Thermo Scientific

Dionex IonPac AS19 Columns

Product Manual

P/N: 065003-08 December 2013

Thermo SCIENTIFIC

PRODUCT MANUAL

DIONEX IONPAC AG19 GUARD COLUMN

(4 x 50 mm, P/N 062887)
(2 x 50 mm, P/N 062888)
(0.4 x 50 mm, P/N 072065)

DIONEX IONPAC AS19 ANALYTICAL COLUMN

(4 x 250 mm, P/N 062885)
(2 x 250 mm, P/N 062886)
(0.4 x 250 mm, P/N 072064)

Document No. 065003
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SECTION 1 - INTRODUCTION

The IonPac® AS19 Analytical Column in combination with the AG19 Guard Column is designed for the analysis of inorganic anions and oxyhalides including fluoride, chlorite, bromate, chloride, nitrite, chlorate, bromide, nitrate, sulfate, and phosphate. The selectivity of the IonPac AS19 Guard plus Analytical Column set has been designed to retain fluoride well out of the water dip (system dip) and to separate oxyhalides and the common anions using hydroxide gradients. The AS19 is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS19 can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor 300 (ASRS 300). The IonPac AS19 has nominal efficiency for sulfate using standard operating conditions of at least 7,000 plates/column.

The IonPac AS19 Capillary Column (0.4 x 250 mm) is packed with the same material as the equivalent standard bore version (producing the same performance as a 4 mm column), but requires only 1/100th the eluent flow rate. The capillary format offers the advantage of less eluent consumption providing reduced costs.

Table 1
IonPac AS19/AG19 Packing Specifications

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column [a]	Functional Group	Hydrophobicity
AS19 4 x 250 mm	7.5	55	240	Alkanol quaternary ammonium	Ultralow
AG19 4 x 50 mm	11	55	6	Alkanol quaternary ammonium	Ultralow
AS19 2 x 250 mm	7.5	55	60	Alkanol quaternary ammonium	Ultralow
AG19 2 x 50 mm	11	55	1.5	Alkanol quaternary ammonium	Ultralow
AS19 Capillary 0.4 x 250 mm	7.5	55	2.4	Alkanol quaternary ammonium	Ultralow
AG19 Capillary 0.4 x 50 mm	11	55	0.06	Alkanol quaternary ammonium	Ultralow

a Analytical Column resin composition: supermacroporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene. Guard Column resin composition: microporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene.

Table 2
AS19/AG19 Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS19 4-mm Analytical	≤ 1800 (12.41)	1.0	2.0
AG19 4-mm Guard	≤ 300 (2.07)	1.0	2.0
AS19 + AG19 4-mm columns	**≤ 2100 (14.48)**	**1.0**	**2.0**
AS19 2-mm Analytical	≤ 1800 (12.41)	0.25	0.5
AG19 2-mm Guard	≤ 300 (2.07)	0.25	0.5
AS19 + AG19 2-mm columns	**≤ 2100 (14.48)**	**0.25**	**0.5**
AS19 0.4-mm Capillary	≤ 2200 (15.17)	0.010	0.020
AG19 2-mm Guard Capillary	≤ 330 (2.28)	0.010	0.020
AS19 + AG19 0.4-mm Capillary	**≤ 2530 (17.45)**	**0.010**	**0.020**

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of Dionex instrumentation and columns through the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the Dionex Offices listed in, "Dionex Worldwide Offices."

SECTION 2 - 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.

- For an ICS in 2-mm format, Dionex recommends a microbore isocratic pump, standard bore isocratic pump, microbore gradient pump, or standard bore gradient pump .
- For an ICS in 4-mm format, Dionex recommends a standard bore isocratic pump or standard bore gradient pump.
- For an ICS in 0.4-mm format, Dionex recommends a Capillary IC System such as the ICS-5000 System.

See Appendix C, Comparison of Ion Chromatography Systems for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor (SRS), Capillary Electrolytic Suppressor (CES), MicroMembrane Suppressor (MMS), injection loop, system void volume, detectors, and tubing back pressure.

SECTION 3 - INSTALLATION

3.1 System Requirements

3.1.1 System Requirements for 0.4 mm Operation

The IonPac AS19 0.4 mm Capillary Guard and Capillary Column are designed to be run on a capillary ion chromatograph system equiped with suppressed conductivity detection. It is recommended to run the capillary column only on the ICS-5000 capillary system for best performance.

3.1.2 System Requirements for 2-mm Operation

The IonPac AS19 2-mm Guard and Analytical Columns are designed to run on Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min or greater can be performed on a pump with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a microbore pump (1/16" pistons) must be employed.

3.1.3 System Requirements for 4-mm Operation

The IonPac AS19 4-mm Guard and Analytical Columns are designed to run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a pump with a standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons).

3.1.4 System Void Volume

When using 2-mm columns, it is particularly important to minimize system void volume. The system void volume should be scaled down to at least 1/4 of the system volume in a standard 4-mm system. For best performance, all of the tubing installed between the injection valve and detector should be 0.005" (P/N 044221) ID PEEK tubing. 0.010" ID PEEK tubing (P/N 042260) or 0.012" Tefzel tubing may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

3.2 The Sample Concentrator

The Trace Anion Concentrator Low Pressure Column (TAC-LP1, P/N 046026), the Trace Anion Concentrator Ultra Low Pressure Column (TAC-ULP1, P/N 061400), the Ultra Trace Anion Concentrator Low Pressure Column (UTAC-LP1, P/N 063079) or (UTAC-LP2, P/N 079917), the Ultra Trace Anion Concentrator Ultra Low Pressure Column (UTAC-ULP1, P/N 063475) or (UTAC-ULP2, P/N 079918), the Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459) or (UTAC-XLP2, P/N 072781), or the IonPac AG19 Guard Column can be used for trace anion concentration work with the 2 mm and 4 mm AS19 columns. The function of a concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the concentrator column, lowering detection limits by 2-5 orders of magnitude. The concentrator column is used in lieu of the sample loop. Pump the sample onto the concentrator column in the **OPPOSITE** direction of the eluent flow. When using concentration techniques, do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column. For Trace Anion Concentration work with the AS19 0.4 mm column use the AG19 0.4 mm Capillary Guard Column or the IonSwift MAC-100 Column.

For more detailed information on sample concentration techniques for high sensitivity work and a detailed discussion of anion concentration techniques refer to:

- Section 3, "Operation," of the Trace Anion Concentrator Low Pressure (TAC-LP1) and Ultra Low Pressure (TAC-ULP1) Column Product Manual (Document No. 034972),
- Section 3, "Operation," of the Ultra Trace Anion Concentrator Low Pressure (UTAC-LP1), Ultra Low Pressure (UTAC-ULP1), and Extremely Low Pressure (UTAC-XLP1) Column Product Manual (Document No. 065091.)
- Section 4, "Operation," of the Ultra Trace Anion Concentrator 2 Low Pressure (UTAC-LP2), Ultra Low Pressure (UTAC-ULP2), and Extremely Low Pressure (UTAC-XLP2) Column Product Manual (Document No. 065376.)

These techniques can also be applied to the AG19.

CAUTION **IonPac Trace Anion Concentrator (TAC-2) Column (P/N 043101) is *not* optimized for use with hydroxide eluents and should *not* be used for concentrator work with the IonPac AS19. Use the AG19 4-mm or AG19 2-mm guards.**

3.3 The Injection Loop

3.3.1 The 2-mm System Injection Loop, 2 - 15 µL

For most applications on a 2-mm analytical system, a 2 - 15 µL injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2-mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. The AS19 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4-mm analytical system (Section 2, "Comparison of 2-mm and 4-mm Ion Chromatography Systems").

3.3.2 The 4-mm System Injection Loop, 10 - 50 µL

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop is sufficient. Generally, you should not inject more than 50 nanomoles of any one analyte onto the 4-mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity.

3.3.3 The 0.4-mm System Injection Loop, 0.4 µL Internal Loop

For most applications on a 0.4-mm capillary system, a 0.4 µL injection loop is sufficient. Generally, you should not inject more than 0.5 nanomoles total anion concentration onto a 0.4-mm capillary column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity.

3.4 The IonPac AG19 Guard Column

An IonPac AG19 Guard Column is normally used with the IonPac AS19 Analytical Column. Retention times will increase by approximately 5% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column. Replacing the AG19 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS19 Analytical Column.

3.5 Installing the CR-ATC Trap Column for Use with EGC II KOH Cartridge

For IonPac AS19 applications using the EGC KOH cartridge, a CR-ATC Continuously Regenerated Trap Column (P/N 060477 or 072078) should be installed at the EGC eluent outlet to remove trace level anionic contaminants from the carrier deionized water. See the CR-TC Product Manual (Document No. 031910) for instructions.
As an alternative, the ATC-HC Trap Column (P/N 059604) should be installed between the pump outlet and the inlet of the EluGen Cartridge in the Module to remove anionic contaminants from the carrier deionized water. See the ATC-HC Product Manual (Document No. 032697) for instructions.

If the lower capacity ATC-3 Trap Column (P/N 059660 and 079932) is used, it should be installed between the gradient pump and the injection valve to remove anionic contaminants from the eluent. The ATC-3 column is used when performing sodium hydroxide gradient anion exchange applications using hand-prepared bottled eluents. See the ATC-3 Product Manual (Document No. 032697) for instructions.

