 100 mM TBAOH
 Sample loop: 10 µL
 Expected Background Conductivity: ≤ 5 µS

Analyte	mg/L
1. Sodium	5
2. Ammonium	5
3. Potassium	5
4. Magnesium	10
5. Calcium	10
6. Strontium	10
7. Barium	40

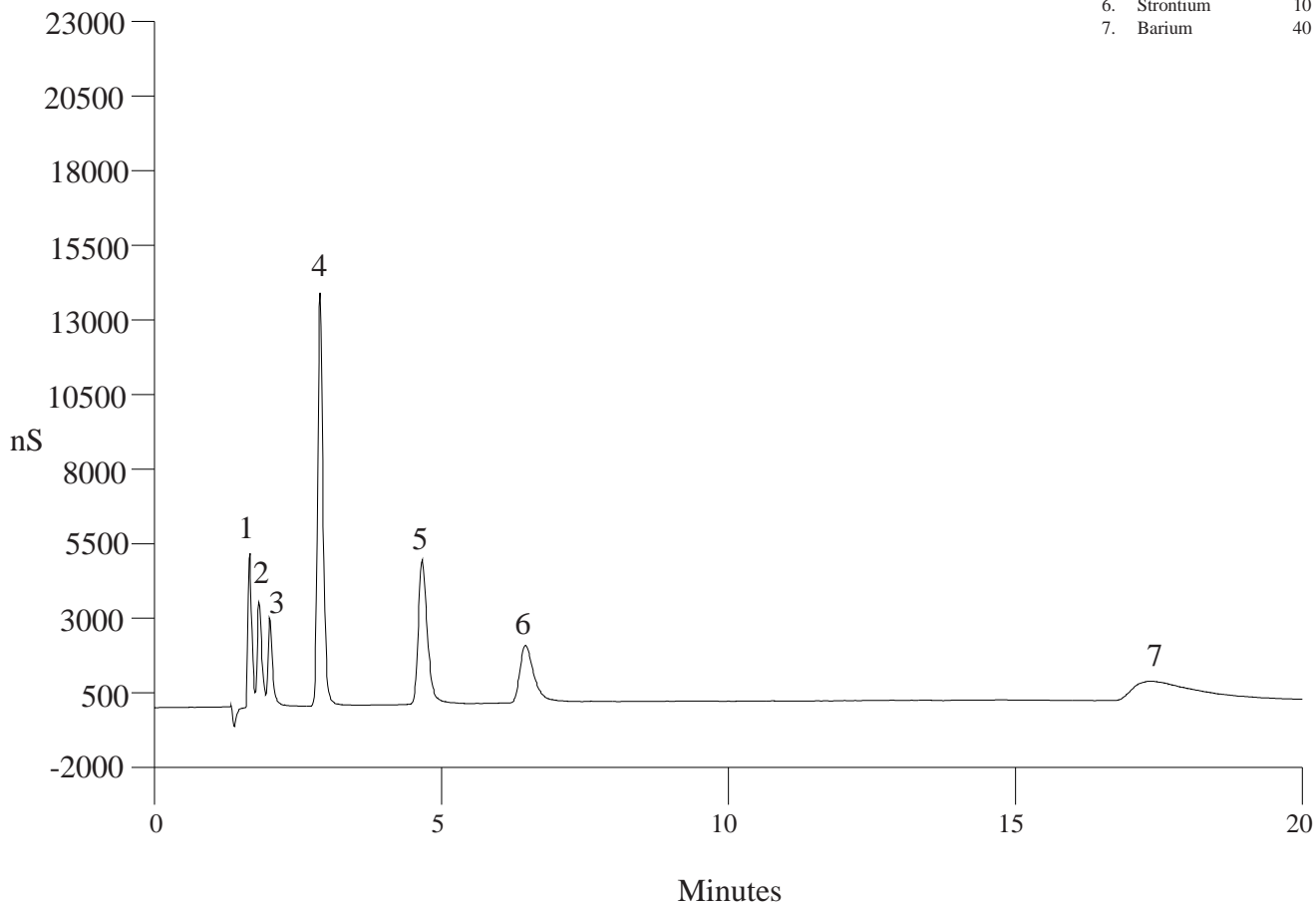


Figure 5
Isocratic Elution of Ammonia, Alkali Metals and Alkaline Earth Metals
 (Na⁺, NH₄⁺, K⁺, Mg²⁺, Ca²⁺)

