



PRODUCT MANUAL

IonPac[®] CS18
IonPac[®] CG18

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PRODUCT MANUAL

for the

IONPAC® CS18 Analytical Column
2 x 250 mm, P/N 062878

IONPAC® CG18 Guard Column
2 x 50 mm, P/N 062880

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SECTION 1 - INTRODUCTION

The IonPac CS18 analytical column can be used with either nonsuppressed or suppressed conductivity detection for the analyses of the common inorganic cations (Lithium, Sodium, Ammonium, Potassium, Magnesium, and Calcium) as well as amines. Its selectivity is particularly useful in the analysis of small, hydrophilic amines such as ethanolamines and methylamines.

NOTE: If the column is used in the nonsuppressed mode, the use of the EG 40/50 Eluent Generator is not recommended because noise will be much higher than when using pre-made eluent from a single bottle.

The CS18 stationary phase is a moderate capacity weak cation exchanger functionalized with carboxylic acid groups. It has a 6 μm polymeric substrate. It is compatible with up to 20% organic solvents (such as acetonitrile and acetone). Alcohols should be avoided as eluent components. They will form esters in the CS18 column, thus reducing the cation exchange capacity of the column.

The CS18 columns can be used without loss of performance at 50 °C with 100 mM methanesulfonic acid eluent. However, the CS18 columns should not be used with basic eluents. The column backpressure increases too much, disrupting the packing.

When used in the nonsuppressed mode, the CS18 should be used on an ICS chromatography system equipped with microbore capability and a heater option. An IonPac Mixer (P/N 063443) is required to minimize system noise levels. The isocratic eluent should be delivered from a bottle. In the case of the nonsuppressed mode, the use of the EG40/50 Eluent Generator 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.

When used in the nonsuppressed conductivity mode, the expected background conductivity under the “standard operating conditions” of the CS18 column (4 mM methanesulfonic acid, 30 °C, 0.25 mL/min) is typically between 1380 and 1480 μS . At this background level the sample peaks have lower conductance than the background, and are displayed as negative peaks. To display positive peaks in the ICS 2000, 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. In the case of the ICS 3000 in the Chromeleon configuration, under "detectors" in "signals," the factor is set to -1.0 to invert the negative peaks.

NOTE: This manual is easier to understand with familiarity of the installation and operation of the Dionex Ion Chromatograph (IC) and the suppressor. Be sure to learn all of the various system components before beginning an analysis. All instrument manuals are available on the Dionex Reference Library CD-ROM (P/N 053891).

Table 1
IonPac CS18/CG18 Packing Specifications

Column	Particle Diameter μm	Column Capacity μeq/column	Functional Group	Hydrophobicity
IonPac CS18 analytical column 2 x 250 mm	6.0	TBD	Carboxylic acid	Medium
IonPac CG18 guard column 2 x 50 mm	7.0	TBD	Carboxylic acid	Medium

Table 2
IonPac CS18/CG18 Operating Parameters

Column	Typical Back Pressure at Standard Flow Rate psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
IonPac CS18 2-mm analytical column	≤ 2,100 (14.46)	0.25	0.35
IonPac CG18 2-mm guard column	≤ 250 (1.72)	0.25	0.35
IonPac CS18 + CG18 2-mm columns	≤ 2,350 (16.18)	0.25	0.35

NOTE: 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.

SECTION 2 - ION CHROMATOGRAPHY SYSTEMS

The proper configuration of an Ion Chromatography System (ICS) in 2-mm or 4-mm format is based on the ratio of the 2-mm to 4-mm column cross-sectional area (a factor of 1/4). The selected format will affect the type of pump recommended. A gradient pump is designed to blend and pump isocratic, linear, or gradient mixtures of up to four mobile phase components at precisely controlled flow rates. An isocratic pump is for applications not requiring gradient and multi-eluent proportioning capabilities. Both are offered in either standard bore or microbore options.

See Appendix B, "Configuration" for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor (SRS), MicroMembrane Suppressor (MMS), Atlas Electrolytic Suppressor (AES), injection loop, system void volume, detectors, and tubing back pressure.

WARNING: Be sure to use PEEK red tubing, 0.005" i.d. (P/N 044221) for the tubings used from injection valve-to-column, from column-to-suppressor, and from suppressor-to-cell. The tubing lengths should be minimized, and the tubing should be cut with a straight edge, NOT slanted. Failure to do this will result in poor chromatographic peak efficiencies. Also, be sure the tubing protrudes from the ferrule when making the connections. Any void volumes and eddies will result in analyte dispersion, which in turn translates into poor efficiencies.

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 2-mm Operation

The IonPac CG18 2-mm Guard Column (P/N 062880) and CS18 2-mm Analytical Column (P/N 062878) are designed to be run on any Dionex ICS Ion Chromatograph equipped with suppressed conductivity detection. For nonsuppressed conductivity detection, use the Dionex ICS Ion Chromatograph compatible with microbore capability and heater option. See the appropriate Operator's Manuals. All plumbing from the injection valve to the column, from the column to the suppressor and from the suppressor to the cell **INLET** should be done with 0.005" i.d. RED PEEK tubing (See Table 7 of Appendix B).

3.1.2 System Requirements for 2-mm Operation in Nonsuppressed Mode

The Dionex IonPac Mixer (P/N 063443) is required for use with 2-mm CS18 columns operated in the nonsuppressed conductivity mode. 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 when using the nonsuppressed conductivity mode. A column heater is required for the 2-mm operation in the nonsuppressed mode. Variations in column or eluent temperature will result in increased noise levels.

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

For IonPac CS18 applications using the EG40 or EG50 with EGC II MSA cartridge, a CR-CTC Continuously Regenerated Trap Column (P/N 060478) may be installed at the EGC eluent outlet to remove trace level cationic contaminants such as ammonium from the carrier deionized water. See the CR-TC Product Manual (Document No. 031910) for instructions. As an alternative, the CTC-1 Trap Column (P/N 040192) can be used. The CTC-1 Trap Column will require off-line regeneration. To use the CTC Cation Trap Column, see Section 3.2.

3.2.1 Installing the CTC-1 Cation Trap Column for Eluent Step Change or Gradient Operation

For gradient operation, an IonPac Cation Trap Column (CTC (2-mm), P/N 043132 for 2-mm CS18) is installed between the Gradient Pump and the injection valve. Remove the high pressure Gradient Mixer if present. The CTC is filled with high capacity cation exchange resin which helps to minimize the baseline shift caused by increasing cationic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis. To install the CTC, complete the following steps:

- A. **Remove the Gradient Mixer.** It is installed between the gradient pump pressure transducer and the injection valve.
- B. **Connect the gradient pump directly to the CTC.** Connect a waste line to the CTC outlet and direct the line to a waste container.
- C. **Flush the CTC.** Note that with the guard and analytical columns out of line, there is no need for flow rate restrictions. For the CTC (2-mm), use 50 mL of a 10x eluent concentrate of the strongest eluent required by the application at a flow rate of 0.5 mL/min.
- D. **Rinse the CTC.** Use the strongest eluent that will be used during the gradient analysis.
- E. **Reconnect the CTC.** Connect the CTC to the eluent line that is connected to the injection valve inlet.

The background conductivity of your system should be less than 0.5 μS when 5 mM 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 0.1 μS during a gradient concentration ramp from 3 to 30 mM methanesulfonic acid (MSA). If the baseline shifts are greater than 0.2 μS after equilibration, the CTC should be cleaned using steps A - E above.

3.3 The Injection Loop

For most applications on a 2-mm analytical system, a 5 to 25 μL injection loop will be sufficient. When samples are unknown and of expected varied concentrations, Dionex recommends that a 5 μL injection loop be used to avoid overloading the 2-mm Analytical Column. Generally, do not inject more than 4 nanomoles (10–100 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.

3.4 Sample Concentration

The TCC-LP1 (P/N 046027), TCC-ULP1 (P/N 063783), and TCC-XLP1 (P/N 063889) should be used for trace cation pre-concentration work on the 2-mm CS18 columns. The CG18 2-mm guard column can be used as well for preconcentration.

See Section 4.5, “Sample Concentration” for details on sample concentration.

3.5 IonPac CG18 Guard Column

An IonPac CG18 Guard Column is normally used with the IonPac CS18 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.

3.6 Eluent Storage

The recommended column storage solution is 5 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.

3.7 Cation Self-Regenerating Suppressor Requirements

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

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

When using an IonPac CS18 2-mm Analytical Column, use a CSRS ULTRA II 2-mm, P/N 061564.

For detailed information on the operation of the Cation Self-Regenerating Suppressor, see Document No. 031956, the “Product Manual for the Self-Regenerating Suppressor.”

3.8 Cation Atlas Electrolytic Suppressor Requirements

A Cation Atlas Electrolytic Suppressor, CAES (P/N 056118), may be substituted for the CSRS ULTRA for applications up to 25 µeq/min. For detailed information on the operation of the Cation Atlas Electrolytic Suppressor, see Document No. 031770, the “Product Manual for the Cation Atlas Electrolytic Suppressor.”

3.9 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.” This suppressor can only be used in the Chemical Suppression Mode.

If you are installing an IonPac CS18 2-mm Analytical Column, use a CMMS III 2-mm, P/N 056753.

3.10 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.

3.11 Using AutoRegen[®] with the Chemical Suppression Mode

Dionex recommends using an AutoRegen Accessory (P/N 039594) with eluents that do not contain acetonitrile. It should be used with the CMMS III. 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.

SECTION 4 - OPERATION

4.1 General Operating Conditions

Column: CS18 2-mm Analytical Column (+ CG18 2-mm Guard Column)

Sample Volume:	5 μ L Loop + 0.8 μ L Injection valve dead volume (2-mm)
Eluent:	5 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	0.25 mL/min (2-mm)
Temperature:	30 °C
CSRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS ULTRA II (2-mm) AutoSuppression Recycle Mode
or CAES Suppressor:	Cation Atlas Electrolytic Suppressor, CAES
or CMMS Suppressor:	Cation MicroMembrane Suppressor, CMMS III (2-mm)
CMMS Regenerant:	TBAOH
CMMS Mode:	Displacement Chemical Regeneration (DCR)
Expected Background Conductivity:	< 0.2 μ S in the suppressed mode
Storage Solution:	Eluent

4.2 IonPac CS18 Operating Precautions

CAUTION: *Operate below 4,000 psi (27.57 MPa). Filter and Degas Eluents. Filter Samples*

4.3 Chemical Purity Requirements

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

4.3.1 Deionized Water

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

4.3.2 Inorganic Chemicals

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

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

4.3.3 Eluents with Solvents

Solvents can be added to the ionic eluents used with IonPac CS18 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, the column, the temperature, 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 CS18 columns is 4,000 psi (27.57 MPa). **The IonPac CS18 is compatible with the HPLC solvents listed in Table 3, "HPLC Solvents for Use with the CS18 Columns."** Solvents and water should be premixed in concentrations which allow proper mixing by the pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

NOTE: *At a characteristic concentrate range of organic solvent concentration in the eluent, the column back pressure 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.*

Table 3	
HPLC Solvents for Use with IonPac CS18 Columns	
Solvent	Maximum Operating Concentration
Acetonitrile	20%
Acetone	20%

CAUTION: *Do NOT use alcohols with the CS18 column.*

4.4 Making and Using Eluents that Contain Solvents

SAFETY: *When purging or degassing eluents containing solvents, do not purge or degas the eluent excessively since it is possible that a volatile solvent can be "boiled" off from the solution. Always degas and store all eluents in glass or plastic eluent bottles pressurized with helium. Only helium can be used to purge and degas ionic eluents containing solvents, since nitrogen is soluble in solvent containing eluents.*

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 20% acetonitrile, prepare the eluent by adding 200 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water at a time to the acetonitrile in the reservoir and fill it up to the 1 liter mark. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.