The ATC-HC (P/N 059604) and ATC-3 Trap Columns will require off-line regeneration. To use the ATC-HC or ATC- 3 Anion Trap Columns, refer to the Product Manuals.

3.6 Eluent Storage

IonPac AS19 columns are designed to be used with hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

3.7 Anion Self-Regenerating Suppressor and Anion Capillary Electrolytic Suppressor Requirements

An Anion Self-Regenerating Suppressor should be used for 2-mm and 4-mm applications that require suppressed conductivity detection. It is compatible with solvent containing eluents and aqueous ionic eluents of all concentrations with which the systems and columns are compatible. Aqueous ionic eluents can be used in all ASRS 300 modes of operation.

CAUTION

Solvent containing eluents should be used in the AutoSuppression External Water Mode.

For IonPac AS19 0.4-mm Capillary Column, use the ACES 300 (0.4-mm, P/N 072052).
For IonPac AS19 4-mm Analytical Column, use the ASRS 300 (4-mm, P/N 064554).
For IonPac AS19 2-mm Analytical Column, use the ASRS 300 (2-mm, P/N 064555).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see Document No. 031956, the "Product Manual for the Anion Self-Regenerating Suppressor 300, the ASRS 300."

For detailed information on the operation of the Anion Capillary Electrolytic Suppressor, see Document No. 065386, the "Product Manual for the Anion Capillary Electrolytic Suppressor 300, the ACES 300."

3.8 Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS 300) may be used instead of an ASRS 300(4-mm) for applications that require suppressed conductivity detection. Use an AMMS 300 4-mm (P/N 064558) with the IonPac AS19 4-mm Analytical Column. It is compatible with all solvents and concentrations with which the systems and columns are compatible. For 2-mm operation, use the AMMS 300 2-mm (P/N 064559).

For detailed information on the operation of the Anion MicroMembrane Suppressor, see Document No. 031727, the "Product Manual for the Anion MicroMembrane Suppressor 300, the AMMS 300."

3.9 Using Displacement Chemical Regeneration (DCR) in the Chemical Suppression Mode

Dionex recommends using the Displacement Chemical Regeneration (DCR) Mode for chemical suppression using sulfuric acid and the Anion MicroMembrane Suppressor (AMMS 300). See the DCR kit manual, Document P/N 031664, for details.

SAFETY

Use proper safety precautions in handling acids and bases.

3.10 Detector Requirements

See Section 2, "Comparison of Ion Chromatography Systems," for 2-mm, 4-mm and 0.4-mm system detector, cell and thermal stabilizer requirements.

3.11 Using the EGC-KOH with AS19

Please refer to the EG40 manual, Document No. 031373, for information on the operation of the EG40. Please refer to the EG50 Product Manual, Document No. 031908, for information on the operation of the EG50. The AS19 column is recommended for use with ICS-2000, or ICS-3000 IC Systems equipped with an Eluent Generator. The AS19 can be used with older Dionex IC Systems equipped with an EG40 Eluent Generator or an RFC-30 Reagent Free Controller. The Eluent Generator is used to automatically produce potassium hydroxide gradients from deionized water.

3.12 Detector Requirements

See Section 2, "Comparison of Ion Chromatography Systems," for 2-mm, 4-mm and 0.4-mm system detector, cell and thermal stabilizer requirements.

3.13 Installation of the Capillary Column

1. Before installing the new separator column, tear off the column label and slide it into the holder on the front of the cartridge (see Figure 1).

2. For reference, Figure 1 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 2 shows the column cartridge after installation of only a capillary separator column.

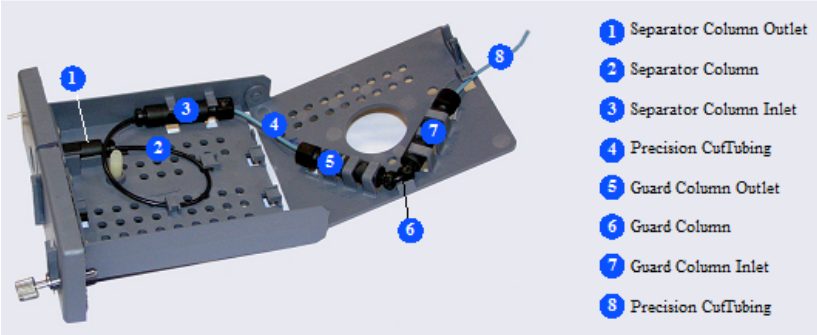


Figure 1
Separator and Guard Columns Installed in Column Cartridge

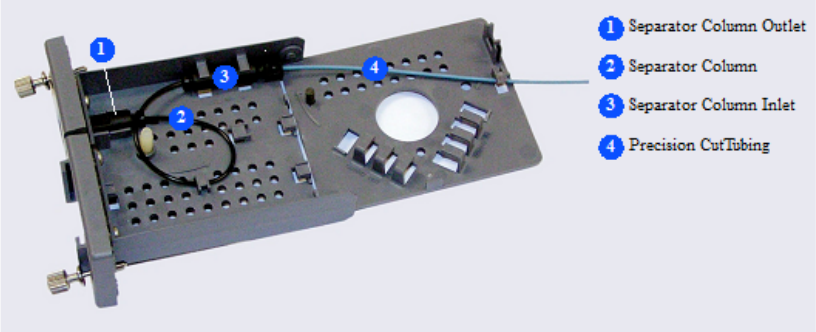


Figure 2
Separator Column Only Installed in Column Cartridge

3. Locate the IC Cube Tubing Kit (P/N 072186) that is shipped with the IC Cube. The tubing kit includes the following items:

Table 3
Contents of the IC Cube Tubing Kit (P/N 072186)

Part	Length / Quantity	Part Number	Used To Connect…
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue	65 mm (2.56 in)	072188	50 mm guard column outlet to 250 mm separator column inlet
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	115 mm (4.53 in)	072189	Guard column inlet to injection valve
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue	75 mm (2.93 in)	074603	35 mm guard column outlet to 150 mm separator column inlet
Precision cut 0.062-mm (0.0025-in) ID PEEK tubing, blue, labeled VALVE PORT 3	210 mm (8.27 in)	072187	Separator column inlet to injection valve (if a guard column is not present)
0.25-mm (0.010-in) ID PEEK tubing, black	610 mm (24 in)	042690	EG degas cartridge REGEN OUT to waste (if an EG is not present)
Fitting bolt, 10-32 hex double-cone (smaller), black	3	072949	Connect precision cut 0.062-mm (0.0025-in) ID PEEK tubing
Fitting bolt, 10-32 double-cone (larger), black	1	043275	Connect 0.25-mm (0.010-in) ID PEEK tubing (black)
Ferrule fitting, 10-32 double-cone, tan	4	043276	Use with both sizes of fitting bolts

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

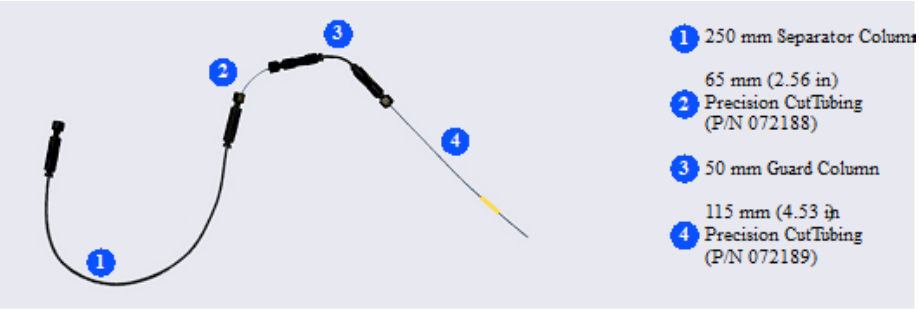


Figure 3
Tubing Connections for 250-mm Separator Column and 50-mm Guard Column

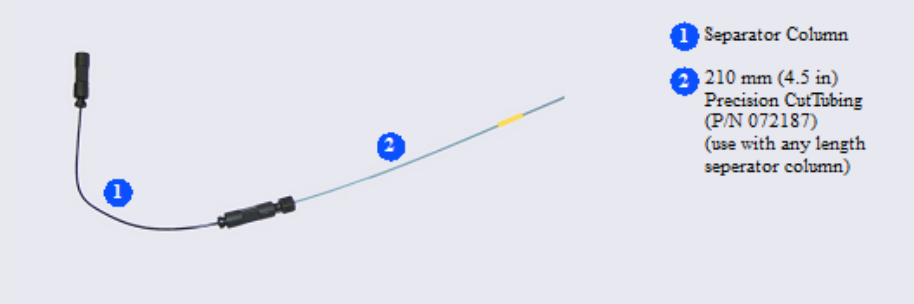


Figure 4
Tubing Connections for Separator Column Only

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

Figure 5
Column Outlet Fitting Installed in Column Cartridge

7. Coil the separator column tubing inside the cartridge as shown in Figure 1 or Figure 2. Secure the column tubing and the inlet fitting in the clips on the column cartridge.

8. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.

9. Route the guard column inlet tubing (if used) or the separator column inlet tubing through the clip on the top edge of the column cartridge lid.

10. Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see Figure 6).

