# PRODUCT MANUAL

## IonPac® SCS 1
## IonPac® SCG 1

DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

# PRODUCT MANUAL

**for the**

**IONPAC® SCG 1 Guard Column**
**2 x 50 mm, P/N 061522**
**4 x 50 mm, P/N 061523**

**IONPAC® SCS 1 Analytical Column**
**2 x 250 mm, P/N 061520**
**4 x 250 mm, P/N 061521**

©Dionex Corporation, 2006
Document No. 031948-05
August 2006

# TABLE OF CONTENTS

## SECTION 1 - INTRODUCTION

The IonPac SCS 1 analytical column should only be used with nonsuppressed conductivity detection, or Single Column Ion Chromatography (SCIC), for the analyses of the common inorganic cations such as Lithium, Sodium, Ammonium, Potassium, Magnesium and Calcium, as well as some amines such as Ethanolamine.

**WARNING 1:** The SCS 1 columns will contaminate the eluent suppressor and should therefore not be used with a suppressor in line.

**WARNING 2:** The SCS 1 column may contaminate the tubing after the column and the conductivity cell. If the system is used with suppressed conductivity, after having been used with the SCS 1 column, you should clean or replace the tubing and the conductivity cell. Refer to the troubleshooting section for further details.

***NOTE:*** ***The use of the EG40/50 Eluent Generator with this column is not recommended because noise will be much higher than when using pre-made eluent from a single bottle.***

The SCS 1 stationary phase is a low capacity weak cation exchanger functionalized with carboxylic acid groups. Its substrate is 4.5 µm silica, and therefore the pH of the eluents with which it can be used is limited to between pH 2 and 7. It is compatible with typical HPLC organic solvents such as acetonitrile, THF, and acetone. Alcohols should be avoided as eluent components as they will form esters in the SCS 1 column, thus reducing the cation exchange capacity of the column.

The SCS 1 columns can be used without loss of performance at 30 °C with a 3 mM methanesulfonic acid (pH 2.5) eluent. Prolonged use at temperatures higher than 35 °C may deteriorate the column.

The SCS 1 4-mm column should only be used on the following chromatographic systems: ICS 2000, ICS 1500, and ICS 1000, with a heater option. The SCS 1 2-mm column should be used on the following chromatographic systems: ICS 2500 IC System and the DX-800 Process Analyzer. An IonPac Mixer is required to minimize system noise levels. These columns should be used isocratically with eluent delivered from a bottle. The use of the EG40/50 Eluent Generator with this column is not recommended because noise will be much higher than by using pre-made eluent from a single bottle. Methods that require eluent gradients or proportioning from two or more eluent bottles are not recommended because without a suppressor the background change and the noise are very high, making quantitation difficult.

The expected background conductivity under the “Standard Operating Conditions” of the SCS 1 column is typically between 950 and 1100 µS. At this background level the sample peaks have lower conductance than the background, and are displayed as negative peaks. To display positive peaks, the “Conductivity Polarity” in the Detector screen should be set to “INVERTED.” Use of the “AUTOZERO” command in the program will set the background to “zero” at the beginning of a run when collecting data.

The Standard Operating Conditions for a 4 mm column is 3 mM methanesulfonic acid at 30 °C, flowing at 1 mL/min or 0.25 mL/min.

### 1.1 Column Packing Specifications (Table 1)

**Table 1**
**IonPac SCS 1/SCG 1 Packing Specifications**

| Column | Particle Diameter µm | Column Capacity µeq/column | Functional Group | Hydrophobicity |
|---|---|---|---|---|
| SCS 1 (2 x 250 mm) | 4.5 | 80 | Carboxylic acid | Medium |
| SCG 1 (2 x 50 mm) | 4.5 | 16 | Carboxylic acid | Medium |
| SCS 1 (4 x 250 mm) | 4.5 | 318 | Carboxylic acid | Medium |
| SCS 1 (4 x 50 mm) | 4.5 | 63 | Carboxylic acid | Medium |

### 1.1 Column Packing Specifications (Table 2)

**Table 2**
**SCS 1/SCG 1 Operating Parameters**

| Column | Typical Back Pressure at Standard Flow Rate psi (MPa) | Standard Flow Rate mL/min | Maximum Flow Rate mL/min |
|---|---|---|---|
| SCS 1 2-mm Analytical | ≤ 1,900 (13.09) | 0.25 | 0.40 |
| SCG1 2-mm Guard | ≤ 400 (2.75) | 0.25 | 0.40 |
| **SCS 1 + SCG 1 2-mm columns** | **≤ 2,300 (15.85)** | **0.25** | **0.40** |
| SCS 1 4-mm Analytical | ≤ 1,900 (13.09) | 1.0 | 1.60 |
| SCG 1 4-mm Guard | ≤ 400 (3.44) | 1.0 | 1.60 |
| **SCS 1 + SCG 1 4-mm columns** | **≤ 2,400 (16.54)** | **1.0** | **1.60** |

***NOTE:*** ***Column backpressures are measured by connecting the column directly to the pump and the column effluent goes directly to waste.***

**Read the system manuals.** This manual assumes that you are familiar with the installation and operation of the Dionex Ion Chromatograph (IC). If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis. All instrument manuals are available on the Dionex Reference Library CD-ROM (P/N 053891) supplied with this column.

**Assistance is available for any problem 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, P/N 053891).**

# SECTION 2 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

## 2.1 Configuration (Table 3)

**Table 3**
**Configuration**

| CONFIGURATION | 2-mm | 4-mm |
|---|---|---|
| **IC System** | ICS-2500<br>DX-800 | ICS-2000<br>ICS-1500<br>ICS-1000 (with heater option) |
| **Column Heater** | Required | Required |
| **Mixer** | Required | Required |

## 2.2 Tubing Back Pressures (Table 4)

**Table 4**
**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 - IONPAC SCS 1 AND IONPAC SCG 1 QUICKSTART

**WARNING: Be sure to wash new SCS 1 or SCG 1 columns (and columns which have not been used for more than 4 weeks) before connecting to an Ion Chromatography (IC) system.**

## 3.1 Column Washing Steps

Follow these steps to wash SCS 1 or SCG 1 columns.

1. Connect the inlet of the column (SCS 1 or SCG 1), one column at a time, to the pump, with the outlet to waste.

2. With the pump set at an initial reduced flow rate (see Table 5), pump 3 mM MSA eluent through the column to waste for approximately 30 minutes.

**WARNING: Failure to do this initial wash at low flow rates may result in very high pressure on the column. This could damage the column or result in high background levels.**

3. Increase the flow rate slowly to the standard flow rate so that the backpressure of the column does not exceed its recommended maximum column backpressure (see Table 5). Standard backpressures should appear, during operation at standard flow rates, after no more than 90 minutes for separator columns and 20 minutes for guard columns.

## 3.2 Start-up Parameters (Table 5)

**TABLE 5**
**IonPac SCS 1 and IonPac SCG 1 Start-up Parameters**

| Column | Initial Reduced Flow Rate mL/min | Recommended Maximum Column Backpressure Psi (MPa) | Standard Flow Rate mL/min | Standard Backpressure Psi (MPa) |
|---|---|---|---|---|
| SCS 1 2x250 mm | 0.1 | ≤3000 (20.67) | 0.25 | ≤1900 (13.09) |
| SCG 1 2x50 mm | 0.1 | ≤900 (6.20) | 0.25 | ≤550 (3.79) |
| SCS 1 4x250 mm | 0.4 | ≤3000 (20.67) | 1.0 | ≤1900 (13.09) |
| SCG 1 4x50 mm | 0.4 | ≤900 (6.20) | 1.0 | ≤550 (3.79) |

# SECTION 4 - INSTALLATION

## 4.1 System Requirements

### 4.1.1 System Requirements for 2-mm Operation

The IonPac SCG 1 2-mm Guard Column (P/N 061522) and Analytical Column (P/N 061520) are designed to be run on the Dionex ICS-2500 Ion Chromatograph or the DX-800 Process Analyzer. See the ICS-2500 or the DX-800 Operator's Manual. All plumbing from the injection valve to the column and from the column to the cell **INLET** should be done with 0.005" i.d. RED PEEK tubing, see Table 3.