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 2% 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.

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.

4.5 Sample Concentration

The IonPac CG18 Guard Column or the Trace Cation Concentrator Low-Pressure (TCC-LP1, P/N 046027), should be used for trace cation concentration. The Trace Cation Concentrator Ultra Low-Pressure (TCC-ULP1, P/N 063783) and Trace Cation Concentrator Extremely Low-Pressure (TCC-XLP1, P/N 063889) can also be used. 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, TCC-ULP1, TCC-XLP1, or the CG18) leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the trace cation concentrator (TCC-LP1, TCC-ULP1, TCC-XLP1, or the CG18) 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 CG18 2-mm Guard Column (P/N 062880), the Trace Cation Concentrator Low-Pressure (TCC-LP1, P/N 046027), the Trace Cation Concentrator Ultra Low-Pressure (TCC-ULP1, P/N 063783), and Trace Cation Concentrator Extremely Low-Pressure (TCC-XLP1, P/N 063889) should be used for sample concentration with the IonPac CS18 2-mm Analytical Column.

The advantage of the TCC-LP1, TCC-ULP1, and TCC-XLP1 is that because of their low backpressure, samples can be preconcentrated using a hand-held syringe.

WARNING: *The Trace Cation Concentrator (TCC-2, P/N 043103) should not be used for sample concentration with the IonPac CS18 column. The TCC-2 column packing is a strong cation exchange resin functionalized with sulfonic acid. The recommended IonPac CS18 eluents will not properly elute ions concentrated on this column.*

SECTION 5 - EXAMPLE APPLICATIONS

5.1 Nonsuppressed Conductivity Detection

5.1.1 Separation of Six Common Cations Plus Ethanolamine

The chromatogram below shows a nonsuppressed conductivity detection separation of the common Group I and Group II cations plus ammonium ion from ethanolamine. Monoethanolamine (or ethanolamine) is one of the most important corrosion inhibitors utilized in the Power Industry, and generally needs to be quantified together with very low levels of sodium and high or low levels of ammonium ion.

The IonPac CS18 offers good selectivity for sodium, ammonium and ethanolamine. Using an isocratic eluent containing 4 mM MSA will resolve these ions.

The IonPac CS18 column can be used with this weak isocratic eluent in the nonsuppressed conductivity mode. Minimum detection levels will be inferior than with suppressed conductivity, as the noise is much higher (approximately 5 nS versus 0.2 nS for the suppressed conductivity mode). In nonsuppressed IC, the higher the eluent concentration the higher the noise level will be. This is why the eluent concentration has been decreased to 4 mM instead of 5 mM MSA. Broader linear calibration curves for weak bases such as ammonium and ethanolamine are possible in the nonsuppressed conductivity mode.

Column:	IonPac CS18 (2 x 250 mm) Analytical column		
	IonPac CG18 (2 x 50 mm) Guard column		
Eluent:	4 mM methanesulfonic acid	Analyte	mg/L
Eluent Source:	Bottle-made eluent	1. Lithium	0.1
Eluent Flow Rate:	0.25 mL/min	2. Sodium	0.4
Temperature:	30 °C	3. Ammonium	0.5
Sample Loop:	5 µL	4. Ethanolamine	0.5
Detection:	Nonsuppressed conductivity	5. Potassium	1.0
Background:	1430 µS	6. Magnesium	0.5
		7. Calcium	1.0

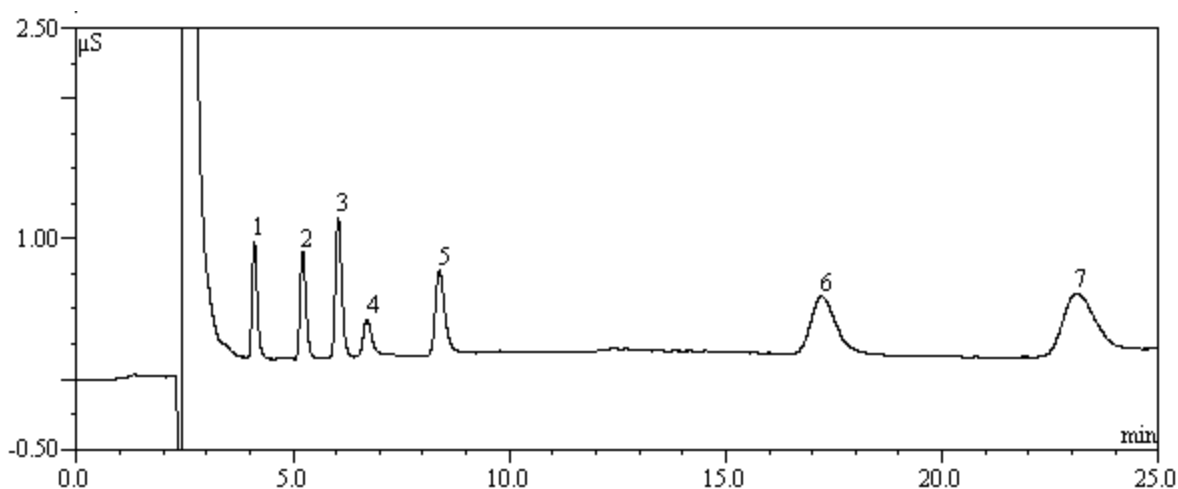


Figure 1
Six Common Cations Plus Ethanolamine with Nonsuppressed Conductivity

5.1.2 Separation of Six Common Cations Plus Alkanolamines

The chromatogram below shows the separation of the common Group I and Group II cations plus ammonium ion from monoethanolamine, diethanolamine and triethanolamine using nonsuppressed conductivity detection. These amines can be found together in the Chemical and Power Industries, as impurities or as decomposition products of other larger amines.

Selectivity of the IonPac CS18 column can separate these isocratically. Either suppressed or nonsuppressed conductivity detection can be used.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Analyte	m/L
	IonPac CG18 (2 x 50 mm) Guard column	1. Lithium	0.1
Eluent:	4 mM methanesulfonic acid	2. Sodium	0.4
Eluent Source:	Bottle-made eluent	3. Ammonium	0.5
Eluent Flow Rate:	0.25 mL/min	4. Ethanolamine	0.5
Temperature:	30 °C	5. Diethanolamine	1.0
Sample Volume:	5 µL	6. Potassium	1.0
Detection:	Nonsuppressed conductivity	7. Triethanolamine	4.5
Background:	1430 µS	8. Magnesium	0.5
		9. Calcium	1.0

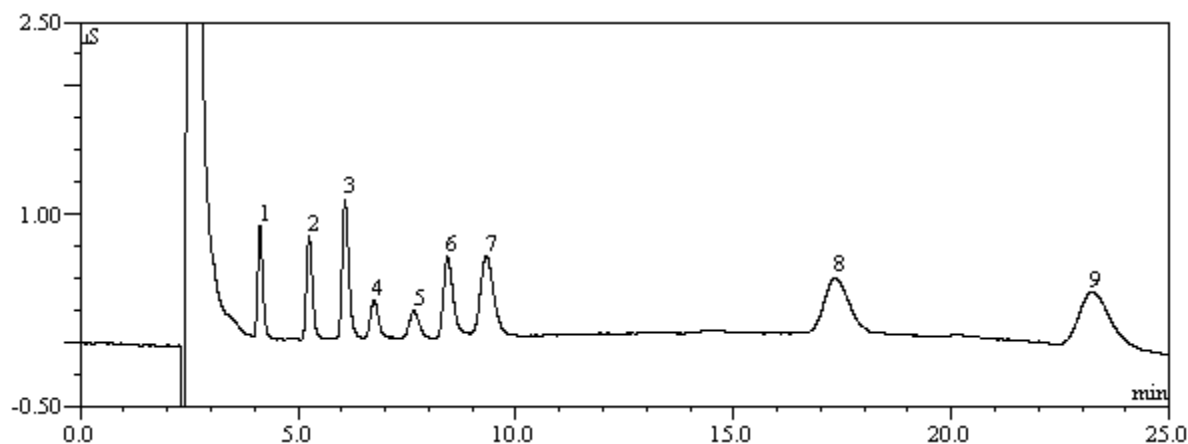


Figure 2
Separation of Six Common Cations Plus Alkanolamines with Nonsuppressed Conductivity

5.2 Suppressed Conductivity Detection

5.2.1 Separation of Six Common Cations Plus Ethanolamine

The chromatograms below show the separation of the common Group I and Group II cations plus ammonium ion from ethanolamine using suppressed conductivity detection. Monoethanolamine (or ethanolamine) is one of the most important corrosion inhibitors utilized in the Power Industry, and generally needs to be quantified together with very low levels of sodium and high or low levels of ammonium ion. The IonPac CS18 offers good selectivity for sodium, ammonium and ethanolamine. Using an isocratic eluent containing 5 mM MSA will resolve these ions.

The CS18 column can be used also with a weaker isocratic eluent in the nonsuppressed conductivity mode (see Figure 1). Minimum detection levels will be superior than with nonsuppressed conductivity, as the noise is much higher for nonsuppressed conductivity (approximately 5 nS vs. 0.2 nS for the suppressed mode).

Column: IonPac CS18 (2 x 250 mm) Analytical column
 Eluent: 5 mM methanesulfonic acid
 Eluent Flow Rate: 0.25 mL/min
 Eluent Source: EGC II MSA cartridge
 Temperature: 30 °C
 Sample Volume: 5 µL
 Detection: Suppressed conductivity with Cation Atlas Electrolytic Suppressor, CAES or with CSRS ULTRA II 2-mm
 Background: < 0.2 µS

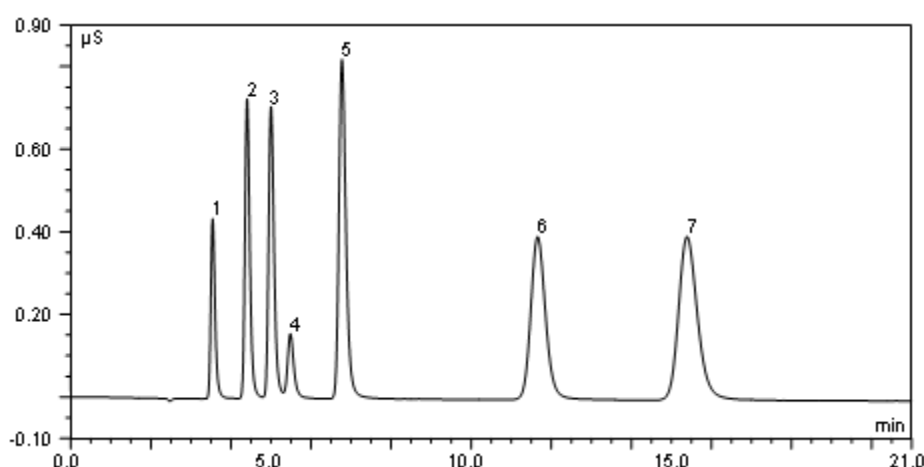


Figure 3a

Separation of Six Common Cations Plus Ethanolamine with IonPac CS18

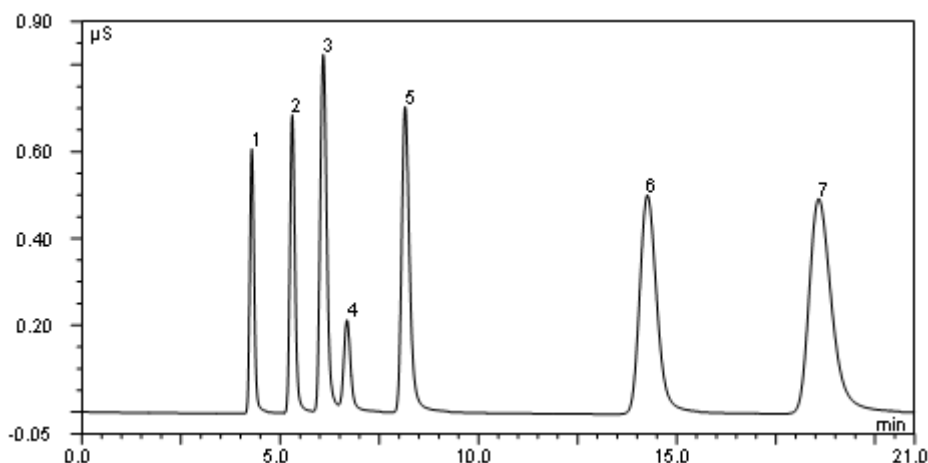


Figure 3b

Separation of Six Common Cations Plus Ethanolamine with IonPac CS18 and CG18

5.2.2 Isocratic Separation of Six Common Cations Plus Alkanolamines

The chromatogram below shows suppressed conductivity detection and separation of the common Group I and Group II cations plus ammonium ion from monoethanolamine, diethanolamine and triethanolamine. These amines can be found together in the chemical and power industries, as impurities or as decomposition products of other larger amines.

