

Errata

Product Manual for Dionex IonPac™ AS18 and AG18 Columns
031878-08

For new orders of the following parts discussed in this manual, please use the updated part numbers listed below.

Part	Old Part Number in this manual	Updated Part Number to use for new orders
PROD,COL,IP,ATC-3,4X35MM	059661	079932
PROD,COL,IP,UTAC-LP2,4X35MM	072779	079917
PROD,COL,IP,UTAC-ULP2,5X23MM	072780	079918



Thermo Scientific

Dionex IonPac AS18 Fast

Product Manual

P/N: 031878-08

June 2012

Product Manual

for

IonPac[®] AG18 Guard Column

(4 x 50 mm, P/N 060551)

(2 x 50 mm, P/N 060555)

IonPac[®] AS18 Analytical Column

(4 x 250 mm, P/N 060549)

(2 x 250 mm, P/N 060553)

IonPac[®] AG18-Fast Capillary Guard Column

(0.4 x 35 mm, P/N 072063)

IonPac[®] AS18-Fast Capillary Column

(0.4 x 150 mm, P/N 072062)

IonPac[®] AG18-Fast Guard Column

(4 x 30 mm, P/N 075762)

(2 x 30 mm, P/N 075761)

IonPac[®] AS18-Fast Analytical Column

(4 x 150 mm, P/N 075760)

(2 x 150 mm, P/N 075759)

IonPac[®] AG18 Capillary Guard Column

(0.4 x 50 mm, P/N 075773)

IonPac[®] AS18 Capillary Column

(0.4 x 250 mm, P/N 075772)

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SECTION 1 – INTRODUCTION

The IonPac® AS18 Analytical Column in combination with the AG18 Guard Column is designed for the analysis of inorganic anions including fluoride, chloride, nitrite, bromide, nitrate, sulfate, and phosphate in less than 10 minutes using an isocratic hydroxide eluent delivered with an Eluent Generator. The selectivity of the IonPac AS18 Guard plus Analytical/Capillary Column set has been designed to retain fluoride well out of the water dip (system dip). Using an isocratic hydroxide eluent, the inorganic anions can easily be separated in a variety of sample matrices including drinking water, ground water, waste water, process streams, and scrubber solutions. The AS18 is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 to 100% in concentration. The AS18 can be used with any suppressible ionic eluent that does not exceed the capacity of the suppressor. The IonPac AS18 has nominal efficiency of at least 11,000 plates/column for chloride using standard operating conditions. The maximum operating pressure should be less than 4,000 psi. The AS18-Fast column series (0.4 x 150 mm, 4 x 150 mm and 2 x 150 mm) offers the same selectivity as the AS18 analytical/capillary scale columns, but with a reduced length of 150 mm. These columns are designed for fast separation of 7 common anions in significantly less time. The AS18 capillary series (0.4 x 150 mm and 0.4 x 250 mm) offers the advantages of less eluent consumption, providing reduced operating costs and excellent reproducibility.

Table 1
IonPac AS18/AG18 and AS18-Fast/AG18-Fast Packing Specifications

Column	Particle Diameter µm	Substrate X-linking %	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column ^b	Functional Group	Hydrophobicity
AS18* 4 x 250 mm	7.5	55	65	0 ^a	285	Alkanol quaternary ammonium	Low
AG18** 4 x 50 mm	13	55	65	0 ^a	10	Alkanol quaternary ammonium	Low
AS18* 2 x 250 mm	7.5	55	65	0 ^a	75	Alkanol quaternary ammonium	Low
AG18** 2 x 50mm	13	55	65	0 ^a	2.5	Alkanol quaternary ammonium	Low
AS18-Fast* 0.4 x 150 mm	7.5	55	65	0 ^a	1.71	Alkanol quaternary ammonium	Low
AG18-Fast** 0.4 x 35 mm	13	55	65	0 ^a	0.07	Alkanol quaternary ammonium	Low
AS18-Fast* 4 x 150 mm	7.5	55	65	0 ^a	171	Alkanol quaternary ammonium	Low
AG18-Fast** 4 x 30 mm	1.3	55	65	0 ^a	6	Alkanol quaternary ammonium	Low
AS18-Fast* 2 x 150 mm	7.5	55	65	0 ^a	45	Alkanol quaternary ammonium	Low
AG18-Fast** 2 x 30 mm	1.3	55	65	0 ^a	1.5	Alkanol quaternary ammonium	Low
AS18* 0.4 x 250 mm	7.5	55	65	0 ^a	2.85	Alkanol quaternary ammonium	Low

^a The effective X-Linking after functionalization is 8%.

* Analytical/Capillary 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
AS18/AG18 and AS18-Fast/AG18-Fast Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS18 4-mm Analytical	≤ 1,500 (10.34)	1.0	3.0
AG18 4-mm Guard	≤ 300 (2.07)	1.0	3.0
AS18 + AG18 4-mm columns	≤ 1,800 (12.41)	1.0	3.0
AS18-Fast 4-mm Analytical	≤ 1100 (7.59)	0.6	3.0
AG18-Fast 4-mm Guard	≤ 180 (1.24)	0.6	3.0
AS18-Fast + AG18-Fast 4-mm columns	≤ 1280 (8.83)	0.6	3.0
AS18 2-mm Analytical	≤ 1,500 (10.34)	0.25	0.75
AG18 2-mm Guard	≤ 300 (2.07)	0.25	0.75
AS18 + AG18 2-mm columns	≤ 1,800 (12.41)	0.25	0.75
AS18-Fast 2-mm Analytical	≤ 1100 (7.59)	0.25	0.75
AG18-Fast 2-mm Guard	≤ 180 (1.24)	0.25	0.75
AS18-Fast + AG18-Fast 2-mm columns	≤ 1280 (8.83)	0.25	0.75
AS18-Fast 0.4-mm Capillary	≤ 1,200 (8.27)	0.010	0.030
AG18-Fast 0.4-mm Capillary Guard	≤ 300 (2.07)	0.010	0.030
AS18-Fast +AG18-Fast 0.4- mm columns	≤ 1,500 (10.34)	0.010	0.030
AS18 0.4-mm Capillary	≤1,500 (10.34)	0.010	0.030
AS18 0.4-mm Capillary Guard	≤300 (2.07)	0.010	0.030
AS18 + AG18 0.4-mm columns	≤1800(12.41)	0.010	0.030

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.

- 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, Suppressor, 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 AS18 and AS18-Fast 0.4-mm Guard and Capillary Columns are designed to run on a Dionex Capillary Ion Chromatograph equipped with suppressed conductivity detection such as an ICS-5000 Capillary IC System. It is recommended to run the capillary column on a Dionex Capillary IC System for best performance.

3.1.2. System Requirements for 2-mm Operation

The IonPac AS18 and AS18-Fast 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 AS18 and AS18-Fast 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 (TAC-LP1, P/N 046026), the Trace Anion Concentrator Ultra Low Pressure (TAC-ULP1, P/N 061400), the Ultra Trace Anion Concentrator Low Pressure (UTAC-LP1, P/N 063079) or (UTAC-LP2, P/N 072779), the Ultra Trace Anion Concentrator Ultra Low Pressure (UTAC-ULP1, P/N 063475) or (UTAC-ULP2, P/N 072780), the Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459) or (UTAC-XLP2, P/N 072781), or the IonPac AG18/AG18-Fast Guard Column can be used for trace anion concentration work with the 2-mm and 4-mm AS18/AS18-Fast 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 AS18/AS18-Fast 0.4-mm column, use the AG18/AG18-Fast 0.4-mm column or the IonSwift MAC-100 or MAC-200 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 Low Pressure (UTAC-LP2), Ultra Low Pressure (UTAC-ULP2), and Extremely Low Pressure (UTAC-XLP2) Column Product Manual (Document No. 065376).
- Section 3, "Operation" of the Monolith Anion Concentrator Column (MAC-100 / MAC-200) Column Manual (Document No. 065387).

These techniques can also be applied to the AG18.



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 AS18/AS18-Fast.

CAUTION

3.3. The Injection Loop

3.3.1. The 2-mm System Injection Loop, 2 - 15 μ L

For most applications on a 2-mm analytical system, a 2 - 15 μ L 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 AS18/AS18-Fast 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less ($<15 \mu$ L) 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 μ L

For most applications on a 4-mm analytical system, a 10 - 50 μ L 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 μ L Internal Loop

For most applications on a 0.4-mm capillary system, a 0.4 μ L injection loop is sufficient. Generally, you should not inject more than 0.5 nanomoles total anion concentration onto the 0.4-mm capillary column. Injecting a 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 AG18/AG18-Fast Guard Column

An IonPac AG18/AG18-Fast Guard Column is normally used with the IonPac AS18/AS18-Fast Analytical/Capillary Column. Retention times will increase by approximately 3% when a guard column is placed in-line prior to the analytical/capillary column. A guard is placed prior to the analytical/capillary column to prevent sample contaminants from eluting onto the analytical/capillary column. It is easier to clean or replace a guard column than it is an analytical/capillary column. Replacing the AG18/AG18-Fast Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS18/AS18-Fast Analytical/Capillary Column.

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

For IonPac AS18/AS18-Fast 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 EGC Cartridge in the EG 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 059661) 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 AS18/AS18-Fast 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. An Anion Capillary Electrolytic Suppressor should be used with a 0.4 mm AS18/ AG18-Fast capillary column.



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

CAUTION

IonPac AS18/AS18-Fast 0.4-mm Capillary Column	ACES 300 (P/N 072052).
IonPac AS18/AS18-Fast 4-mm Analytical Column	ASRS 300 (4-mm, P/N 064554).
IonPac AS18/AS18-Fast 2-mm Analytical Column	ASRS 300 (2-mm, P/N 064555).

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see Document No. 031367, 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.

3.8. The Anion Atlas Electrolytic Suppressor Requirements

An Anion Atlas® Electrolytic Suppressor (AAES) may be used instead of an ASRS 300 for applications that require suppressed conductivity detection. The AAES (P/N 056116) can be used for 2-mm and 4-mm IonPac AS18/AS18-Fast applications using eluents up to 25 µeq/min.

For detailed information on the operation of the Anion Atlas Electrolytic Suppressor, see Document No. 031770, the “Product Manual for the Anion Atlas Electrolytic Suppressor.”

3.9. 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 AS18 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.”

3.10. 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.

3.11. 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.

3.12. Using the EGC-KOH with AS18 and AS18-Fast

Please refer to the EG40 manual, Document No. 031373, for information on the operation of the EG40. For the EG50, please refer to the EG50 Product Manual, Document No. 031908, for information on the operation of the EG50.



Use proper safety precautions in handling acids and bases.

SAFETY

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.