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

Figure 6
Column Cartridge Closed

SECTION 4 - OPERATION

4.1 General Operating Conditions

Sample Volume:	2-mm: 5 µL Loop + 0.8 µL Injection valve dead volume 4-mm: 25 µL Loop + 0.8 µL Injection valve dead volume 0.4-mm: 0.4 µL Loop
Column:	2-mm: AS19 2-mm Analytical Column + AG19 2-mm Guard Column 4-mm: AS19 4-mm Analytical Column + AG19 4-mm Guard Column 0.4-mm: AS19 0.4-mm Capillary Column + AG19 0.4-mm Capillary Guard Column
Eluent:	20 mM KOH
Eluent Source:	EGC-KOH
Eluent Flow Rate:	2-mm: 0.25 mL/min 4-mm: 1.0 mL/min 0.4-mm: 0.010 mL/min
Temperature:	30 °C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 (2-mm or 4-mm) Anion Capillary Electrolytic Suppressor, ACES 300 (0.4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300 (2-mm or 4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 1 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2 IonPac AS19 Operation Precautions

CAUTIONS
Filter and Degas Eluents
Filter Samples
Eluent pH betwee n 0 and 14
Sample pH between 0 and 14
0.5 mL/min Maximum Flow Rate for 2-mm Columns
2.0 mL/min Maximum Flow Rate for 4-mm Columns
Maximum Operating Pressure = 3,000 psi (20.68 MPa)

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 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.

4.3.2 Deionized Water

The deionized water used to prepare eluents should be Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 µm. 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.

4.3.3 Solvents

Solvents can be added to the ionic eluents used with IonPac AS19 columns to modify the ion exchange process or improve sample solubility. The solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultra high purity solvents that are compatible for HPLC and spectrophotometric applications. These ultra high purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column generated back pressures will depend on the solvent used, concentration of the solvent, the ionic strength of the eluent and the flow rate used. The column back pressure will vary as the composition of water-methanol and water-acetonitrile mixture varies. The practical back pressure limit for the IonPac AS19 columns is 3,000 psi (20.68 MPa).

The AS19 can withstand common HPLC solvents in a concentration range of 0–100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 4
HPLC Solvents for Use with IonPac AS19 Columns

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

*Higher concentration may only be used for limited duration applications such as column clean up at pressures < 2000 psi.

CAUTION

The Anion Self-Regenerating Anion Suppressor 300 (ASRS 300) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents. Do not use > 40% solvent on the ASRS-300 in the electrolytic mode (power on).

4.4 Making Eluents that Contain Solvents

When mixing solvents with water remember to mix solvent with water on a volume to volume basis. If a procedure requires an eluent of 90% acetonitrile, prepare the eluent by adding 900 mL of acetonitrile to an eluent reservoir. Then add 100 mL of deionized water or eluent concentrate to the acetonitrile in the reservoir. Using this procedure to mix solvents with water will ensure that a consistent true volume/volume eluent is obtained. Premixing water with solvent will minimize the possibility of outgassing.

NOTE

When purging or degassing eluents containing solvents, do not purge or degas the eluent excessively since it is possible that a volatile solvent can be "boiled" off from the solution.

Always degas and store all eluents in glass or plastic eluent bottles pressurized with helium. Only helium can be used to purge and degas ionic eluents containing solvents, since nitrogen is soluble in solvent containing eluents.

Acetonitrile (ACN) hydrolyzes to ammonia and acetate when left exposed to basic solutions. To prevent eluent contamination from acetonitrile hydrolysis, always add acetonitrile to basic aqueous eluents by proportioning the acetonitrile into the basic eluent with the gradient pump. Keep the acetonitrile in a separate eluent bottle containing only acetonitrile and water.

SAFETY

Never add the acetonitrile directly to the basic carbonate or hydroxide eluent bottle.

4.5 Eluent Preparation

Sodium Hydroxide Eluent Concentration

Weight Method

When formulating eluents from 50% sodium hydroxide, Dionex recommends weighing out the required amount of 50% sodium hydroxide. Use Fisher Grade 50% sodium hydroxide. Do not use pellets.

Example: To make 1 L of 20 mM NaOH use 1.60 g of 50% sodium hydroxide:
(as used in Appendix A, Quality Assurance Report)

$$\text{For 20 mM:} \quad \frac{0.020 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\%} = 1.6 \text{ g diluted to 1 L}$$

Volume Method

Although it is more difficult to make precise carbonate-free eluents for gradient analysis volumetrically, you may choose to use the following formula to determine the correct volume of 50% sodium hydroxide to be diluted.

$$\mathbf{g = dvr}$$

Where: **g = weight of sodium hydroxide required (g)**
d = density of the concentrated solution (g/mL)
v = volume of the 50% sodium hydroxide required (mL)
r = % purity of the concentrated solution

Example: To make 1 L of 20 mM NaOH use 1.04 mL of 50% sodium hydroxide:
(as used in Appendix A, Quality Assurance Report)

$$\text{For 20 mM:} \quad \frac{0.020 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\% \times 1.53 \text{ g/mL}} = 1.04 \text{ mL diluted to 1 L}$$

* This density applies to 50% NaOH. If the concentration of the NaOH solution is significantly different from 50%, the upper (weight method) calculation should be used instead.

Sodium Hydroxide Eluents

Dilute the amount of 50% (w/w) NaOH (in water) specified in Table 6, "Dilution of 50% (w/w) NaOH to Make Standard AS19 Eluents" with degassed, deionized water (having a specific resistance of 18.2 megohm-cm) to a final volume of 1,000 mL using a volumetric flask. Avoid the introduction of carbon dioxide from the air into the aliquot of 50% (w/w) NaOH or the deionized water being used to make the eluent. Do not shake the 50% (w/w) NaOH or pipette the required aliquot from the top of the solution where sodium carbonate may have formed.

Table 5

Dilution of 50% (w/w) NaOH to Make Standard AS19 Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
1.6 (1.04)	20
8.00 (5.25)	100
160.00 (104.6)	2 M

4.6 Regenerant Preparation for the AMMS 300

The Anion MicroMembrane Suppressor 300 (AMMS 300) requires the use of a regenerant solution. If you are using the AMMS 300 instead of the Anion Self-Regenerating Suppressor 300 (ASRS 300), see Document No. 031727, the "Product Manual for the Anion MicroMembrane Suppressor 300, the AMMS 300."

SECTION 5 - EXAMPLE APPLICATIONS

5.1 Recommendations for Optimum System Performance

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

The IonPac AS19 is designed for the determination of oxyhalides and the common anions and in less than 30 minutes using an gradient hydroxide eluent delivered with an EGC-KOH cartridge. Resolution of specific analytes can be further optimized if necessary by using gradient elution. In any type of gradient elution system it is important to use eluents that produce a minimum shift in baseline conductivity during the run, as well as a fast equilibration time from one run to the next. Because sodium or potassium hydroxide is converted to water in the suppressor, it is the best choice for an eluent. As long as the capacity of the suppressor is not exceeded, the eluent hydroxide concentration has little effect on background conductivity. For example, a gradient run could begin at 1.0 mM KOH and end at 80 mM KOH, with a resulting total baseline change of 1 to 2 µS.

Ensure that adequate equilibration time is allowed between runs. If downward shift in baseline is observed during the isocratic section of the chromatogram, increase the equilibration time.

You can increase the sensitivity of your system by using sample concentration techniques (see Section 3.3, "The Sample Concentrator").

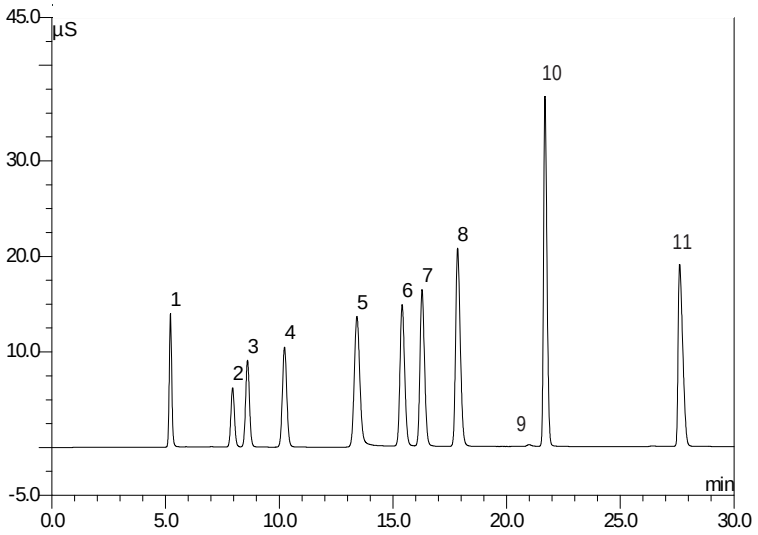
NOTE

Carbon dioxide readily dissolves in dilute basic solutions, forming carbonate. Carbonate contamination of eluents can effect the retention times of the anions being analyzed. Eluents should be maintained under an inert helium atmosphere to avoid carbonate contamination.

5.2 Separation of Oxyhalides and the Inorganic Anions Using Hydroxide Gradient

The following chromatogram demonstrates the separation of oxyhalides and inorganic anions using a hydroxide gradient. As illustrated in Figure 7, a simple hydroxide gradient will resolve chlorite from bromate and easily separates the common inorganic anions.