The selectivity of the IonPac CS18 column can separate these isocratically. Either suppressed or nonsuppressed conductivity detection can be used. This same application is shown in Figure 2 with nonsuppressed conductivity detection and a weaker acidic eluent.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Analyte	mg/L
Eluent:	5 mM methanesulfonic acid	1. Lithium	0.1
Eluent Flow Rate:	0.25 mL/min	2. Sodium	0.4
Eluent Source:	EGC II MSA cartridge	3. Ammonium	0.5
Temperature:	30 °C	4. Ethanolamine	0.5
Sample Volume:	5 µL	5. Diethanolamine	1.0
Detection:	Cation Atlas Electrolytic Suppressor (CAES) or CSRS ULTRA II 2-mm	6. Potassium	1.0
Background:	< 0.2 µS	7. Triethanolamine	4.5
		8. Magnesium	0.5
		9. Calcium	1.0

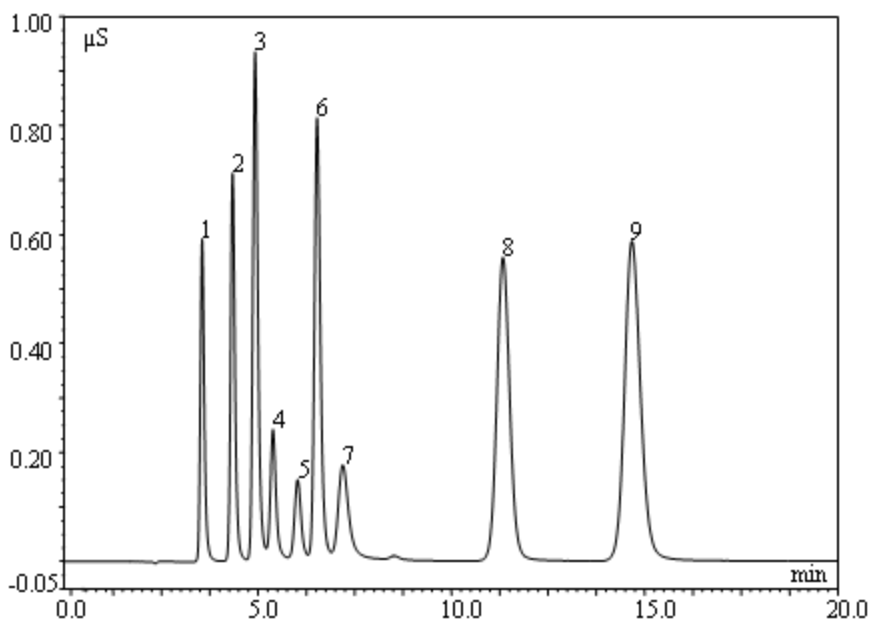


Figure 4
Six Common Cations Plus Alkanolamines with IonPac CS18 and CG18

5.2.3 Separation of Six Common Cations and Methylamines

Using the “standard eluent conditions”, the six common cations can be separated from methylamine and trimethylamine, however potassium and dimethylamine overlap.

To resolve potassium from dimethylamine, the column temperature needs to be increased to 40 °C. The effect of temperature on potassium is higher than on dimethylamine, causing it to elute earlier and thus be resolved from it. Thus, it is possible to resolve the common six cations from methyl-, dimethyl- and trimethylamine on the CS18 using an isocratic eluent at 40 °C.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Analyte	mg/L
	IonPac CG18 (2 x 50 mm) Guard column	1. Lithium	0.1
Eluent:	4 mM methanesulfonic acid	2. Sodium	0.4
Eluent Flow Rate:	0.25 mL/min	3. Ammonium	0.5
Eluent Source:	EGC II MSA cartridge	4. Methylamine	0.5
Temperature:	40 °C	5. Potassium	1.0
Sample Volume:	5 µL	6. Dimethylamine	0.4
Detection:	Suppressed Conductivity,	7. Trimethylamine	1.5
	Cation Atlas Electrolytic Suppressor, CAES or CSRS ULTRA II 2-mm	8. Magnesium	0.5
Background:	< 0.2 µS	9. Calcium	1.0

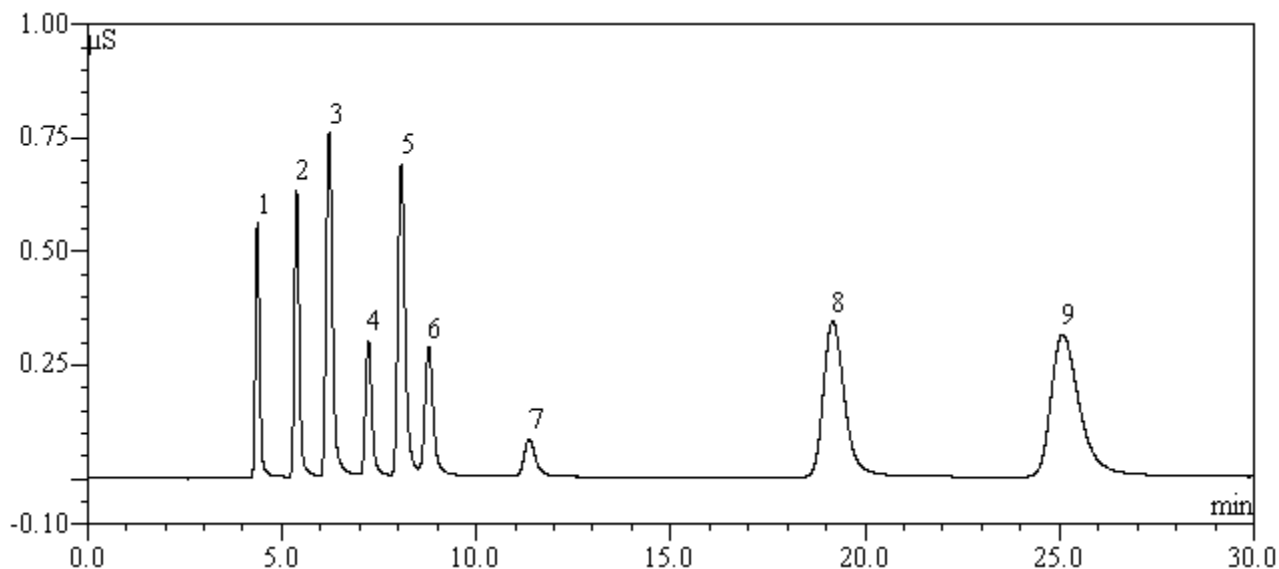


Figure 5
Separation of Six Common Cations Plus Methylamines with IonPac CS18 and CG18

5.2.4 Gradient Separation of Methylamines, Alkanolamines, and the Six Common Cations

The IonPac CS18 column, because of its relative hydrophobicity, is especially suited to separate the smaller, more hydrophilic amines such as methyl-, dimethyl- and trimethylamine, and ethanol-, diethanol- and triethanolamine from the common six cations. The column separates these small monovalent amines through slight differences in their hydrophobicity.

A modest eluent gradient and temperature elevation to 35 °C are necessary to achieve this separation. Notice that the baseline essentially remains unchanged during the gradient, even without a cation trap column in line. See Figure 6.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Analyte	mg/L
Eluent:	0.5 mM methanesulfonic acid (MSA), Gradient to 1 mM MSA at 20 minutes, Gradient to 6.0 mM MSA at 22.4 minutes, Gradient to 8.0 mM MSA at 28 minutes, Step back to 0.5 mM MSA at 28.1 minutes.	1. Lithium	0.1
Eluent Source:	EGC II MSA cartridge	2. Sodium	0.4
Eluent Flow Rate:	0.3 mL/min	3. Ammonium	0.5
Temperature:	35 °C	4. Ethanolamine	1.0
Sample Volume:	5 µL	5. Methylamine	1.0
Detection:	Suppressed Conductivity	6. Diethanolamine	2.0
	CSRS ULTRA II 2-mm	7. Potassium	1.0
Background:	< 0.2 µS	8. Dimethylamine	0.8
		9. Triethanolamine	9.0
		10. Trimethylamine	3.0
		11. Magnesium	0.5
		12. Calcium	1.0

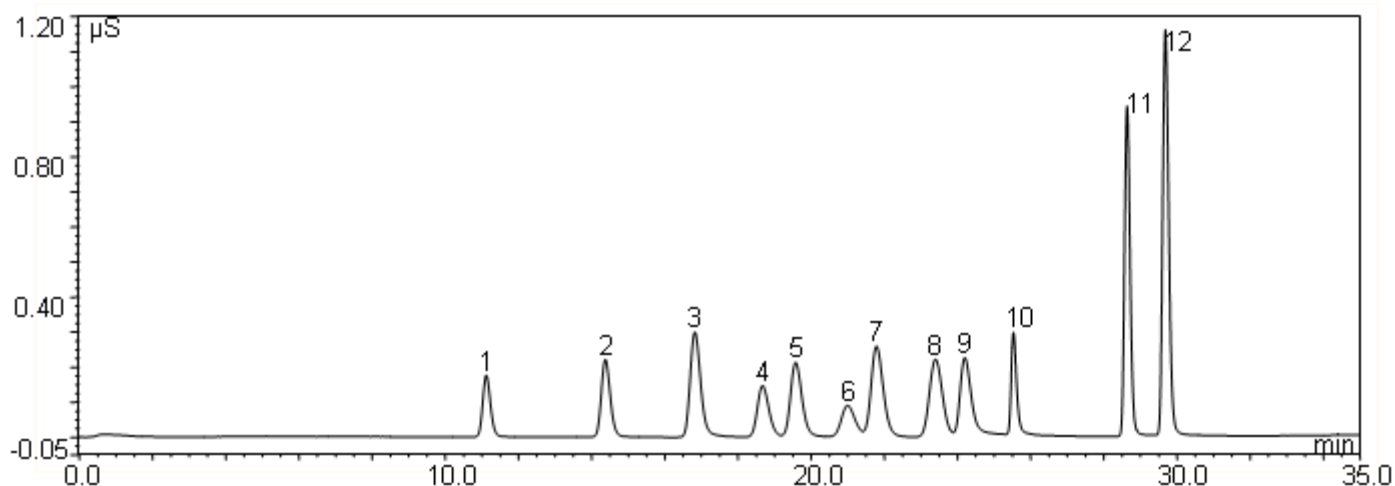


Figure 6
Six Cations, Methylamines, and Alkanolamine with IonPac CS18

5.2.5 TRIS and the Six Common Cations

TRIS, or tris(hydroxymethyl)aminomethane, is a weak base, very hydrophilic, and only lightly retained in most cation exchange columns. In most stationary phases it co-elutes with sodium. With an eluent gradient at elevated temperature, it is baseline resolved from sodium on the CS18 column.