### 4.1.2 System Requirements for 4-mm Operation

The IonPac SCG 1 4-mm Guard Column (P/N 061523) and SCS 1 Analytical Column (P/N 061521) are designed to be run on Dionex ICS Ion Chromatographs equipped with 4-mm Precell Heat Exchanger. See the ICS 1000, 1500, 2000 Operator's Manual. All plumbing from the injection valve to the column and from the column to the cell **INLET** should be done with 0.010" i.d. BLACK PEEK tubing, see Table 3.

### 4.1.3 System Requirements for 4-mm and 2-mm Operation

The Dionex IonPac Mixer (Part Number 063443) is required for use with 2-mm and 4-mm SCS 1 columns. The IonPac Mixer is placed before the eluent inlet of the injection valve. This mixer “averages” or “homogenizes” any eluent concentration changes due to temperature or pump pulsations. The eluent is “mixed” in this device before reaching the column. It is thus instrumental in minimizing the background noise.

## 4.2 Installing the CTC-1 Cation Trap Column

To remove cationic impurities from the eluent, if required, an IonPac Cation Trap Column (CTC) (4-mm P/N 040192) is installed between the pump and the injection valve. The CTC is filled with high capacity cation exchange resin which traps cationic contaminants present in the eluent. To install the CTC complete the following steps:

a. Connect the gradient pump directly to the CTC
b. Connect a waste line to the CTC outlet
c. Connect a direct the line to a waste container
d. Flush the CTC.

***NOTE:*** ***With the guard and analytical columns out of line, there is no need for flow rate restrictions. Use 50 mL of a 10x eluent concentrate of the strongest eluent required by the application at a flow rate of 2.0 mL/min.***

e. Rinse the CTC with eluent
f. Reconnect the CTC
g. Connect the CTC to the eluent line that is connected to the injection valve inlet

## 4.3 The Injection Loop

For most applications on a 2-mm analytical system, a 2.5 to 50 µL injection loop will be sufficient. When samples are unknown and of expected varied concentrations, Dionex recommends that a 6.25 µL injection loop be used to avoid overloading the 2 mm Analytical Column. Generally, do not inject more than 1 nanomole (8–55 ppm) of any one analyte onto the 2-mm analytical column. Injecting larger volumes of samples can result in overloading the column, affecting peak symmetry and detection linearity. This phenomenon will be more prevalent at higher concentrations of the analytes of interest.

4.3.1 The IonPac Mixer Use Instructions

***NOTE 1:*** ***The IonPac Mixer (P/N 063443) is completely assembled and ready for use.***

***NOTE 2:*** ***The IonPac Mixer is designed to be used with plastic fittings only.***

a. Use finger tight fittings to connect the tubing to both ends of the IonPac Mixer.
b. To replace the frit (P/N 062781) in the IonPac Mixer
   1. Disconnect the tubing
   2. Unscrew one filter end fitting
   3. Remove the old frit
   4. Place a new frit into the cavity of this end fitting
   5. Re-tighten the filter end fitting with a wrench, ¼ turn past hand tight
   6. Reconnect the tubing.

## 4.4 Sample Concentration

The TCC-LP1, P/N 046027, should be used for trace cation pre-concentration work on the 4-mm and 2-mm SCS 1 columns when the eluent consists only of an acid (such as methanesulfonic acid, tartaric acid, or oxalic acid) in water. If PDCA (pyridine-2, 6-dicarboxylic acid) needs to be added to the eluent, then the respective 2-mm or 4-mm SCG 1 Guard Column should be used for pre-concentration.

***NOTE 1:*** ***MSA, oxalic acid, tartaric acid, or PDCA may be used with the SCG 1 column.***

***NOTE 2:*** ***See Section 4.5, "Sample Concentration" for details on sample concentration.***

## 4.5 IonPac SCG 1 Guard Columns

An IonPac SCG 1 Guard Column is normally used with the IonPac SCS 1 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. Cleaning or replacing a guard column is more economical than replacing an analytical column. For maximum life of the analytical column, the guard column should be changed or replaced as part of a regular maintenance schedule, or at the first sign of performance deterioration. Use the test chromatogram that is shipped with the analytical column, or the initial application run, as a performance benchmark.

## 4.6 Eluent Storage

The column's storage solution should be 3 mM MSA. 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 and cap both ends securely, using the plugs supplied with the column.

# SECTION 5 - OPERATION

## 5.1 General Operating Conditions

| | |
|---|---|
| Column: | SCS 1 2-mm Analytical Column (+ SCG 1 2-mm Guard Column)<br>SCS 1 4-mm Analytical Column (+SCG 1 4-mm Guard Column) |
| Sample Volume: | 6.2 µL Loop + 0.8 µL Injection valve dead volume (2-mm)<br>25 µL Loop (4-mm) |
| Eluent: | 3 mM Methanesulfonic acid (MSA) |
| Eluent Flow Rate: | 0.25 mL/min (2-mm), 1.0 mL/min (4-mm) |
| Temperature: | 30 °C |
| Expected Background Conductivity: | 950–1100 µS |
| Storage Solution: | Eluent |

## 5.2 IonPac SCS 1 Operating Precautions

1. Operate below 4,000 psi (27.57 MPa)
2. Filter and degas all eluents
3. Filter all samples
4. Do NOT use in-line with a suppressor!

## 5.3 Chemical Purity Requirements

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

### 5.3.1 Deionized (DI) Water

The DI water used to prepare eluents should be Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The DI 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.

### 5.3.2 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. Fluka or Aldrich Methanesulfonic Acid (MSA) >99% pure, or Dionex Methanesulfonic Acid Concentrate (0.4 M) P/N 057562, or Dionex Methansulfonic Acid (15.4 M) P/N 033478.
b. Use DI water with a specific resistance of 18.2 megohm-cm to make all standards and eluents.

5.3.3 Eluents with Solvents

Solvents can be added to the ionic eluents used with IonPac SCS 1 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 make ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific. When using a solvent in an ionic eluent, column back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-acetonitrile mixture varies. The practical back pressure limit for the IonPac SCS 1 columns is 4,000 psi (27.57 MPa). The IonPac SCS 1 is compatible with the HPLC solvents listed in Table 6, "HPLC Solvents for Use with the SCS 1 Columns." Solvents and water should be premixed in concentrations which allow proper mixing by the pump and to minimize out gassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

***NOTE 1:*** ***At a characteristic concentrate range of organic solvent concentration in the eluent, the column backpressure may more than double. If this is the case, you should decrease the eluent flow rate to allow use of the eluent containing solvent in this concentration range.***

***NOTE 2:*** ***Do NOT use alcohols with the SCS 1 column.***

**Table 6 - HPLC Solvents for Use with IonPac SCS 1 Columns**

| Solvent | Maximum Operating Concentration |
|---|---|
| Acetonitrile | 100% |
| Acetone | 100% |
| Tetrahydrofuran | 20% |

## 5.4 Making and Using Eluents that Contain Solvents

5.4.1 Mixing Eluents

When mixing solvents with water, remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 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 out gassing.

5.4.2 Purging or Degassing Eluents

When purging or degassing eluents containing solvents, do not purge or degas the eluent excessively. 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.

5.4.3 Avoiding High Viscosity Pressure Fronts

Avoid creating high viscosity pressure fronts that may disrupt the column packing when the eluent solvent component is changed. To do this, wash the column to waste for approximately 15 minutes with an eluent containing 10% of the new solvent type. Exchange this eluent for the final desired eluent ion concentration and composition, and let the column wash to waste for 15 minutes before re-connecting.