Injection volume:	25 µL
Column:	AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 10 min and 10 to 45 mM from 10 to 25 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression Recycle



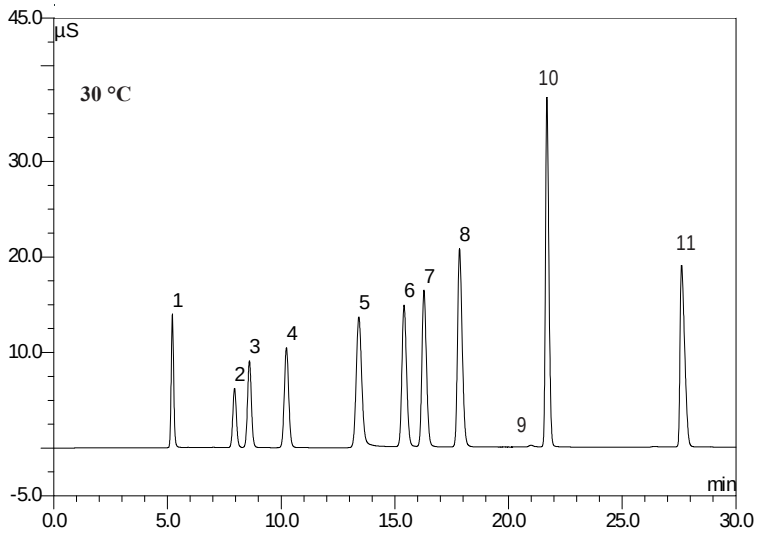
	Peaks	ppm
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Nitrite	15
6.	Chlorate	25
7.	Bromide	25
8.	Nitrate	25
9.	Carbonate	-
10.	Sulfate	25
11.	Phosphate	40

Figure 7
Separation of Oxyhalides and the Inorganic Anions Using Hydroxide Gradient

5.3 Effect of Temperature on Column Selectivity and Capacity

The recommended operating temperature for the IonPac AS19 column is 30 °C. However, if necessary, it is possible to operate this column at 35 °C, but notice that the resolution of chlorate and bromide decreases at 35 °C as compared to 30 °C. In addition, retention time will decrease at a faster rate when the column is exposed to 35 °C over a long period of time. If 35 °C is required for the application, it is recommended to reduce the column temperature to 30 °C when not in use, or store the column in the refrigerator when not in use.

Injection volume:	25 µL
Column:	AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 10 min and 10 to 45 mM from 10 to 25 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	See chromatograms
Suppressor:	Anion Self-Regenerating Suppressor (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression Recycle



	Peaks	ppm
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Nitrite	15
6.	Chlorate	25
7.	Bromide	25
8.	Nitrate	25
9.	Carbonate	-
10.	Sulfate	25
11.	Phosphate	40

Figure 8a
Temperature at 30 °C

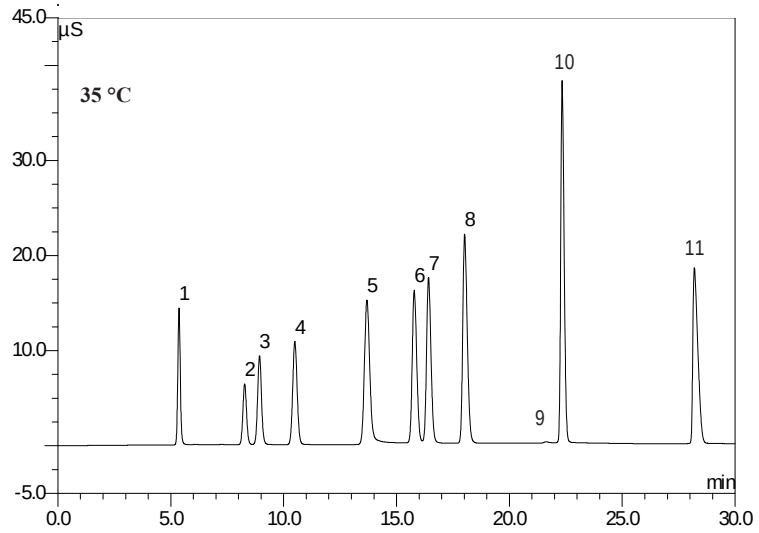


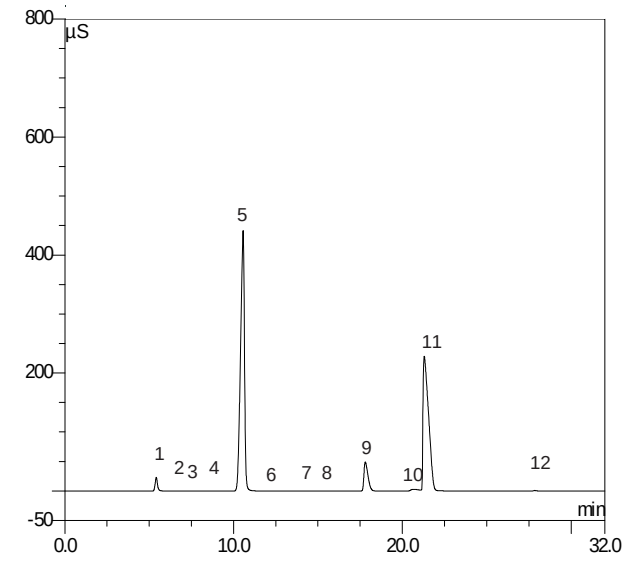
Figure 8b
Temperature at 35 °C

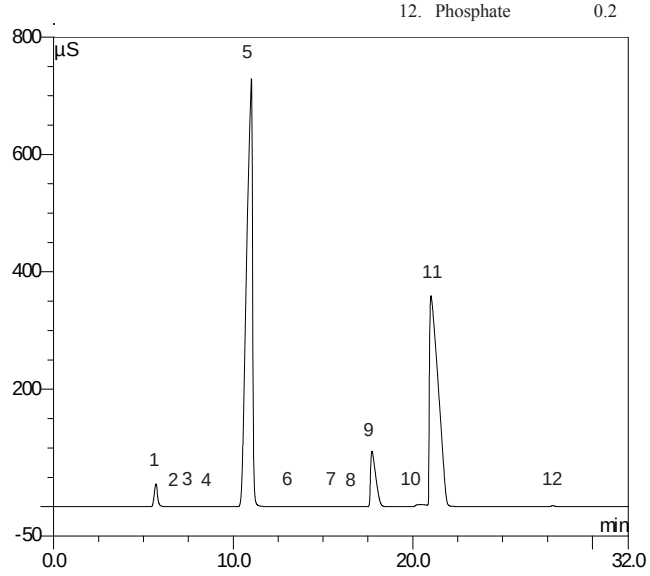
5.4 Separation of Anions in Simulated Drinking Water

The following chromatogram shows the analysis of a simulated drinking water sample using the IonPac AS19 column and a 200 µL injection volume. Notice the excellent separation of chlorite, bromate, chlorate, and bromide in the presence of high levels of chloride, nitrate, and sulfate. Also notice that the response for ions like chlorite, bromate, chlorate, and bromide can be increased by increasing the sample volume.

Injection volume:	See chromatogram
Column:	AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 10 minutes and 10 to 45 mM from 10 to 25 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor II (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression External Water

	Peaks	mg/L (ppm)
1.	Fluoride	1
2.	Formate	-
3.	Chlorite	0.005
4.	Bromate	0.005
5.	Chloride	50
6.	Nitrite	0.005
7.	Chlorate	0.005
8.	Bromide	0.005
9.	Nitrate	10
10.	Carbonate	25
11.	Sulfate	50
12.	Phosphate	0.2





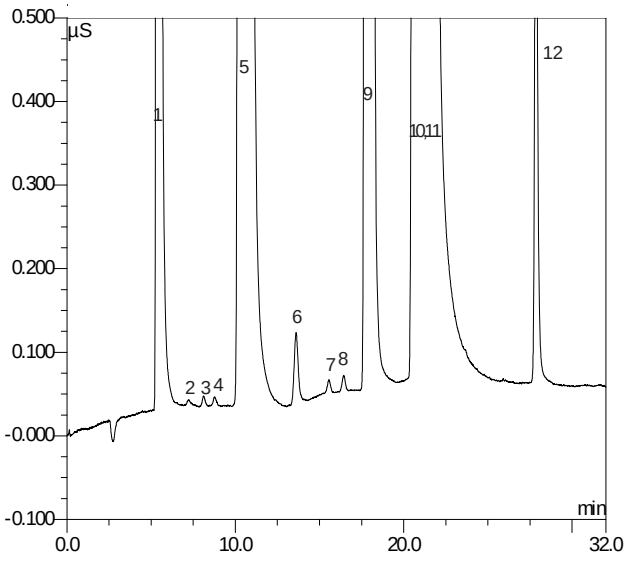


Figure 9a
Simulated Drinking Water
Injection Volume 200 µL

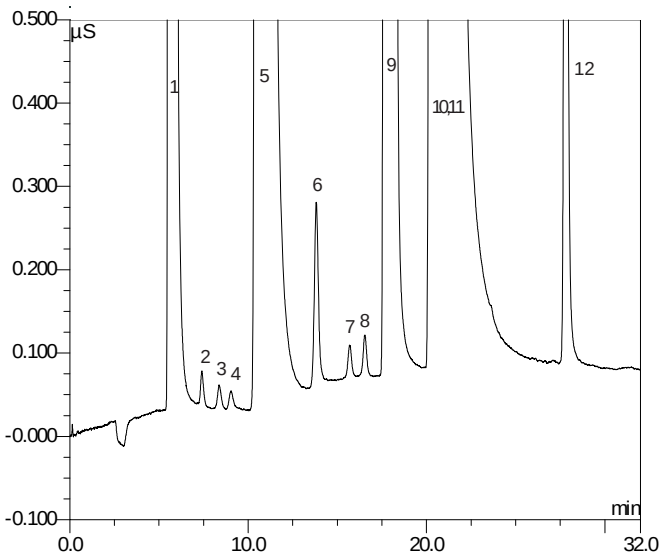


Figure 9b
Simulated Drinking Water
Injection Volume 500 µL

5.5 Separation of Anions in Sunnyvale Drinking Water Spiked with Surrogate Anion

The following chromatogram shows the analysis of a drinking water sample using the IonPac AS19 column and a 500 µL injection volume. Notice the excellent separation of trichloroacetate (surrogate anion) from nitrate and carbonate when the drinking water was spiked with 1 ppm of trichloroacetate.