Column: IonPac CS18 (2 x 250) Analytical column
IonPac CG18 (2 x 50) Guard column
Eluent: 0.5 mM MSA, gradient to 1.0 mM at 24 min,
Gradient to 6.0 mM at 26.9 min,
Gradient to 8.0 mM at 33.6 min,
Back to 0.5 mM MSA at 33.7 minutes.
Eluent Source: EGC II MSA cartridge
Flow Rate: 0.30 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Injection Vol.: 5 µL

Peaks:	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. TRIS	3.00
4. Ammonium	0.25
5. Potassium	0.50
6. Magnesium	0.25
7. Calcium	0.50

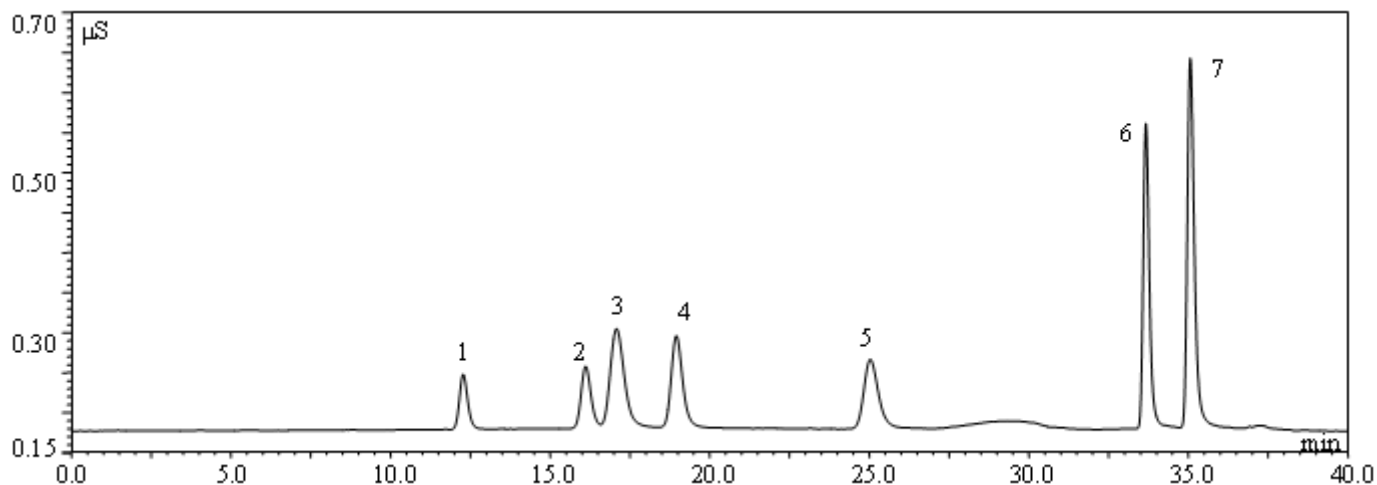


Figure 7
Tris and the Six Common Cations with IonPac CS18 and CG18

5.2.6 Power Amines and the Six Common Cations

Today, most US Power Plants use one or more "advanced amines" as additives for secondary system pH control. The proportions and amounts of these have to be carefully monitored to be effective in corrosion prevention. The amines separation shown in this chromatogram represents the most widely used amines in the Power Industry.

Column: IonPac CS18 (2x250 mm) Analytical column
Eluent: 0.5 mM MSA, gradient to 3 mM at 16 min,
Gradient to 25 mM at 30 min, back to 0.5 mM MSA at 30.1 minutes.
Eluent Source: EGC II MSA cartridge
Flow Rate: 0.30 mL/min
Temperature: 40 °C
Detection: Suppressed Conductivity
Injection Vol.: 5 µL

Peaks:	mg/L
1. Lithium	0.05
2. Sodium	0.20
3. Ammonium	0.25
4. Ethanolamine	2.0
5. Potassium	0.50
6. 2(2-aminoethoxy)ethanol	3.0
7. 5-amino-1-pentanol	3.0
8. Morpholine	3.0
9. 3-methoxypropylamine	3.0
10. 2-diethylaminoethanol	3.0
11. 3-quinuclidinol	2.0
12. Magnesium	0.25
13. Calcium	0.50
14. Ethylenediamine	2.0
15. Cyclohexylamine	3.0

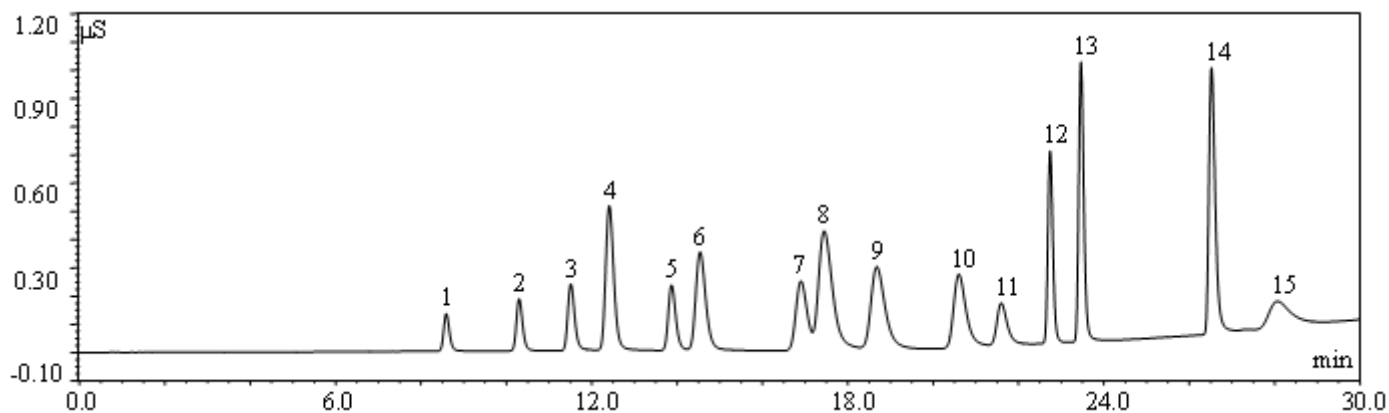
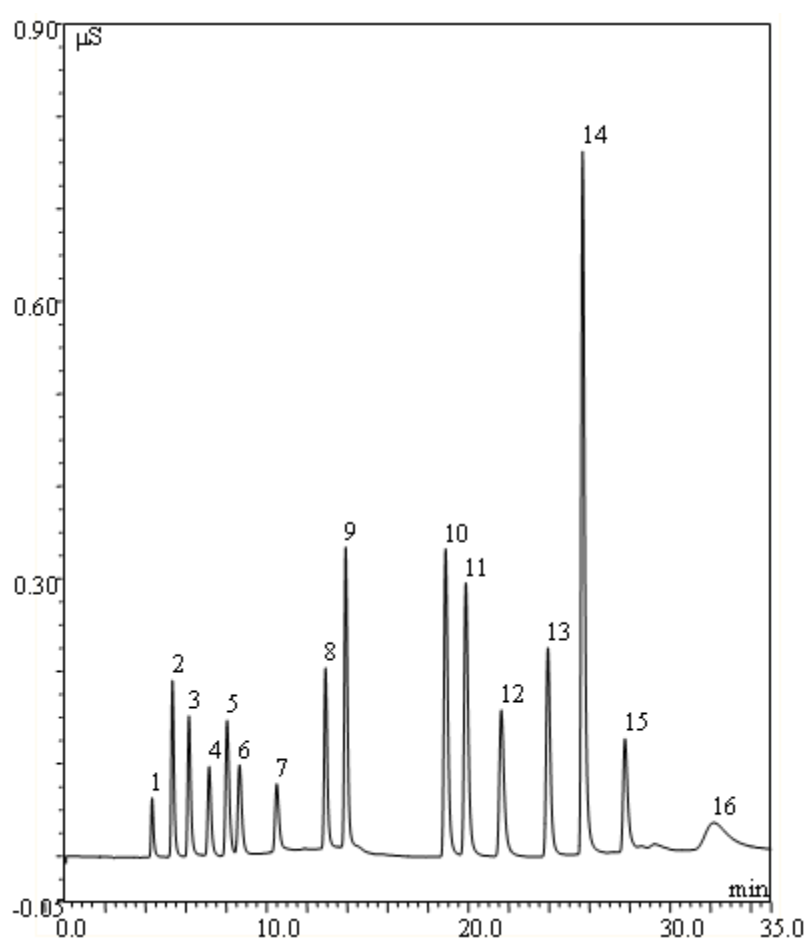


Figure 8
Power Amines and the Six Common Cations with IonPac CS18

5.2.7 Biogenic Amines, Methylamines, and the Six Common Cations

The amines shown here separated on the CS18 are of interest in the Food Industry. For example, histamine is formed by bacterial decomposition of histidine, and is important to determine its content in wine. The freshness of seafood and meat products is determined by the amounts of biogenic amines present. An organic-solvent-free eluent is used to elute the polyvalents spermidine and spermine from this column, with the help of a higher acidic eluent concentration and elevated temperature. Eluent gradient conditions require the use of a suppressor.

Column: IonPac CS18 (2x250 mm) Analytical column
 Eluent: 3 mM MSA, isocratic to 5 min,
 Gradient to 18 mM at 20 min,
 Gradient to 45 mM at 25 min,
 Isocratic to 35 min,
 Back to 3 mM MSA at 35.1 min
 Eluent Source: EGC II MSA cartridge
 Flow Rate: 0.25 mL/min
 Temperature: 40 °C
 Detection: Suppressed Conductivity
 Injection Vol.: 5 µL



Peaks:	mg/L
1. Lithium	0.02
2. Sodium	0.08
3. Ammonium	0.10
4. Methylamine	0.25
5. Potassium	0.20
6. Dimethylamine	0.20
7. Trimethylamine	0.75
8. Magnesium	0.1
9. Calcium	0.2
10. Putrescine	1.1
11. Cadaverine	1.1
12. Histamine	1.6
13. Agmatine	1.4
14. Spermidine	4.0
15. Spermine	0.4
16. Phenethylamine	1.5

Figure 9

Biogenic Amines, Methylamines, and the Six Common Cations with IonPac CS18

5.2.8 Amines of Interest in the Petrochemical Industry

The selectivity of the CS18 has been found to be especially suited for the analysis of small water soluble amines in refinery processes. It is excellent for analyzing amine-scrubbing solutions in the sulfur removal units and to analyze amine-based additives used for pH, corrosion and foam control. At 40 °C, peaks 7 and 8 (potassium and ethylamine) and peaks 11 and 12 (morpholine and 1-dimethylamino-2-propanol) co-elute. At 42 °C, resolution of these two pairs improve, with potassium, the most temperature-sensitive of the analytes, eluting earlier between diethanolamine and ethylamine. At 50 °C, there is baseline resolution of peaks 11 and 12, and potassium co-elutes completely with diethanolamine.

