



PRODUCT MANUAL

for

IonPac[®] AG17-C

IonPac[®] AS17-C



IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

IONPAC® AG17-C GUARD COLUMN

4 x 50 mm, (P/N 066295)

2 x 50 mm, (P/N 066297)

IONPAC® AS17-C ANALYTICAL COLUMN

4 x 250 mm, (P/N 066294)

2 x 250 mm, (P/N 066296)

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SECTION 1 - INTRODUCTION TO IONPAC AS17-C / AG17-C CHROMATOGRAPHY

The IonPac® AS17-C Analytical Column in combination with the AG17-C Guard Column is designed for the analysis of inorganic anions including fluoride, chloride, nitrite, bromide, nitrate, sulfate, and phosphate in less than ten minutes using a hydroxide gradient delivered with an Eluent Generator. The key application for the AS17-C column is the determination of common inorganic anions in high purity water matrices. This column is designed primarily to eliminate or minimize the sulfate blanks during trace level analysis of anions. The selectivity of the IonPac AS17-C Guard plus Analytical Column set has been designed to retain fluoride well out of the water dip (system dip). Using gradient conditions, the inorganic anions can easily be separated in a variety of sample matrices including high purity water drinking water, ground water, waste water, process streams, and scrubber solutions. The AS17-C is compatible with pH 0-14 eluents and eluents containing organic solvents from 0-100% in concentration. The AS17-C can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor. The IonPac AS17-C has nominal efficiency of at least 28,000 plates/meter for sulfate using standard operating conditions.

Table 1
IonPac AS17-C / AG17-C Packing Specifications

Column	Particle Diameter μm	Substrate X-linking %	Column Capacity μeq/column	Functional Group	Hydrophobicity
AS17-C 4 x 250 mm	10.5	55	30	Alkanol quaternary ammonium	Low
AG17-C 4 x 50 mm	10.5	55	6	Alkanol quaternary ammonium	Low
AS17-C 2 x 250 mm	10.5	55	7.5	Alkanol quaternary ammonium	Low
AG17-C 2 x 50 mm	10.5	55	1.5	Alkanol quaternary ammonium	Low

Resin composition: microporous polyvinylbenzyl ammonium polymer cross-linked with divinylbenzene

Table 2
AS17-C / AG17-C Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS17-C 4-mm Analytical	≤ 1200 (5.17)	1.0	3.0
AG17-C 4-mm Guard	≤ 200 (1.03)	1.0	3.0
AS17-C + AG17-C 4-mm columns	≤ 1400 (6.20)	1.0	3.0
AS17-C 2-mm Analytical	≤ 1200 (5.17)	0.25	0.75
AG17-C 2-mm Guard	≤ 200 (1.03)	0.25	0.75
AS17-C + AG17-C 2-mm columns	≤ 1400 (6.20)	0.25	0.75

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.

See Appendix B, Comparison of Ion Chromatography Systems for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor (SRS), 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 2-mm Operation

The IonPac AS17-C 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) is recommended.

3.1.2 System Requirements for 4-mm Operation

The IonPac AS17-C 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 standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons).

3.1.3 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" ID PEEK tubing (P/N 044221). 0.010" ID PEEK tubing (P/N 042260) may be used but peak efficiency will be compromised which may also result in decreased peak resolution. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

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), the Ultra Trace Anion Concentrator Ultra Low Pressure Column (UTAC-ULP1, P/N 063475), the Ultra Trace Anion Concentrator Extremely Low Pressure Column (UTAC-XLP1, P/N 063459), or the IonPac AG17-C Guard Column can be used for trace anion concentration work. 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 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.)

These techniques can also be applied to the AG17-C.



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 AS17-C.

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 AS17-C 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. For typical low to sub ppb samples, you can inject up to 2-4 mL.

3.4 The IonPac AG17-C Guard Column

An IonPac AG17-C Guard Column is normally used with the IonPac AS17-C Analytical Column. Retention times will increase by approximately 20% when a guard column is placed in-line prior to the analytical column. A guard is placed prior to the analytical column to prevent sample contaminants from eluting onto the analytical column. It is easier to clean or replace a guard column than it is an analytical column. Replacing the AG17-C Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS17-C Analytical Column.

3.5 The Anion Trap Column, CR-ATC

Several IonPac AS17-C applications consist of the EG40 or EG50 with an EGC II KOH cartridge. In this case, a Continuously Regenerated AnionTrap Column, CR-ATC (P/N 060477) 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).

Alternatively, the ATC-HC Trap Column (P/N 059604) can be installed between the pump outlet and the inlet of the EluGen Cartridge in the EG40 or EG50 Module to remove anionic contaminants from the carrier deionized water. The ATC-HC is for use with EGC II KOH or EGC II NaOH cartridge in the EG40 and EG50 Eluent Generators (see the ATC-HC Product Manual (Document No. 032697) for instructions).

3.6 Eluent Storage

IonPac AS17-C 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).



CAUTION

DO NOT USE GLASS BOTTLES for either stock solution bottles or eluent bottles!
Base slowly dissolves glass, releasing impurities that adversely effect the IonPac AS17-C column performance.

3.7 Anion Self-Regenerating Suppressor Requirements

An Anion Self-Regenerating Suppressor should be used for applications that require suppressed conductivity detection. It is compatible with solvent (up to 40%) 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® ULTRA II modes of operation.



NOTE

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

If you are installing an IonPac AS17-C 4 mm Analytical Column, use an ASRS® ULTRA II (4 mm, P/N 061561).

If you are installing an IonPac AS17-C 2 mm Analytical Column, use an ASRS® ULTRA II (2 mm, P/N 061562).

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-ULTRA II, the ASRS® ULTRA II.”

3.8 Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS III) may be used instead of an ASRS® ULTRA II (4 mm) for applications that require suppressed conductivity detection. Use an AMMS III 4 mm (P/N 056750) with the IonPac AS17-C 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® III 2 mm (P/N 056751).

For detailed information on the operation of the Anion MicroMembrane Suppressor, see Document No. 031727, the “Product Manual for the Anion MicroMembrane Suppressor III, the AMMS® III.”

3.9 Using Displacement Chemical Regeneration (DCR) with 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® III). See the DCR kit manual, Document P/N 031664, for details.



SAFETY

Use proper safety precautions in handling acids and bases.