5.4 Gradient Separation of Amines and Inorganic Cations using the CS10 (4-mm) Column

Sample Loop Volume: 10 µL
 Guard Column: IonPac CG10 Guard Column
 Analytical Column: IonPac CS10 Analytical Column
 Eluents: Eluent 1: 200 mM HCl
 Eluent 2: Acetonitrile (95%)
 Eluent 3: Deionized Water (having a specific resistance of 18.2 megohm-cm)
 Eluent 4: 20 mM DAP
 Eluent Flow Rate: 1.0 mL/min
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA (4-mm)
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III (4-mm)
 SRS or MMS Regenerant: 0.1 M Tetrabutylammonium hydroxide
 Expected Background Conductivity: ≤ 4 µS
 Expected System Operating Backpressure: 1,600 - 2,200 psi

Gradient Program

Time (min)	Flow	%1	%2	%3	%4	Analyte	mg/L
0.0	1.0	5	5	85	5	1. Lithium	1
0.1	1.0	5	5	85	5	2. Sodium	5
4.1	1.0	5	5	85	5	3. Ammonium	5
25.1	1.0	40	15	0	45	4. Methylamine	5
25.2	1.0	5	5	85	5	5. Potassium	5
						6. Trimethylamine	5
						7. Morpholine	20
						8. Diethylamine	10
						9. Pyrrolidine	10
						10. Triethylamine	10
						11. Piperidine	10
						12. Tetraethylamine	10
						13. Magnesium	5
						14. Cyclohexylamine	15
						15. Calcium	10