5.4.4 Changing to a Solvent-Free Eluent System after Using Eluents Containing Solvent

Properly equilibrate the column when changing to a solvent-free eluent system after using eluents containing solvent. First equilibrate the column with 5 percent of the current solvent for approximately 15 minutes. Exchange this eluent for the new solvent free aqueous eluent.

## 5.5 Sample Concentration

The IonPac SCG 1 Guard Column or the Low-Pressure Trace Cation Concentrator, TCC-LP1, should be used for trace cation concentration. Trace cation concentrators are used primarily in high purity water analysis. The function of the trace cation concentrator in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This can be accomplished by replacing the sample loop with the concentrator column, then pumping (and concentrating) large volumes of the sample onto a concentrator column. The sample should be pumped into the concentrator column in the opposite direction of the eluent flow; otherwise the chromatography will be compromised. This process "concentrates" all cationic analyte species onto the trace cation concentrator (the TCC-LP1 or the SCG 1) leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the trace cation concentrator (TCC-LP1 or the SCG 1) for the analytical chemist in these applications is the capability of performing routine trace analyses of sample matrix ions at ng/L levels without extensive and laborious sample pretreatment.

The IonPac SCG 1 4-mm Guard Column (P/N 061523), or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027), should be used for sample concentration with the IonPac SCS 1 4-mm Analytical Column.

The IonPac SCG 1 2-mm Guard Column (P/N 061522), or the Low-Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027), should be used for sample concentration with the IonPac SCS 1 2-mm Analytical Column. The advantage of the TCC-LP1 is that because of its low backpressure, samples can be preconcentrated using a hand-held syringe.

**WARNING 1: The Trace Cation Concentrator (TCC-2, P/N 043103) should not be used for sample concentration with the SCS 1 column. The TCC-2 column packing is a strong cation exchange resin functionalized with sulfonic acid. The recommended IonPac SCS 1 eluents will not properly elute ions concentrated on this column.**

**WARNING 2: Do NOT use the TCC-LP1 for pre-concentration when the eluent contains PDCA (pyridine-2, 6-dicarboxylic acid), also called dipicolinic acid. In this case, use the SCG 1 Guard Column. The PDCA is retained in the TCC-LP1, its elution later causes a "hump" in the baseline which may interfere with the analyte peaks.**

# SECTION 6 - EXAMPLE APPLICATIONS

## 6.1 Determination of Trace Ammonium in the Presence of High Concentration of Sodium

An important application for the environmental industry is the ability to determine trace concentrations of ammonium in the presence of high concentrations of sodium. Figure 1 illustrates the determination of trace level ammonium in the presence of high sodium using 3 mM MSA eluent on the SCS 1 column. The maximum ratio determined for this column is 1,000:1 sodium to ammonium.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 3 mM Methanesulfonic acid (MSA) |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Loop: | 25 µL |
| Detection: | Nonsuppressed conductivity |
| Background: | ~1100 µS |

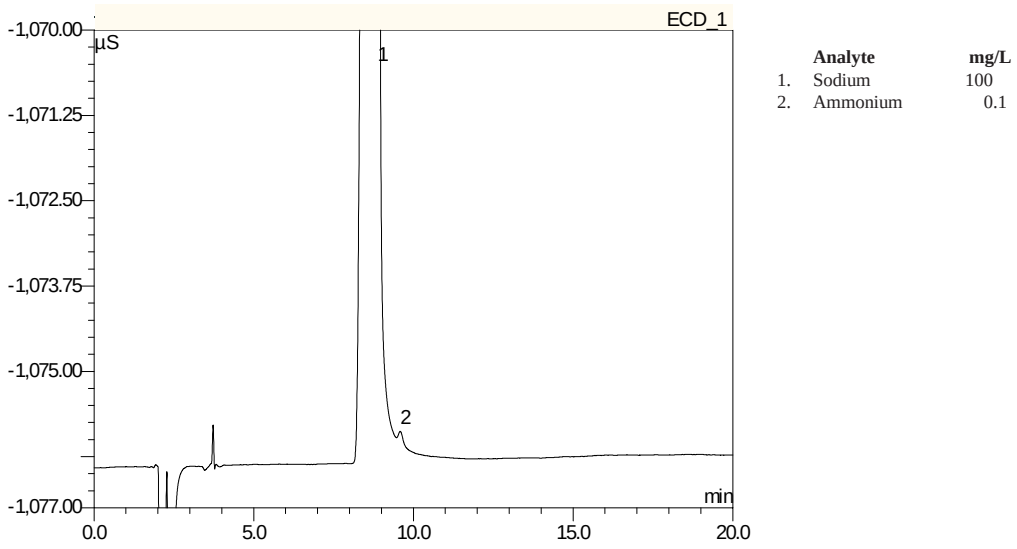



**Figure 1**
**Determination of Trace Ammonium in the Presence of High Concentration of Sodium**

## 6.2 Determination of Trace Sodium in the Presence of High Concentration of Ammonium

An important application for the power industry is the ability to determine trace concentrations of sodium in the presence of high concentrations of ammonium. The sample contained 1000 ppb of ammonium and was spiked with 1 ppb of sodium. A 1.5 mL sample was loaded onto a TCC-LP1 concentrator column using a DXP single piston pump. Figure 2 illustrates the determination of trace level sodium in the presence of high ammonium using the SCS 1 column. The sodium peak is well resolved from the ammonium peak using 3 mM MSA eluent. Also, the peak shape for the trace sodium is very good.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 3 mM MSA |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| DXP Flow Rate | 2 mL/min |
| Sample Volume: | 1.5 mL |
| Concentrator Column: | TCC-LP1 |
| Detection: | Nonsuppressed conductivity |
| Background: | ~1100 µS |

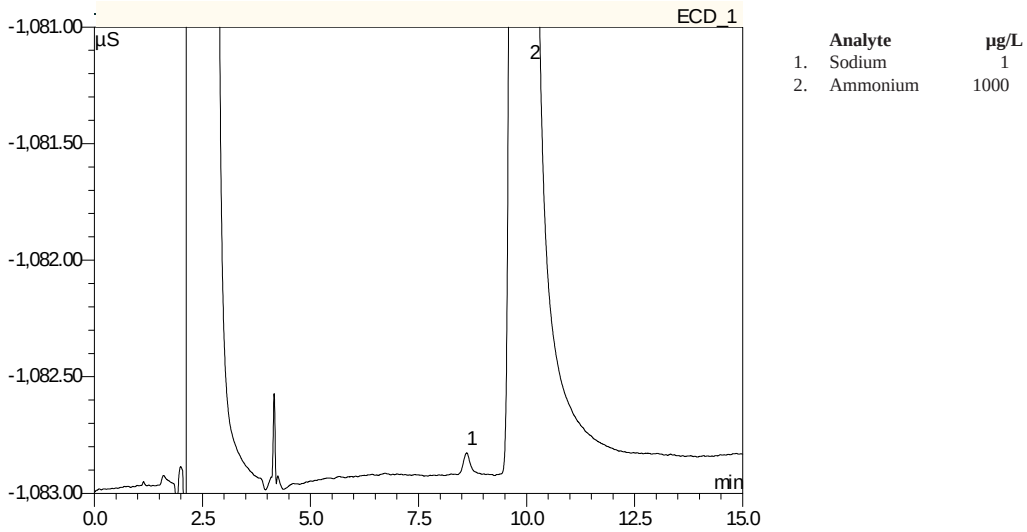



**Figure 2**
**Determination of Trace Sodium in the Presence of High Concentration of Ammonium**

## 6.3 Determination of Trace Sodium in the Presence of High Concentration of Ethanolamine

An important application for the power generation industry is the ability to determine trace concentrations of sodium in the presence of high concentrations of ethanolamine. Figure 3 shows a simulated sample which contains 3000 ppb ethanolamine spiked with 0.250 ppb sodium. A 3.0 mL sample was loaded onto a TCC-LP1 concentrator column using a Dionex DXP single piston pump. The sodium peak is well resolved from the ethanolamine peak using 3 mM MSA eluent on the SCS 1 column.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 3 mM MSA |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| DXP Flow Rate: | 2 mL/min |
| Sample Volume: | 3 mL |
| Concentrator Column: | TCC-LP1 |
| Detection: | Nonsuppressed conductivity |
| Background: | ~1100 µS |