3.10 Using AutoRegen with the ASRS® ULTRA II in the Chemical Suppression Mode or the AMMS® III

To save regenerant preparation time and reduce regenerant consumption and waste, DIONEX recommends using an AutoRegen® Accessory (P/N 039594). For more detailed information on the use of the AutoRegen Accessory see the AutoRegen Accessory manual (Document No. 032853). For more detailed information on the use of AutoRegen Regenerant Cartridges, see the “Product Manual for the AutoRegen Regenerant Cartridge Refills” (Document No. 032852).

3.11 Detector Requirements

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

3.12 Using the EGC-KOH with AS17-C

Please refer to ICS-3000 manual for operation of ICS-3000 systems document No. EG-065031. Please refer to the EG50 Product Manual, Document No. 031908, for information on the operation of the EG50. The AS17-C column is recommended for use with ICS-2000, or ICS-3000 IC Systems equipped with an Eluent Generator. The AS17-C 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 Eluents or Sodium Hydroxide Eluent from deionized water.

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
Column:	2 mm: AS17-C 2 mm Analytical Column + AG17-C 2 mm Guard Column 4 mm: AS17-C 4 mm Analytical Column + AG17-C 4 mm Guard Column
Eluent:	15 mM KOH
Eluent Flow Rate:	2 mm: 0.25 mL/min 4 mm: 1.0 mL/min
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (2 mm or 4 mm), AutoSuppression Recycle Mode
SRS Optimal Current Settings or MMS Suppressor:	38 mA (4 mm) Anion MicroMembrane Suppressor, AMMS® III (2 mm or 4 mm)
MMS Regenerant:	50 mM NH_2SO_4
Expected Background	
Conductivity:	$\leq 2 \mu\text{S}$
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2 IonPac AS17-C 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- 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 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 AS17-C 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 AS17-C columns is 4,000 psi (27.57 MPa).

The AS17-C 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 5
HPLC Solvents for Use with IonPac AS17-C 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 (ASRS® ULTRA II) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents. Do not use > 40% solvent on the ASRS® ULTRA II 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.

**CAUTION**

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.

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 15 mM NaOH use 1.20 g of 50% sodium hydroxide:
(as used in Section 5.3, “Production Test Chromatogram”)

$$\text{For 15 mM:} \quad \frac{0.015 \text{ mole/L} \times 40.01 \text{ g/mole}}{50\%} = 1.20 \text{ g diluted to 1 L}$$

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.

$$g = dvr$$

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 15 mM NaOH use 0.78 mL of 50% sodium hydroxide:
(as used in Section 5.3, "Production Test Chromatogram")

$$\text{For 15 mM: } \frac{0.015 \text{ mole/L} \times 40.01 \text{ g/mole}}{50\% \times 1.53 \text{ g/mL}} = 0.78 \text{ mL diluted to 1 L}$$

* 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 AS17-C 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 6
Dilution of 50% (w/w) NaOH to Make
Standard AS17-C Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
1.20 (0.78)	15
8.00 (5.25)	100
160.04 (104.6)	2 M

4.6 Regenerant Preparation for the AMMS® III

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

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 Section 5.3. “Production Test Chromatogram”) on optimized Ion Chromatographs (see Section 3. “Installation”). 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 AS17-C is designed for the determination of inorganic anions in less than ten minutes using a hydroxide gradient delivered with an Eluent Generator. 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 only a resulting 1 to 2 μ S total baseline change.

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 effect the retention times of the anions being analyzed. Eluents should be maintained under an inert helium atmosphere to avoid carbonate contamination.

5.2 Production Test Chromatogram

To guarantee that all IonPac AS17-C 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. Phosphate is not used in this quality assurance test. Optimized operating conditions for analysis of common inorganic anions, including phosphate, are given in Sections 5.5. and 5.6.

Sample Volume:	2 mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume 4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	See Chromatogram
Eluent:	15 mM KOH
Eluent Flow Rate:	0.25 mL/min (2 mm), 1.0 mL/min (4 mm)
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (2 mm or 4 mm), AutoSuppression® Recycle Mode, 50mA
SRS Optimal Current Settings or MMS Suppressor:	38 mA (4 mm), 10 mA (2 mm)
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (2 mm or 4 mm)
Expected Background	50 mN H ₂ SO ₄
Conductivity:	≤ 2 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

Analyte	mg/L (ppm)
1. Fluoride	0.5
2. Chloride	1.0
3. Nitrite	1.0
4. Bromide	3.0
5. Nitrate	3.0
6. Sulfate	3.0