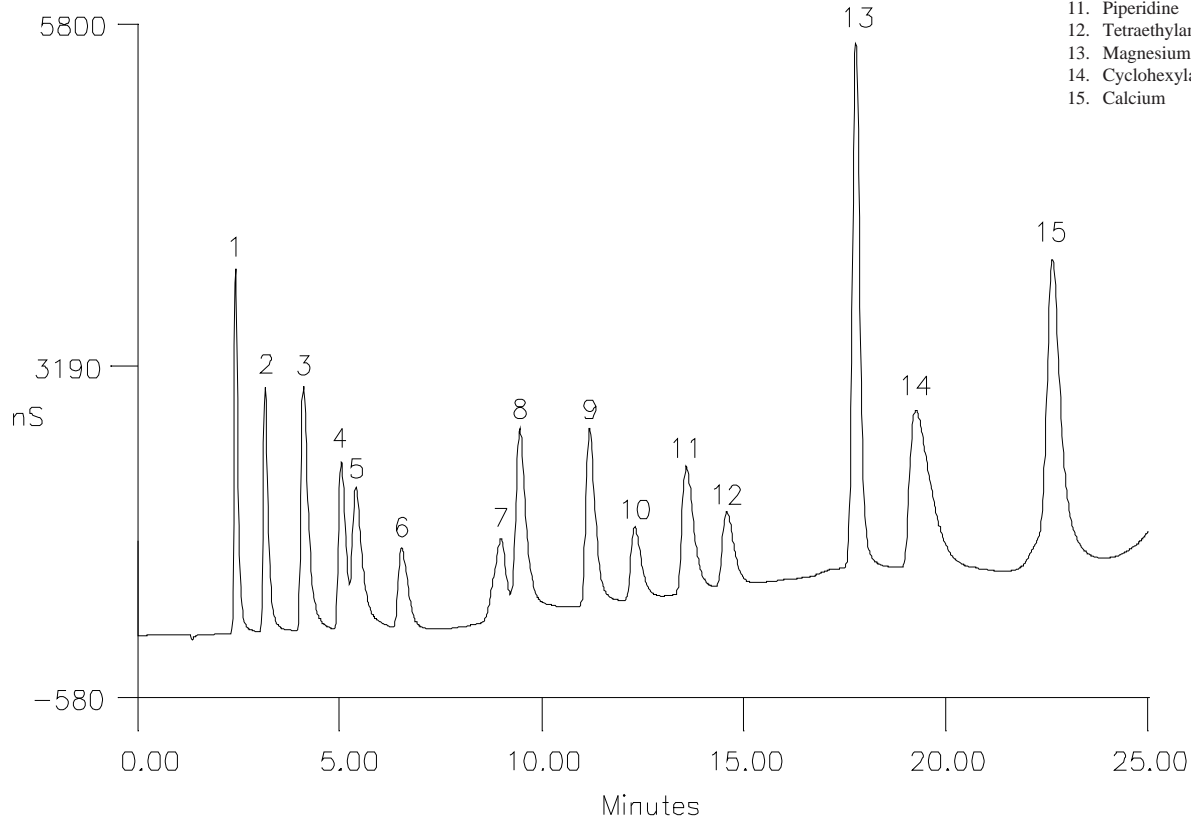


Figure 6
Gradient Separation of Amines and Inorganic Cation

5.5 Elution of Lithium, Sodium, Ammonium, Potassium, Magnesium and Calcium

The separation and elution of the 6 inorganic cations of most interest on the IonPac CS11 2-mm Analytical Column utilizing a hydrochloric acid/DL-2,3-diaminopropionic acid (DAP) eluent is shown in this example. By using this eluent, monovalent and divalent cations can be isocratically separated and quantitated in a single injection in less than 15 minutes.

Sample Loop Volume: 2.5 µL
 Analytical Column: IonPac CS11 2-mm Analytical Column
 Eluent: 50 mM HCl/7 mM DAP·HCl
 Eluent Flow Rate: 0.25 mL/min
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA (2-mm)
 MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III (2-mm)
 SRS or MMS Regenerant: 100 mM TBAOH
 Expected Background Conductivity: ≤ 5 µS
 Column Storage Solution: Eluent (50 mM HCl/7 mM DAP)

Analyte	mg/L
1. Lithium	5
2. Sodium	5
3. Ammonium	5
4. Potassium	5
5. Magnesium	10
6. Calcium	10