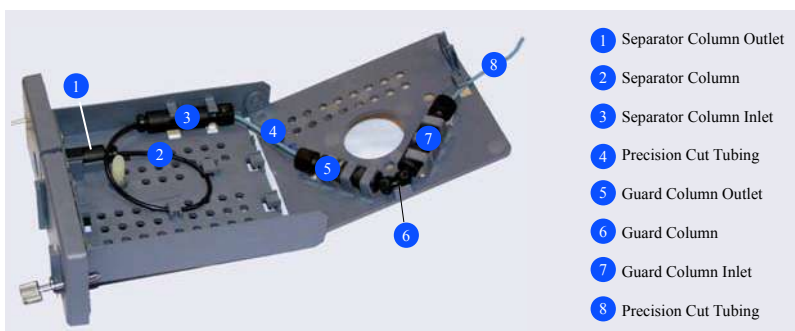


Figure 1
Separator and Guard Columns Installed in Column Cartridge

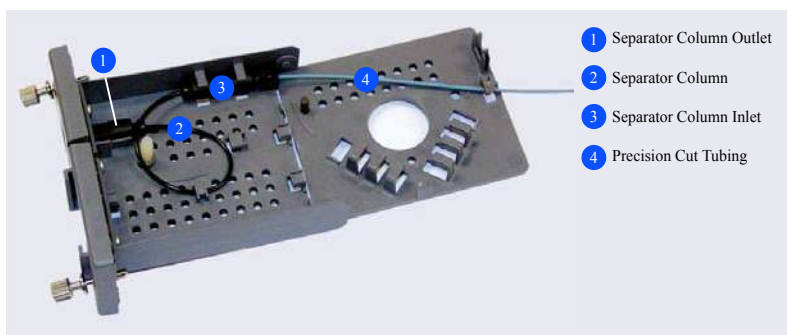


Figure 2
Separator Column Only Installed in Column Cartridge

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:

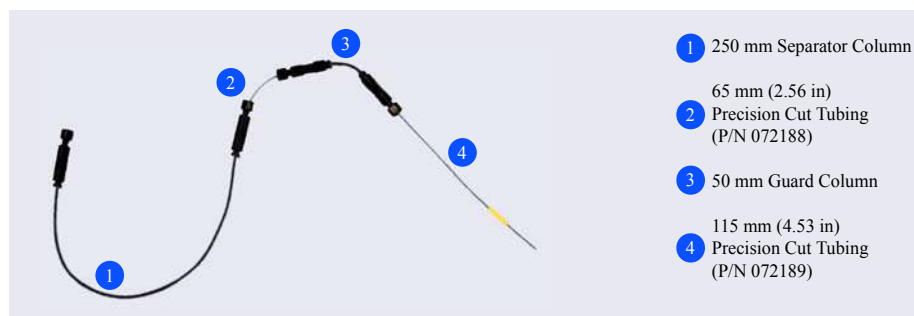


Figure 3
Tubing Connections for 250-mm Separator Column and 50-mm Guard Column

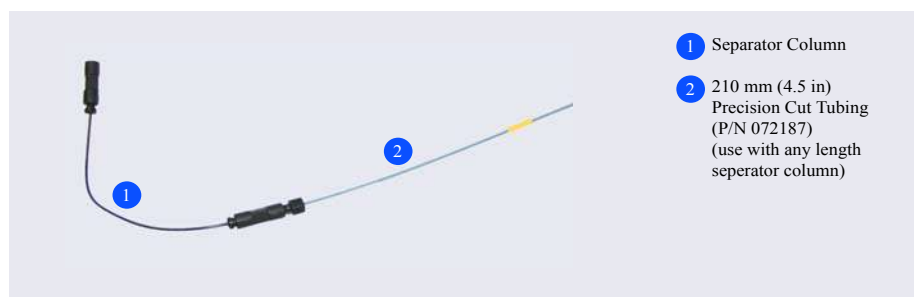


Figure 4
Tubing Connections for Separator Column Only

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.

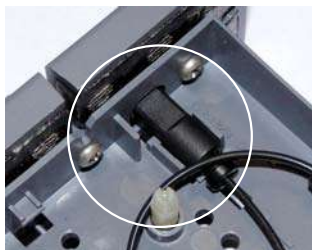


Figure 5
Column Outlet Fitting Installed in Column Cartridge

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: 2.5 μ L Loop + 0.8 μ L Injection valve dead volume 4-mm: 10 μ L Loop + 0.8 μ L Injection valve dead volume 0.4-mm: 0.4 μ L Loop
Column:	2-mm: AS18/AS18-Fast 2-mm Analytical Column + AG18/AG18-Fast 2-mm Guard Column 4-mm: AS18/AS18-Fast 4-mm Analytical Column + AG18/AG18-Fast 4-mm Guard Column 0.4-mm: AS18/AS18-Fast 0.4-mm Capillary Column + AG18/AG18-Fast 0.4-mm Capillary Guard Column
Eluent:	23 mM KOH
Eluent Source:	Eluent Generator
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 ₂ SO ₄
Expected Background Conductivity:	< 2 μ S
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2. IonPac AS18 Operation Precautions



CAUTION

Filter and Degas Eluents

Filter Samples

Eluent pH between 0 and 14

Sample pH between 0 and 14

0.75 mL/min Maximum Flow Rate for 2-mm Columns

3.0 mL/min Maximum Flow Rate for 4-mm Columns

0.03 mL/min Maximum Flow Rate for 0.4-mm Columns

Maximum Operating Pressure = 4,000 psi (27.57 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 μ m. Bottled HPLC-Grade Water (with the exception of Burdick & Jackson) should not be used since most bottled water contains an unacceptable level of ionic impurities.

4.3.3. Solvents

Solvents can be added to the ionic eluents used with IonPac AS18/AS18-Fast 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 AS18/AS18-Fast columns is 4,000 psi (27.57 MPa).

The AS18/AS18-Fast 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 AS18 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 ASRS 300 and the ACES 300 must be operated in the AutoSuppression External Water Mode when using eluents containing solvents. Do not use > 40% solvent on the ASRS 300 and the ACES 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.



NOTE

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.



NOTE

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 Solutions.

4.5. Eluent Preparation

4.5.1. Sodium Hydroxide Eluent Concentration

4.5.1.1. 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 39 mM NaOH use 3.12 g of 50% sodium hydroxide:
(as used in Section 5.2, “Production Test Chromatogram”)

$$\text{For 39 mM: } \frac{0.039 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\%} = 3.12 \text{ g diluted to 1 L}$$

4.5.1.2. 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.

$g = dvr$ 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 39 mM NaOH use 2.04 mL of 50% sodium hydroxide:
(as used in Section 5.2, “Production Test Chromatogram”)

$$\text{For 39 mM: } \frac{0.039 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\% \times 1.53 \text{ g/mL}} = 2.04 \text{ mL diluted to 1 L}$$

* 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.

4.5.1.3. Sodium Hydroxide Eluents

Dilute the amount of 50% (w/w) NaOH (in water) specified in Table 5, “Dilution of 50% (w/w) NaOH to Make Standard AS18 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 AS18 Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
2.8 (1.83)	35
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 AS18/AS18-Fast is designed for the determination of inorganic anions in less than ten minutes using an isocratic hydroxide eluent delivered with an Eluent Generator. 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 NaOH and end at 80 mM NaOH, with a resulting total baseline change of 1 to 2 μ S.

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”).



CAUTION

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

5.2. Fast Isocratic Separation of the Common Anions Using the IonPac AS18-Fast Capillary Column

The AS18-Fast Capillary Column (0.4 x 150 mm) offers the same selectivity as the AS18 Analytical Columns, but with a reduced length of 150 mm. This column can separate the 7 common Anions in significantly less time as shown in Figure 7.

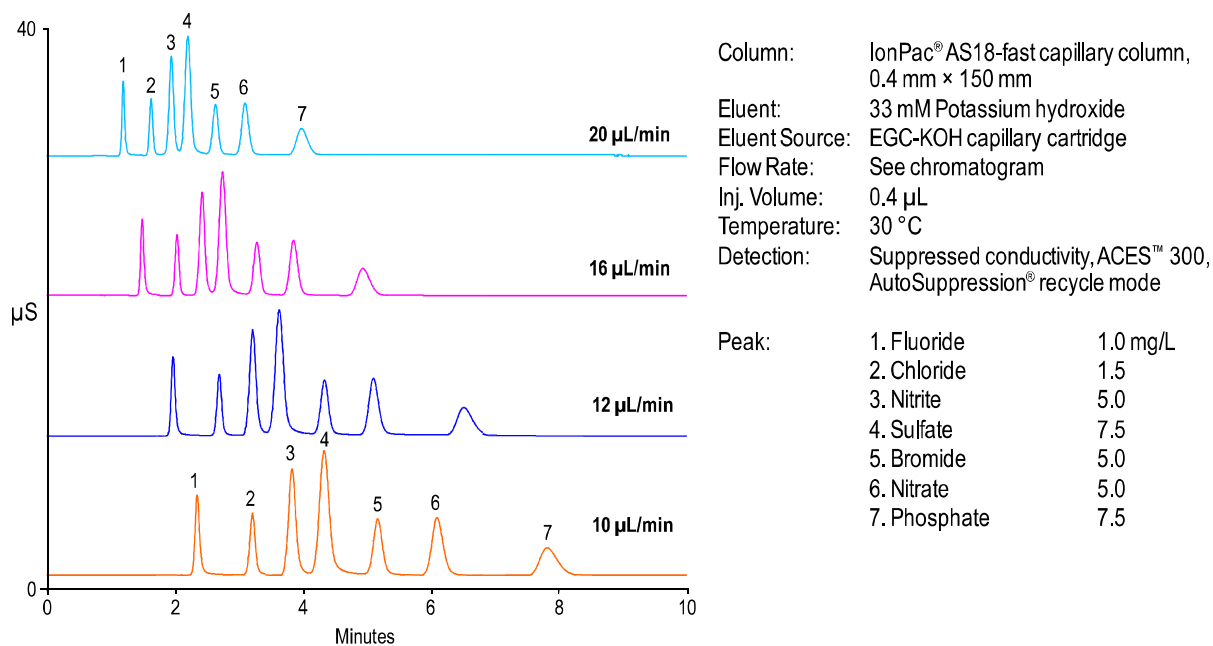


Figure 7
Fast Isocratic Separation of the Common Anions
Using the IonPac AS18-Fast Capillary Column

5.3. Fast Isocratic Separation of the Common Anions Using the IonPac AS18-Fast Column

The AS18-Fast Column (4 x 150 mm) offers the same selectivity as the AS18 Analytical Columns, but with a reduced length of 150 mm. This column can separate the 7 common Anions in significantly less time. With an increased flow rate of 1.5 mL/min, the 7 common Anions can be eluted in approximately 5 minutes as shown in Figure 8.