Column:	AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 13 minutes and 10 to 45 mM from 13 to 28 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Injection volume:	500 µL
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor II (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression External Water

Peaks

1. Fluoride
2. Acetate
3. Formate
4. Chlorite
5. Chloride
6. Nitrite
7. UNK
8. Chlorate
9. Bromide
10. Nitrate
11. Trichloroacetate
12. Carbonate
13. Sulfate
14. Oxalate
15. Phosphate

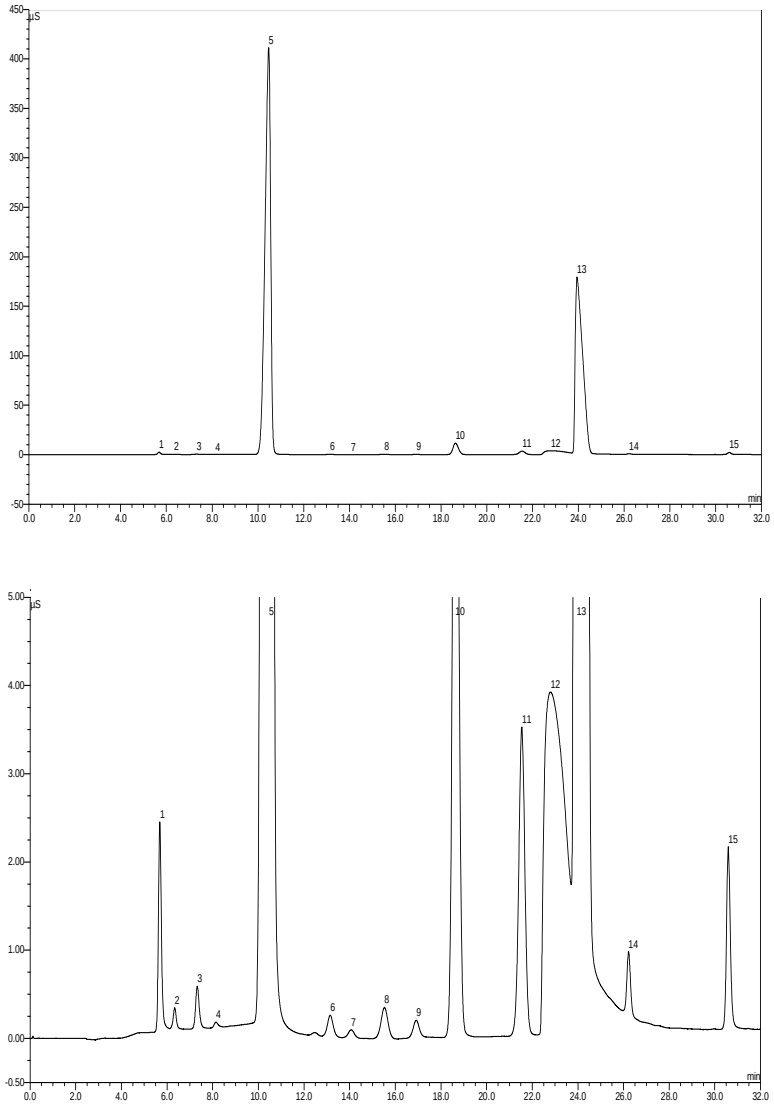


Figure 10
Sunnyvale Drinking Water Spiked with Surrogate Anion

5.6 Determination of Trace Nitrite and Nitrate in High Ionic Strength Matrix

The following chromatograms demonstrate the determination of trace nitrite and nitrate in high ionic strength matrices. Notice that a 100 µL injection volume is optimum for quantitation of traces of nitrite and nitrate in the presence of large amounts of salt (see Figure 11b.) Nitrite quantitation may be difficult with 200 µL of injection volume, as there is an overloading of chloride (see Figure 11a.)

Injection volume:	See Chromatograms
Column:	IonPac AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 10 min and 10 to 45 mM from 10 to 25 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression Recycle

	Peaks	mg/L (ppm)
1.	Chloride	800
2.	Nitrite	0.1
3.	Nitrate	0.1
4.	Carbonate	-
5.	Sulfate	100
6.	Phosphate	100

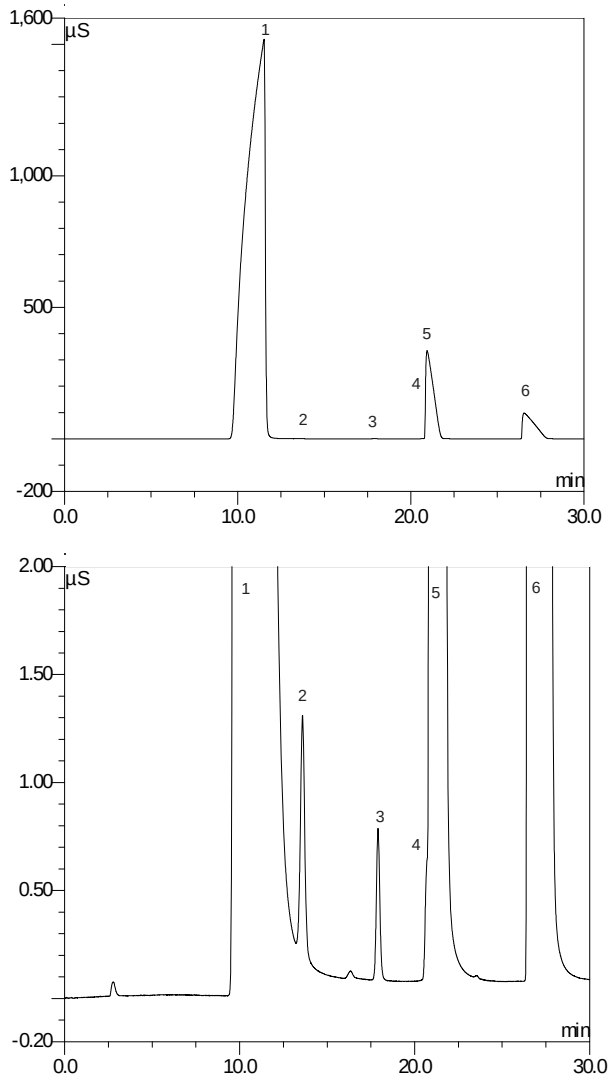


Figure 11a
Injection Volume: 200 µL

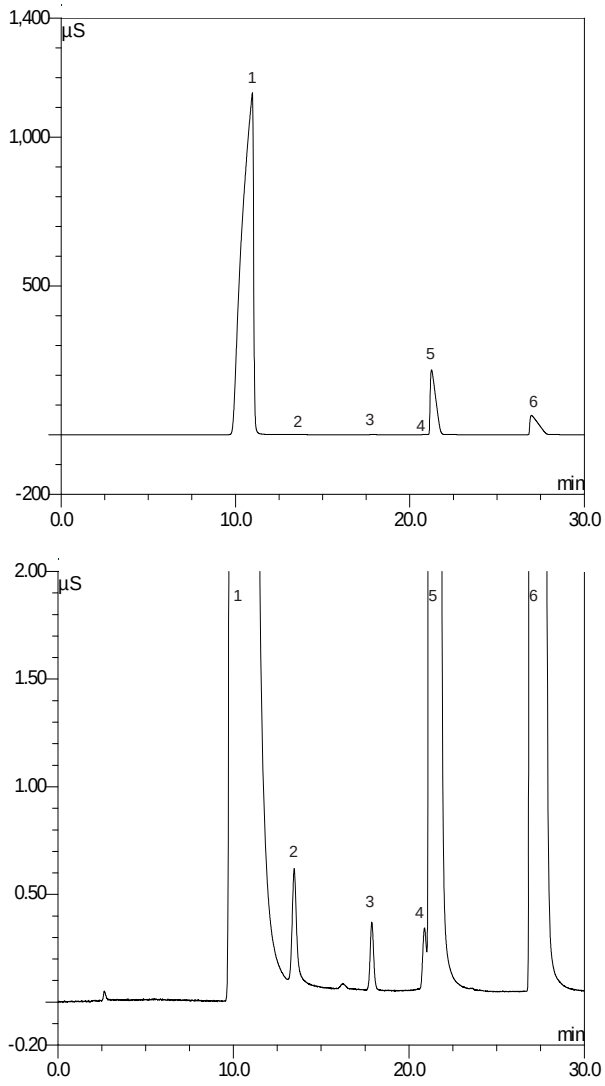
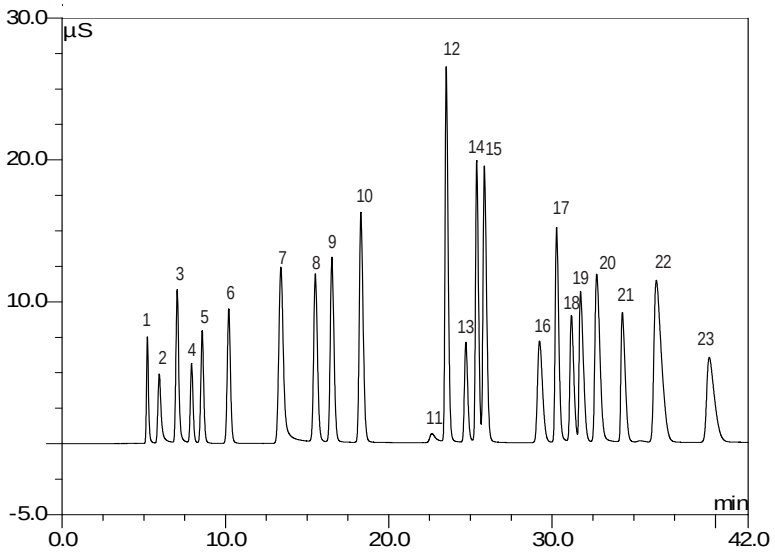


Figure 11b
Injection Volume: 100 µL

5.7 Analysis of Twenty-three Environmental Anions on the AS19 Analytical Column

The IonPac AS19 column provides excellent separation of a variety of environmental anions including inorganic anions, oxyhalides, oxyanions, and organic acids.