Column: IonPac CS18 (2 x 250 mm) Analytical column
 Eluent: 0.5 mM MSA, gradient to 1 mM at 20 min,
 Gradient to 4 mM at 28 min,
 Gradient to 11 mM at 34 min,
 Isocratic to 40 min,
 Back to 0.5 mM MSA at 40.1 minutes.
 Eluent Source: EGC II MSA cartridge
 Flow Rate: 0.30 mL/min
 Detection: Suppressed Conductivity
 Injection Vol.: 5 µL

Peaks:	mg/L
1. Lithium	0.05
2. Sodium	0.2
3. Ammonium	0.25
4. Ethanolamine	3.0
5. Methylamine	3.6
6. Diethanolamine	3.6
7. Potassium	0.5
8. Ethylamine	3.0
9. Dimethylamine	1.4
10. N-methyldiethanolamine	3.0
11. Morpholine	3.2
12. 1-dimethylamino-2-propanol	3.7
13. N-methylmorpholine	7.5
14. Butylamine	1.5
15. Magnesium	0.25
16. Calcium	0.5
17. Strontium	0.5
18. Barium	0.5

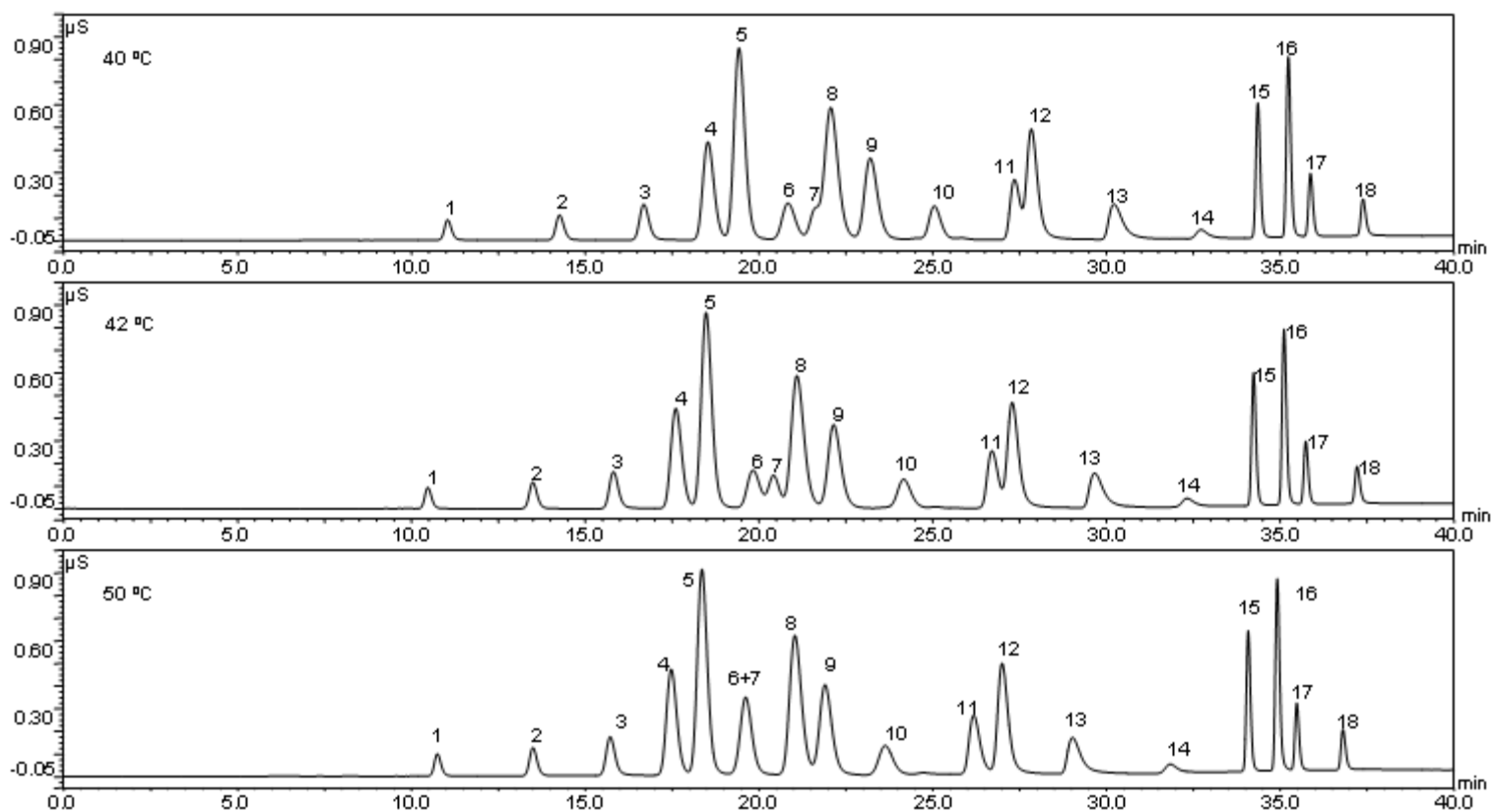


Figure 10
Amines of Interest in the Petrochemical Industry with IonPac CS18

5.2.9 Group I and Group II Cations Plus Ammonium

These three chromatograms were run isocratically. At 5 mM methanesulfonic acid concentration, monovalents elute first followed by divalents. At 7 mM acid, all peaks elute sooner with a reversal of elution order for peaks 6 and 7, cesium and magnesium. At 8 mM MSA, elevated temperature and higher flow rate, the common six cations can elute within 6 minutes to provide a fast run for high sample throughput.

Column: IonPac CS18 (2 x 250 mm) Analytical column
 Eluent Source: EGC II MSA cartridge
 Detection: Suppressed Conductivity
 Injection Vol.: 5 μ L

Peaks:	mg/L
1. Lithium	0.10
2. Sodium	0.40
3. Ammonium	0.50
4. Potassium	1.00
5. Rubidium	0.60
6. Cesium	0.60
7. Magnesium	0.50
8. Calcium	1.00
9. Strontium	0.60
10. Barium	0.60

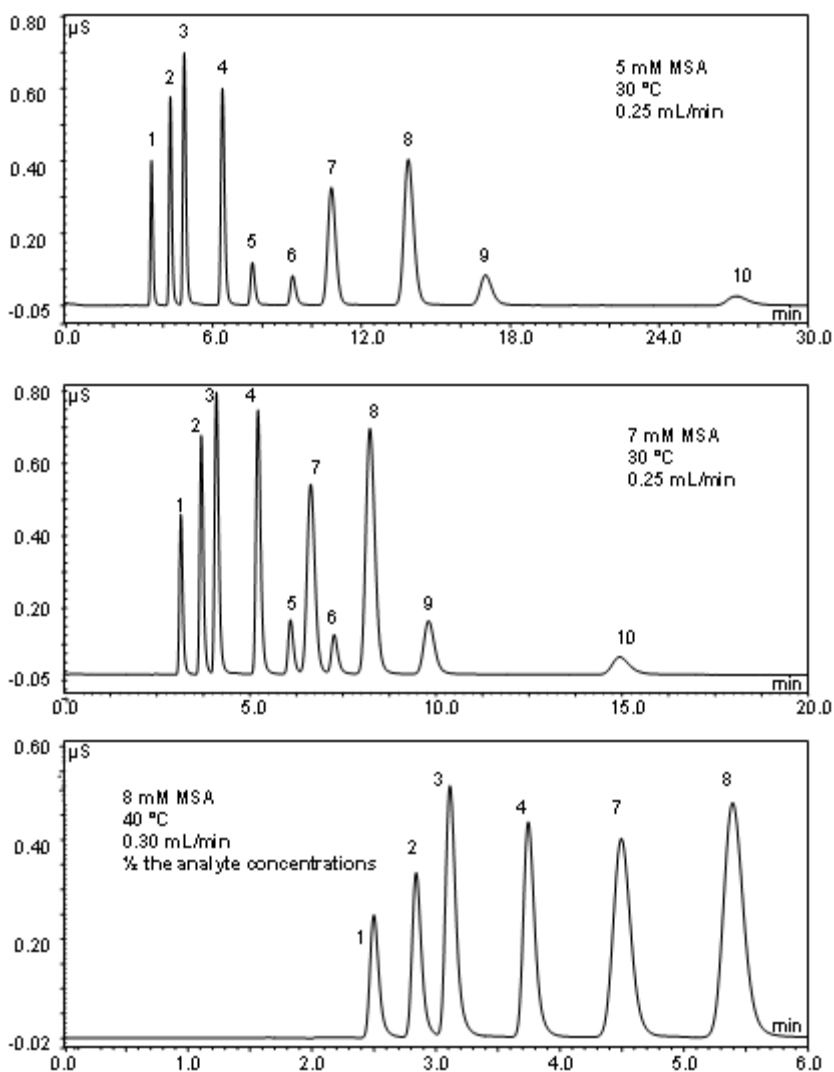


Figure 11
Group I and Group II Cations Plus Ammonium with IonPac CS18

5.2.10 Alkyl Diamines and the Six Common Cations

Alkyl diamines are well separated on the CS18 by using a methanesulfonic acid gradient at 40 °C. Up to a chain length of ten carbons, no organic solvent is required in the eluent.

Column: IonPac CS18 (2 x 250 mm) Analytical column
 Eluent: 3 mM MSA, gradient to 10 mM at 20 min,
 Gradient to 40 mM at 30 min,
 Gradient to 45 mM at 45 min,
 Back to 3 mM MSA at 45.1 min
 Eluent Source: EGC II MSA cartridge
 Flow Rate: 0.30 mL/min
 Temperature: 40 °C
 Detection: Suppressed Conductivity
 Injection Vol.: 5 µL

Peaks:	mg/L
1. Lithium	0.02
2. Sodium	0.08
3. Ammonium	>> 0.10
4. Potassium	0.20
5. Magnesium	0.10
6. Calcium	0.20
7. Ethylenediamine	2.0
8. Putrescine	2.0
9. Cadaverine	2.0
10. 1,6-Hexanediamine	2.0
11. 1,7-Heptanediamine	2.0
12. 1,8-Octanediamine	2.0
13. 1,9-Nonanediamine	6.0
14. 1,10-Decanediamine	10.0