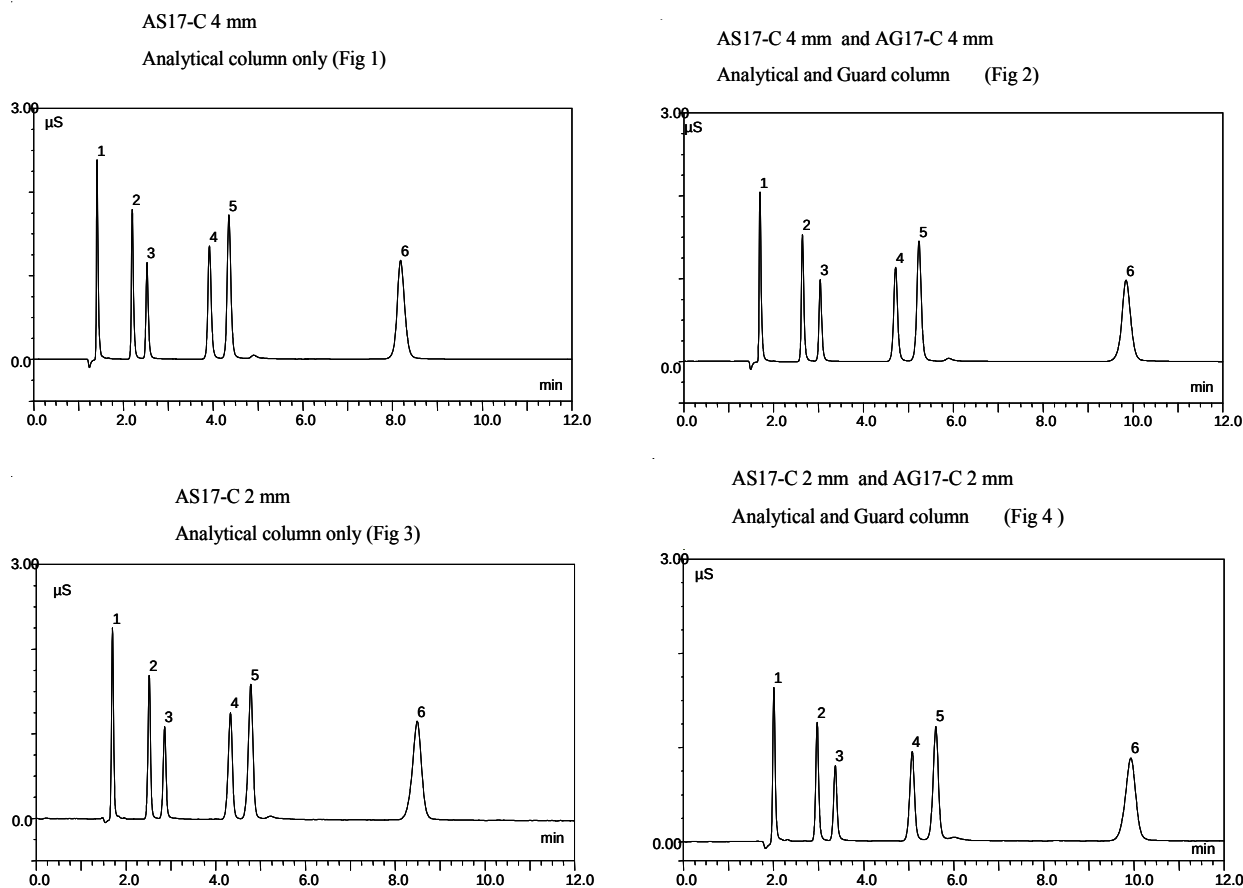


Figure 1
IonPac AS17-C Production Test Chromatograms

5.3 Production Test Chromatograms at Room Temperature

The quality assurance test for the IonPac AS17-C analytical column has been optimized at 30°C. However, the column can be operated at room temperature. Notice at room temperature the divalent ion (sulfate) has a shorter retention time with 15 mM KOH. Phosphate is not used in this quality assurance test. Optimized operating conditions for analysis of common inorganic anions, including phosphate, are given in Sections 5.5. and 5.6.

Sample Volume:	4-mm: 10 μ L Loop + 0.8 μ L Injection valve dead volume
Column:	IonPac AG17-C, AS17-C 4 mm
Eluent:	15 mM KOH
Eluent Flow Rate:	1.0 mL/min
Temperature:	See Chromatogram
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode, 50mA
SRS Optimal Current Settings or MMS Suppressor:	38 mA (4 mm)
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)
MMS Regenerant:	50 mN H ₂ SO ₄
Expected Background	
Conductivity:	$\leq 2 \mu$ S
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	0.5
2.	Chloride	1.0
3.	Nitrite	1.0
4.	Bromide	3.0
5.	Nitrate	3.0
6.	Sulfate	3.0

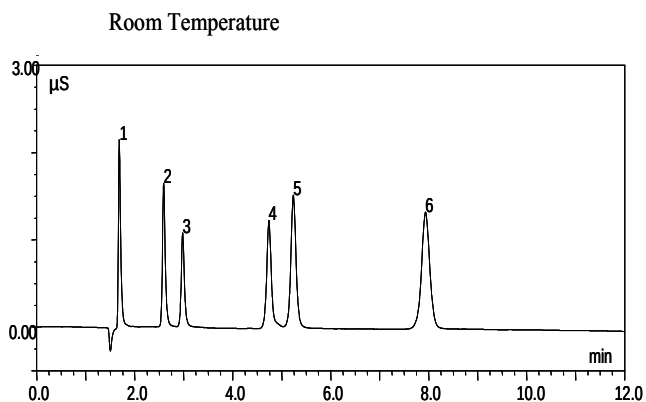
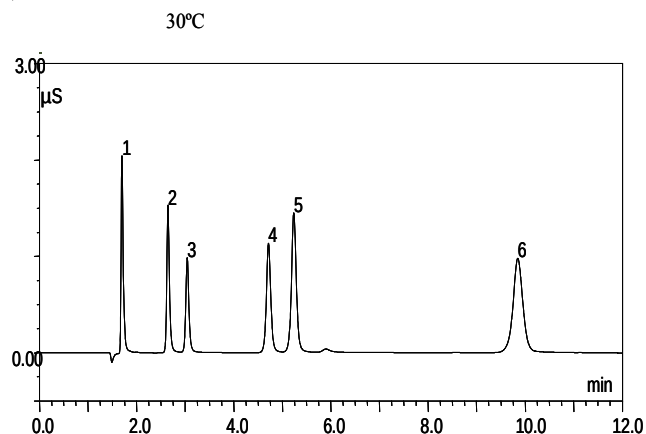


Figure 2
IonPac AS17-C Production Test Chromatograms at Room Temperature

5.4 Analysis of Common Inorganic Anions Using Eluent Generator Delivery System

The IonPac AS17-C analytical column in combination with the AG17-C guard column is designed for the analysis of inorganic anions including fluoride, chloride, nitrite, bromide, nitrate, sulfate, and phosphate in less than ten minutes using a hydroxide gradient delivered with an Eluent Generator. The following chromatograms illustrate the analysis of common inorganic anions using the EG eluent generator eluent delivery system at 30°C and at room temperature. Notice at room temperature a lower eluent concentration is required to achieve similar separation and retention times.

Trap Column:	CR-ATC is installed after the EGC-KOH Cartridge
Sample Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	IonPac® AG17-C, AS17-C 4 mm
Eluent Source:	Eluent Generator
Eluent:	See Table
Eluent Flow Rate:	2.0 mL/min (4-mm)
Temperature:	See Chromatogram
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode
SRS Optimal Current Settings or MMS Suppressor:	198 mA (4 mm)
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)
Expected Background	50 mN H ₂ SO ₄
Conductivity:	≤ 1.0 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

Analyte	mg/L (ppm)
1. Fluoride	2.0
2. Acetate	5.0
3. Chloride	3.0
4. Nitrite	5.0
5. Bromide	10.0
6. Nitrate	10.0
7. Carbonate	—
8. Sulfate	5.0
9. Phosphate	10.0

EG eluent generator gradient for 30°C

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 2 min
2.0	1.0	
Analysis		
2.1	1.0	Start isocratic analysis
2.5	1.0	Inject valve to load position
3.5	1.0	Begin gradient analysis
7.0	20	
9.0	40	