Injection volume:	25 µL
Column:	IonPac AS19 Analytical column + AG19 Guard column
Eluent :	Potassium hydroxide: 10 mM from 0 to 10 min and 10 to 58 mM from 10 to 40 minutes
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression Recycle



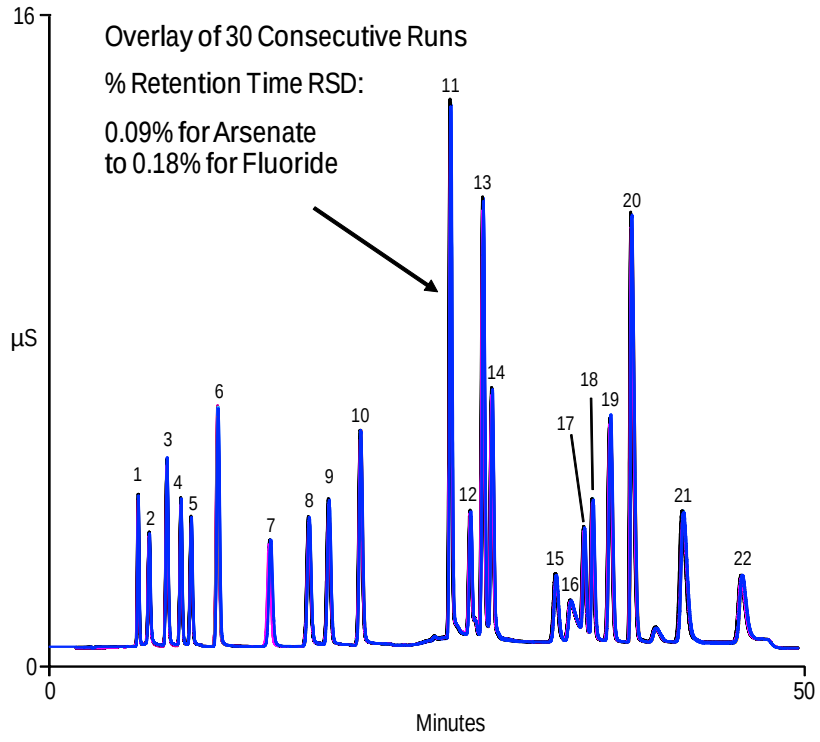
	Peaks	mg/L (ppm)
1.	Fluoride	2
2.	Acetate	10
3.	Formate	10
4.	Chlorite	10
5.	Bromate	20
6.	Chloride	6
7.	Nitrite	15
8.	Chlorate	25
9.	Bromide	25
10.	Nitrate	25
11.	Carbonate	25
12.	Sulfate	25
13.	Malonate	25
14.	Selenate	25
15.	Oxalate	25
16.	Iodide	30
17.	Thiosulfate	25
18.	Chromate	25
19.	Phosphate	30
20.	Fumarate	30
21.	Arsenate	30
22.	Thiocyanate	30
23.	Perchlorate	30

Figure 12
Analysis of Twenty-three Environmental Anions

5.8 Analysis of Twenty-two Environmental Anions on the AS19 Capillary Column

The IonPac AS19 capillary column provides excellent separation of a variety of environmental anions including inorganic anions, oxyhalides, oxyanions, and organic acids.

Injection volume:	1.0 µL
Column:	IonPac AS19 Capillary column (0.4 mm x 250 mm)
Eluent :	10 mM KOH (0 to 10 min), 10 to 52 mM KOH (10 to 42 min), 52 to 70 mM (42 to 45 min), 10 mM 45 to 50 min)
Eluent Source:	EGC KOH Capillary Cartridge
Flow Rate:	10 µL/min
Temperature:	30 °C
Suppressor:	Anion Capillary Electrolytic Suppressor (ACES 300)
Suppressor Mode:	AutoSuppression Recycle



Peaks

1. Fluoride
2. Acetate
3. Formate
4. Chlorite
5. Bromate
6. Chloride
7. Nitrite
8. Chlorate
9. Bromide
10. Nitrate
11. Sulfate
12. Malonate
13. Selenate
14. Oxalate
15. Iodide
16. Thiosulfate
17. Chromate
18. Phosphate
19. Fumarate
20. Arsenate
21. Thiocyanate
22. Perchlorate

Figure 13
Analysis of Twenty-three Environmental Anions

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS19 columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

Table 6
AS19/AG19 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2
		Filter Eluents	6.1.3
		Filter Samples	6.1.4
	Other System Components	Unplug, Replace	Component Manual
High Background Conductivity	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Trap Column	Clean Trap Column	6.2.2, Component Manual
	Contaminated ASRS or AMMS	Clean Suppressor	6.2.4, Component Manual
	Contaminated Hardware	Clean Component	Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.1.A, Component Manual
	Column Headspace	Replace Column	6.3.1.B
Short Retention Times	Flow Rate Too fast	Recalibrate Pump	6.3.2.A
	Conc. Incorrect Eluents	Remake Eluents	6.3.2.B
	Column Contamination	Clean Column	6.3.2.C, 6.3.2.D,
Poor Front End Resolution	Conc. Incorrect Eluents	Remake Eluents	6.3.3.A
	Column Overloading	Reduce Sample Size	6.3.3.B, 3.3.1, 3.3.2
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual
	Large System Void Volumes	Replumb System	6.3.3.D, Component Manual
Spurious Peaks	Sample Contaminated	Pretreat Samples	6.3.4.A, 6.3.4.B
	Sluggish Injection Valve	Service Valve	6.3.3.C, Component Manual

6.1 High Back Pressure

6.1.1 Finding the Source of High System Pressure

Total system pressure for the IonPac AG19 (4-mm) Guard Column plus the AS19 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,100 psi. If the system pressure is higher than 2,100 psi, it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated. The maximum flow rate is 2 mL/min and the maximum pressure is 3,000 psi (20.68 MPa).

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

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

To determine which part of the chromatographic system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure; it should not exceed 50 psi. 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 7, "Typical AS19/AG19 Operating Back Pressures").

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

Table 7
Typical AS19/AG19 Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS19 4-mm Analytical	≤ 1800 (12.41)	1.0
AG19 4-mm Guard	≤ 300 (2.07)	1.0
AS19 + AG19 4-mm columns	**≤ 2100 (14.48)**	**1.0**
AS19 2-mm Analytical	≤ 1800 (12.41)	0.25
AG19 2-mm Guard	≤ 300 (2.07)	0.25
AS19 + AG19 2-mm columns	**≤ 2100 (14.48)**	**0.25**
AS19 0.4-mm Capillary	≤ 2200 (15.17)	0.01
AG19 0.4-mm Capillary Guard	≤ 330 (2.28)	0.01
AS19 + AG19 0.4-mm columns	**≤ 2530 (17.45)**	**0.01**

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. 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.

Product	IonPac AS19 4-mm Columns (P/N)	IonPac AS19 2-mm Columns (P/N)	IonPac AS19 0.4-mm Columns (P/N)
Analytical Column	062885	062886	072064
Guard Column	062887	062888	072065
Bed Support Assembly	042955	044689	N/A
End Fitting	052809	043278	N/A

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

E. Screw the end fitting back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in. x lb.). Tighten further only if leaks are observed.

F. Reconnect the column to the system and resume operation.

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

6.1.3 Filter Eluent

Eluents containing particulate material or bacteria may clog the column inlet bed support. Filter water used for eluents through a 0.45 µm filter.

6.1.4 Filter Samples

Samples containing particulate material may clog the column inlet bed support. Filter samples through a 0.45 µm filter prior to injection.

6.2 High Background or Noise

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

ELUENT	EXPECTED BACKGROUND CONDUCTIVITY
20 mM NaOH	≤ 3 µS
20 mM KOH (EGC)	≤ 1.0 µS

6.2.1 Preparation of Eluents

A. Make sure that the eluents and the regenerant (if used) are made correctly.

B. Make sure that the eluents are made from chemicals with the recommended purity.

C. Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

6.2.2 A Contaminated Trap Column

High background may be caused by contamination of the ATC-HC or ATC-3 with carbonate or other anions from the eluent. Clean the ATC-HC or 4-mm ATC-3 with 100 mL of 2.0 M NaOH or 50 mL for the 2-mm ATC-3. Rinse the ATC-HC or 4-mm ATC-3 immediately with 20 mL of eluent or 10 mL of eluent for the 2-mm ATC-3 into a beaker prior to use.