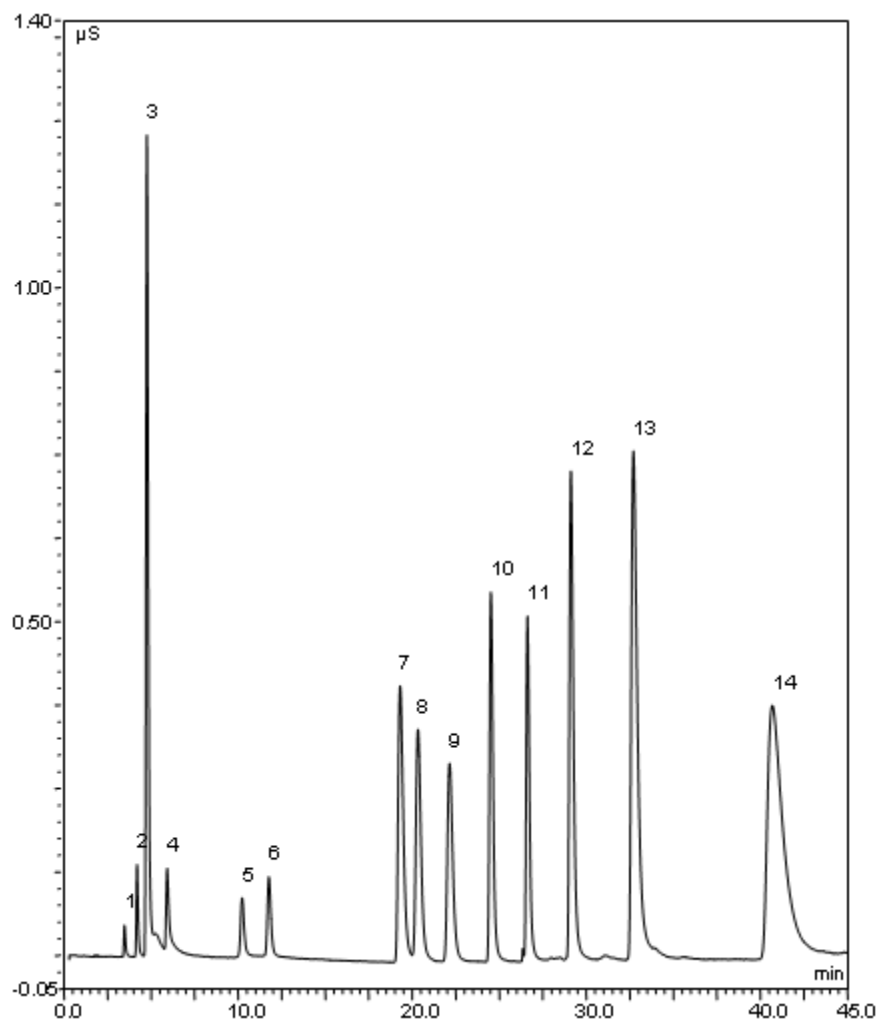


Figure 12
Alkyl Diamines and the Six Common Cations with IonPac CS18

5.2.11 Diamines and the Six Common Cations

This chromatogram shows the separation of substituted diamines together with the six common cations.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Peaks:	mg/L
Eluent:	3 mM MSA, gradient to 25 mM at 30 mins, Back to 3 mM MSA at 30.1 min	1. Lithium	0.05
Eluent Source:	EGC II MSA cartridge	2. Sodium	0.20
Flow Rate:	0.30 mL/min	3. Ammonium	0.25
Temperature:	40 °C	4. Potassium	0.50
Detection:	Suppressed Conductivity	5. Magnesium	0.25
Injection Vol.:	5 µL	6. Calcium	0.50
		7. 1,2-Propanediamine	3.0
		8. 3-Dimethylaminopropylamine	3.0
		9. N,N-Dimethyl-1,3-Propanediamine	3.0
		10. N,N,N,N-Tetramethylethylenediamine	3.0
		11. N,N,N,N-Tetramethyl-1,4-butanediamine	3.0
		12. 3,3-Diaminodipropylamine	3.0
		13. N,N,N,N-Tetramethyl-1,6-hexanediamine	3.0

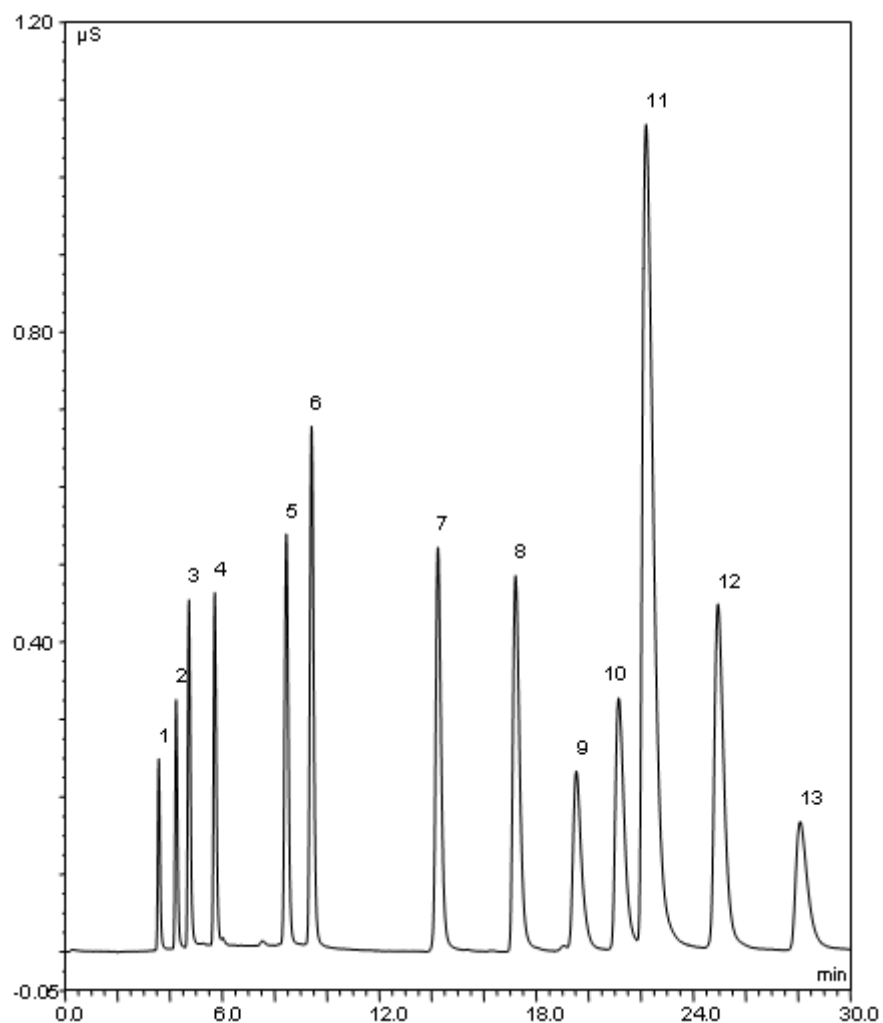


Figure 13
Diamines and the Six Common Cations with IonPac CS18

5.2.12 Polyamines and the Six Common Cations

Polyvalent amines can be eluted off the CS18 stationary phase by using a methanesulfonic acid gradient at 40 °C.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Peaks:	mg/L
Eluent:	3 mM MSA, gradient to 10 mM at 20 min, Gradient to 40 mM at 30 min, Gradient to 45 mM at 45 min, Back to 3 mM MSA at 45.1 minutes.	1. Sodium	-
Eluent Source:	EGC II MSA cartridge	2. Ammonium	-
Flow Rate:	0.30 mL/min	3. Ethylamine	2
Temperature:	40 °C	4. Calcium	-
Detection:	Suppressed Conductivity	5. Ethylenediamine	2
Injection Vol.:	5 µL	6. 3-Dimethylaminopropylamine	2.4
		7. Diethylethylenediamine	2.4
		8. Bis(2-aminopropyl)amine	2.4
		9. Diethylenetriamine	2
		10. Triethylenetetramine	2
		11. Paraquat	2

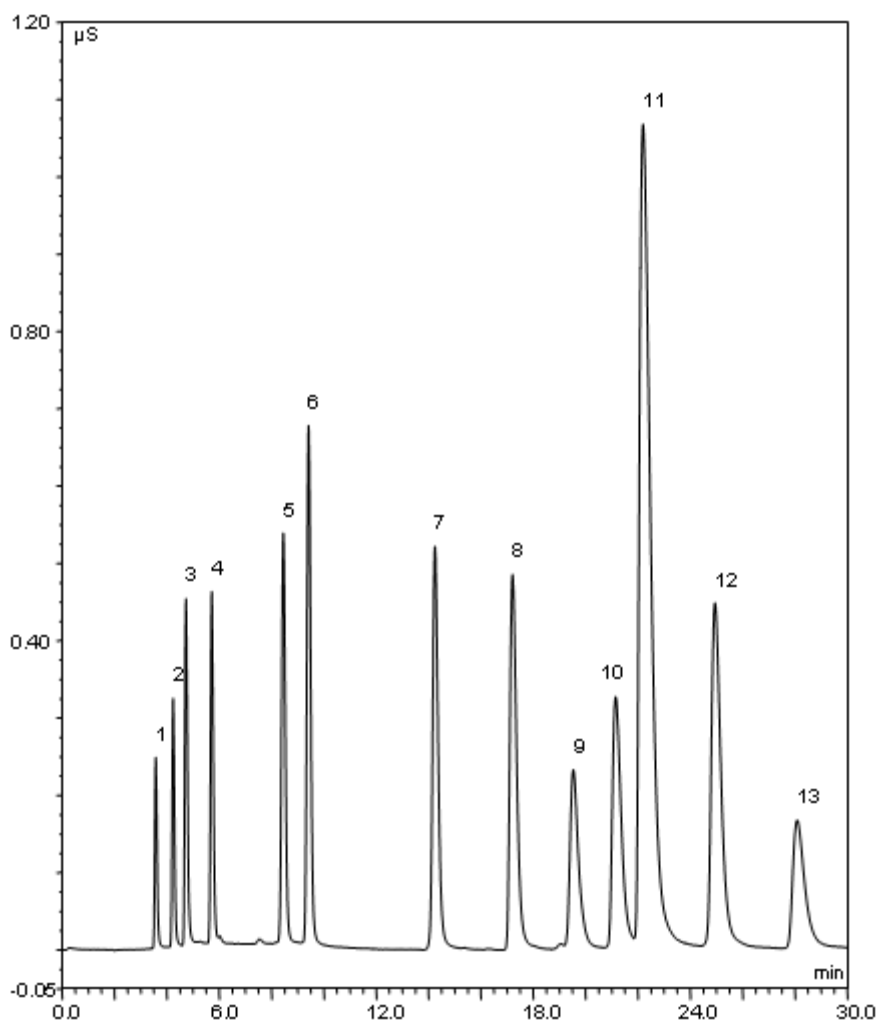


Figure 14
Polyamines and the Six Common Cations with IonPac CS18

5.2.13 Ethylamines and the Six Common Cations

A gradient and elevated temperature are needed to separate potassium from ethylamine. At lower temperatures, these two co-elute.

Column:	IonPac CS18 (2 x 250 mm) Analytical column IonPac CG18 (2 x 50 mm) Guard column	Peaks:	mg/L
Eluent:	0.5 mM MSA, gradient to 1 mM at 24 minutes, Gradient to 9 mM at 26.9 minutes, Isocratic at 9 mM till 33.6 minutes, Back to 0.5 mM at 33.7 minutes.	1. Lithium	0.05
Flow Rate:	0.30 mL/min	2. Sodium	0.20
Eluent Source:	EGC II MSA cartridge	3. Ammonium	0.25
Temperature:	50 °C	4. Potassium	0.50
Detection:	Suppressed Conductivity	5. Ethylamine	0.50
Injection Vol.:	5 µL	6. Diethylamine	0.50
		7. Magnesium	0.25
		8. Calcium	0.50
		9. Triethylamine	5.00