EG eluent generator gradient for room temperature

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 2 min
2.0	1.0	
Analysis		
2.1	1.0	Start isocratic analysis
2.5	1.0	Inject valve to load position
3.5	1.0	Begin gradient analysis
7.0	15	
9.0	40	

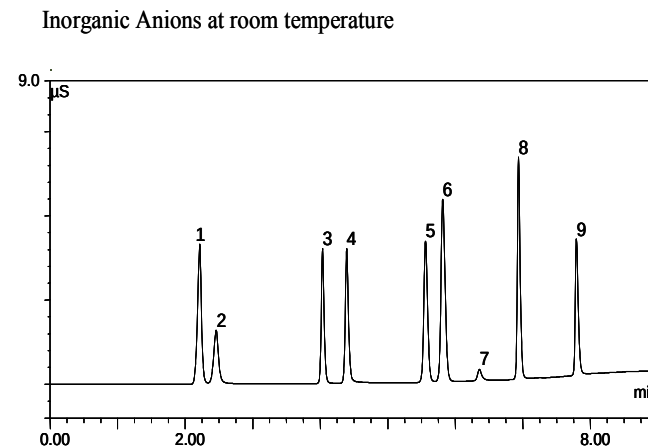


Figure 3
Analysis of Common Inorganic Anions using EG Eluent Delivery System

5.5 Gradient Analysis of Common Inorganic Anions at Room Temperature

The following chromatograms compare the use of the Eluent Generator delivery system vs. the bottle eluent delivery system for the determination of common inorganic anions at room temperature. Notice that with the manually prepared eluents, longer equilibration time is required before injection.

Trap Column:	Bottle Eluent System, ATC-3 located after pump		
	EG Eluent System, CR-ATC is installed after the EGC-KOH Cartridge		
Sample Volume:	4 mm: 10 μ L Loop + 0.8 μ L Injection valve dead volume		
Column:	IonPac® AG17-C, AS17-C 4 mm		
Eluent Source	See table		
Eluent:	See table		
Eluent Flow Rate:	2.0 mL/min (4 mm)		
Temperature:	Room temperature		
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode		
SRS Optimal Current Settings or MMS Suppressor:	198 mA (4 mm)		
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)		
Expected Background	50 mN H ₂ SO ₄		
Conductivity:	≤ 1.0 μ S		
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate		
Short-term Storage Solution (< 1 week):	Eluent		

Analyte	mg/L (ppm)
1. Fluoride	2.0
2. Acetate	5.0
3. Chloride	3.0
4. Nitrite	5.0
5. Bromide	10.0
6. Nitrate	10.0
7. Carbonate	—
8. Sulfate	5.0
9. Phosphate	10.0

EG Gradient

Eluent: Deionized water

Offset volume = 0.0 μ L

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 2 min
2.0	1.0	
Analysis		
2.1	1.0	Start isocratic analysis
2.5	1.0	Inject valve to load position
3.5	1.0	Begin gradient analysis
7.0	15	
9.0	40	End gradient

Manually Prepared Eluent Gradient Conditions

E1: 5 mM NaOH
E2: DI water
E3: 100 mM NaOH

TIME (min)	%E1	%E2	%E3	Comments
Equilibration				
0	20	80	0	1.0 mM NaOH for 3 min
3.0	20	80	0	
Analysis				
3.1	20	80	0	Start isocratic analysis
3.5	20	80	0	Inject valve to load position
4.0	20	80	0	Begin gradient analysis
7.5	0	85	15	
9.5	0	60	40	End gradient

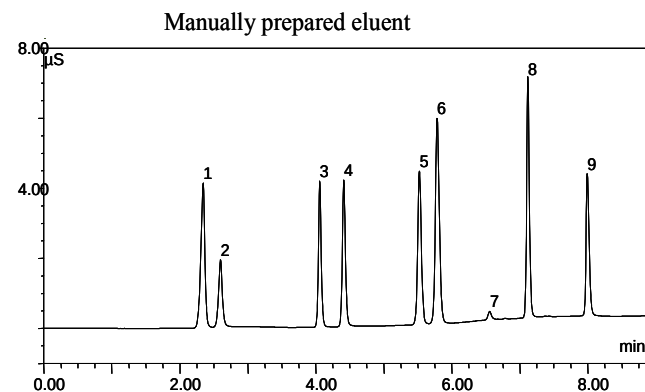
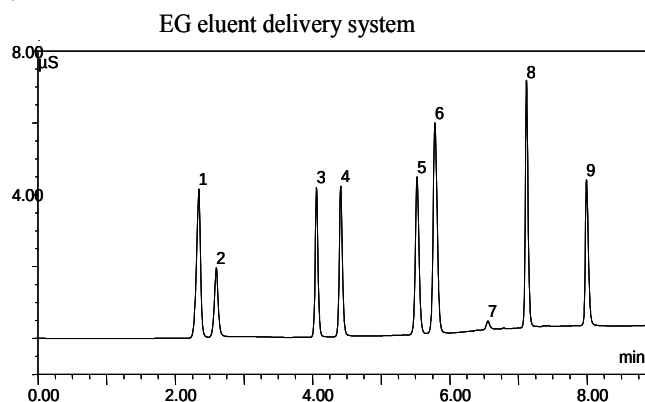


Figure 4
Analysis of Common Inorganic Anions at Room Temperature

5.6 Determination of Inorganic Anions, Oxyhalides, and Organic Acids Using EGC Eluent Delivery System

The following chromatograms show the separation of a large number of inorganic anions, oxyhalides, and organic acids on the IonPac AS17-C using a hydroxide gradient delivered with the Eluent Generator. The following chromatograms demonstrate the effect of temperature on the separation. Notice at 30°C, the resolution of fluoride/acetate and bromate/chloride is improved.