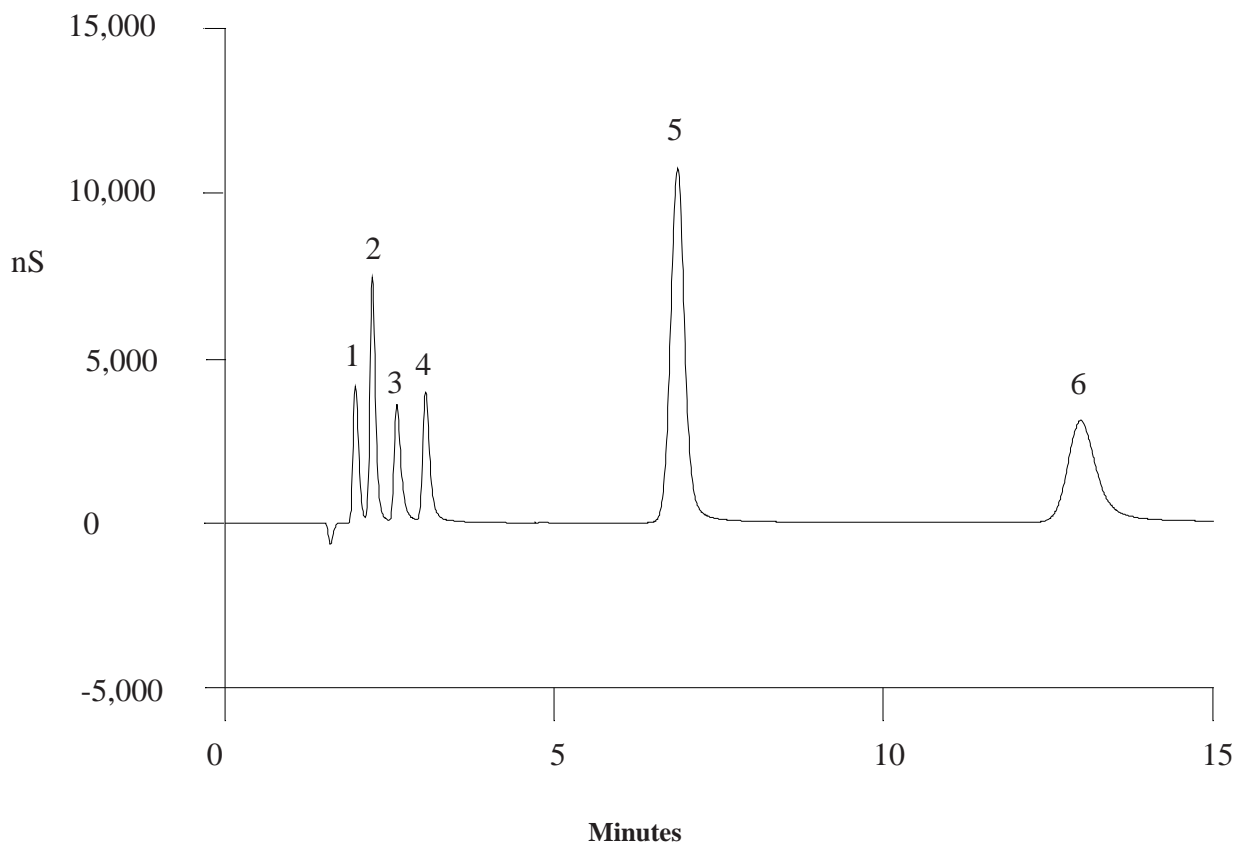


Figure 7
Elution of Lithium, Sodium, Ammonium, Potassium, Magnesium and Calcium

5.6 Rapid Elution of Lithium, Sodium, Ammonium, Potassium, Rubidium, Cesium, Magnesium, Strontium and Barium

The separation and elution of the 10 inorganic cations on the IonPac CS11 2-mm Analytical Column utilizing a hydrochloric acid/DL-2,3-diaminopropionic acid (DAP) eluent is shown in this example. By using this eluent, monovalent and divalent cations can be isocratically separated and quantitated in a single injection in less than 30 minutes.

Sample Loop Volume: 2.5 μ L
 Analytical Column: IonPac CS11 2-mm Analytical Column
 Eluent: 60 mM HCl/12 mM DAP-HCl
 Eluent Flow Rate: 0.25 mL/min
 SRS Suppressor: Cation Self-Regenerating Suppressor, CSRS ULTRA (2-mm)
 or MMS Suppressor: Cation MicroMembrane Suppressor, CMMS III (2-mm)
 SRS or MMS Regenerant: 100 mM TBAOH
 Expected Background Conductivity: $\leq 5 \mu$ S
 Column Storage Solution: Eluent (60 mM HCl/12 mM DAP)

Analyte	mg/L
1. Lithium	5
2. Sodium	5
3. Ammonium	5
4. Potassium	5
5. Rubidium	10
6. Cesium	10
7. Magnesium	10
8. Calcium	10
9. Strontium	25
10. Barium	50