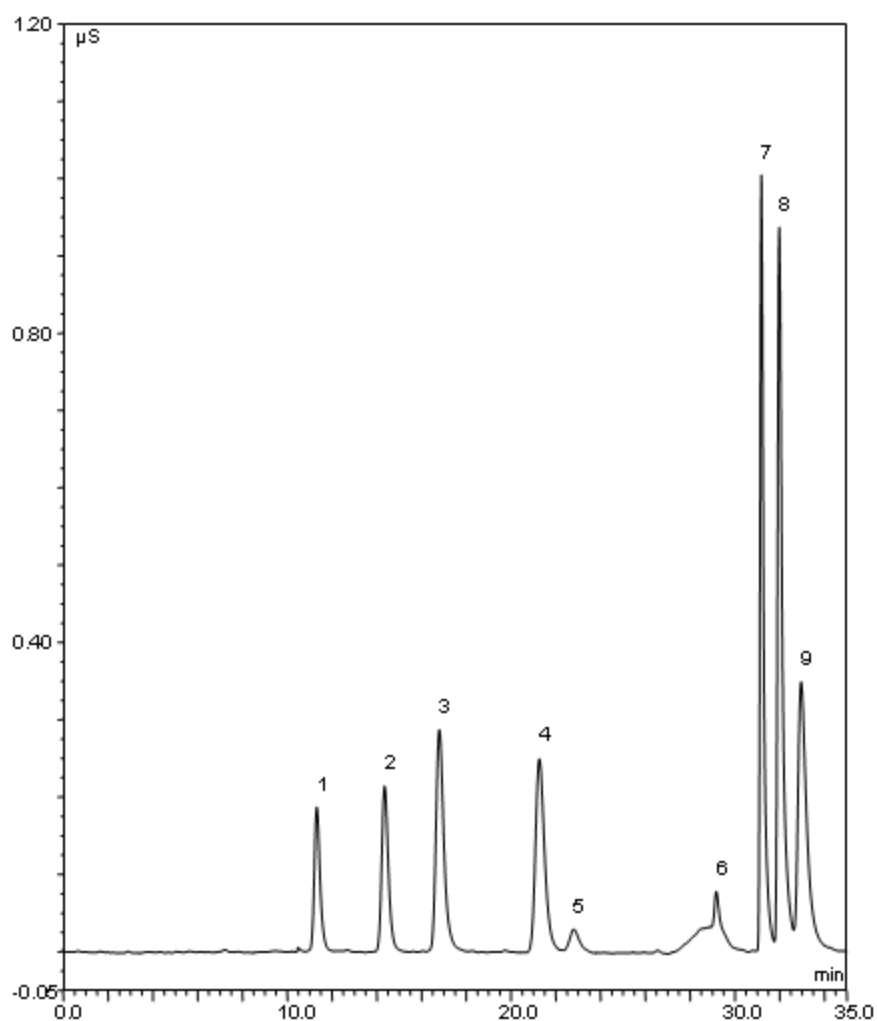


Figure 15
Ethylamines and the Six Common Cations with IonPac CS18 and CG18

5.2.14 Comparison of the IonPac CS17 with the IonPac CS18 and Temperature Effect on the IonPac CS18

The IonPac CS17 is the predecessor amine column to the CS18. Chromatograms A and B show the two columns run under the same conditions, and the difference in selectivities can be seen here. The CS17 is a more hydrophilic column, and therefore peak shape for the most hydrophobic of the analytes shown here, triethylamine, is better on it than on the CS18. Notice also that except for the potassium/dimethylamine pair, there is better monovalent peak selectivity on the CS18. The co-eluting pair can be resolved by decreasing the eluent concentration and changing the temperature of the column, as is shown in chromatograms C and D. At the higher temperature, potassium elutes first, and peak efficiencies are better. At the lower temperature, the elution order switches. The column was run in an ICS 3000 chromatographic system, which, besides heating, has the ability of cooling the column below ambient temperatures.

Detection: Suppressed Conductivity
Injection Vol.: 5 μ L

Peaks:	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Potassium	1.0
5. Dimethylamine	1.0
6. Triethylamine	7.0
7. Magnesium	0.5
8. Calcium	1.0

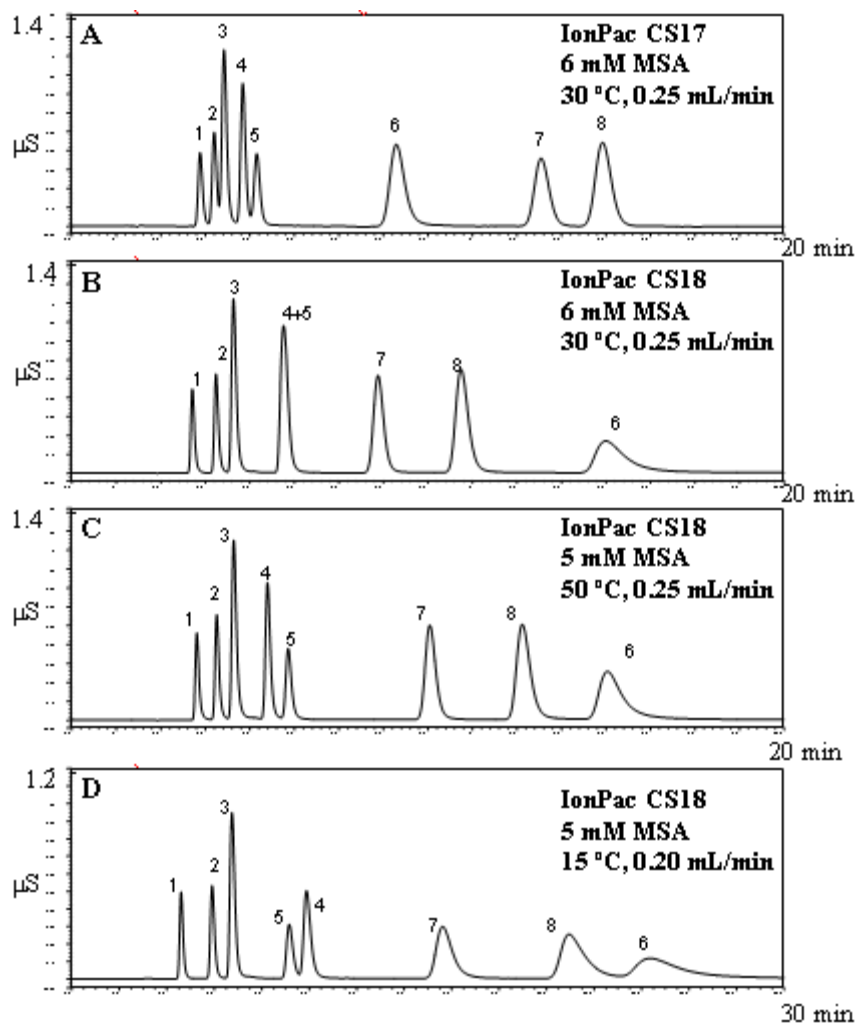


Figure 16
Comparison with IonPac CS17 and Temperature Effect on IonPac CS18

5.2.15 Effect of Sample pH

The IonPac CS18 resin contains weak carboxylic acid cation exchange sites. The ionization of the sites are dependent on the eluent and on the sample pH. Furthermore, because these cation exchange sites are hydronium-selective, the sample pH will have an impact on the elution of the analyte cations from such sites. Due to the nature of the cation exchange sites as well as to the moderate cation exchange capacity, as the sample pH decreases, so do peak efficiencies and asymmetries. Samples of low pH can be pre-treated before injection with an OnGuard II A cartridge, shipped in the bicarbonate form. Anions in the sample will be exchanged for the bicarbonate in the OnGuard resin. The bicarbonate ions neutralize the hydronium ions in the sample.

If the sample injection volume is large, the sample pH effects will be more severe than if the sample injection volume is small.

Column:	IonPac CS18 (2 x 250 mm) Analytical column	Peaks:	mg/L
Eluent	5 mM MSA	1. Lithium	0.1
Eluent Source:	EGC II MSA cartridge	2. Sodium	0.4
Flow Rate:	0.25 mL/min	3. Ammonium	0.5
Temperature:	30 °C	4. Ethanolamine	0.5
Detection:	Suppressed Conductivity	5. Potassium	1.0
Injection Vol.:	5 µL	6. Magnesium	0.5
		7. Calcium	1.0

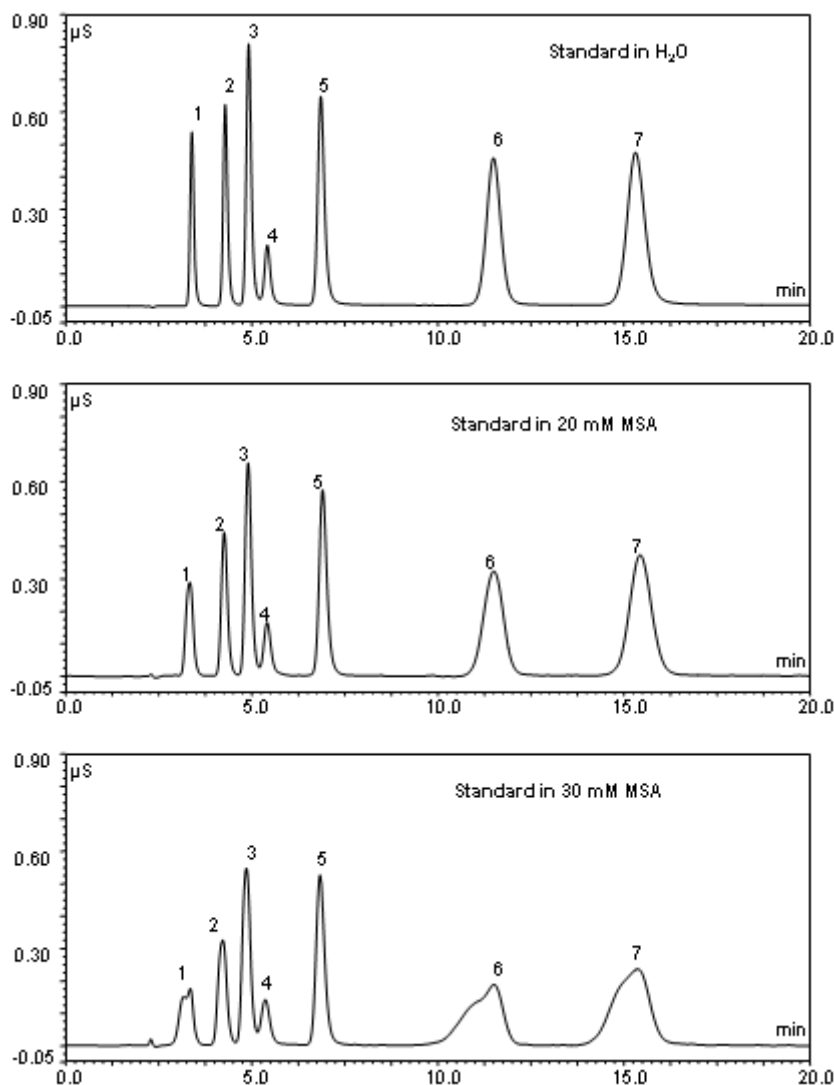


Figure 17
Effect of Sample pH on IonPac CS18

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac CS18 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").

Table 4
CS18/CG18 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
Suppressed Conductivity Improper Suppressor Operation	CSRS or CAES	Check Current	6.5, Component Manual
	Not Suppressing	Check "REGEN OUT" Flow	6.5, Component Manual
		Check for leaks	6.5, Component Manual
	CMMS Not Suppressing	Check Regenerant	6.5, Component Manual
		Check AutoRegen Cartridge	6.5.C, Component Manual
Hardware Operation	Air Bubble Trapped in CSRS or CAES	Remove Bubble by Loosening Fittings	6.4
	Proportioning Valve	Service Valve	Component Manual
	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	
		Dilute Sample	
		Use OnGuard II A	
Fronting Peaks	Low Sample pH	Reduce Sample Size	
		Dilute Sample	
		Use OnGuard II A	
	Column Overloading	Reduce Sample Size	5.18
	Contaminated or Deformed Bed Support	Replace Bed Support	6.1.2
Tailing Peaks	Column Headspace	Replace Column	6.6.1.A
	Column Overloading	Reduce Sample Size	
	Column Contaminated	Clean Column	Column Care B.4.2
	Contaminated Suppressor	Clean Suppressor	6.3.1.A, 6.5, Component Manual

Observation	Cause	Action	Reference Section
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
	First Peaks Elute Too Fast	Equilibrate to First Eluent	6.6.3.A
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	
	Contaminated Suppressor	Clean Suppressor	
	Contaminated Inlet Bed Support in Column	Replace Inlet Clean Suppressor	6.1.2

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac CG18 Guard Column plus the CS18 Analytical Column when using the test chromatogram conditions should be equal or less than 2,350 psi (16.18 MPa). See Appendix A. If the system pressure is much higher than 2,450 psi, determine the cause of the high pressure immediately. The system should be operated with a High-Pressure In-Line Filter (P/N 044105), 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, suppressor, and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 2, "IonPac CS18/CG18 Operating Parameters").

Refer to the appropriate manual for cleanup or replacement of the problem component.