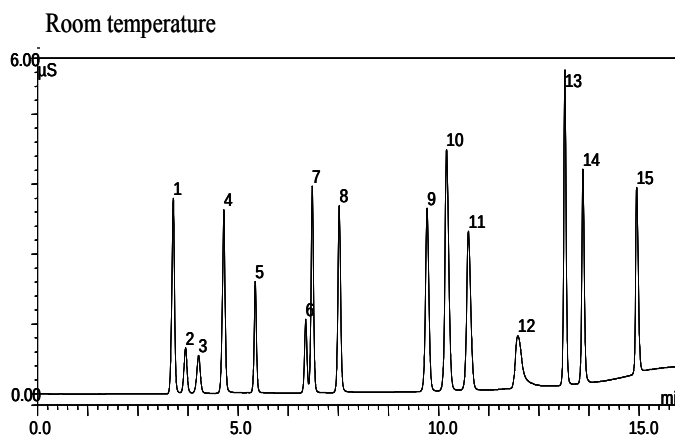
Trap Column:	Bottle Eluent System, ATC-3 located after pump		
	EG Eluent System, CR-ATC is installed after the EGC-KOH cartridge		
Sample Volume:	4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume		
Column:	IonPac® AG17-C, AS17-C 4 mm		
Eluent Source	See table	Analyte	mg/L(ppm)
Eluent:	See table	1. Fluoride	2.0
Eluent Flow Rate:	1.5 mL/min (4-mm)	2. Acetate	5.0
Temperature:	Room temperature	3. Propionate	5.0
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode	4. Formate	5.0
SRS Optimal Current Settings	130 mA	5. Chlorite	5.0
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)	6. Bromate	5.0
MMS Regenerant:	50 mN H ₂ SO ₄	7. Chloride	3.0
Expected Background		8. Nitrite	5.0
Conductivity:	≤ 1.0 µS	9. Bromide	10.0
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate	10. Nitrate	10.0
Short-term Storage Solution (< 1 week):	Eluent	11. Chlorate	10.0
		12. Carbonate	20.0
		13. Sulfate	5.0
		14. Oxalate	5.0
		15. Phosphate	10.0

EG eluent generator gradient at room temperature

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 4 min
4.0	1.0	
Analysis		
4.1	1.0	Start isocratic analysis
4.5	1.0	Inject valve to load position
7.0	1.0	Begin gradient analysis
14.0	10.0	
18.0	35.0	End gradient



EG eluent generator gradient at 30 °C

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 4 min
4.0	1.0	
Analysis		
4.1	1.0	Start isocratic analysis
4.5	1.0	Inject valve to load position
7.0	1.0	Begin gradient analysis
14.0	10.0	
18.0	35.0	End gradient

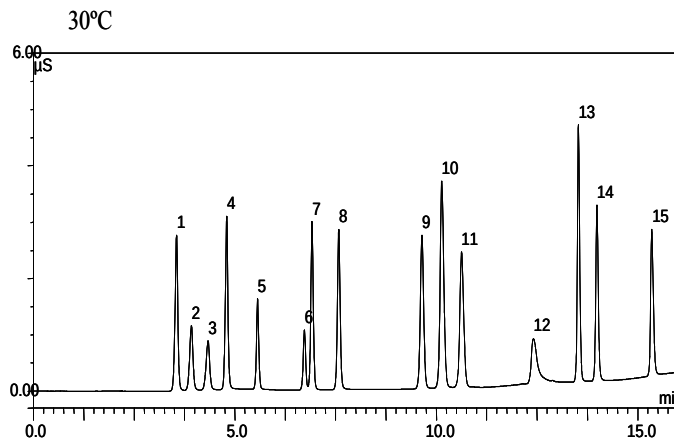


Figure 5
Determination of Inorganic Anions, Oxyhalides, and Organic Acids
using Eluent Generator Delivery System

5.7 Analysis of a High Purity Water Spiked with Anions Using the AS17-C Column

The following chromatogram illustrates the analysis of a simulated high purity sample spiked with anions using the AS17-C column with a KOH gradient.

Trap Column:	CR-ATC is installed after the EGC-KOH cartridge
Sample Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	IonPac AG17-C, AS17-C 4 mm
Eluent:	See table
Eluent Flow Rate:	1.5 mL/min (4 mm)
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode
SRS Optimal Current Settings or MMS Suppressor:	130 mA
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)
Expected Background	50 mN H ₂ SO ₄
Conductivity:	≤ 1.0 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

Eluent: Deionized water
Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments	Analyte	µg/L (ppb)
Equilibration			1. Fluoride	1
0	1.0	1.0 mM KOH for 4 min	2. Acetate	10
4.0	1.0		3. Formate	10
Analysis			4. Acrylate	10
4.1	1.0	Start isocratic analysis	5. Methacrylate	10
4.5	1.0	Inject valve to load position	6. Chloride	5
7.0	1.0	Begin gradient analysis	7. Nitrite	5
14.0	12.0		8. Bromide	5
18.0	35.0	End gradient	9. Nitrate	5
			10. Benzoate	20
			11. Carbonate	—
			12. Sulfate	5
			13. Oxalate	10
			14. Phthalate	10
			15. Phosphate	10

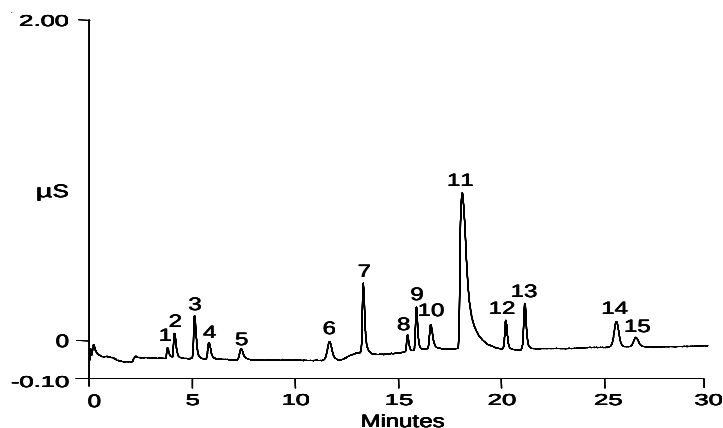


Figure 6
Analysis of Simulated Drinking Water Sample

5.8 Analysis of Municipal and Bottled Drinking Water Samples Using AS17-C

The following chromatograms illustrate the analysis of a municipal drinking water sample and a bottled drinking water sample using the AS17-C column with a KOH gradient.