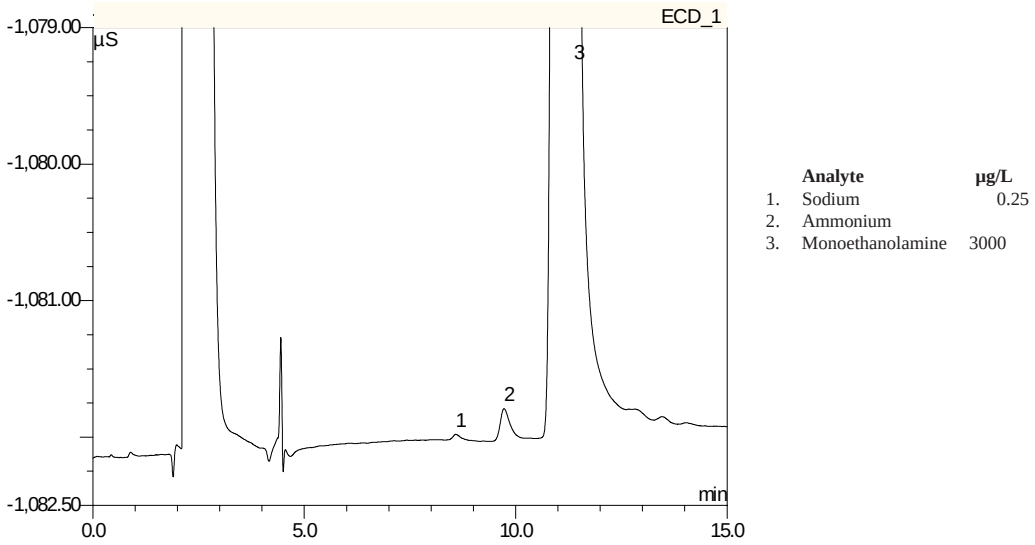



**Figure 3**
**Determination of Trace Sodium in the Presence of High Concentration of Ethanolamine**

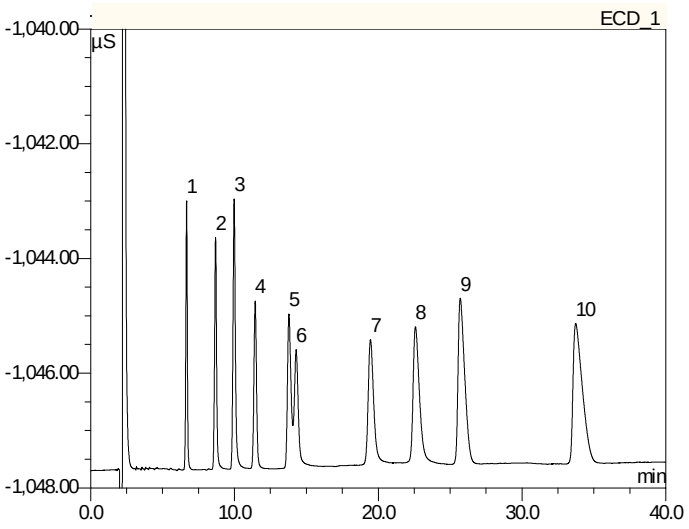
## 6.4 Separation of the Common Inorganic Cations and Alkanolamines

Alkanolamines including monoethanolamine, diethanolamine, and triethanolamine are most commonly used individually or in any combination to optimize the efficiency of the scrubber treatment for a specific chemical process. In large plants, different alkanolamines may be used in adjacent units as tracers for leakage problems. The SCS 1 resolves all combinations of these priority scrubber amines using a 3 mM MSA eluent as illustrated in Figure 4.

***NOTE:*** ***Under these conditions potassium and diethanolamine are not completely resolved. Separation can be improved by adding solvent (see Figure 5 in section 5.5) or 18-crown-6 to the eluent.***

***NOTE:*** ***Even a small amount of 18-crown-6 can significantly increase the retention time of potassium.***

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 3 mM MSA |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Loop: | 25 µL |
| Detection: | Nonsuppressed conductivity |
| Background: | ~1050 µS |



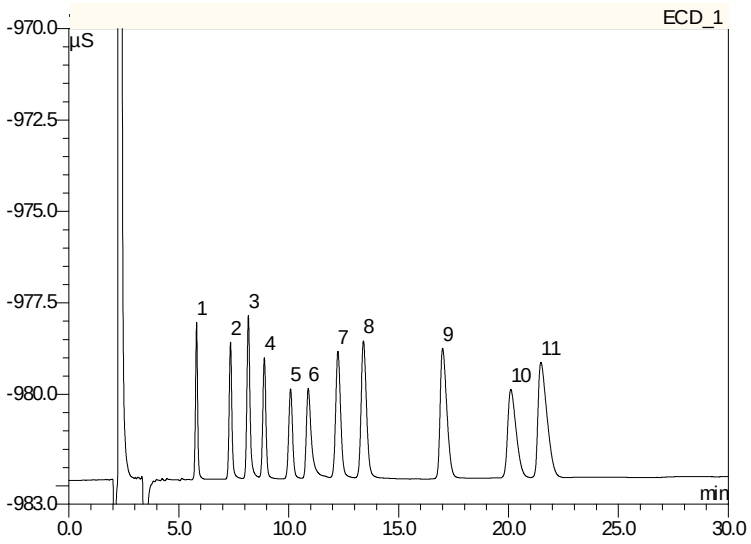

| | Analyte | mg/L |
|---|---|---|
| 1. | Lithium | 0.5 |
| 2. | Sodium | 2 |
| 3. | Ammonium | 2.5 |
| 4. | Monoethanolamine | 5 |
| 5. | Potassium | 5 |
| 6. | Diethanolamine | 10 |
| 7. | Triethanolamine | 20 |
| 8. | Methyldiethanolamine | 20 |
| 9. | Magnesium | 2.5 |
| 10. | Calcium | 5 |

**Figure 4**
**Separation of the Common Inorganic Cations and Alkanolamines**

## 6.5 Separation of the Common Inorganic Cations, Alkanolamines and DIPA

This chromatogram shows the separation of the common inorganic cations, alkanolamines and diisopropylamine (DIPA). In Figure 4 of Section 6.4, potassium and diethanolamine are not resolved. By adding 10% acetonitrile to the 3 mM MSA eluent, potassium and diethanolamine are baseline resolved. Also, peak efficiencies are improved for the divalent inorganic cations and amines.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 3 mM MSA/10% ACN |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Loop: | 25 µL |
| Detection: | Nonsuppressed conductivity |
| Background: | ~980 µS |



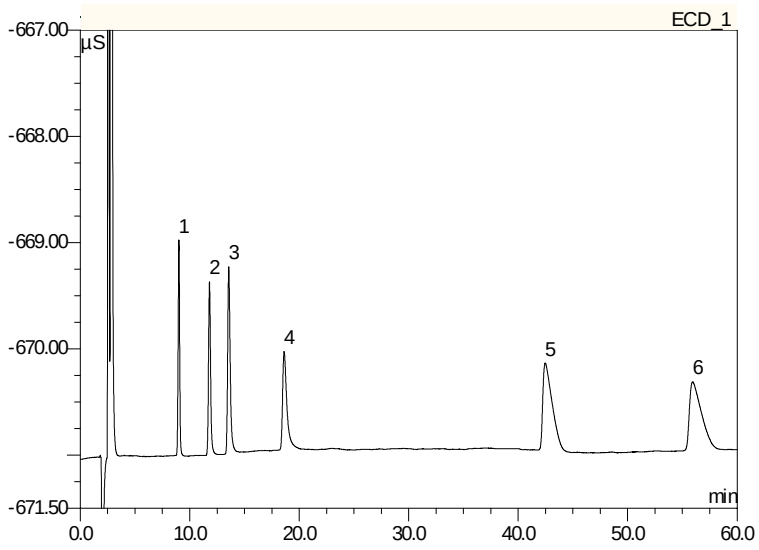

| | Analyte | mg/L |
|---|---|---|
| 1. | Lithium | 0.5 |
| 2. | Sodium | 2 |
| 3. | Ammonium | 2.5 |
| 4. | Monoethanolamine | 5 |
| 5. | Diethanolamine | 10 |
| 6. | Potassium | 5 |
| 7. | Triethanolamine | 20 |
| 8. | Methyldiethanolamine | 20 |
| 9. | Magnesium | 2.5 |
| 10. | Diisopropylamine | 20 |
| 11. | Calcium | 5 |