Column: IonPac AS18-Fast (4 x 150 mm)
Eluent: 23mM KOH
Eluent Source: KGC-KOH cartridge
Flow rate: See chromatogram
Inj. Volume: 20 μ L
Temperature: 30 $^{\circ}$ C
Detection: Suppressed Conductivity, ASRS 300 4-mm, 57 mA
Current: AutoSuppression recycle mode

Peaks

1. Fluoride
2. Chlorite
3. Chloride
4. Nitrite
5. Carbonate
6. Bromide
7. Sulfate
8. Nitrate
9. Chlorate

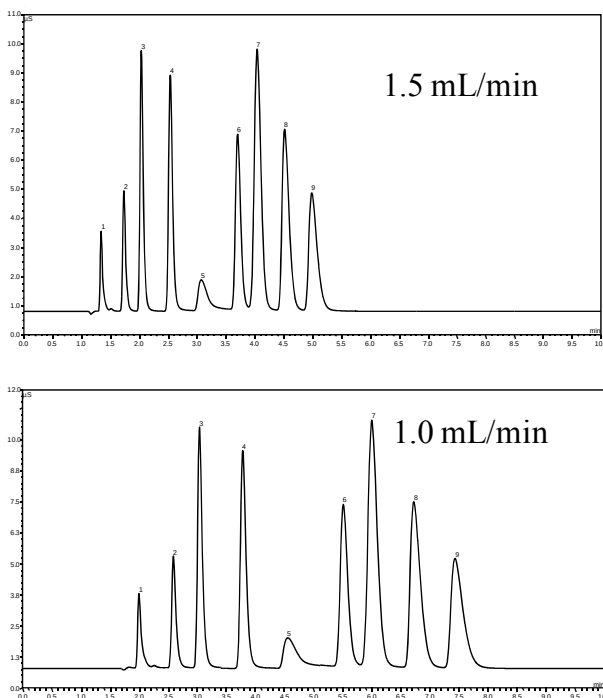


Figure 8
Fast Isocratic Separation of the Common Anions
Using the IonPac AS18-Fast Column

5.4. Isocratic Separation with the IonPac AS18

The AS18 provides the fast separation of fluoride, acetate, formate, chlorite, chloride, nitrite, sulfate, bromide, nitrate, and phosphate using an isocratic potassium hydroxide eluent and suppressed conductivity detection. Using a 39 mM potassium hydroxide eluent delivered by an Eluent Generator, these anions can easily be separated in 9 minutes as illustrated in Figure 9.

Inj. Volume: 25 μ L
 Column: IonPac AS18 4-mm Analytical Column
 Eluent: 39 mM KOH
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30 $^{\circ}$ C
 Detection: Suppressed Conductivity
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H_2SO_4

Peaks	mg/L
1. Fluoride	1
2. Acetate	5
3. Formate	2
4. Chlorite	5
5. Chloride	2
6. Carbonate	4
7. Nitrite	5
8. Sulfate	6
9. Bromide	10
10. Nitrate	10
11. Phosphate	20

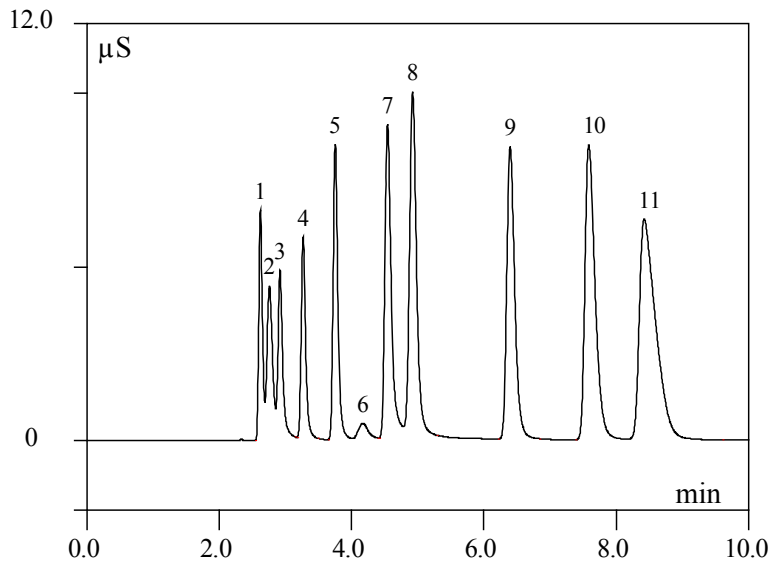


Figure 9
Isocratic Anion Standard Separation with the IonPac AS18

5.5. Isocratic Separation of Anions in 12-Anion Standard

The isocratic hydroxide eluent can be easily modified to resolve the common inorganic anions plus low molecular weight organic acids and oxyhalides on the IonPac AS18 column. Figure 10 shows the separation of these analytes eluting in 11 minutes using an optimized 32 mM potassium hydroxide eluent.

Inj. Volume: 25 μ L
Column: IonPac® AS18 Analytical Column and AG18 Guard Column (4-mm)
Eluent: 32 mM KOH
Eluent Source: EG with CR-ATC
Eluent Flow Rate: 1 mL/min
Temperature: 22 °C (ambient)
Detection: Suppressed Conductivity
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
MMS Regenerant: 50 mN H₂SO₄

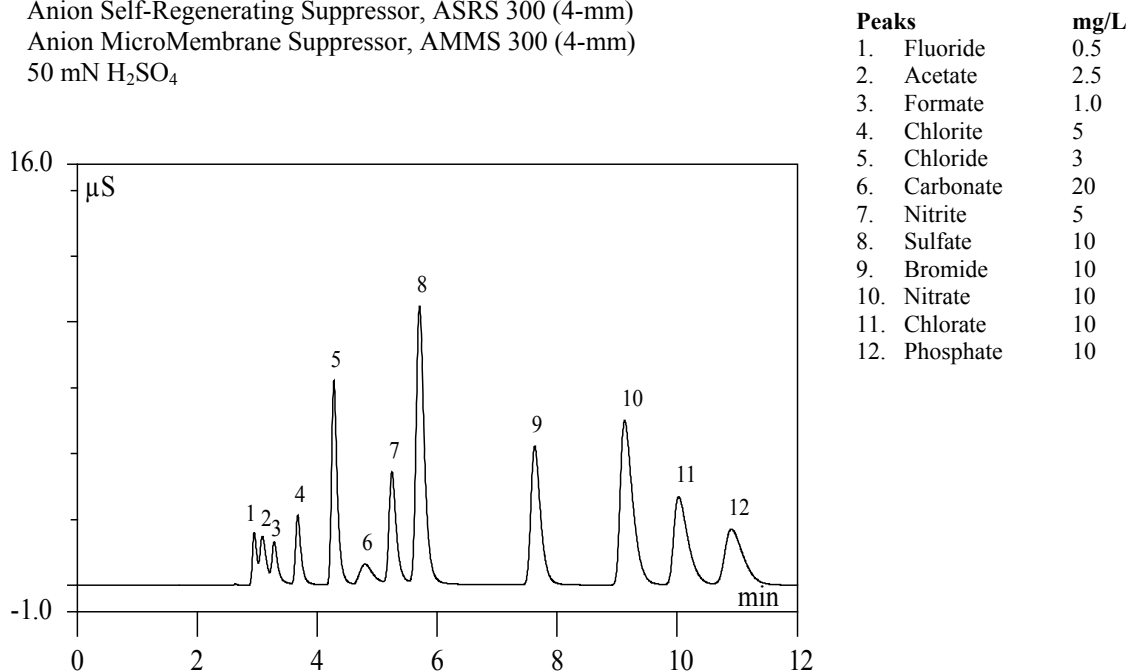


Figure 10
Isocratic Anion Standard Separation with the IonPac AS18 at 22°C

5.6. Two Municipal Drinking Water Samples Separated on AS18-Fast Using the ICS-5000 Analytical System

Figure 11 Shows the analysis of two municipal drinking water samples separated on the AS18-Fast 2 mm column using an isocratic eluent, the analytes are eluted in less than 5 minutes.

Column: IonPac® AG18-Fast, AS18-Fast, 2 mm
Eluent Source: EGC III KOH Cartridge
Eluent: 23 mM Potassium Hydroxide
Column Temp.: 30 °C
Flow Rate: 0.55 mL/min
Inj. Volume: 5 µL
Detection: ASRS 300®, 2 mm, 32 mA, recycle mode
Samples: A: Drinking Water A
B: Drinking Water B

Peaks:	mg/L	
	A	B
1. Fluoride	0.6	0.5
2. Chloride	8.5	11
3. Carbonate	46	46
4. Sulfate	10	11
5. Nitrate	0.1	--
6. Chlorate	--	0.4

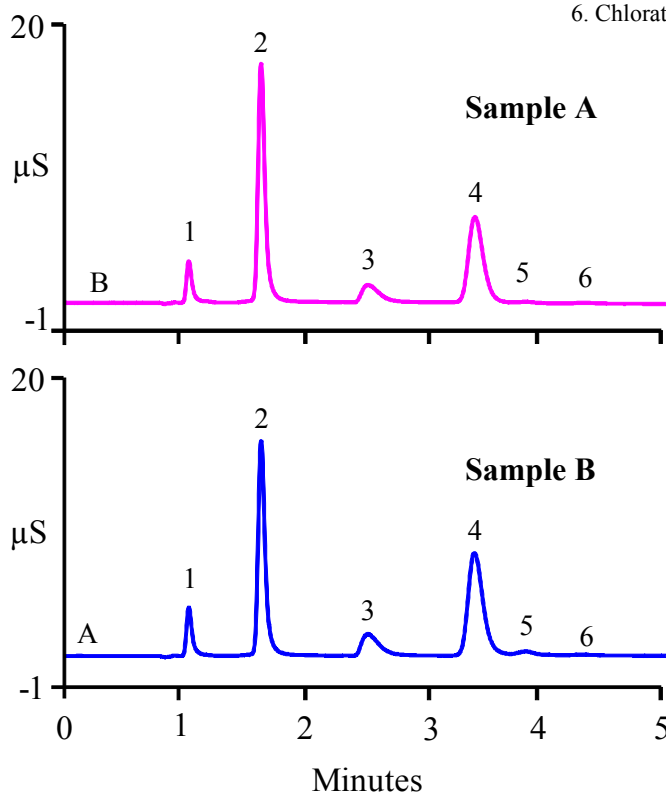


Figure 11
Two Municipal Drinking Water Samples Separated
on AS18 Fast Using the ICS-5000 Analytical System

5.7. Two Municipal Wastewater Effluent Samples Separated on AS18-Fast Using the ICS-5000 Analytical System

Waste water samples can often contain disparate analyte concentrations. The AS18-Fast column can separate analytes on diverse sample matrices in less than 4 minutes using an isocratic eluent as shown in Figure 12.