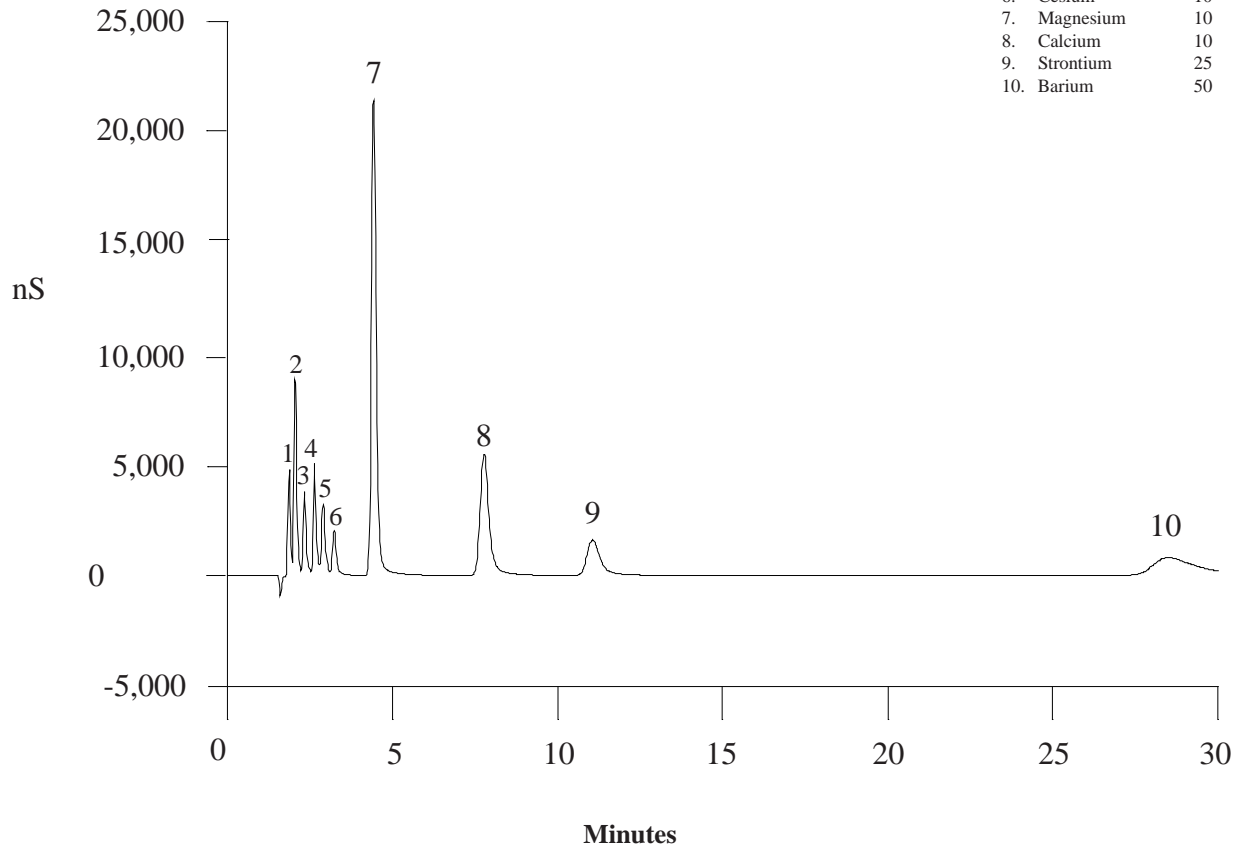


Figure 8
Rapid Elution of Lithium, Sodium, Ammonium, Potassium, Rubidium, Cesium, Magnesium, Calcium, Strontium and Barium

SECTION 6 - TROUBLESHOOTING

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the IonPac CS10 or the IonPac CS11 Analytical Column. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

Table 6
CS10/CG11 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown Component	Isolate Blockage	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2
	Plugged System Hardware	Unplug, Replace	Component Manual
High Background Conductivity and/or High Noise			
Improper Suppressor Operation	CSRS Not Suppressing	Check Current	6.3.1, Component Manual
		Check REGEN OUT Flow	6.3.1, Component Manual
		Check for leaks	6.3.1, Component Manual
	CMMS Not Suppressing	Check Regenerant	6.3.1, Component Manual
		Check AutoRegen Cartridge	6.3.1, Component Manual
		Air Bubbles	Back Pressure Coils or loosen fitting
Contamination	Bad Eluents	Remake Eluents	6.2
	Contaminated Column	Clean Column	6.3.3, Column Care
	Contaminated Suppressor	Clean Suppressor	6.3.1, 6.3.2
	Contaminated Hardware		6.3
Hardware Operation	Proportioning Valve	Service Valve	Component Manual
Poor Peak Resolution			
Poor Efficiency	Large System Void Volumes	Replumb System	6.5.1, Component Manual
	Sluggish Injection Valve	Service Valve	Component Manual
	Contaminated or Deformed Bed Support	Replace Bed Support	6.1.2
	Column Headspace	Replace Column	6.5.1 A
	Column Overloading	Reduce Sample Size	3.4
	Low sample pH	Reduce Sample Size	3.4
		Dilute Sample	6.6.2 D
Fronting Peaks	Low Sample pH	Reduce Sample Size	3.4
		Dilute Sample	6.6.2 D
	Column Overloading	Reduce Sample Size	3.4
	Contaminated or Deformed Bed Support	Replace Bed Support	6.1.2
	Column Headspace	Replace Column	6.5.1
Tailing Peaks	Contaminated Suppressor	Clean Suppressor	6.3.1, Component Manual
	Column Overloading	Reduce Sample Size	3.4
Short Retention Times	Flow Rate Too Fast	Recalibrate Pump	6.1.1 A
	First Peaks Elute Too Fast	Equilibrate to First Eluent	6.5.2 A
	Bad Eluents	Remake Eluents	6.5.2 B
	Column Contamination	Clean Column	6.5.3, Column Care
Spurious Peaks	Column Contamination	Pretreat Samples	6.5.3, 6.7
	Sluggish Injection Valve	Service Valve	6.7 C, Component Manual
Poor Quantification of Divalents	Sample Loop Contamination	Flush or Replace	

6.1 High Backpressure

6.1.1 Finding the Source of High System Pressure

Total system pressure when using either the IonPac CS10 Analytical Column and Guard (at 1.0 mL/min) or the IonPac CG11 2-mm Analytical Column and Guard (at 0.25 mL/min) should be less than 1,500 psi. Refer to Section 4.1.3, "Solvents," to see how solvent concentration can affect the column operating pressure. If the system pressure is higher than 2,000 psi, it is advisable to find out what is causing the high system pressure. The system should be used with a High-Pressure In-Line Filter (P/N 035331) for the eluents which is positioned between the 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. Find out what part of the system is causing the high pressure. It could be a piece of tubing that has plugged or whose walls are collapsed, an injection valve with a plugged port, a column with particulates plugging the bed support, a plugged High-Pressure In-Line Filter, the suppressor or the detector.