6.2.3 Contaminated CR-ATC Column

For RFIC-EG operation, use a CR-ATC Trap Column. Install a CR-TC Anion Trap Column (P/N 060477 or 072078) if using an Eluent Generator with EGC KOH cartridge. If the CR-ATC becomes contaminated, please refer to Section 6, Clean-Up, in the CR-ATC Product Manual (Document No. 031910).

6.2.4 A Contaminated Guard or Analytical Column

Remove the IonPac AG19 Guard and AS19 Analytical Columns from the system. Install a back pressure coil that generates approximately**.** 1,500 psi and continue to pump eluent. If the background conductivity decreases, the column(s) is (are) the cause of the high background conductivity. Clean or replace the AG19 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").

6.2.5 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the columns and the suppressor. Install a back pressure coil that generates approximately. 1,500 psi and continue to pump eluent. Pump deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

6.2.6 A Contaminated ASRS 300, ACES 300 or AMMS 300 Suppressor

If the above items have been checked and the problem persists, the Anion Self-Regenerating Suppressor, Anion Capillary Electrolytic Suppressor or the Anion MicroMembrane Suppressor is probably causing the problem. For details on Anion Self-Regenerating Suppressor operation., refer to the Anion Self-Regenerating Suppressor 300 Product Manual (Document No. 031956). For details on Anion Membrane Suppressor 300 operation, refer to the Product Manual (Document No. 031727) for assistance. For details on Anion Capillary Electrolytic Suppressor (ACES 300) operation, refer to the Product Manual (Document No. 065386) for assistance.

A. Check the power level and alarms on the SRS Control.

B. Check the regenerant flow rate at the REGEN OUT port of the ASRS or ACES if operating in the AutoSuppression External Waster mode or the Chemical Suppression mode or the AMMS.

C. Check the eluent flow rate.

D. If you are using an AutoRegen Accessory with the ASRS in the Chemical Suppression Mode or the AMMS, 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 ASRS or AMMS.

2. **If the background conductivity is low when freshly prepared regenerant is run through the ASRS or AMMS 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.

6.3 Poor Peak Resolution

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

6.3.1 Loss of Column Efficiency

A. Peak Fronting: Check to see if headspace has developed in the guard or analytical column. This is usually due to improper use of the column such as submitting it to high pressures. Remove the column's top end fitting (see Section 6.1.2, "Replacing Column Bed Support Assemblies"). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

B. Symmetric Inefficient Peaks: Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.010" for 4-mm systems or no greater than 0.005" for 2-mm systems to make all eluent liquid line connections between the injection valve and the detector cell inlet. Cut the tubing lengths as short as possible. Check for leaks. Do not cut tubing for 0.4 mm Capillary Systems, always use the pre-cut tubing provided by Dionex.

6.3.2 Poor Resolution Due to Shortened Retention Times

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

A. Check the flow rate. See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder. **Check the flow rate of the pump. If the flow rate is higher than the set flow rate, it will cause longer run time as it will dilute the eluent generated by the eluent generator. If the flow rate is lower than the set flow rate, it will cause short run as eluent will be more concentrate than needed for the separation.**

B. Check to see if the eluent compositions and concentrations are correct. An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent, components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

C. Column contamination can lead to a loss of column capacity. This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to, "Column Cleanup" (see "Column Care"), for recommended column cleanup procedures.

Possible sources of column contamination are impurities in chemicals and in the deionized water used for eluents or components of the sample matrix. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

D. Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times. If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see, "Column Cleanup" in "Column Care").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. No water wash is necessary. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column. If you need assistance in solving resolution problems, contact the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) ore the nearest Dionex Office (see, "Dionex Worldwide Offices").

6.3.3 Loss of Front End Resolution

If poor resolution or efficiency is observed for the peaks eluting near the system void volume compared to the later eluting peaks, check the following:

A. Improper eluent concentration may be the problem. Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity. Check the flow rate of the pump as it effects the concentration generated by the eluent concentrator.

B. Column overloading may be the problem. Reduce the amount of sample ions being injected onto the analytical column by either diluting the sample or injecting a smaller volume onto the column.

C. 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.

D. 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. Use the shortest tubing lengths possible.

6.3.4 Spurious Peaks

A. The columns may be contaminated. If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system, the retention times for the analytes will then decrease and be spurious, inefficient (broad) peaks that can show up at unexpected times. Clean the column as indicated in "Column Cleanup" (see "Column Care").

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS19 columns, contact the North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest Dionex Office (see, "Dionex Worldwide Offices").

B. The injection valve may need maintenance. When an injection valve is actuated, the possibility of creating a baseline disturbance exists. This baseline upset can show up as a peak of varying size and shape. This will occur when the injection valve needs to be cleaned or retorqued (see valve manual). Check to see that there are no restrictions in the tubing connected to the valve. Also check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures. Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

6.3.5 Poor Efficiency Using Capillary Columns

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

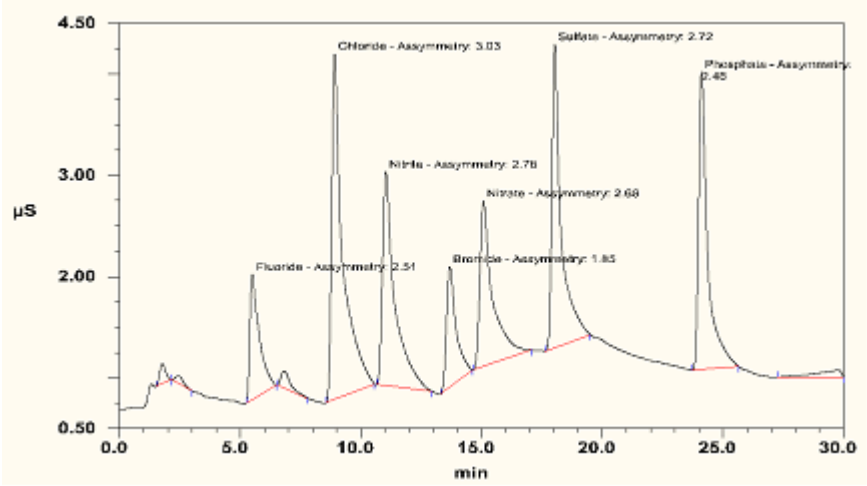


Figure 14
Tailing Peaks Caused by Incorrectly Installed Capillary Tubing Fittings

When connecting a capillary tube fitting, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing. Figure 15 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

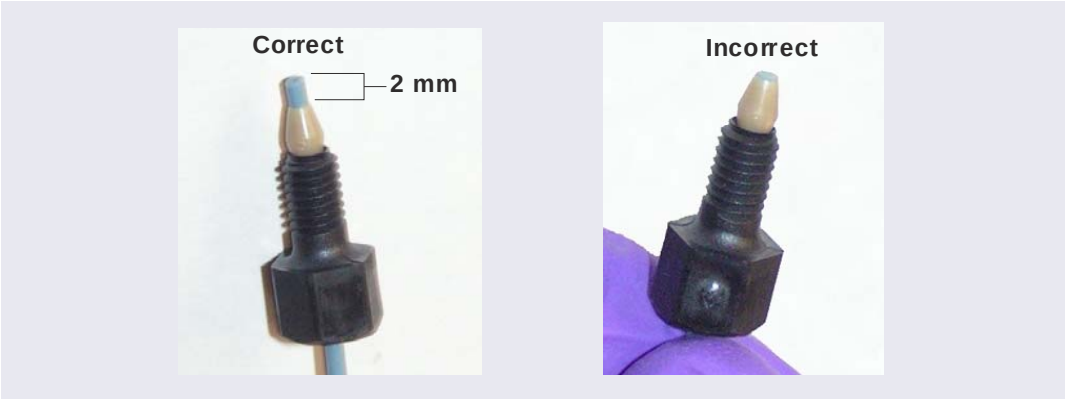


Figure 15
Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

APPENDIX A - QUALITY ASSURANCE REPORT

Quality Assurance Report - IonPac AS19 Analytical Column - 4-mm

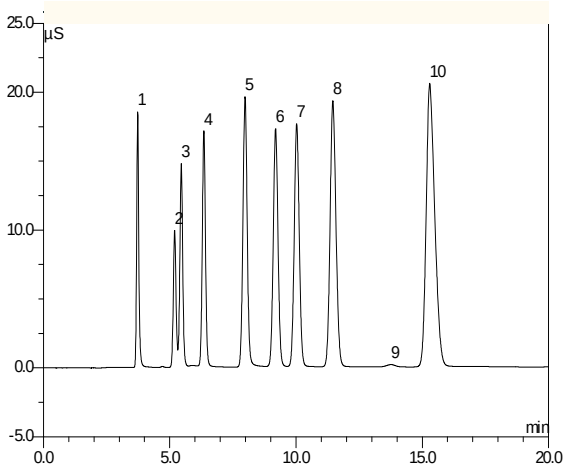
Quality Assurance Report - IonPac AS19 Analytical Column - 2-mm

Quality Assurance Report - IonPac AS19 Capillary Column - 0.4-mm

A.1 Quality Assurance Report - IonPac AS19 Analytical Column - 4-mm

To guarantee that all IonPac AS19 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. An operating temperature of 30 °C is used to ensure reproducible resolution and retention time.