Trap Column:	CR-ATC is installed after the ECG-KOH cartridge	Analyte 1. Fluoride 2. Chlorite 3. Chloride 4. Nitrite 5. Bromide 6. Nitrate 7. Chlorate 8. Carbonate 9. Sulfate 10. Oxalate 11. Phosphate
Sample Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume	
Column:	IonPac AG17-C, AS17-C 4-mm	
Eluent:	See table	
Eluent Flow Rate:	2 mL/min (4 mm)	
Temperature:	30°C	
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode	
SRS Optimal Current Settings or MMS Suppressor:	198 mA	
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)	
MMS Regenerant:	50 mN H ₂ SO ₄	
Expected Background		
Conductivity:	≤ 1.0 µS	
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate	
Short-term Storage Solution (< 1 week):	Eluent	

EG eluent generator gradient at 30°C

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 2 min
2.0	1.0	
Analysis		
2.1	1.0	Start isocratic analysis
2.5	1.0	Inject valve to load position
3.5	1.0	Begin gradient analysis
7.0	20	
9.0	40	End gradient

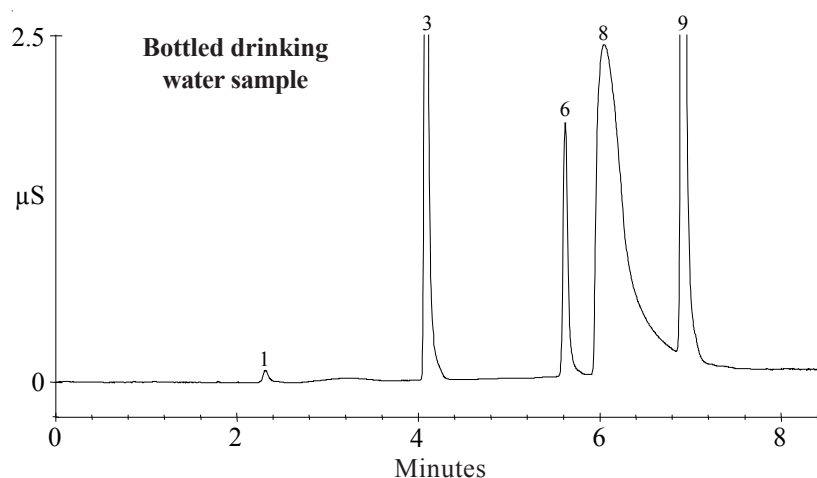
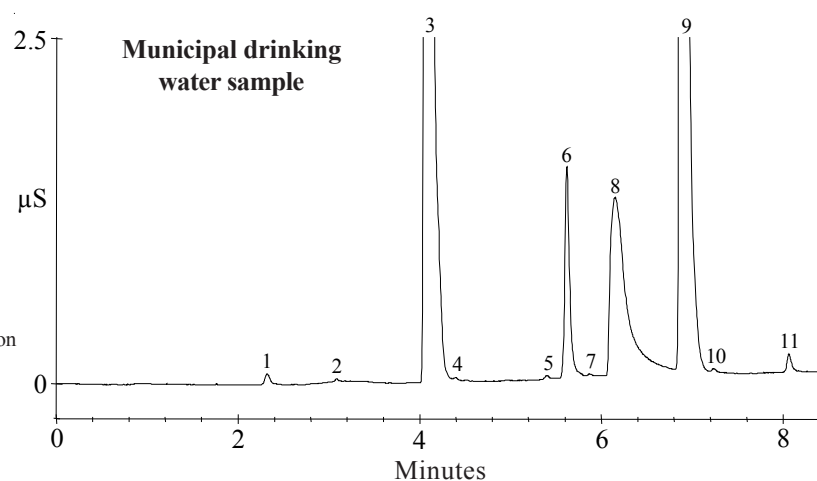


Figure 7
Analysis of Municipal and Bottled Drinking Water Sample

5.9 Determination of Sulfur Species in a Simulated Industrial Waste Water Sample

Figure 8 illustrates the separation of sulfur species, organic acids, and inorganic anions found in a simulated industrial waste water sample on the IonPac AS17-C using a hydroxide gradient delivered with the Eluent Generator. The simulated industrial waste sample was diluted 1:100 with deionized water and filtered through a 0.45 µm syringe filter. Notice the separation of sulfite, sulfate, and thiosulfate within ten minutes. A controlled temperature of 30°C is used in order to ensure reproducible retention times.

Trap Column:	CR-ATC is installed after the EGC-KOH cartridge
Sample Volume:	4 mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	IonPac® AG17-C, AS17-C 4 mm
Eluent Source	Eluent Generator
Eluent:	See Table
Eluent Flow Rate:	2.0 mL/min (4 mm)
Temperature:	30°C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm), AutoSuppression® Recycle Mode
SRS Optimal Current Settings or MMS Suppressor:	198 mA
MMS Regenerant:	Anion MicroMembrane Suppressor, AMMS® III (4 mm)
Expected Background	50 mN H ₂ SO ₄
Conductivity:	≤ 1.0 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

EG eluent generator gradient at 30°C

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments	Analyte	mg/L (ppm)
Equilibration			1. Acetate	2.0
0	1.0	1.0 mM KOH for 2 min	2. Glycolate	2.0
2.0	1.0		3. Formate	2.0
Analysis			4. Chloride	20.0
2.1	1.0	Start isocratic analysis	5. Nitrate	1.0
2.5	1.0	Inject valve to load position	6. Carbonate	50.0
3.5	1.0	Begin gradient analysis	7. Sulfite	4.0
7.0	20		8. Sulfate	40.0
9.0	40	End gradient	9. Thiosulfate	12.0

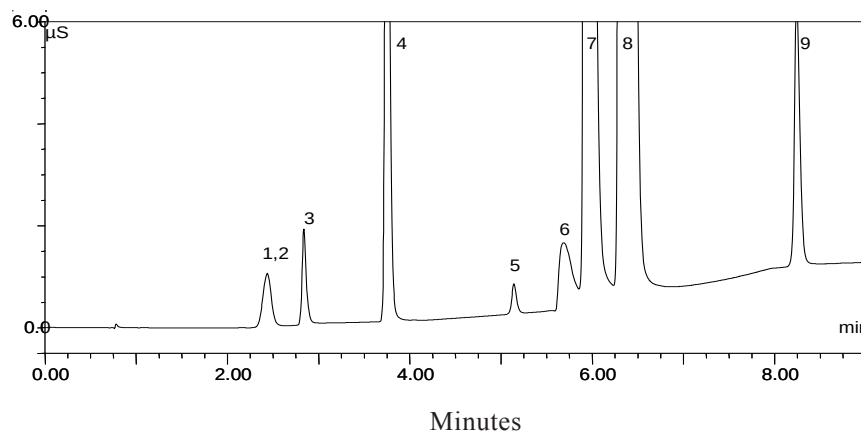


Figure 8
Determination of Sulfite Species in a Simulated Industrial Waste Water Sample

5.10 Separation of Anionic Additives in Toothpaste

The excellent retention of fluoride on the IonPac AS17-C makes it ideal for the determination of fluoride and monofluorophosphate in dental care products. Figure 9 illustrates the separation of organic acids and inorganic anions found in dental care products. Notice, due to the high sensitivity offered by hydroxide eluent system due to lower background, very low levels of organic acids can be determined along with good separation of benzoate, a preservative used in dental care products. The toothpaste sample was prepared by dissolving 0.5 g toothpaste in 100 g deionized water and filtered through a 0.45 µm syringe filter before injecting on to an AS17-C column.