**Figure 5**
**Separation of the Common Inorganic Cations, Alkanolamines and DIPA**

## 6.6 Determination of the Common Inorganic Cations Using a Tartaric Acid/PDCA Eluent

Figure 6 shows the separation of common inorganic cations using an alternate eluent consisting of 4 mM tartaric acid and 0.75 mM PDCA. Compared to the recommended 3 mM MSA eluent, the significant increase in run times results from the higher capacity of the SCS 1 column. The optimum eluent for the SCS 1 column is 3 mM MSA as specified in the previous examples.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 4 mM tartaric acid/ 0.75 mM PDCA |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Loop: | 25 µL |
| Detection: | Nonsuppressed conductivity |
| Background: | ~670 µS |



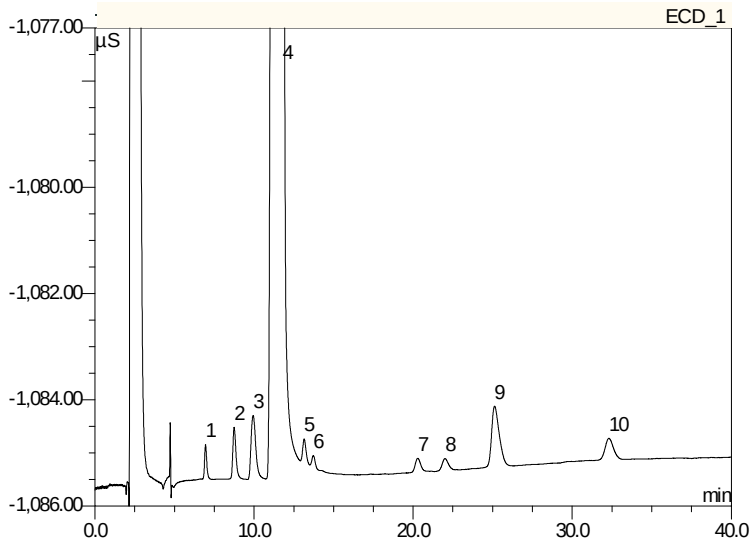

| | Analyte | mg/L |
|---|---|---|
| 1. | Lithium | 0.5 |
| 2. | Sodium | 2 |
| 3. | Ammonium | 2.5 |
| 4. | Potassium | 5 |
| 5. | Calcium | 2.5 |
| 6.. | Magnesium | 5 |

**Figure 6**
**Determination of the Common Inorganic Cations Using a Tartaric Acid/PDCA Eluent**

## 6.7 Separation of Common Inorganic Cations, Alkanolamines, and Transition Metals

Figure 7 shows an example of a 3 mL simulated feedwater sample containing 7 ppm ethanolamine spiked with sub-ppm levels of common cations, diethanolamine, zinc, cobalt, and manganese. The majority of the cations are well separated, with the exception of magnesium and manganese. For this separation, the eluent is modified to contain 0.8 mM oxalic acid and 2.5 mM MSA to achieve the appropriate selectivity for the transition metals.

| | |
|---|---|
| Column: | IonPac SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 2.5 mM MSA /0.8 mM oxalic acid |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Volume: | 3 mL |
| Concentrator Column: | TCC-LP1 |
| Detection: | Nonsuppressed conductivity |
| Background: | ~1100 µS |



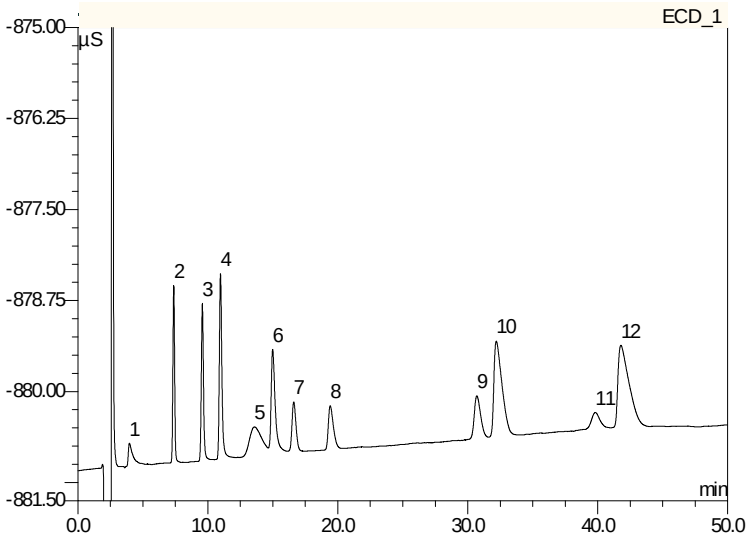

| | Analyte | µg/L |
|---|---|---|
| 1. | Lithium | 0.76 |
| 2. | Sodium | 6.9 |
| 3. | Ammonium | 6.9 |
| 4. | Monoethanolamine | 7000 |
| 5. | Potassium | 5.6 |
| 6. | Diethanolamine | 6.9 |
| 7. | Zinc | 7.1 |
| 8. | Cobalt | 6.6 |
| 9. | Magnesium | 9.3 |
| | Manganese | 6.9 |
| 10. | Calcium | 7.2 |

**Figure 7**
**Separation of Common Inorganic Cations, Alkanolamines, and Transition Metals**

## 6.8 Separation of the Common Inorganic Cations and Transition Metals

Figure 8 shows a separation of the six transition metals and common cations in a single analysis at ppm concentrations using a 25 µL loop injection. The eluent was modified to 4.0 mM tartaric acid and 2.0 mM oxalic acid to achieve the selectivity shown in the example.

| | |
|---|---|
| Column: | SCG 1 Guard Column (4-mm) + IonPac SCS 1 Analytical Column (4-mm) |
| Eluent: | 4 mM Tartaric acid /2 mM Oxalic acid |
| Eluent Flow Rate: | 1 mL/min |
| Temperature: | 30 °C |
| Sample Loop: | 25 µL |
| Detection: | Nonsuppressed conductivity |
| Background: | ~880 µS |




| | Analyte | mg/L |
|---|---|---|
| 1. | Copper | 10 |
| 2. | Lithium | 0.5 |
| 3. | Sodium | 2 |
| 4. | Ammonium | 2.5 |
| 5. | Nickel | 10 |
| 6 | Potassium | 5 |
| 7. | Zinc | 10 |
| 8. | Cobalt | 10 |
| 9. | Manganese | 10 |
| 10. | Magnesium | 2.5 |
| 11. | Cadmium | 10 |
| 12. | Calcium | 5 |

**Figure 8**
**Separation of the Common Inorganic Cations and Transition Metals**

## SECTION 7 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac SCS 1 columns. For more information on problems that originate with the Ion Chromatograph (IC), 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" on the Dionex Reference Library CD-ROM, P/N 053891).