To find out 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. Continue adding the system's components (injection valve, column(s), suppressor and the detector) one by one, while watching the system pressure. The pressure should increase up to a maximum of 2,000 psi when the column(s) are connected. The suppressor will add up to 100 psi. No other components should add more than 100 psi of pressure. Refer to the appropriate manual for cleanup or replacement of the problem.

Table 7
CS10/CS11 Typical Operating Back Pressures

Column	Typical Back Pressure psi (MPa) at 30°C	Standard Flow Rate mL/min
CS10 Analytical (4 x 250 mm)	1,200 (8.27)	1.0
CS10 Guard (4 x 50 mm)	350 (2.41)	1.0
CS10 Analytical + Guard	1,550 (10.69)	1.0
CS11 Analytical (2 x 250 mm)	1,200 (8.27)	0.25
CS11 Guard (2 x 50 mm)	350 (2.41)	0.25
CS11 Analytical + Guard	1,550 (10.69)	0.25

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high backpressure, 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. Using two open end wrenches, carefully unscrew the inlet (top) column fitting.
- C. 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 CS10 4-mm Columns (P/N)	IonPac CS11 2-mm Columns (P/N)
Analytical Column	043015	043127
Guard Column	043016	043128
Bed Support Assembly	041375	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 High Background Noise

In a properly working system, the background conductivity level for the standard eluent system is shown below:

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
40 mM HCl/4 mM DAP	1-5 μ S
40 mM HCl/12 mM DAP	1-5 μ S
60 mM HCl/6 mM DAP	1-5 μ S

The background conductivity typically increases between 1 and 5 μ S when running a gradient. Ensure that the Cation Micromembrane Suppressor is operating properly. See Document No. 031728, "Product Manual for the Cation MicroMembrane Suppressor, CMMS III," or Document No. 031370, "Product Manual for the Cation Self-Regenerating Suppressor, CSRS ULTRA," for complete details.

A system with a high background will probably also have high noise, resulting in increased detection limits.

- A. Make sure that the eluents and regenerant are correctly formulated.
- B. Make sure that the eluents are made from chemicals with the recommended purity.
- C. Make sure that deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.
- D. Remove the column from the system. Is the background still high? 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 2 μ S.
- E. If the above items have been checked and the problem still persists, the suppression system is probably causing the problem. See the suppressor and AutoRegen Accessory Product Manuals for assistance.

6.3 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the suppressor and pump degassed, deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 μ S. If it is not, check the detector/conductivity cell calibration by injecting degassed, deionized water directly into it.

6.3.1 CSRS ULTRA 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 Product Manual (Document No. 031370) or to the Cation MicroMembrane Suppressor Product Manual (Document No. 031728) for detailed troubleshooting assistance.

- 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 in the CSRS ULTRA.
- D. Check the regenerant flow rate at the REGEN OUT port of the CSRS.** Turn the power to the CSRS off. Measure the regenerant flow rate. If it is being used in the recycle mode, it should be the same flow rate as the eluent (typically 1 mL/min for 4-mm operation). If it is used in the AutoSuppression External Water Mode, it should be at least 5 mL/min for non-solvent containing eluents. When solvents are used in the eluent, the regenerant flow rate should be at least 10 mL/min.
- E. Check the eluent flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. Refer to the Cation Self-

Regenerating Suppressor Product Manual (Document No. 031370) for assistance in determining if the eluent is within suppressible limits.

- F. If you are using an AutoRegen Accessory with the CMMS or 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.
- G.** All detector cells require enough back pressure to prevent eluent in the cell from degassing due to abrupt volume changes between the small inner diameter of the connecting tube and the relatively larger volume of the cell. Degassing creates bubbles in the cell and disrupts detector responsiveness. Back pressure coils help to prevent gases generated during AutoSuppression from coming out of solution and forming bubbles in the detector cell.