6.1.2 Replacing Column Bed Support Assemblies

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

- A. Disconnect the column from the system.**
- B. Carefully unscrew the inlet (top) column fitting.** Use two open-end wrenches.
- C. Remove the bed support.** Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you **do not scratch the walls of the end fitting**. Discard the old bed support assembly.
- D. Place a new bed support assembly into the end fitting.** Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

IonPac CS18 2-mm Column Part	Part Number (P/N)
Analytical Column	062878
Guard Column	062880
Bed Support Assembly	044689
End Fitting	043278

CAUTION: *If the column tube end is not clean when inserted into the end fitting, particulate matter may obstruct a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.*

- E. Screw the end fitting back onto the column.** Tighten it fingertight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.
- F. Reconnect the column to the system and resume operation.**

NOTE: *Replace the outlet bed support ONLY if high pressure persists after replacement of the inlet fitting.*

6.2 Preparation of Eluents

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

6.3 Contamination

6.3.1 A Contaminated Guard or Analytical Column

Determine if the column is contaminated. Column contamination can lead to a loss of column capacity since all of the cation exchange sites will no longer be available for the sample ions. Polyvalent cations may be concentrating on the column over a series of runs. Remove the IonPac CG18 Guard and CS18 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 CG18 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. In a suppressed conductivity system, a 5 mM MSA eluent should give a background < 0.5 μ S. The background with a 4 mM MSA eluent should be 1380–1480 μ S in a nonsuppressed conductivity system, and with deionized water < 0.5 μ S. If it is not, check the detector/conductivity cell calibration. See the appropriate manual for details.

- A. **Use chemicals and deionized water of the proper purity.** Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.
- B. **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. **Glass eluent reservoirs can be a source of sodium contamination in the eluent.** Two-liter polyethylene eluent reservoirs (P/N 039163) are recommended.
- D. **For EG50 or EG40 operation, use a CR-CTC Trap Column.** Install a CR-CTC Cation Trap Column (P/N 060478) if using an Eluent Generator with EGC II MSA cartridge.

6.3.2 Sample Loop and/or Tubing Contamination

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

A. Weak Cation Exchangers

Carboxylated stationary phases used in the IonPac CS12, CS12A, CS14, CS15, CS16, CS17, SCS 1 and the CS18 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 ($\text{pH} \leq 4$), these sites are protonated by the sample and rendered inactive, so that the divalent quantification is not affected.

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

A simple test can be performed (when using a column such as the IonPac CS18 which contains a carboxylated resin) with methanesulfonic acid or sulfuric acid to see if the sample loop has been contaminated:

1. Prepare a standard containing 0.5 ppm of calcium and add a small amount of 0.2 mM sodium hydroxide so that the final pH of the standard is between 6.5 - 7.5.
2. With the sample loop in the load position, flush the loop with just enough standard to rinse and fill the loop (e.g. if the loop is 25 μL , flush it with no more than 100 μL).
3. Run the standard and record the peak area.
4. Repeat steps 2 and 3, but this time flush the loop with about 5 mL of standard.
5. If after repeating steps 2 through 4, the peak area for calcium recorded in 4 is significantly larger than that in 3, then the sample loop is contaminated and acting as a concentrator.
6. Replace the sample loop with new tubing and repeat this test.
7. If there is still a quantification problem, check other components of the system (tubing, injection valve, detector cell) or call your Dionex representative.

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

1. Your application involves high levels of divalent cations e.g. > 5 ppm calcium; the “concentration error” is small, percentage-wise.
2. The pH of your samples and standards is < 4 .
3. A constant volume of sample (and standard), only slightly larger than the sample loop, is flushed through the loop at a constant sampling flow rate.

6.4 High Background or Noise

In a properly working nonsuppressed conductivity system, the background conductivity using the operating conditions described in Section 4, “Operation,” should be 1380–1480 μS . In a suppressed conductivity system, the background should be < 0.5 μS . If the background is low but the system is noisy, an air bubble may be trapped in the system. In suppressed IC, with the system running, disconnect the ELUENT OUT line from the suppressor and allow bubbles to escape. Reconnect the line. Do not take too long to do this, as the current is still being applied to the CSRS ULTRA and the eluent flow is required to produce regenerant.

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

A system with a high background ($> 1500 \mu\text{S}$ in the nonsuppressed mode, > 0.5 in the suppressed mode) will probably also have higher noise, resulting in increased detection limits.

- A. Make sure that the eluent is prepared correctly (see Section 4, Operation).**
- B.** Determine if the columns or system are contaminated (see Section 6.3, “A Contaminated Guard or Analytical Column”).
- C. Determine if the Suppressor is the cause of the high background and/or noise.** If the above items have been checked and the problem still persists, the suppression system is causing the problem. See Section 6.5, “A Cation Self-Regenerating Suppressor (CSRS ULTRA), Cation MicroMembrane Suppressor (CMMS III), or Cation Atlas Eletrolytic Suppressor (CAES) that Does Not Suppress Properly.”

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

<u>ELUENT</u>	<u>EXPECTED BACKGROUND CONDUCTIVITY</u>
4 mN Methanesulfonic acid	1380–1480 μS (nonsuppressed system) < 0.2 μS (suppressed system)
Deionized Water	< 0.5 μS (nonsuppressed system) < 0.2 μS (suppressed system)

6.5 A Suppressor Not Suppressing Properly

If the Cation Self-Regenerating Suppressor, Cation Atlas Electrolytic Suppressor, or the Cation MicroMembrane Suppressor is causing the problem, refer to the Cation Self-Regenerating Suppressor Product Manual (Document No. 031139), to the Cation Atlas Electrolytic Suppressor Product Manual (Document No. 031770), or to the Cation MicroMembrane Suppressor Product Manual (Document No. 034359) 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 correct back pressure tubing is properly installed after the CSRS ULTRA, 40 psi is ideal.
- 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 0.25 mL/min for 2-mm operation). If it is used in the AutoSuppression External Water Mode, refer to appropriate suppressor manual for recommended regenerant flow rates.
- E. Check the eluent flow rate.** See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. Refer to the Cation Self-Regenerating Suppressor Product Manual (Document No. 031139) or to the Cation MicroMembrane Suppressor Product Manual (Document No. 034359) for assistance in determining if the eluent is within suppressible limits.
- F. If you are using an AutoRegen Accessory with the CSRS (in the Chemical Suppression Mode) or the CMMS, prepare fresh regenerant solution.** Test both the suppressor and the AutoRegen Regenerant Cartridge for contamination.
 - 1. If the background conductivity is high after preparing fresh regenerant and bypassing the AutoRegen Regenerant Cartridge, you probably need to clean or replace your CSRS or CMMS.**

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

NOTE: *Do not recycle the regenerant through the Cation Regenerant Cartridge if the eluent contains acetonitrile. Refer to the Cation Atlas Electrolytic Suppressor Product Manual (Document No. 031771) for trouble shooting assistance.*

6.6 Poor Peak Resolution

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

6.6.1 Loss of Peak Efficiency Throughout the Chromatogram

- A. **Extra-column effects can result in sample band dispersion, causing loss of peak efficiencies.** Make sure you are using PEEK tubing with an i.d. of no greater than 0.005" 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 6.1.2, “Replacing Column Bed Support Assemblies”). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

6.6.2 Loss of Resolution Throughout the Chromatogram Due to Shortened Retention Times

Even with adequate system and column efficiency, resolution of peaks will be compromised if analytes elute too fast.

- A. **Check the flow rate.** 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").

6.6.3 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.
- C. **Be sure that the column is equilibrated to the initial eluent.** Typically gradient or step change applications require approximately 10 minutes to equilibrate to the initial eluent. The minimum equilibration time can be determined by making successive runs with increasing equilibration times. The column is equilibrated to the initial eluent when additional equilibration time does not increase the runtime of the first eluting peaks.

6.7 Spurious Peaks

- A. **Eluents made with chemicals lacking the required purity will contaminate columns rapidly.** Remake all stock solutions and eluents using chemicals that meet the chemical requirements specified in Section 4.3, "Chemical Purity Requirements." Clean the column as indicated in "Column Cleanup" (see, "Column Care").
- B. **Spurious peaks may be due to column contamination.** If the samples contain an appreciable level of polyvalent cations, polyvalent cations may contaminate the column. As a result, the retention times for the analytes will decrease, and spurious, inefficient peaks can show up at unexpected times. This problem may be solved by increasing the time between analyses or by adding a regeneration step between successive runs to elute polyvalent cationic contaminants off the column before the next sample injection takes place.
- C. **An injection valve that needs service may produce baseline upsets.** This baseline upset can show up as one or multiple peaks of varying size(s) and shape(s). Typically this will occur when the particular valve needs to be cleaned or torqued (see the system manual). Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

APPENDIX A - COLUMN CARE

A.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 CS18 Analytical or Guard Column is 4,000 psi (27.57 MPa).

A.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. 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.

A.3 Column Storage

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

A.4 Column Cleanup

The following column cleanup protocols have been divided into two general isocratic protocols to remove acid-soluble or organic contaminants. They can be combined into one gradient protocol if desired but the following precautions should be observed.

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

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to $\leq 5\%$ levels and the ionic strength of the eluent to ≤ 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

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

A. Prepare a 500 mL of 100 mM HCl for the cleanup solution.

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

B. Disconnect the CSRS ULTRA, CAES, or CMMS III from the IonPac CS18 Analytical Column.

If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.

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

- C. **Set the pump flow rate** to 0.25 mL/min for a CS18 2-mm Analytical or Guard Column.
- D. **Pump the cleanup solution (100 mM HCl) through the column for 60 minutes.**
- E. **Equilibrate the column(s) with eluent** before resuming normal operation for at least 30 minutes.
- F. **Reconnect the CSRS ULTRA, CAES, or CMMS III to the CS18 Analytical Column and place the guard column in line** between the injection valve and the analytical column if your system was originally configured with a guard column.

A.4.2 Hydrophobic Cations and Organic Contaminants

Install the analytical and guard columns for cleanup following the steps below:

- A. Disconnect the analytical column from the injection valve and the suppressor.
Connect the IonPac CS18 Analytical Column directly to the gradient pump.
Place the guard after the analytical column.
Direct the effluent from the analytical column directly to a waste container.
- B. **Set the flow rate to 0.25 mL/min.**
- C. **Use the following gradient program to remove hydrophobic cations and organic contaminants.**

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

Time (min)	%E1	%E2
0.0	100	0
30.0	80	20
35.0	80	20
65.0	100	0
70.0	100	0

- D. **Reconnect the IonPac CS18.** Connect the guard and analytical columns. Connect the analytical column OUTLET to the suppressor. Connect the analytical column INLET to the IonPac CG18 2-mm.
- E. **Equilibrate the column with eluent before resuming normal operation.**

APPENDIX B - CONFIGURATION

B.1 Configuration of IC Systems

Table 6
Configuration of Ion Chromatography Systems

CONFIGURATION	2-mm
Eluent Flow Rate	0.25 mL/min
SRS Suppressor	CSRS® ULTRA II (2-mm) (P/N 061564)
MMS Suppressor	CMMS III (2-mm) (P/N 056753)
CAES Suppressor	CAES® (P/N 056158)
	NOTE Do not run suppressors over 40°C. If an application requires a higher temperature, place suppressor outside of chromatographic oven.
Regenerant Flow Rate	Refer to appropriate suppressor manual.
Injection Loop	2 - 25 µL Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the Microbore GM-4 (2-mm) Mixer (P/N 049135).
Pumps	Use the DP/SP/GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. The GPM-2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2-mm gradient chromatography. NOTE: Use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II MSA cartridge (P/N 053922) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.
Detectors	AD20 Cell (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL) (P/N 043120) DC/CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 (P/N 044130) or Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-2 (P/N 043117) is optimized for 2-mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi

B.2 Tubing Back Pressures

Table 7
Tubing Back Pressures for Suppressed and Non-suppressed IC

Color	Dionex P/N	I.D. inch	I.D. cm	Volume mL/ft	Back pressure Psi/ft. at 1 mL/min	Back pressure Psi/ft. at 0.25 mL/min	Back pressure Psi/cm at 1 mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642