Injection volume:	25 µL
Column:	IonPac AS19 Analytical column
Eluent:	20 mM KOH
Eluent Source:	EGC KOH Cartridge
Flow Rate:	1 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor (ASRS 300, 4-mm)
Suppressor Mode:	AutoSuppression Recycle
Applied Current:	100 mA



	Peaks	ppm
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Nitrite	15
6.	Chlorate	25
7.	Bromide	25
8.	Nitrate	25
9.	Carbonate	-
10.	Sulfate	30

Figure 16
IonPac AS19 Quality Assurance Report - Analytical 4-mm

A.2 Quality Assurance Report - IonPac AS19 Analytical Column - 2-mm

To guarantee that all IonPac AS19 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. An operating temperature of 30 °C is used to ensure reproducible resolution and retention time.

Injection volume:	5 µL
Column:	IonPac AS19 Analytical column (2-mm)
Eluent:	20 mM KOH
Eluent Source:	EGC KOH Cartridge
Flow Rate:	0.25 mL/min
Temperature:	30 °C
Suppressor:	Anion Self-Regenerating Suppressor (ASRS ULTRA, 2-mm)
Suppressor Mode:	AutoSuppression Recycle
Applied Current:	100 mA

	Peaks	ppm
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Nitrite	15
6.	Chlorate	25
7.	Bromide	25
8.	Nitrate	25
9.	Carbonate	-
10.	Sulfate	30

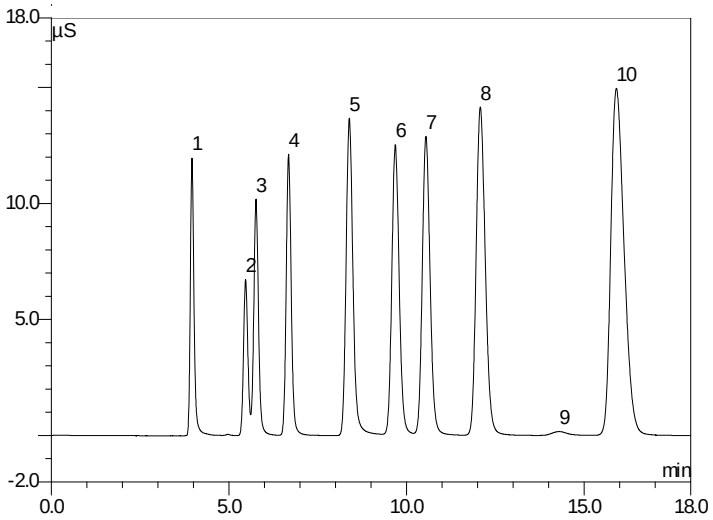


Figure 17
IonPac AS19 Quality Assurance Report - Analytical, 2-mm

A.3 Quality Assurance Report - IonPac AS19 Capillary Column - 0.4-mm

To guarantee that all IonPac AS19 Capillary Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test. An operating temperature of 30 °C is used to ensure reproducible resolution and retention time.

Injection volume:	400 nL
Column:	IonPac AS19 Capillary column (0.4 x 250 mm)
Eluent:	20 mM KOH
Eluent Source:	EGC KOH Capillary Cartridge
Flow Rate:	0.01 mL/min
Temperature:	30 °C
Suppressor:	Anion Capillary Electrolytic Suppressor (ACES 300)
Suppressor Mode:	AutoSuppression Recycle

	Peaks	ppm
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Nitrite	15
6.	Chlorate	25
7.	Bromide	25
8.	Nitrate	25
9.	Sulfate	30

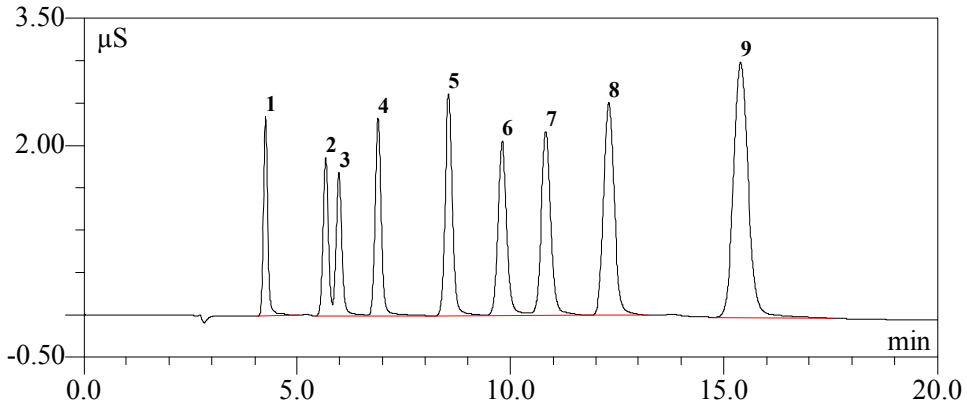


Figure 18
IonPac AS19 Quality Assurance Report - Capillary, 0.4-mm

APPENDIX B - COLUMN CARE

B.1 Recommended Operation Pressures

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS19 columns is 3,000 psi (20.68 MPa).

B.2 Column Start-Up

The column is shipped using the column test eluent as the storage solution.

Prepare the eluent shown on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Continue making injections of the test standard until consecutive injections of the standard give reproducible retention times. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

B.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), use 100 mM Sodium Borate for the column storage solution. Flush the column for a minimum of 30 minutes with the storage solution. 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. 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 ≤ 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.

B.4.1 Choosing the Appropriate Cleanup Solution

A. Concentrated hydroxide solutions such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.

B. Concentrated acid solutions such as 1 to 3 M HCl, remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.

C. Metal contamination often results in asymmetric peak shapes and/or variable analyte recoveries. For example, iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.

D. Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents listed in Table 3, HPLC Solvents for Use with IonPac AS19 Columns.

E. Concentrated acid solutions such as 1 to 3 M HCl can be used with compatible organic solvents to remove contamination that is ionic and hydrophobic. The acid suppresses ionization and ion exchange interactions of the contamination with the resin. The organic solvent then removes the subsequent nonionic and hydrophobic contamination. See Section D above.

A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

F. Regardless of the cleanup solution chosen, use the following cleanup procedure in, "Column Cleanup Procedure", to clean the AG19 and AS19.

B.4.2 Column Cleanup Procedure

A. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in, "Choosing the Appropriate Cleanup Solution".

B. Disconnect the ASRS 300, ACES 300 or AMMS 300 from the IonPac AS19 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 or capillary and capillary guard column in series, ensure that the guard column is placed after the analytical or capillary column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical capillary column and irreversibly damage it. If in doubt, clean each column separately.**

C. Set the pump flow rate to 1.0 mL/min for an AS19 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS19 2-mm Analytical or Guard Column. Set the pump flow rate to 0.010 mL/min for an AS19 0.4 mm Capillary or Capillary Guard Column.

D. Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column.

E. Pump the cleanup solution through the column for at least 60 minutes.

F. Rinse the column for 10 minutes with deionized water before pumping eluent over the column.

G. Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.

H. Reconnect the ASRS 300, ACES 300 or AMMS 300 to the AS19 Analytical or Capillary Column and place the guard column in line between the injection valve and the analytical or capillary column if your system was originally configured with a guard column.

APPENDIX C - ION CHROMATOGRAPHY SYSTEMS

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

CONFIGURATION	2-mm	4-mm	0.4-mm
Eluent Flow Rate	0.25 mL/min	1.0 µL/min	0.010 µL/min
SRS Suppressor	ASRS 300 (P/N 061562)	ASRS 300 (P/N 061561)	N/A
MMS Suppressor	AMMS 300 (P/N 056751)	AMMS 300 (P/N 056750)	N/A
ACES Suppressor	N/A	N/A	ACES 300 (P/N 072052)
NOTE: ***Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven.***			
Injection Loop	2 - 15 µL Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL.	10 - 50 µL	0.4 µL (typical)
System Void Volume	Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the 2 mm GM-4 Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2, GM-3 or recommended gradient mixers.	Use only in an IC system equipped for capillary analysis.
Pumps	Use the GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2 mm) Gradient Mixer. The GPM-2 can be used for 2 mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2 mm gradient chromatography	Use the GP40/GP50/IP20/IP25 in Standard-Bore Configuration. The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP50. The GP40 has an active mixer.	Use only a pump designed for capillary flow rates such as the ICS-5000 capillary pump.
NOTE: ***Use of an EGC-KOH cartridge (P/N 074532 or 072076 in conjunction with a CR-ATC P/N 060477 or 072078) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***			
Chromatographic Module	A thermally controlled column oven such as theLC25,LC30,ICS-10,11,15,16,20,2100,3000,5000 DC	A thermally controlled column oven such as theLC25,LC30,ICS-10,11,15,16,20,2100,3000,5000 DC	A thermally controlled column compartment such as the ICS-5000 DC or IC-Cube.

CONFIGURATION	2-mm	4-mm	0.4-mm
Detectors	AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL) (P/N 043120) CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. Ensure 30–40 psi back pressure.	AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) VDM-2 Cell (6-mm, 10 µL) P/N 043113 CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Either the TS-1 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. Ensure 30–40 psi back pressure.	Use only a conductivity detector designed for capillary flow rates such as the ICS-5000 Capillary CD.

Table 8
Tubing Back Pressures

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
Light Blue		0.0025					