Column: IonPac® AG18-Fast, AS18-Fast, 2 mm
 Eluent Source: EGC III KOH Cartridge
 Eluent: 23 mM Potassium Hydroxide
 Column Temp.: 30 °C
 Flow Rate: 0.55 mL/min
 Inj. Volume: 5 µL
 Detection: ASRS 300®, 2 mm, 32 mA, recycle mode
 Samples: A: Drinking Water A
 B: Drinking Water B
 Sample Prep.: Filtered, 0.45 µm

Peaks:	mg/L	
	A	B
1. Fluoride	0.2	0.4
2. Formate	--	--
3. Chloride	169	281
4. Nitrite	0.2	0.8
5. Carbonate	--	--
6. Bromide	--	1.1
7. Sulfate	113	106
8. Nitrate	62	90

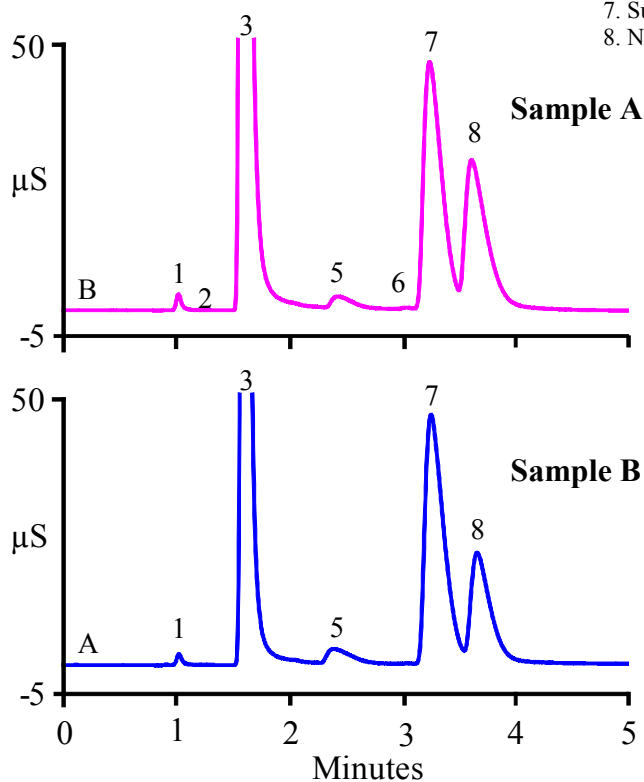


Figure 12
 Two Municipal Wastewater Effluent Samples Separated
 on AS18 Fast Using the ICS-5000 Analytical System

5.8. Step Change Separation with the IonPac AS18

Figure 13 illustrates the use of a potassium hydroxide step change to separate these anions in approximately 22 minutes. Fluoride, acetate and formate are resolved using a 19 mM potassium hydroxide eluent. At 10 minutes the concentration of the eluent steps to 30 mM potassium hydroxide to elute off the late eluting anions.

Inj. Volume: 25 μ L
 Column: IonPac AS18 Analytical Column and AG18 Guard Column (4-mm)
 Eluent: 19 to 30mM KOH at 10 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 22 °C (ambient)
 Detection: Suppressed conductivity
 AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES
 or SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H₂SO₄

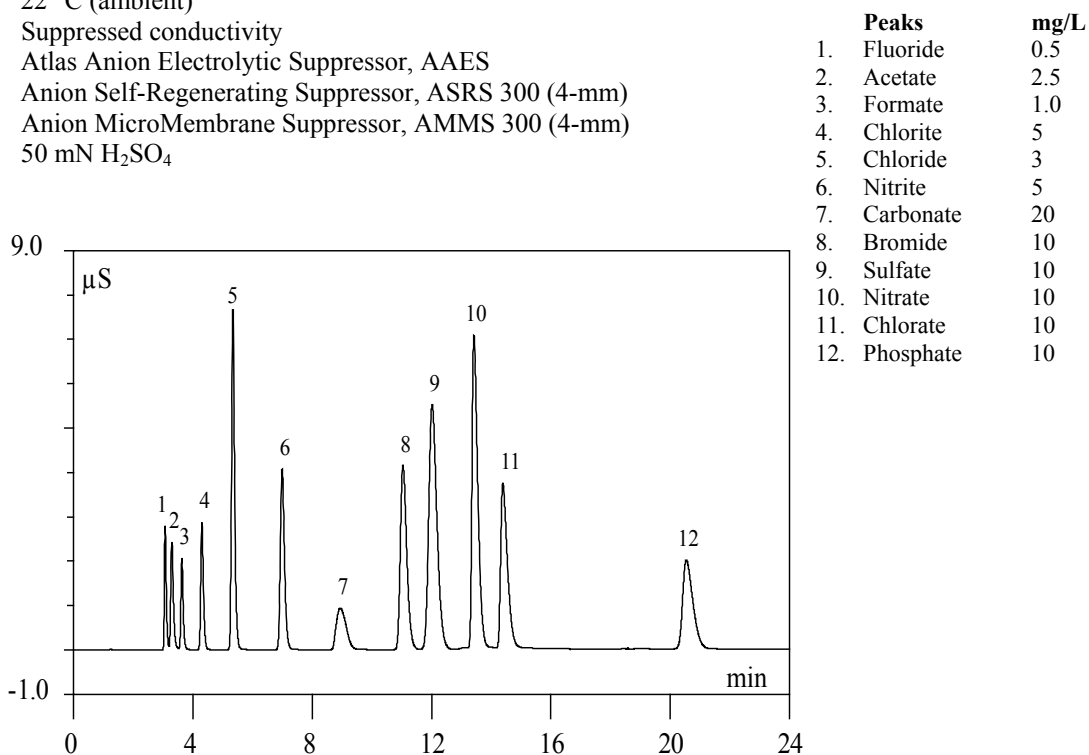
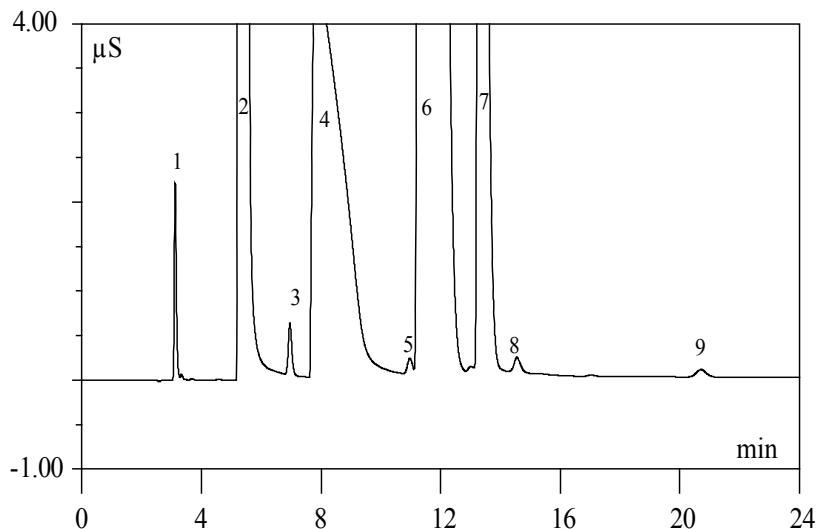


Figure 13
 Step Change Separation with the IonPac AS18

5.9. Step Change Separation of Anions in Simulated Drinking Water

The following chromatogram illustrates the analysis of a simulated drinking water sample using the AS18 column with a potassium hydroxide step change.

Inj. Volume: 100 μ L
 Column: IonPac AS18 Analytical Column and AG18 Guard Column(4-mm)
 Eluent: 19 to 30mM KOH at 10 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Detection: Suppressed conductivity
 Temperature: 22 °C (ambient)
 AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES
 or SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mM H₂SO₄



Peaks	mg/L
1. Fluoride	0.1
2. Chloride	100
3. Nitrite	0.1
4. Carbonate	100
5. Bromide	0.1
6. Sulfate	50
7. Nitrate	5.0
8. Chlorate	0.1
9. Phosphate	0.1

Figure 14
 Step Change Separation of Anions in Simulated Drinking Water with the IonPac AS18

5.10. Hydroxide Gradient Separation of Inorganic Anions

The AS18 can be operated with hydroxide gradients for optimum separation of inorganic anions in complex sample matrices. Simple hydroxide gradients can substantially improve the ability of the column to handle diverse analyte concentrations. As illustrated in Figure 15, a simple hydroxide gradient greatly improves the resolution of all analytes and limits the total analysis time to approximately 11 minutes.

Inj. Volume: 25 µL
 Column: IonPac AS18 Analytical Column and AG18 Guard Column(4-mm)
 Eluent: 22 mM from 0-7 min and 22 to 40 mM from 7-8 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30 °C
 Detection: Suppressed Conductivity
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H₂SO₄

	Peaks	mg/L
1.	Fluoride	0.5
2.	Acetate	2.5
3.	Formate	1.0
4.	Chlorite	5
5.	Chloride	3
6.	Nitrite	5
7.	Carbonate	20
8.	Bromide	10
9.	Sulfate	10
10.	Nitrate	10
11.	Chlorate	10
12.	Phosphate	10

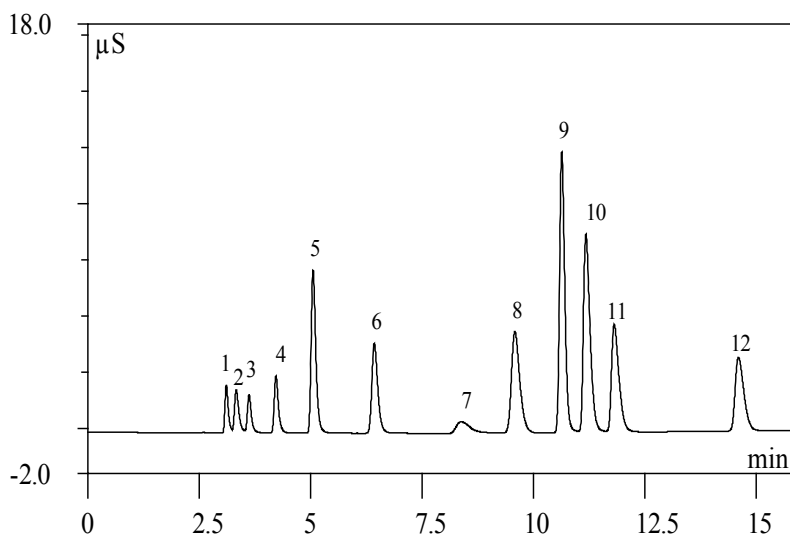


Figure 15
Gradient Separation with the IonPac AS18

5.11. Gradient Separation of Anions in Simulated Drinking Water

The following chromatogram illustrates the analysis of a simulated drinking water sample using the AS18 column with a simple potassium hydroxide gradient.