**Table 7**
**SCS 1/SCG 1 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** | | | |
| **Contamination** | Bad Eluents | Remake Eluents | 6.2, 6.4 |
| | Contaminated Column | Clean Column | 6.3.1, Column Care |
| | Cell | Check Cell Calibration | |
| **Poor Peak Resolution** | | | |
| **Poor Efficiency** | Large System Void Volumes | Replumb System | 6.6.3.B, Component Manual |
| | Sluggish Injection Valve | Service Valve | 6.6.3.A, Component Manual |
| | Contaminated or Deformed Bed Support | Replace Bed Support | 6.1.2 |
| | Column Headspace | Replace Column | 6.6.1.A |
| | Column Overloading | Reduce Sample Size | |
| | Low sample pH | Reduce Sample Size<br>Dilute Sample<br>Use OnGuard II A | |
| | Tubing and Cell | Clean and/or replace tubing | 6.3.3 |
| **Fronting Peaks** | Low Sample pH | Reduce Sample Size<br>Dilute Sample<br>Use OnGuard II A | |
| | Column Overloading | Reduce Sample Size | 5.18 |
| | Contaminated or Deformed Bed Support | Replace Bed Support | 6.1.2 |
| | Column Headspace | Replace Column | 6.6.1.A |
| **Tailing Peaks** | | | |
| | Column Overloading | Reduce Sample Size | |
| | Column Contaminated | Clean Column | Column Care B.4.2 |
| | Tubing and Cell | Clean and/or replace tubing | 6.3.3 |
| **Short Retention Times** | Flow Rate Too Fast | Recalibrate Pump | 6.6.2.A |
| | Bad Eluent | Remake Eluent | 6.6.2.B |
| | Column Contamination | Clean Column | Column Care B.4.2 |
| **Spurious Peaks** | Column Contamination | Pretreat Samples, Clean column | 6.3.1, 6.6 |
| | Sluggish Injection Valve | Service Valve | 6.7.C, Component Manual |
| **Poor Quantification of Divalents** | Sample Loop Contamination | Flush or Replace | 6.3.2 |
| | Contaminated Cell | Clean Cell | |

## 7.1 High Back Pressure

### 7.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac SCG 1 Guard Column plus the SCS 1 Analytical Column when using the test chromatogram conditions should be equal or less than 2,400 psi (16.54 MPa), see Appendix A. If the system pressure is higher than 2,400 psi, determine the cause of the high pressure immediately. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) followed by a CTC-1, if your eluents require it.

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, 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), 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 SCS 1/SCG 1 Operating Back Pressures”.

**Table 8**
**Typical SCS 1/SCG 1 Operating Back Pressures**

| Column | Typical Back Pressure at Standard Flow Rate psi (MPa) | Standard Flow Rate mL/min | Maximum Flow Rate mL/min |
|---|---|---|---|
| SCS 1 2-mm Analytical | ≤ 1,900 (13.09) | 0.25 | 0.40 |
| SCG 1 2-mm Guard | ≤ 400 (2.75) | 0.25 | 0.40 |
| **SCS 1 + SCG 1 2-mm columns** | **≤ 2,300 (15.85)** | **0.25** | **0.40** |
| SCS 1 4-mm Analytical | ≤ 1,900 (13.09) | 1.0 | 1.60 |
| SCG 1 4-mm Guard | ≤ 500 (3.44) | 1.0 | 1.60 |
| **SCS 1 + SCG 1 4-mm columns** | **≤ 2,400 (16.54)** | **1.0** | **1.60** |

***NOTE:*** ***Column backpressures are measured by connecting the column directly to the pump and the column's effluent going directly to waste.***

### 7.1.2 Replacing Column Bed Support Assemblies

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

a. Disconnect the column from the system.
b. Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.
c. Remove the bed support.
d. Turn the end fitting over and tap it against a bench-top 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.
e. Discard the old bed support assembly.
f. 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. .
g. Place a new bed support assembly into the end fitting. Use the end of the column to carefully start the bed support assembly into the end fitting.

**Table 9 Parts List**

| Part | IonPac SCS 1 2-mm Column (P/N) | IonPac SCS 1 4-mm Column (P/N) |
|---|---|---|
| Analytical Column | 061520 | 061521 |
| Guard Column | 061522 | 061523 |
| Bed Support Assembly | 044689 | 042955 |
| End Fitting | 043278 | 052809 |

**WARNING: 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.**

h. Screw the end fitting back onto the column. Tighten it finger-tight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.
i. 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.***

## 7.2 Preparation of Eluents

A. Ensure that the eluent is made correctly.
B. Ensure that the eluents are made from chemicals with the recommended purity.
C. Ensure the deionized (DI) water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

## 7.3 Contamination

### 7.3.1 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 SCG 1 Guard and SCS 1 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 SCG 1 at the first sign of column performance degradation (compared to the original test chromatogram) to eliminate downtime. Clean the column(s) as instructed in, "Column Cleanup" (See, "Column Care"). To make sure that contaminated hardware is not causing the high background, use deionized water with a specific resistance of 18.2 megohm-cm as eluent. The background with a 3 mM MSA eluent should be 950–1100 µS, and with deionized water < 0.5 µS. If it is not, check the detector/conductivity cell calibration. See the appropriate manual for details.

a. **Chemicals and DI Water Contamination:** use chemicals and DI water of the proper purity. Be especially careful to make sure that the recommended chemicals are used. The DI water should have a specific resistance of 18.2 megohm-cm.
b **Metal Contamination:** the system should be as metal-free as possible. Gripper tubing fittings used in older systems are a potential source for metal contamination of the column. The new Dionex ThermoFlare or PEEK ferrule fittings are preferred. Inspect the eluent pumps periodically for any signs of leakage. Stainless steel HPLC pumps are a potential source of metal contamination.
c. **Sodium Contamination:** glass eluent reservoirs can be a source of sodium contamination in the eluent. Two-liter polyethylene eluent reservoirs (P/N 039163) are recommended.

### 7.3.2 Sample Loop and/or Tubing Contamination

Eluents made with DI water that are contaminated with bacteria and samples such as humic acids and soil extracts can potentially contaminate eluent lines and sample loops. Weak cation exchange sites are created on, or attached to, the tubing. This can happen to either Tefzel or PEEK tubing. Thus, the sample loop itself can act as a concentrator and, depending on the pH of the sample or the standard and the method of introduction, inaccurate readings for divalent analytes on weak cation exchange resins may be observed.

### 7.3.3 Tubing and Cell Contamination after Use with SCS 1 Column

The SCS 1 column can potentially contaminate the tubing after the column and the conductivity cell. If the system is used with suppressed conductivity after having been used with the SCS 1 column it is recommended to clean or replace the tubing and/or the conductivity cell. Poor peak efficiency and peak tailing will be observed if the tubing or conductivity cell is contaminated.

a. Clean the tubing and conductivity cell with the following:
   1. 100% Acetonitrile for 1 hour
   2. DI water for 5 min
   3. 500 mM Sodium Hydroxide for 1 hour
   4. DI water for 5 min
   5. 200 mM Methanesulfonic Acid for 1 hour
   6. DI water for 5 min

If the problem persists, replace the tubing and conductivity cell.

***NOTE:*** ***If using the system with both nonsuppressed and suppressed conductivity it is recommended to have sets of tubing and conductivity cells for each mode of operation.***

### 7.3.4 Weak Cation Exchangers:

Carboxylated stationary phases used in the IonPac CS12, CS12A, CS14, CS15, CS16, CS17, and in the silica-based SCS 1 are weak acid cation exchangers. These packings have high selectivity for hydronium ion and are used with weak acid eluents. When the sample pH is high (pH 5), the weak cation exchange sites on the contaminated tubing are ionized and divalent cations are preferentially retained. When the sample pH is low (pH < 4), these sites are protonated by the sample and rendered inactive, so that the divalent quantification is not affected.

#### 7.3.4.1 Testing for Loop Contamination when Using Carboxylated Cation Exchange Columns

A simple test can be performed, when using a column such as the IonPac SCS 1 which contains a carboxylated resin, with methanesulfonic acid or sulfuric acid to see if the sample loop has been contaminated:

a. 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.
b. 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).
c. Run the standard and record the peak area.
d. Repeat steps b and c, but this time flush the loop with about 5 mL of standard.
e. If after repeating steps b through d, the peak area for calcium recorded in d is significantly larger than that in c, then the sample loop is contaminated and acting as a concentrator.
    1. Replace the sample loop with new tubing and repeat this test.
f. If there is still a quantification problem, check other components of the system (tubing, injection valve, detector cell) or call your Dionex representative.