Trap Column: CR-ATC is installed after the EGC-KOH cartridge
 Sample Volume: 10 µL
 Column: IonPac® AG17-C, AS17-C 4 mm
 Eluent Source: Eluent Generator
 Eluent: See Table
 Eluent Flow Rate: 1.5 mL/min (4 mm)
 Temperature: 30°C
 SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS® ULTRA II (4 mm),
 AutoSuppression® Recycle Mode
 SRS Optimal Current Settings or MMS Suppressor: 130 mA
 Anion MicroMembrane Suppressor, AMMS® III (4 mm)
 MMS Regenerant: 50 mN H₂SO₄
 Expected Background
 Conductivity: ≤ 1.0 µS
 Long-term Storage Solution (> 1 week): 100 mM Sodium Borate
 Short-term Storage Solution (< 1 week): Eluent

Analyte

1. Fluoride
2. Acetate
3. Formate
4. Chloride
5. Nitrite
6. Bromide
7. Benzoate
8. Carbonate
9. Monofluorophosphate
10. Sulfate
11. Phosphate

EG eluent generator gradient at 30°C

Eluent: Deionized water

Offset volume = 0.0 µL

Time (min)	Eluent Conc. (mM)	Comments
Equilibration		
0	1.0	1.0 mM KOH for 4 min
4.0	1.0	
Analysis		
4.1	1.0	Start isocratic analysis
4.5	1.0	Inject valve to load position
7.0	1.0	Begin gradient analysis
14.0	12.0	
18.0	35.0	End gradient

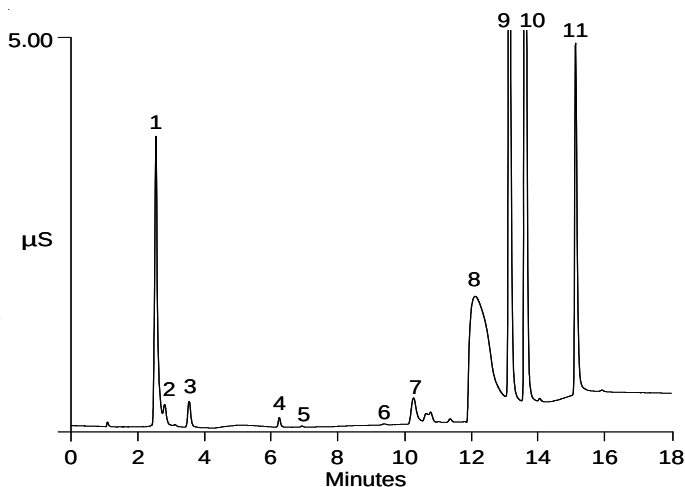


Figure 9
Separation of Anionic Additives in toothpaste

5.11 Clean-up After Humic Acid Samples

Solvent compatibility of the IonPac AS17-C permits the use of organic solvents to effectively remove organic contaminants from the column. An AS17-C column, after losing over 29% of its original capacity due to fouling with humic acid samples, can easily be restored to 100% of its original performance by cleaning for 3 hours with 80% tetrahydrofuran (THF)/20% 1.0 M HCl.

Sample Loop: 10 μ L
Column: IonPac AS17-C 4 mm
Eluent: 15 mM KOH
Eluent Flow Rate: 1.0 mL/min
Operating Temp: 30°C
SRS Suppressor: Anion Self-Regenerating Suppressor, ASRS® ULTRA II
(4 mm), AutoSuppression® Recycle Mode
SRS Optimal Current Setting 38 mA (4 mm)
or MMS Suppressor: Anion MicroMembrane Suppressor, AMMS® III (4 mm)
MMS Regenerant: 50 mN H₂SO₄
Expected Background
Conductivity: ≤ 1.0 μ S
Long-term Storage Solution (> 1 week): 100 mM Sodium Borate
Short-term Storage Solution (< 1 week): Eluent

Analyte	mg/L (ppm)
1. Fluoride	0.5
2. Chloride	1.0
3. Nitrite	1.0
4. Bromide	3.0
5. Nitrate	3.0
6. Sulfate	3.0

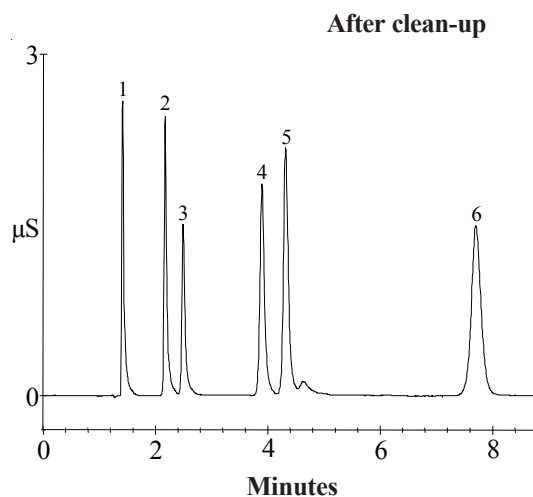
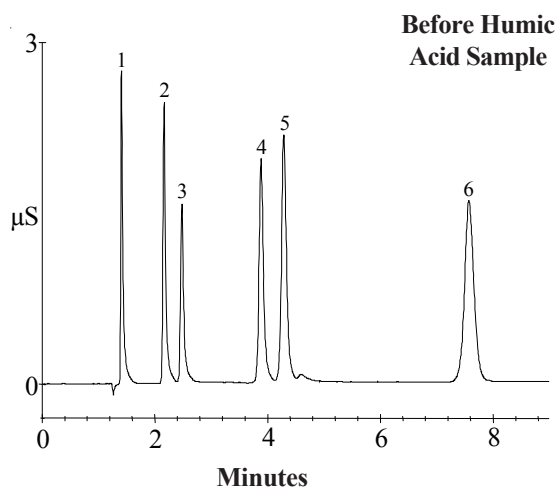
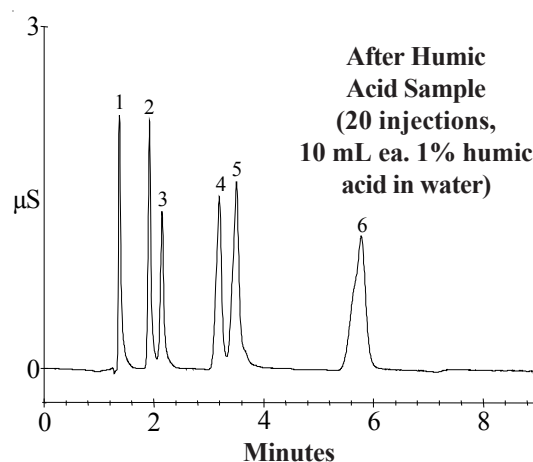