Inj. Volume: 100 μ L
 Column: IonPac AS18 Analytical Column and AG18 Guard Column (4-mm)
 Eluent: 22 mM from 0-7 min and 22 to 40 mM from 7-8 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30 $^{\circ}$ C
 Detection: Suppressed conductivity
 AES Suppressor: Atlas Anion Electrolytic Suppressor, AAES
 or SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H_2SO_4

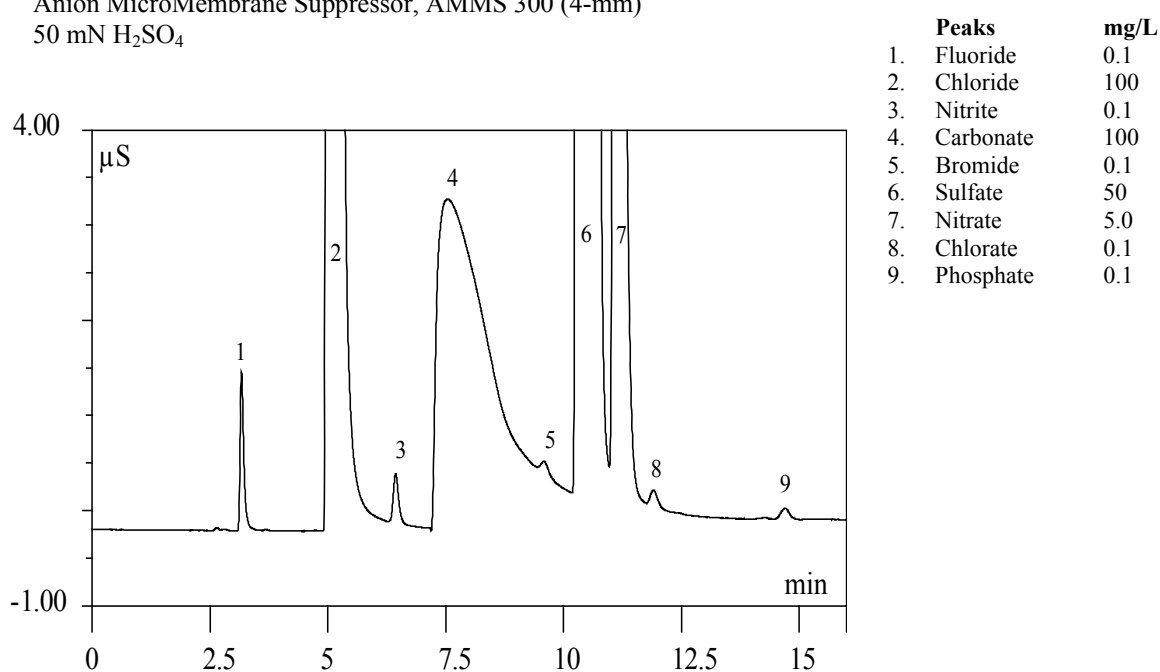


Figure 16
Gradient Separation of Anions in Simulated Drinking Water with the IonPac AS18

5.12. Gradient Separation of Anions in Sunnyvale Drinking Water

Figure 17 illustrates the application of this potassium hydroxide gradient to a real drinking water sample. In this figure, one can see not only the resolution of the analytes commonly found in drinking water, but also a number of less frequently identified impurities, such as acetate and formate.

Inj. Volume: 100 μ L
Column: IonPac AS18 Analytical Column and AG18 Guard Column
Eluent: 22 mM from 0-7 min and 22 to 40 mM from 7-8 min
Eluent Source: EG with CR-ATC
Eluent Flow Rate: 1 mL/min
Temperature: 30°C
Detection: Suppressed conductivity
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
MMS Regenerant: 50 mN H_2SO_4

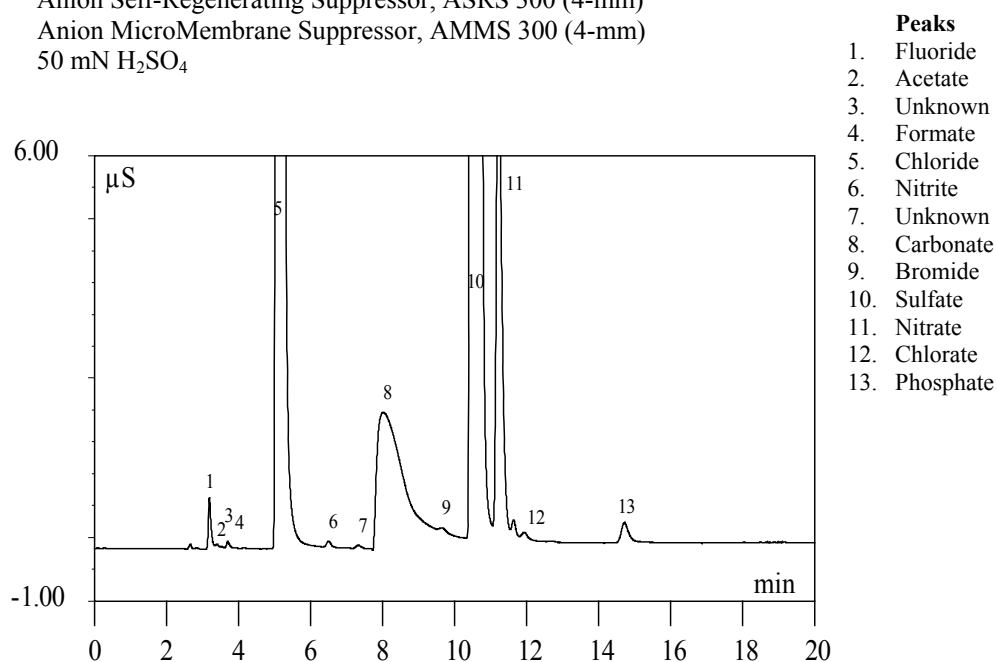


Figure 17
Gradient Separation of Anions in Sunnyvale Drinking Water with the IonPac AS18

5.13. Fast-Dual KOH Gradient Separation

The capacity and selectivity of the AS18 column provides the flexibility to modify the eluent conditions for optimum separation of the anions of interest. Figure 18 illustrates the use of a fast-dual potassium hydroxide gradient to resolve these anions in less than 11 minutes.

Inj. Volume: 25 μ L
 Column: IonPac® AS18 Analytical Column and AG18 Guard Column (4-mm)
 Eluent: 12 to 44 mM KOH from 0-5 min, 44 mM from 5-8 min, and 44 - 52 mM from 8-10 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30°C
 Detection: Suppressed conductivity
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H₂SO₄

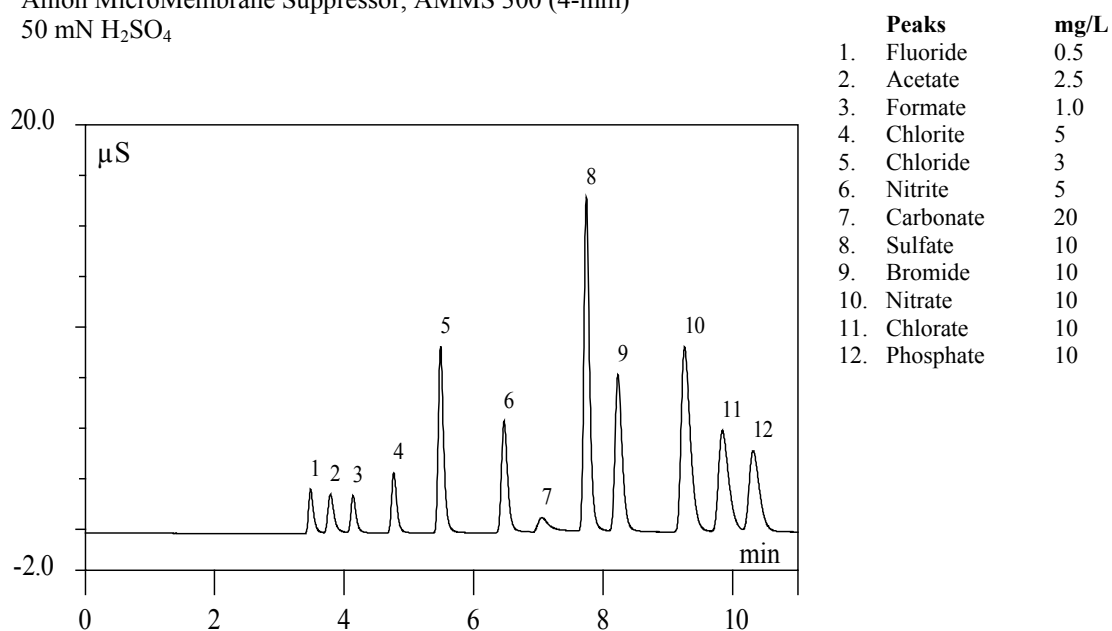


Figure 18
Gradient Separation with the IonPac® AS18 Column

5.14. Fast-Dual KOH Gradient Separation of Anions in Simulated Drinking Water

The following chromatogram illustrates the analysis of a simulated drinking water sample using the AS18 column with a fast-dual potassium hydroxide gradient.

Inj. Volume: 100 μ L
Column: IonPac AS18 Analytical Column and AG18 Guard Column
Eluent: 12 to 44 mM KOH from 0-5 min, 44 mM from 5-8 min, and 44-52 mM from 8-10 min
Eluent Source: EG with CR-ATC
Eluent Flow Rate: 1 mL/min
Temperature: 30 $^{\circ}$ C
Detection: Suppressed conductivity
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
MMS Regenerant: 50 mN H_2SO_4

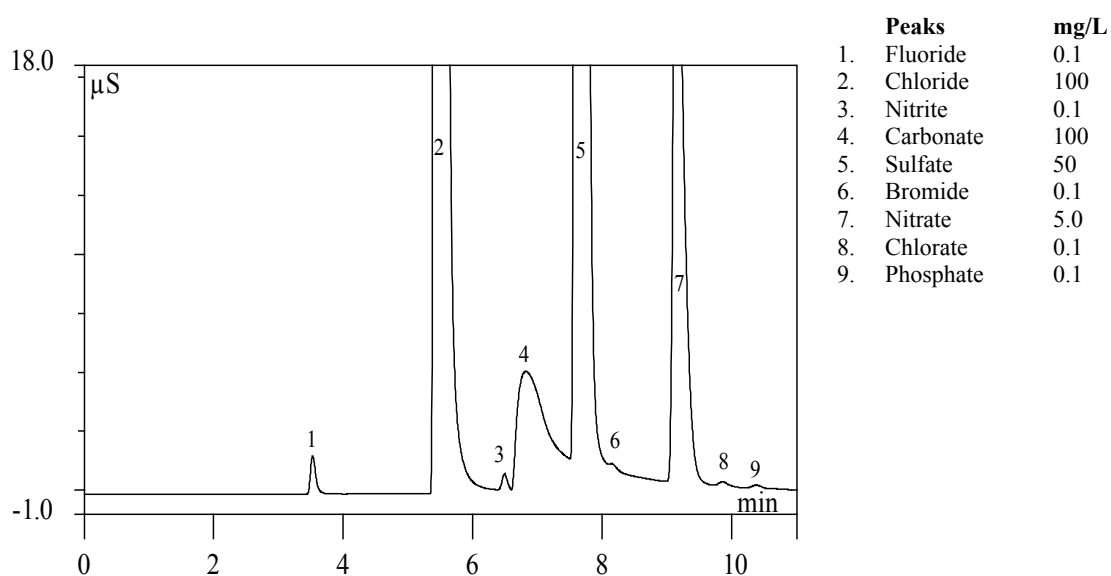


Figure 19
Gradient Separation of Anions in Simulated Drinking with the IonPac AS18

5.15. Optimum Eluent for Environmental Anions on the IonPac AS18

The AS18 column provides excellent separation of a variety of environmental anions including inorganic anions, oxyhalides (excluding bromate), oxyanions, and organic acids. With a fast dual hydroxide gradient, these analytes are easily separated in less than 14 minutes as illustrated in Figure 20.