Back pressure coil components are located in the detector Ship Kits as part of the Gas Separator Waste Tube Assembly/ Back Pressure Coils Kit, P/N 045825; locate assembly P/N 045877. For 2-mm systems, the backpressure coils are available in the microbore tubing kit, P/N 052324; locate assembly P/N 045878. Alternatively, lengths and diameters of tubing necessary for proper back pressure are given in Table 8, “Coils for CSRS ULTRA Back Pressure Requirements” For further information, please consult the Product Manual for the Cation Self-Regenerating Suppressor, CSRS ULTRA (Document No. 031370).

- H.** To free air bubbles trapped in Suppressor, loosen fitting.

Table 8
Coils for CSRS ULTRA Back Pressure Requirements

CSRS ULTRA Size	Flow Rate mL/min	Tubing i.d.	Tubing Color	Length of Each Coil	Number of Coils
4-mm	0.5 - 1.5	0.010"	Black	2.5 feet	2
4-mm	1.5 - 3.0	0.010"	Black	2.5 feet	1
2-mm	0.12 - 0.25	0.005"	Red	1.0 foot	2
2-mm	0.25 - 0.75	0.005"	Red	1.0 foot	1

NOTE

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

6.3.2 CMMS III Not Suppressing Properly

- A. 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 MicroMembrane Suppressor Product Manual (Document No. 031728) for assistance in determining if the eluent is within suppressible limits.
- B. If you are using an AutoRegen Accessory with 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 CMMS.**
 - 2. If the background conductivity is low when freshly prepared regenerant is run through the 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.
- C.** All detector cells require enough back pressure to prevent eluent in the cell from degassing due to abrupt volume changes between the small inner diameter of the connecting tube and the relatively larger volume of the cell. Degassing creates bubbles in the cell and causes noise. Back pressure coils help to prevent gases from coming out of solution and forming bubbles in the detector cell.

Back pressure coil components are located in the detector Ship Kits as part of the Gas Separator Waste Tube Assembly/ Back Pressure Coils Kit, P/N 045825; locate assembly P/N 045877. For 2-mm systems, the backpressure coils are available in the microbore tubing kit, P/N 052324; locate assembly P/N 045878. Alternatively, lengths and diameters of tubing necessary for proper back pressure are given in Table 9, "Coils for CMMS III Back Pressure Requirements." For further information, please consult the Product Manual for the Cation MicroMembrane Suppressor, CMMS III (Document No. 031728).

- D.** To free air bubbles trapped in Suppressor, loosen fitting.

Table 9
Coils for CMMS III Back Pressure Requirements

CMMS III Size	Flow Rate mL/min	Tubing i.d.	Tubing Color	Length of Each Coil	Number of Coils
4-mm	0.5 - 1.5	0.010"	Black	2.5 feet	2
4-mm	1.5 - 3.0	0.010"	Black	2.5 feet	1
2-mm	0.12 - 0.25	0.005"	Red	6 inches	2
2-mm	0.25 - 0.75	0.005"	Red	6 inches	1

NOTE

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

6.3.3 Contaminated Cation Trap Column

If you are doing gradient analyses, has the Cation Trap Column, the CTC, been installed correctly? If it has not, install one as directed in Section 3.2 - Installing the Cation Trap Column, and watch the background conductivity. If the background conductivity is now low, this means that the CTC is trapping contaminants from the eluent. The eluents probably have too many impurities (see items A–C above).

If the CTC is already installed, remove it. Is the background conductivity still high? If the background conductivity decreases, the CTC is the source of the high background conductivity.

- A. Disconnect the CTC from the injection valve and direct the outlet to waste.
- B. Flush the CTC with 200 mL of 200 mM HCl at a flow rate of 2.0 mL/min.
- C. Equilibrate the CTC at 2.0 mL/min for 30 minutes with the strongest eluent used during the chromatographic run.
- D. If the problem persists, replace the CTC

6.3.4 Contaminated Guard or Analytical Column

Remove the IonPac CG10 or CG11 Guard and the IonPac CS10 or CS11 Analytical Column from the system. If the background conductivity still high, the column is the cause of the high background conductivity. Clean the column as instructed in "Column Care."

6.4 Eluent Problems

Also see Section 6.2, items A–C, for information on eluents.