If you have a divalent quantification problem in your system but you neither have the time nor replacement parts, you can still get accurate results for divalent cations if any one of the following applies:

1. Your application involves high levels of divalent cations e.g. > 5 ppm calcium; the "concentration error" is small, percentage-wise.

2. The pH of your samples and standards is < 4.

3. A constant volume of sample (and standard), only slightly larger than the sample loop, is flushed through the loop at a constant sampling flow rate.

## 7.4 High Background or Noise

In a properly working system, the background conductivity using the operating conditions described in Section 5, "Operation," should be 950–1100 µS.

a. Check the conductivity flow cell for bubbles. See the conductivity detector manual for details. A system with a high background (> 1200 µS) will probably also have higher noise, resulting in increased detection limits.
b. Make sure that the eluent is prepared correctly (see Section 5, Operation).
c. Determine if the columns or system are contaminated (see Section 7.3, "A Contaminated Guard or Analytical Column").

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

| ELUENT | EXPECTED BACKGROUND CONDUCTIVITY |
|---|---|
| 3 mN Methanesulfonic acid | 950–1100 µS |
| Deionized Water | < 0.5 µS |

## 7.5 Poor Peak Resolution

Poor peak resolution can be due to any or all of the following factors.

### 7.5.1 Loss of Peak Efficiency throughout the Chromatogram

**A. Extra-column effects can result in sample band dispersion, causing loss of peak efficiencies.** Make sure you are using PEEK tubing with an i.d. of no greater than 0.005" 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.

**B. Check to see if headspace has developed in the guard or analytical column.** This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 7.1.2, "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.

### 7.5.2 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.** Check 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 composition and concentration are correct.** An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent.

**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" on the Reference Library CD-ROM).

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. If you need assistance in solving resolution problems, contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

### 7.5.3 Loss of Early Eluting Peak Resolution

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

a **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.

b. **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.

## 7.6 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 5.3, "Chemical Purity Requirements." Clean the column as indicated in "Column Cleanup" (see, "Column Care").

b. **Spurious peaks may be due to column contamination.** If the samples contain an appreciable level of polyvalent cations, polyvalent cations may contaminate the column. As a result, the retention times for the analytes will decrease, and spurious, inefficient peaks can show up at unexpected times. This problem may be solved by increasing the time between analyses or by adding a regeneration step between successive runs to elute polyvalent cationic contaminants off the column before the next sample injection takes place.

c. **An injection valve that needs service may produce baseline upsets.** This baseline upset can show up as one or multiple peaks of varying size(s) and shape(s). Typically this will occur when the particular valve needs to be cleaned or torqued (see the system manual). Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

# APPENDIX A - QUALITY ASSURANCE REPORTS

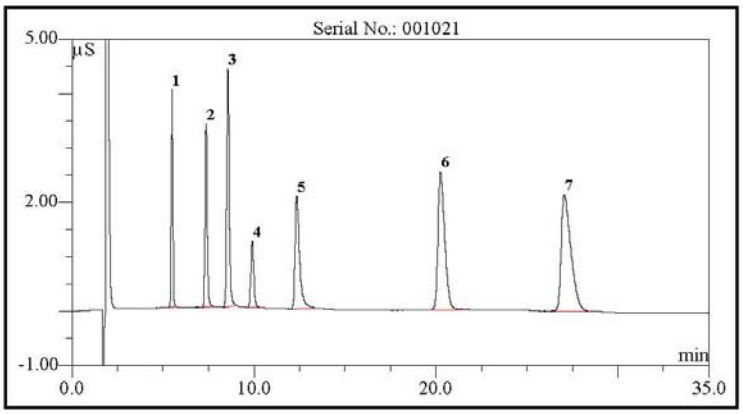
Quality Assurance Reports for the 2-mm IonPac SCS 1 and the 4-mm IonPac SCS 1 columns.

## A.1 IonPac SCS 1 (4 x 250 mm) Quality Assurance Report

**IonPac® SCS 1**
**Analytical (4 x 250 mm)**
**Product No. 061521**

**Date:** 04-Feb-04 11:32
**Serial No. :** 001021
**Lot No. :** 02-19-046

**Eluent:** 3 mM MSA (Methanesulfonic acid)
**Flow Rate:** 1.00 mL/min
**Temperature:** 30° C
**Detection:** Non Suppressed Conductivity
**Injection Volume:** 25 µL
**Storage Solution:** Eluent




| No. | Peak Name | Ret.Time (min) | Asymmetry (EP @ 10%) | Resolution (EP) | Efficiency (EP) | Concentration (mg/L) |
|---|---|---|---|---|---|---|
| 1 | Lithium | 5.50 | 0.99 | 10.13 | 19034 | 0.4 |
| 2 | Sodium | 7.37 | 1.11 | 5.32 | 19570 | 1.6 |
| 3 | Ammonium | 8.57 | 1.18 | 5.04 | 20354 | 2.0 |
| 4 | Ethanolamine | 9.91 | 1.09 | 6.96 | 18307 | 2.0 |
| 5 | Potassium | 12.35 | 1.71 | 14.76 | 14468 | 4.0 |
| 6 | Magnesium | 20.25 | 1.45 | 8.42 | 14969 | 2.0 |
| 7 | Calcium | 27.05 | 1.78 | n.a. | 12736 | 4.0 |

*QA Results:*

| Analyte | Parameter | Specification | Results |
|---|---|---|---|
| Calcium | Efficiency | >=9000 | Passed |
| Calcium | Asymmetry | 1.05-2.42 | Passed |
| Calcium | Retention Time | 25.80-30.20 | Passed |
| Sodium | Efficiency | >=13500 | Passed |
| | Pressure, psi | <=2090 | 1475 |

*Production Reference:*
Datasource: CON_SQL_local
Sequence: SCS-4X250MM_02-04Iqbal
Sample No.: 8

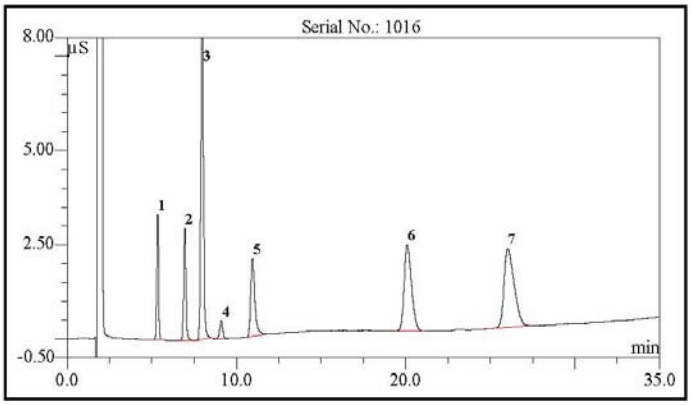
6.50 SP3 Build 980
Chromeleon® Dionex Corp. 1996-2003

## A.2 IonPac SCS 1 (2 x 250 mm) Quality Assurance Report

**IonPac® SCS 1**
**Analytical (2 x 250 mm)**
**Product No. 061520**

**Date:** 19-Apr-04 11:47
**Serial No. :** 1016
**Lot No. :** 02-19-046

**Eluent:** 3 mM MSA (Methanesulfonic acid)
**Flow Rate:** 0.25 mL/min
**Temperature:** 30° C
**Detection:** Non Suppressed Conductivity
**Injection Volume:** 6.2 µL
**Storage Solution:** Eluent




| No. | Peak Name | Ret.Time (min) | Asymmetry (EP @ 10%) | Resolution (EP) | Efficiency (EP) | Concentration (mg/L) |
|---|---|---|---|---|---|---|
| 1 | Lithium | 5.35 | 0.95 | 7.43 | 12717 | 0.4 |
| 2 | Sodium | 6.95 | 1.03 | 4.01 | 13129 | 1.6 |
| 3 | Ammonium | 7.98 | 1.04 | 3.82 | 13955 | 2.0 |
| 4 | Ethanolamine | 9.10 | 1.01 | 5.00 | 12944 | 2.0 |
| 5 | Potassium | 10.95 | 1.43 | 15.17 | 10881 | 4.0 |
| 6 | Magnesium | 20.09 | 1.26 | 6.33 | 10382 | 2.0 |
| 7 | Calcium | 26.03 | 1.45 | n.a. | 9072 | 4.0 |