Inj. Volume: 25 μ L
 Column: IonPac AS18 Analytical Column (4-mm)
 Eluent: 12 to 44 mM KOH from 0-5 min, 44 mM from 5-8 min, and 44-52 mM from 8-10 min
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30 $^{\circ}$ C
 Detection: Suppressed conductivity
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS 300 (4-mm)
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H_2SO_4

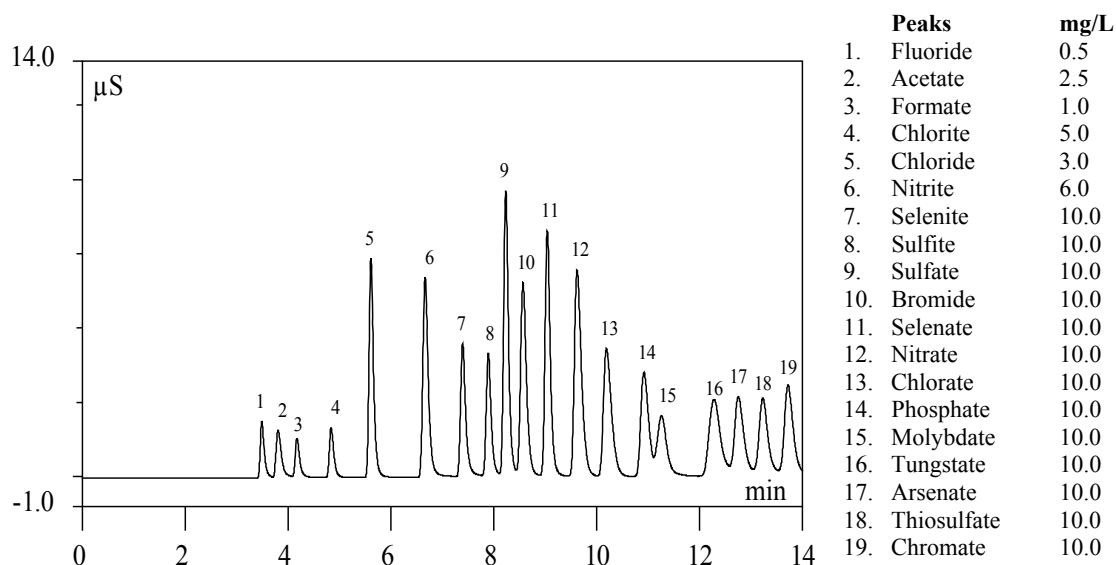


Figure 20
Optimum Eluent for Environmental Anions on the IonPac AS18

5.16. Effect of Temperature on Selectivity

Inj. Volume: 25 μ L
 Column: IonPac AS18 Analytical Column (4-mm)
 Eluent: 23 mM KOH
 Eluent Source: EG with CR-ATC
 Eluent Flow Rate: 1 mL/min
 Temperature: 30 $^{\circ}$ C
 Detection: Suppressed Conductivity
 Suppressor: Anion Self-Regenerating Suppressor, ASRS 300
 or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS 300 (4-mm)
 MMS Regenerant: 50 mN H_2SO_4

Peaks	mg/L
1. Fluoride	0.5
2. Acetate	2.5
3. Formate	1.0
4. Chlorite	5
5. Chloride	3
6. Nitrite	5
7. Carbonate	20
8. Bromide	10
9. Sulfate	10
10. Nitrate	10
11. Chlorate	10

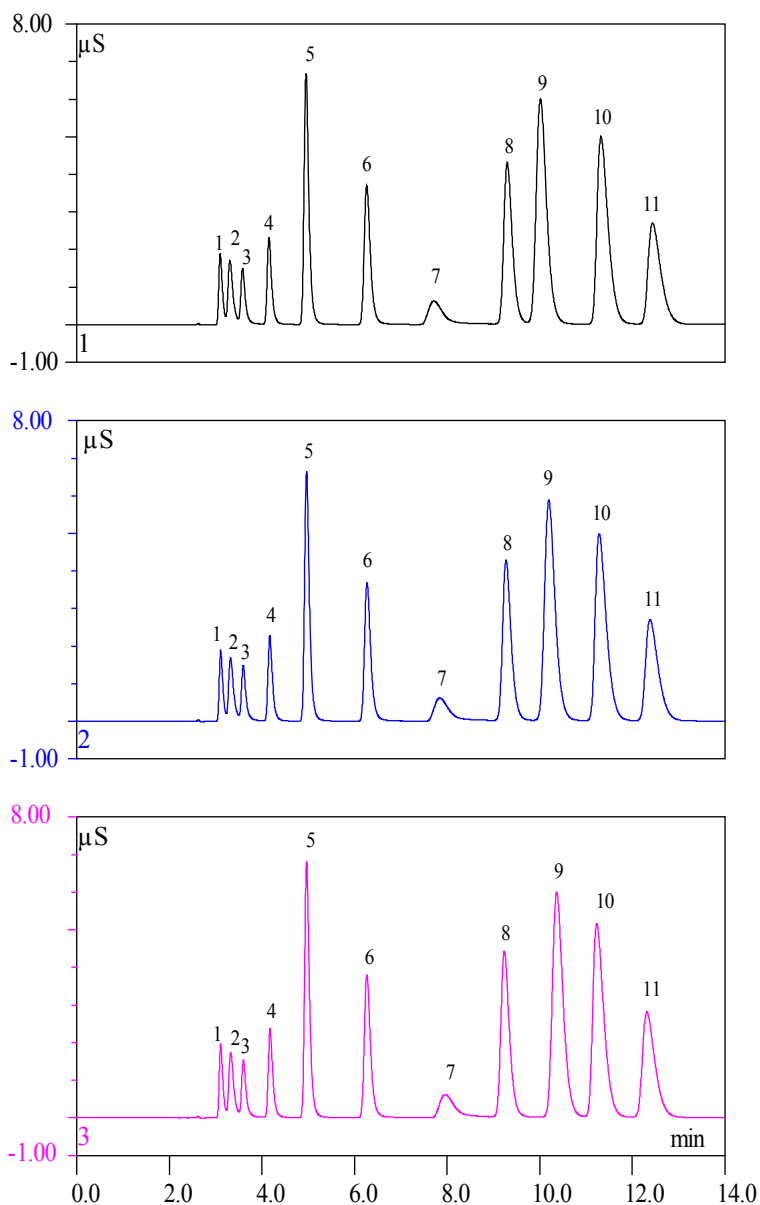


Figure 21
Effect of Temperature on Selectivity

SECTION 6 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS18 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
AS18/AG18 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports, Filter Eluents, and Filter Samples	6.1.2, 6.1.3, 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, ACES or AMMS	Clean Suppressor	6.2.6, Component Manual
	Contaminated Hardware	Clean Component	Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Re-plumb System	6.3.1.A,
	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	Conc. Incorrect Eluents	Remake Eluents	6.3.3.A
Resolution	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	Re-plumb System	6.3.3.D, Component Manual
Spurious Peaks	Sample Contaminated	Pre-treat 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 AG18/AG18-Fast (4-mm) Guard Column plus the AS18/AS18-Fast (4-mm) Analytical/Capillary Column when using the test chromatogram conditions should be equal or less than 1,800 psi. If the system pressure is higher than 1,800 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 3 mL/min and the maximum pressure is 4,000 psi (27.58 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/Capillary columns are connected (see Table 7, "Typical AS18/AG18 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 AS18/AG18/AS18-Fast/AG18-Fast Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS18 4-mm Analytical	≤ 1,500 (10.34)	1.0
AG18 4-mm Guard	≤ 300 (2.07)	1.0
AS18 + AG18 4-mm columns	≤ 1,800 (12.41)	1.0
AS18-Fast 4-mm Analytical	≤ 1100 (7.59)	0.6
AG18-Fast 4-mm Guard	≤ 180 (1.24)	0.6
AS18-Fast + AG18-Fast 4-mm columns	≤ 1280 (8.83)	0.6
AS18 2-mm Analytical	≤ 1,500 (10.34)	0.25
AG18 2-mm Guard	≤ 300 (2.07)	0.25
AS18 + AG18 2-mm columns	≤ 1,800 (12.41)	0.25
AS18-Fast 2-mm Analytical	≤ 1100 (7.59)	0.25
AG18-Fast 2-mm Guard	≤ 180 (1.24)	0.25
AS18-Fast + AG18-Fast 2-mm columns	≤ 1280 (8.83)	0.25
AS18-Fast 0.4-mm Capillary	≤ 1,200 (8.27)	0.010
AG18-Fast 0.4-mm Capillary Guard	≤ 300 (2.07)	0.010
AS18-Fast + AG18-Fast 0.4-mm columns	≤ 1,500 (10.34)	0.010
AS18 0.4-mm Capillary	≤ 1500 (10.34)	0.010
AS18 0.4-mm Capillary Guard	≤ 300 (2.07)	0.010
AS18 + AG18 0.4-mm column	≤ 1800 (12.41)	0.010

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 bench-top or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you do not scratch the walls of the end fitting. 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.