6.4.1 Wrong Selectivity for Ammonia

The wrong DAP level in the eluent described in Section 4.4.2 can cause a shift in the location of ammonia relative to sodium and potassium. If this occurs, remake the eluent, being especially careful that the concentration of DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl) is correct.

6.5 Poor Peak Resolution

Poor peak resolution can be due to the loss of column or system efficiency. It can also be observed if the column loses capacity or selectivity.

6.5.1 Column Headspace

- A. Check to see if headspace has developed in the column (e.g., due to improper use of the column, such as submitting the column to high pressures). Remove the column's inlet end fitting from the column (see Section 5.1.2 for instructions). If the resin does not fill the column body completely to the top, the resin bed has collapsed, creating a headspace. If this is the case, the column will have to be replaced.
- B. Extra column effects can result in sample band dispersion making the peaks look less efficient. Make sure that you are using 0.010" i.d. tubing for 4-mm columns and 0.005" i.d. for 2-mm columns, and that the tubing lengths are as short as practically possible. Critical tubing lengths are from the injection valve to the column, from the column to the suppressor's **ELUENT IN** port, and from the **ELUENT OUT** port of the suppressor to the **CELL IN** of the detector. The tubing cuts should be straight and not at an angle. Make certain that the tubing goes beyond the ferrule before making the connection with the fitting. Once the tubings are correct, check for leaks. Leak-free operation is important for optimal 2-mm operation.

6.5.2 Shortened Solute Retention Times

Shortened solute retention times will compromise resolution. Potential reasons for shorter retention times are the following:

- A. Eluent flow rate is faster than 1.0 mL/min. Check the eluent flow rate after the analytical column.
- B. Incorrect eluent component ratio or concentration will change retention times. An eluent that is too concentrated will make the peaks elute sooner. 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 might not be accurate enough for this application. Use one reservoir containing the correct eluent composition to see if this is the problem.

6.5.3 Column Contamination

Column contamination can lead to a loss of column capacity because 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 (see "Column Care"). Possible sources of column contamination are:

- A. Gradient Mixers (GM-2) in the Gradient Pump Module with serial numbers before 6500 should be removed from the system when using eluents containing DL-2,3-diaminopropionic acid monohydrochloride (DAP-HCl) and replaced with mixers having serial numbers greater than 6500.
- B. Impurities in chemicals and in the deionized water used. 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. The installation of an IonPac Cation Trap Column between the pump and the injection valve is highly recommended for all cation gradient analyses. The CTC-1 strips the eluent of strongly retained 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.

6.5.4 Diluting Eluents

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 Care").

After cleaning the column, install it in the system, and let it equilibrate with eluent for about 30 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should have been restored by this treatment, since the contaminant metals should have been eluted from the column.

6.6 Poor Peak Resolution

6.6.1 Loss of Column Efficiency

- A. Check to see if headspace has developed in the guard or analytical column (e.g., 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 tubing with an i.d. of no greater than 0.012", in all cases, between the injection valve and the detector cell inlet, and that the tubing lengths are as short as possible. Check for leaks. Leak-free operation is especially important for optimal 2-mm operation.

6.6.2 Poor Resolution 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 to see if the eluent flow rate is not faster than 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 eluents' composition and concentration are correct. An eluent that is too strong will cause the peaks to elute sooner. 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 because all of the anion exchange sites will no longer be available for the sample ions. Polyvalent anions might be concentrating on the column. Refer to - Column Care - for recommended column cleanup procedures.

Possible sources of column contamination are impurities in chemicals and in the deionized water being used. 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 Care"). After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. 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.

6.6.3 Loss of Front End Resolution

If poor resolutions and efficiencies are observed for the very early eluting peaks near the system void volume compared to the later eluting peaks, check the following:

- A. Improper eluent E1 concentration may be the problem. Remake the eluent.
- B. The wrong DAP level in the eluent, can cause a shift in the location of ammonia relative to sodium and potassium. If this occurs, remake the eluent, being especially careful that the concentration of DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl) is correct. See Table 5.
- B. Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.
- C. Improperly swept out volumes anywhere in the system prior to the guard and analytical columns may be the problem. See Section 6.1.1 - Finding the Source of High System Pressure.
- D. 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.

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 4.1.2, “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.

For DX-300 systems equipped with a Rheodyne Microinjection Valve, Model 9126 (Dionex P/N 044697), consult the accompanying manual for service instructions.