*QA Results:*

| Analyte | Parameter | Specification | Results |
|---|---|---|---|
| Calcium | Efficiency | >=6750 | Passed |
| Calcium | Asymmetry | 1.05-2.42 | Passed |
| Calcium | Retention Time | 25.80-30.20 | Passed |
| Sodium | Efficiency | >=8100 | Passed |
| | Pressure, psi | <=2090 | 1513 |

*Production Reference:*
Datasource: CON_SQL_local
Sequence: SCS-2X250MM-06-04Iqbal
Sample No.: 4

6.50 SP3 Build 980
Chromeleon® Dionex Corp. 1996-2003

# APPENDIX B - COLUMN CARE

## B.1 Recommended Operating Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the IonPac SCS 1 Analytical or Guard Column is 4,000 psi (27.57 MPa).

## B.2 Column Start-Up

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

## B.3 Column Storage

The column's storage solution should be 3 mM MSA. 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 and cap both ends securely, using the plugs supplied with the column.

## B.4 Column Cleanup

The following column cleanup protocols have been divided into three general isocratic protocols to remove acid-soluble, base-soluble or organic contaminants.

Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column. High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column. High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent with may create a neutralization pressure band. The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to ≤ 5% levels and the ionic strength of the eluent to ≤ 10 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

### B.4.1 Column Cleanup Procedure for Polyvalent Cations and Acid-Soluble Contaminants

a. Prepare 500 mL of 8 mM Oxalic acid for the cleanup solution.

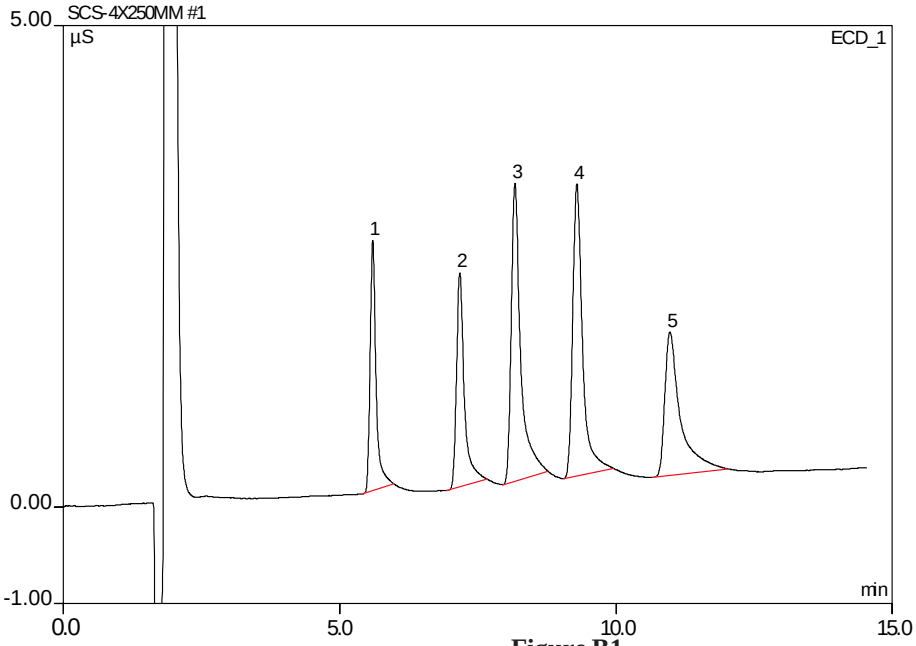
***IMPORTANT NOTE:*** ***Nitric acid should not be used instead of Oxalic acid since nitric acid will not effectively remove iron contaminants. Do not clean the column with basic eluents. The operating pH range for the IonPac SCS 1 is 2–7.***

b. 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.

**WARNING: 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.**

### B.4.3 Clean-up of Contaminated IonPac SCS 1 Column

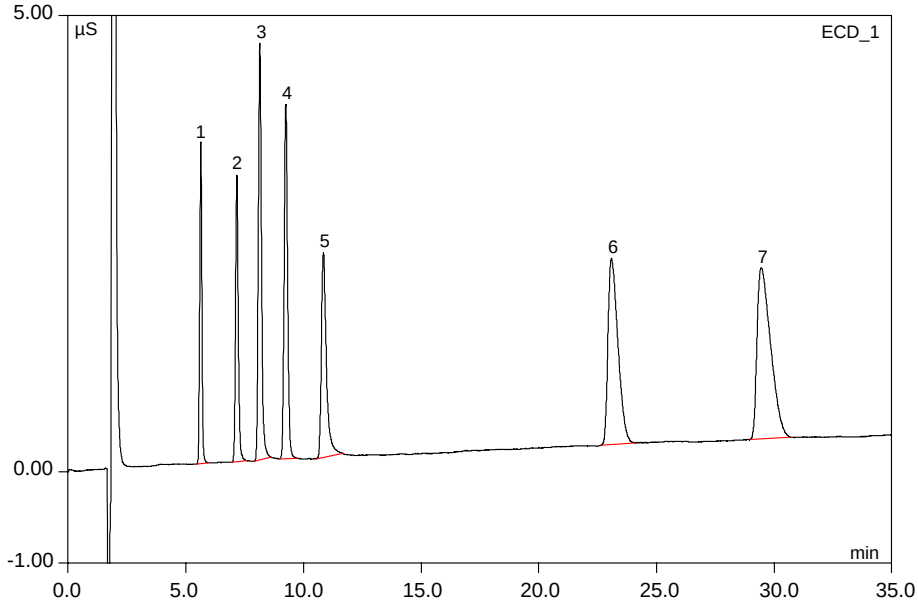
| | |
|---|---|
| Column: | IonPac SCS 1 (4 x 250 mm) |
| Eluent: | 3 mM MSA (Methanesulfonic acid) |
| Flow Rate: | 1.00 mL/min |
| Temperature: | 30 °C |
| Detection: | Non Suppressed Conductivity |
| Injection Volume: | 25 µL |
| Storage Solution: | Eluent |




| | Analyte | mg/L |
|---|---|---|
| 1. | Lithium | 0.4 |
| 2. | Sodium | 2.0 |
| 3. | Ammonium | 4.0 |
| 4. | Ethanolamine | 2.0 |
| 5. | Potassium | 4.0 |

**Figure B1**
**Before Contaminated Column**

Performance of a contaminated IonPac SCS 1 column was restored when cleaned (washed) with 3 mM MSA/50% Acetonitrile, 60 °C, 2 hours.




| | Analyte | mg/L |
|---|---|---|
| 1. | Lithium | 0.4 |
| 2. | Sodium | 1.6 |
| 3. | Ammonium | 2.0 |
| 4. | Ethanolamine | 2.0 |
| 5. | Potassium | 4.0 |
| 6. | Magnesium | 2.0 |
| 7. | Calcium | 4.0 |

**Figure B2**
**After Column Cleanup**

## APPENDIX C - LITERATURE

The following literature is available on the Dionex Reference Library CD-ROM or by contacting your local Dionex representative.

**AN 157**

"Comparison of Suppressed to Nonsuppressed Conductivity Detection for the Determination of Common Inorganic Cations"

**AN 158**

"Determination of Trace Sodium and Transition Metals in the Power Industry by Ion Chromatography with Nonsuppressed Conductivity Detection"