Table 8
Product Information

Product	AS18 4-mm Columns (P/N)	AS18-Fast 4-mm Columns (P/N)	AS18 2-mm Columns (P/N)	AS18-Fast 2-mm Columns (P/N)	AS18-Fast 0.4-mm Columns (P/N)	AS18 0.4-mm Columns (P/N)
Analytical Column	060549	075760	060553	075759	072062	075772
Guard Column	060551	075762	060555	075761	072063	075773
Bed Support Assembly	042955	042955	044689	044689	N/A	N/A
End Fitting	052809	052809	043278	043278	N/A	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 finger-tight, 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:

Table 9
Background Conductivity

Eluent	Expected Background Conductivity
39 mM NaOH	≤ 3 µS
39 mM KOH (EGC)	≤ 1.0 µS

6.2.1. Preparation of Eluents

- Make sure that the eluents and the regenerant (if used) are made correctly.
- Make sure that the eluents are made from chemicals with the recommended purity.
- 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 AG18/AG18-Fast Guard and AS18/AS18-Fast Analytical/Capillary Columns from the system. Install a backpressure coil that generates approx. 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 AG18/AG18-Fast 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 backpressure coil that generates approx. 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 μ S. 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. 031367). 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 operation refer to the Anion Electrolytic Capillary Suppressor ACES 300 Product Manual (Document No. 065386).

Metal Contaminants or Precipitates



NOTE

The suppressor voltage is a good indicator of the resistance across the suppressor. Higher resistance may indicate contamination of the suppressor. For more information regarding monitoring the voltage, see Document No. 031814, "Removal of Iron Contamination from Electrolytic Suppressors."

- 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 if operating in the AutoSuppression External Water mode or the Chemical Suppression mode of 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.

6.2.7. A Contaminated Anion Atlas Electrolytic Suppressor, AAES

If the above items have been checked and the problem persists, the Anion Atlas Electrolytic Suppressor is probably causing the problem. For details on Anion Atlas Electrolytic Suppressor operation, refer to the Product Manual (Document No. 031770) 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

6.3.1.1. Peak Fronting:

- a) **Peak Fronting:** Check to see if headspace has developed in the guard or analytical/capillary 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



NOTE

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 shorter run times as eluent will be more concentrated 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. 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) 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.

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) or 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 affects the concentration generated by the eluent generator.
- 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 AS18 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.

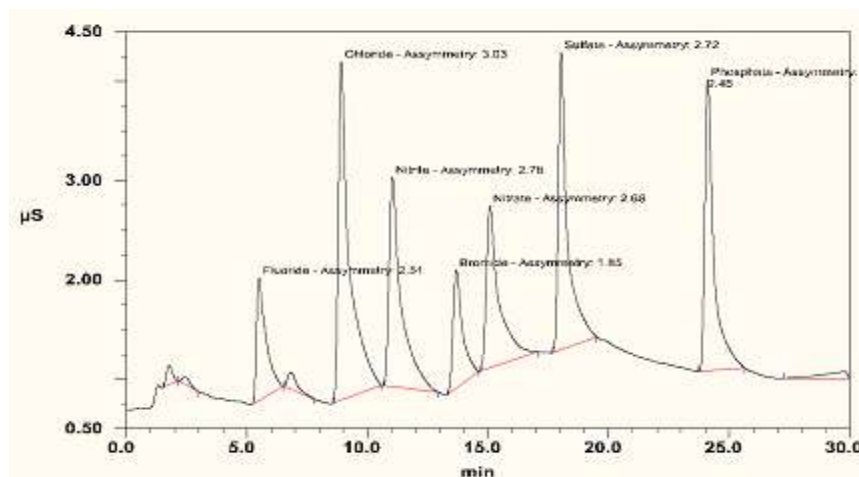


Figure 22
Tailing Peaks Caused by Incorrectly Installed
Capillary Tubing Fittings

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 23 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

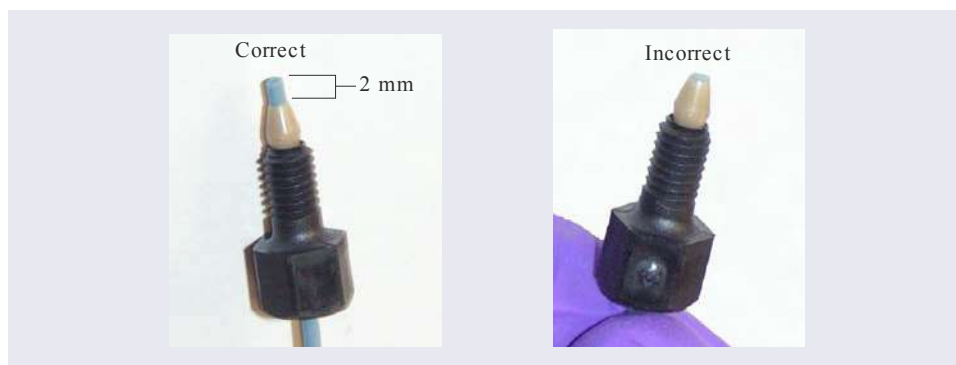


Figure 23
Correct and Incorrect Ferrule and
Fitting Bolt Placement for Capillary Tubing Connections

APPENDIX A - QUALITY ASSURANCE REPORTS

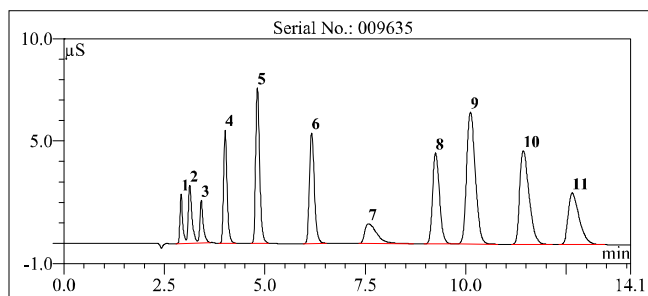
- A.1 Quality Assurance Report - IonPac AS18 Analytical Column (4 x 250 mm)
- A.2 Quality Assurance Report - IonPac AS18 Analytical Column (2 x 250 mm)
- A.3 Quality Assurance Report - IonPac AS18-Fast Capillary Column (0.4 x 150 mm)
- A.4 Quality Assurance Report - IonPac AS18-Fast Analytical Column (4 x 150 mm)
- A.5 Quality Assurance Report – IonPac AS18-Fast Analytical Column (2 x 150 mm)
- A.6 Quality Assurance Report – IonPac AS18 Capillary Column (0.4 x 250 mm)

A.1 Quality Assurance Report - IonPac AS18 Analytical Column (4 x 250 mm)

IonPac® AS18
Analytical (4 x 250 mm)
Product No. 060549

Date: 10-Feb-11 10:26
Serial No. : 009635
Lot No. : 007-08-167D

Eluent: 23 mM KOH
Eluent Source EGC-KOH II Cartridge
Flow Rate: 1 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 4mm)
 AutoSuppression® Recycle Mode
Applied Current: 57 mA
Injection Volume: 20 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.92	1.8	1.60	9899	0.5
2	Acetate	3.13	1.8	1.94	6616	2.5
3	Formate	3.42	1.8	4.12	9618	1.0
4	Chlorite	4.01	1.4	4.98	11265	5.0
5	Chloride	4.82	1.4	6.88	12558	3.0
6	Nitrite	6.16	1.4	3.71	12484	5.0
7	Carbonate	7.58	2.7	3.89	3102	20.0
8	Bromide	9.25	1.4	2.43	13677	10.0
9	Sulfate	10.12	1.4	3.20	10340	10.0
10	Nitrate	11.43	1.9	2.66	11519	10.0
11	Chlorate	12.65	2.0	n.a.	10457	10.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	1.0-2.2	Passed
Sulfate	Retention Time	8.90-11.10	Passed
Chloride	Efficiency	>=9900	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1650	1390

Production Reference:

Datasource: Resin
 Directory: RTB\RTB_5
 Sequence: RQ_AS18_4X250MM_1-27-11
 Sample No.: 9

6.70 Build 1820 (Demo-Installation)

Chromeleon® Dionex Corp. 1994-2011

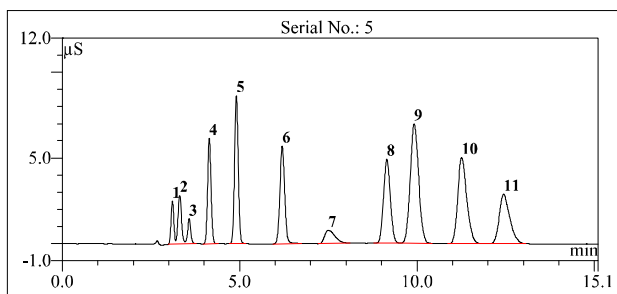
066673-03 (QAR)

A.2 Quality Assurance Report - IonPac AS18 Analytical Column (2 x 250 mm)

IonPac® AS18
Analytical (2 x 250 mm)
Product No. 060553

Date: 31-Jan-11 13:17
Serial No. : 000005
Lot No. : 006-09-088A

Eluent: 23 mM KOH
Eluent Source: EGC-KOH II Cartridge
Flow Rate: 0.25 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 2mm)
 AutoSuppression® Recycle Mode
Applied Current: 15 mA
Injection Volume: 5 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (A1A)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	3.10	1.2	1.33	7951	0.5
2	Acetate	3.31	1.1	1.58	5523	0.5
3	Formate	3.58	0.9	3.35	7909	1.0
4	Chlorite	4.14	1.0	4.12	8655	5.0
5	Chloride	4.91	1.0	5.88	10264	3.0
6	Nitrite	6.20	1.1	3.29	10060	5.0
7	Carbonate	7.50	2.1	3.70	3047	20.0
8	Bromide	9.15	1.1	1.97	10905	10.0
9	Sulfate	9.92	1.2	2.99	8297	10.0
10	Nitrate	11.26	1.5	2.38	9582	10.0
11	Chlorate	12.44	1.6	n.a.	8641	10.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	1.0-2.2	Passed
Sulfate	Retention Time	8.90-11.10	Passed
Chloride	Efficiency	>=9000	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1650	1075

Production Reference:

Datasource: Resin
 Directory: RTB\RTB_5
 Sequence: RQ_AS18_2X250MM_1-28-11
 Sample No.: 12

6.80 DU10a Build 2826 (171948) (Demo-Installation)

Chromelcon® Dionex Corp. 1994-2011

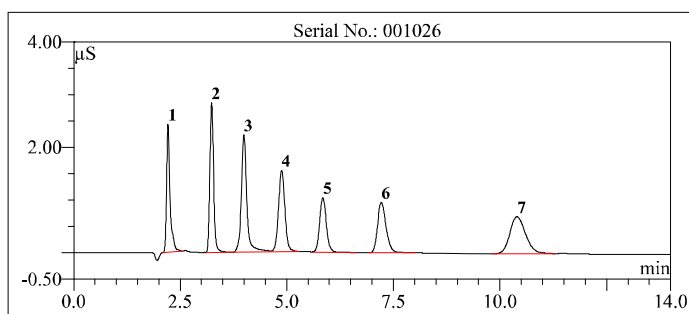
066670-03 (QAR)