Figure 10
Clean-up after Humic Acid Samples

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS17-C 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
AS17-C / AG17-C Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1.
	Plugged Column	Replace Bed Supports	6.1.2.
	Bed Supports	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® ULTRA II or AMMS® III	Clean Suppressor	6.2.5., Component Manual
	Contaminated Hardware	Clean Component	6.2.4., Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.B, Component Manual
	Column Headspace	Replace Column	6.3. A
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 AG17-C (4 mm) Guard Column plus the AS17-C (4 mm) Analytical Column when using the test chromatogram conditions should be equal or less than 1,400 psi. Note: when using EG there is added pressure from other components. If the system pressure is higher than 1,000 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.

- 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 AS17-C / AG17-C Operating Back Pressures").

The Anion Self-Regenerating Suppressor ASRS® ULTRA II 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 AS17-C / AG17-C Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
AS17-C 4 mm Analytical	≤ 1200 (5.17)	1.0
AG17-C 4 mm Guard	≤ 200 (1.03)	1.0
AS17-C + AG17-C 4 mm columns	≤ 1400 (6.20)	1.0
AS17-C 2 mm Analytical	≤ 1200 (5.17)	0.25
AG17-C 2 mm Guard	≤ 200 (1.03)	0.25
AS17-C + AG17-C 2 mm columns	≤ 1400 (6.20)	0.25

6.1.2 Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high 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.

	IonPac AS17-C 4 mm Columns (P/N)	IonPac AS17-C 2 mm Columns (P/N)
Analytical Column	066294	066296
Guard Column	066295	066297
Bed Support Assembly	042955	044689
End Fitting	052809	043278



CAUTION

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

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



CAUTION

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:

<u>ELUENT</u>	<u>EXPECTED BACKGROUND CONDUCTIVITY</u>
15 mM NaOH	≤ 2 µS
15 mM KOH (EG prepared eluents) *refer to section 4.5.	≤ 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 Anion 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

Install a CR-TC Anion Trap Column (P/N 060477) if using an Eluent Generator with EGC II 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 AG17-C Guard and AS17-C Analytical Columns from the system. Install a backpressure 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 AG17-C 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 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® ULTRA II or AMMS® III Suppressor

If the above items have been checked and the problem persists, the Anion Self-Regenerating 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, ASRS® ULTRA II Product Manual (Document No. 031956). For details on Anion Membrane Suppress, AMMS III operation, refer to the Product Manual (Document No. 031727) 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® 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.

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.
- 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) 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.
- 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 AS17-C 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.

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

APPENDIX A

COLUMN CARE

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 AS17-C columns is 4,000 psi (27.57 MPa).

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.

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.

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 $\leq 5\%$ levels and the ionic strength of the eluent to ≤ 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

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 valency 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 AS17-C 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 AG17-C and AS17-C.**

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® ULTRA II or AMMS®III** from the IonPac AS17-C Analytical Column. If your system is configured with both a guard column and an analytical column, reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.



CAUTION

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

- C. **Set the pump flow rate to 1.0 mL/min for an AS17-C 4 mm Analytical or Guard Column** or set the pump flow rate to 0.25 mL/min for an AS17-C 2 mm Analytical or 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® ULTRA II or AMMS®III** to the AS17-C Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

Appendix B

CONFIGURATION	2 mm	4 mm
Eluent Flow Rate	0.30 mL/min	1.20 mL/min
SRS Suppressor	ASRS ULTRA II (2 mm) (P/N 061562)	ASRS ULTRA II (4 mm) (P/N 061561)
MMS Suppressor	AMMS III (2 mm) (P/N 056751)	AMMS III (4 mm) (P/N 056750)
AAE Suppressor	AAES (P/N 056116)	AAES (P/N 056116)
Injection Loop	2 - 15 μ L	10-50 μ L
	Rheodyne Microinjection Valve (P/N 044697) for full loop injections <15 μ L.	
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 volume. Switching valves, couplers can be used. Use the GM-2, GM-3 or recommended gradient mixers.
Pumps	Use the DP/SP/GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2 mm) Gradient Mixer. The GPM-2 can be used for 2 mm isocratic chromatography at flow rates of 0.5 mL/min or greater but cannot be used for 2 mm gradient chromatography.	Use the DP/SP/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. Note: The GP40 has an active mixer.
Detectors	AD20 Cell (6 mm, 7.5 μ L, P/N 046423) VDM-2 Cell (3 mm, 2.0 μ L) (P/N 043120) DC/CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 (P/N 044130) or Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-2 (P/N 043117) is optimized for 2 mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi	AD25 Cell (10 mm, 9 μ L, P/N 049393) VDM-2 Cell (6 mm, 10 μ L) (P/N 043113) DC/CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 (P/N 044130) or Conductivity Cell with Shield (P/N 044132) CDM-2/CDM-3 Cell (P/N 042770) Do not use the TS-1 or TS-2 with ED40/ED50/ED50A or CD20/CD25/CD25A. The TS-1 or TS-2 (P/N 043117) is optimized for 4 mm operation on CDM-2 or CDM-3. Recommended back pressure: 30–40 psi

Table 3
Tubing Back Pressures

Color	Dionex P/N	ID Inches	ID cm	Volume mL/cm	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	4.560	0.086	0.021	0.003
Orange	042855	0.020	0.051	2.027	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.856	2.437	0.609	0.081
Black	042690	0.010	0.025	0.507	6.960	1.740	0.232
Red	044221	0.005	0.013	0.127	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.046	859.259	214.815	28.642