A.3 Quality Assurance Report - IonPac AS18-Fast Capillary Column (0.4 x 150 mm)

IonPac® AS18-Fast
Capillary (0.4 x 150 mm)
Product No. 072062

Date: 26-Aug-10 13:38
Serial No. : 001026
Lot No. : 010-04-090

Eluent: 30 mM KOH
Eluent Source: EGC-KOH (Capillary)
Flow Rate: 0.010 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Capillary Electrolytic Suppressor (ACES 300)
 AutoSuppression® Recycle Mode
Applied Current: 8 mA
Injection Volume: 0.4 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.21	2.0	7.15	4499	0.2
2	Chloride	3.24	1.2	4.20	6957	0.5
3	Nitrite	3.99	1.2	3.74	6017	1.0
4	Sulfate	4.88	1.1	3.52	5252	1.0
5	Bromide	5.85	1.2	4.27	6928	1.0
6	Nitrate	7.22	1.4	6.17	6330	1.0
7	Phosphate	10.40	1.3	n.a.	3834	2.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	0.9-2.1	Passed
Sulfate	Retention Time	4.15-5.25	Passed
Chloride	Efficiency	>=4950	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1650	900

Production Reference:

Datasource: Archive-Con
 Directory: CON-2010\COLUMN\CAPILLARY\CAP_ANION-3
 Sequence: AS18_FAST_P4X150MM_836591
 Sample No.: 7

6.70 Build 1820 (Demo-Installation)

Chromeleon® Dionex Corp. 1994-2011

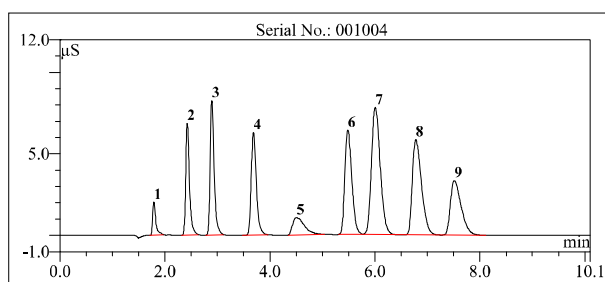
074170-03 (QAR)

A.4 Quality Assurance Report - IonPac AS18-Fast Column (4 x 150 mm)

IonPac® AS18 Fast
Analytical (4 x 150 mm)
Product No. 075760

Date: 04-Mar-11 15:52
Serial No. : 001004
Lot No. : 010-21-113

Eluent: 23 mM KOH
Eluent Source: EGC-KOH II Cartridge
Flow Rate: 1 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 4mm)
 AutoSuppression® Recycle Mode
Applied Current: 57 mA
Injection Volume: 20 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (ATA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	1.79	2.0	5.73	5218	0.5
2	Chlorite	2.43	1.6	3.66	6151	5.0
3	Chloride	2.90	1.5	5.21	7544	3.0
4	Nitrite	3.69	1.5	2.80	7480	5.0
5	Carbonate	4.51	2.6	2.97	1870	20.0
6	Bromide	5.48	1.5	1.90	8165	10.0
7	Sulfate	6.01	1.4	2.45	5980	10.0
8	Nitrate	6.78	1.9	2.11	7092	10.0
9	Chlorate	7.51	2.0	n.a.	6413	10.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	1.0-2.2	Passed
Sulfate	Retention Time	5.34-6.66	Passed
Chloride	Efficiency	>=5940	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1100	892

Production Reference:

Datasource: Resin
 Directory: RTB\RTB_5
 Sequence: RQ_Val_AS18_4X150MM-3-3-11
 Sample No.: 16

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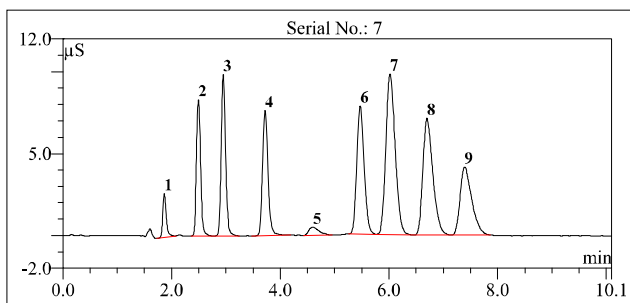
6.70 Build 1820 (Demo-Installation)

A.5 Quality Assurance Report – IonPac AS18-Fast Analytical Column (2 x 150 mm)

IonPac® AS18 Fast
Analytical (2 x 150 mm)
Product No. 075759

Date: 26-Jan-11 15:36
Serial No. : 000007
Lot No. : 010-04-090

Eluent: 23 mM KOH
Eluent Source: EGC-KOH II Cartridge
Flow Rate: 0.25 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 2mm)
 AutoSuppression® Recycle Mode
Applied Current: 15 mA
Injection Volume: 5 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	1.86	1.4	5.00	4264	0.5
2	Chlorite	2.49	1.3	3.25	5279	5.0
3	Chloride	2.95	1.2	4.68	6469	3.0
4	Nitrite	3.72	1.2	3.28	6594	5.0
5	Carbonate	4.60	2.1	2.89	2706	20.0
6	Bromide	5.47	1.3	1.94	7467	10.0
7	Sulfate	6.02	1.3	2.11	5877	10.0
8	Nitrate	6.70	1.6	1.94	6552	10.0
9	Chlorate	7.39	1.8	n.a.	5868	10.0

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	1.0-2.2	Passed
Sulfate	Retention Time	5.34-6.66	Passed
Chloride	Efficiency	>=5400	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1100	874

Production Reference:

Datasource: Resin
 Directory: RTB\RTB_5
 Sequence: 892862_VAL_AS18_2X150MM
 Sample No.: 43

6.80 DU10a Build 2826 (171948) (Demo-Installation)

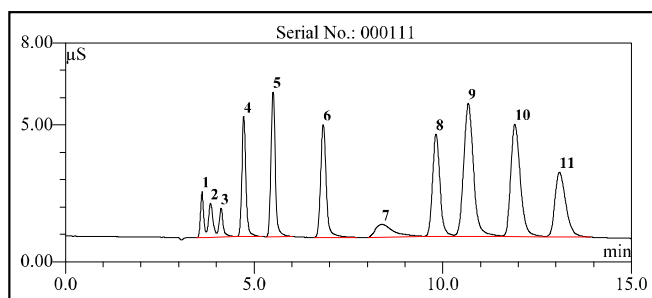
Chromeleon® Dionex Corp. 1994-2011

A.6 Quality Assurance Report – IonPac AS18 Capillary Column (0.4 x 250 mm)

IonPac® AS18
Capillary (0.4 x 250 mm)
Product No. 075772

Date: 09-Mar-11 15:26
Serial No. : 000111
Lot No. : 2010-15-96.1

Eluent: 23 mM KOH
Eluent Source: EGC-KOH (Capillary)
Flow Rate: 0.010 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity
Suppressor: Anion Capillary Electrolytic Suppressor (ACES 300)
 AutoSuppression® Recycle Mode
Applied Current: 8 mA
Injection Volume: 0.4 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret. Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	3.62	n.a.	1.27	9446	0.25
2	Acetate	3.85	n.a.	1.41	5228	1.25
3	Formate	4.12	n.a.	3.33	8850	0.50
4	Chlorite	4.73	1.1	4.02	10106	2.50
5	Chloride	5.50	1.1	5.84	12153	1.50
6	Nitrite	6.83	1.2	2.90	11296	2.50
7	Carbonate	8.39	2.5	2.46	1689	10.00
8	Bromide	9.82	1.2	2.12	12549	5.00
9	Sulfate	10.68	1.2	2.71	8673	5.00
10	Nitrate	11.91	1.5	2.43	10941	5.00
11	Chlorate	13.09	1.6	n.a.	10182	5.00

QA Results:

Analyte	Parameter	Specification	Results
Sulfate	Asymmetry	1.0-2.2	Passed
Sulfate	Retention Time	8.90-11.10	Passed
Chloride	Efficiency	>=9900	Passed
Chloride	Asymmetry	1.0-1.9	Passed
	Pressure	<=1650	1325

Production Reference:

Datasource: Column
 Directory: Capillary\Cap_Anion-1
 Sequence: AS18_P4X250MM_VAL_02-25-11
 Sample No.: 54

6.70 Build 1820 (Demo-Installation)

Chromeleon® Dionex Corp. 1994-2011

076182-01 (QAR)

APPENDIX B - COLUMN CARE

B.1 Recommended Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for IonPac AS18 columns is 4,000 psi (27.57 MPa).

B.2 Column Start-Up

The column is shipped using 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 10 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; however, the following precautions should be observed..



WARNING

- *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

Contamination	Solution
Hydrophilic Contamination of Low Valence	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.
High Valence Hydrophilic Ions Contamination	Concentrated acid solutions such as 1 to 3 M HCl will remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.
Metal Contamination	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.
Nonionic and Hydrophobic Contamination	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.
Ionic and Hydrophobic Contamination	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.
	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.

B.4.2 Column Cleanup Procedure

Use the following cleanup procedures to clean the AG18/AG18-Fast and AS18/AS18-Fast.

- Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in, "Choosing the Appropriate Cleanup Solution".
- Disconnect the ASRS 300, ACES 300 or AMMS 300 from the IonPac AS18 / AS18-Fast Analytical / Capillary 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 / capillary column in the eluent flow path. If not, the 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.

- Set the pump flow rate to 1.0 mL/min for an AS18/AS18-Fast 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS18/AS18-Fast 2-mm Analytical or Guard Column. Set the flow rate to 0.010 mL/min for an AS18-Fast 0.4-mm Capillary or Capillary Guard Column.
- Rinse the column for 10 minutes with deionized water before pumping the chosen cleanup solution over the column
- Pump the cleanup solution through the column for at least 60 minutes.
- Rinse the column for 10 minutes with deionized water before pumping eluent over the column.
- Equilibrate the column(s) with eluent for at least 60 minutes before resuming normal operation.
- Reconnect the ASRS 300, ACES 300 or AMMS 300 to the AS18/AS18-Fast Analytical/Capillary Column
- Place the guard column in line between the injection valve and the analytical/capillary column if your system was originally configured with a guard column.

APPENDIX C - CONFIGURATION

Table C1
Configuration

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: <i>Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven.</i>			
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: <i>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.</i>			
Chromatographic Module	A thermally controlled column oven such as the LC25, LC30, ICS-10, 11, 15, 16, 20, 2100, 3000, 5000 DC	A thermally controlled column oven such as the LC25, 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 μ L, P/N 046423) VDM-2 Cell (3-mm, 2.0 μ L) (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 μ L, P/N 049393) VDM-2 Cell (6-mm, 10 μ L) 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 C2
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	071870	0.0025	0.006	0.0009	1766.0	441.0	58.0