# Errata

***Product Manual for Dionex IonPac™ AS17 and AG17 Columns***
*031529-02*

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* |

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# PRODUCT MANUAL

## IONPAC® AG17 GUARD COLUMN

**(4 x 50 mm, P/N 055684)**
**(2 x 50 mm, P/N 055685)**

## IONPAC® AS17 ANALYTICAL COLUMN

**(4 x 250 mm, P/N 055682)**
**(2 x 250 mm, P/N 055683)**

**QUICKSTART STEPS AND LINKS**
**Click blue text below to get started.**

1. See Section 4, “Operation”. Note operation precautions and chemical purity requirements. Make the required eluents.

2. See “Quality Assurance Reports”. Run the Production Test Chromatogram as a system check.

3. See Section 5, "Example Applications" for example applications.

4. See “Column Care" for column cleanup and long-term storage recommendations.


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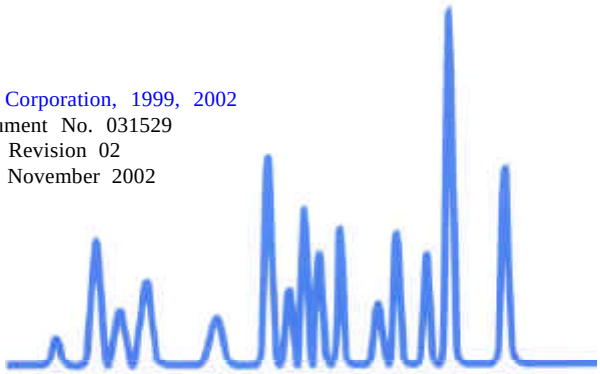

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# SECTION 1 - INTRODUCTION TO IONPAC AS17/AG17 CHROMATOGRAPHY

The IonPac® AS17 Analytical Column in combination with the AG17 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 EG40 Eluent Generator. The selectivity of the IonPac AS17 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 drinking water, ground water, waste water, process streams, and scrubber solutions. The AS17 is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS17 can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor. The IonPac AS17 has nominal efficiency of at least 28,000 plates/meter for sulfate using standard operating conditions.

**Table 1**
**IonPac AS17/AG17 Packing Specifications**

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

| Column | Typical Back Pressure psi (MPa) | Standard Flow Rate mL/min | Maximum Flow Rate mL/min |
|---|---|---|---|
| AS17 4-mm Analytical | < 750 (5.17) | 1.0 | 3.0 |
| AG17 4-mm Guard | < 150 (1.03) | 1.0 | 3.0 |
| **AS17 + AG17 4-mm columns** | **< 900 (6.20)** | **1.0** | **3.0** |
| AS17 2-mm Analytical | < 750 (5.17) | 0.25 | 0.75 |
| AG17 2-mm Guard | < 150 (103) | 0.25 | 0.75 |
| **AS17 + AG17 2-mm columns** | **<900 (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 - COMPARISON OF ION CHROMATOGRAPHY SYSTEMS

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

| CONFIGURATION | 2-mm | 4-mm |
|---|---|---|
| **Eluent Flow Rate** | 0.25 | 1.0 |
| **SRS** | ASRS-ULTRA (2-mm) (P/N 053947) | ASRS-ULTRA (4-mm) (P/N 053946) |
| **MMS** | AMMS III (2-mm) (P/N 056751) | AMMS III (4-mm) (P/N 056750) |
| **NOTE** Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven. | | |
| **Injection Loop** | 2 - 15 µL<br>Use the Rheodyne Microinjection Valve, Model No. 9126 DIONEX P/N 044697) for full loop injections <15 µL. | 10-50 µL |
| **System Void Volume** | Eliminate switching valves, couplers and the GM-3 Gradient Mixer. Use only the 2-mm GM-3 Mixer (P/N 043149). | Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2 , GM-3 or recommended gradient mixers. |
| **Pumps** | Use the GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer.<br><br>No External Gradient Mixer is required for GS50/GP50/GP40 Pump when performing gradient analysis.<br><br>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.<br><br>The GM-3 Gradient Mixer should be used for gradient analysis on systems other than the GP40/GP50/IP20/IP25 and the DX-300 HPLC Pump. |

| CONFIGURATION | 2-mm | 4-mm |
|---|---|---|
| **Detectors** | AD20/AD25 Cell<br>(6-mm, 7.5 µL, P/N 046423)<br><br>VDM-2 Cell<br>(3-mm, 2.0 µL, P/N 043120)<br><br>CD20, CD25, CD25A, ED40, ED50, or ED50A<br>Conductivity Cell with DS3<br>P/N 044130 or<br>Conductivity Cell with shield<br>P/N 044132<br><br>CDM-2/CDM-3 Cell<br>P/N 042770<br>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.<br><br>DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3)<br>Ensure 50-75 psi back pressure. | AD20/AD25 Cell<br>(10-mm, 9 µL, P/N 049393)<br><br>VDM-2 Cell (6-mm, 10 µL)<br>P/N 043113<br><br>CD20, CD25, CD25A, ED40, ED50, or ED50A<br>Conductivity Cell with DS3<br>P/N 044130 or with shield<br>P/N 044132<br><br>CDM-2/CDM-3 Cell<br>P/N 042770<br>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.<br><br>DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3)<br>Ensure 50-75 psi back pressure. |

**Table 3**
**Tubing Back Pressures**

| Tubing ID<br>in | $H_20$ Back Pressure<br>Psi/ft at 1 mL/min |
|---|---|
| 0.005 | 111.4 |
| 0.007 | 29.0 |
| 0.010 | 7.0 |
| 0.012 | 3.4 |

# SECTION 3 - INSTALLATION

## 3.1 System Requirements

### 3.1.1 System Requirements for 2-mm Operation

The IonPac AS17 2-mm Guard and Analytical Columns are designed to be run on the following 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 Gradient Pump Module (GPM-2) an Advanced Gradient Pump (AGP) or a GP40/GP50/GS50 with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a Microbore Advanced Gradient Pump or a microbore IP20/IP25 (1/16" pistons) must be employed.

### 3.1.2 System Requirements for 4-mm Operation

The IonPac AS17 4-mm Guard and Analytical Columns are designed to be 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 Gradient Pump Module (GPM-2) or an Advanced Gradient Pump (AGP) or a GP40/GP50/GS50 with standard 1/8" pump heads. Isocratic analysis can also be performed on an IP20/IP25 with standard bore.

### 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" (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. If you need assistance in properly configuring your system 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”).

## 3.2 The Sample Concentrator

The Low Pressure Trace Anion Concentrator Column (TAC-LP1, P/N 046026) or the IonPac AG17 Guard Column can be used for trace anion concentration work. The function of the TAC-LP1 or the AG17 Guard Column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process “concentrates” all anionic analyte species onto the TAC-LP1 or the AG17 leading to a lowering of 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. The unique advantage to the analytical chemist of the TAC-LP1 or the AG17 in these applications is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

For more detailed information on sample concentration techniques for high sensitivity work refer to Section 3, “Operation,” of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column Product Manual (Document No. 034972). These techniques can also be applied to the AG17.

**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. Use the AG17 4-mm or AG17 2-mm guards.**

## 3.3 The Injection Loop

**Table 4**
**Smallest Injectable Volumes (µL)**

| Valve Type | Using 0.012" ID Tefzel Tubing | Using 0.007" ID Tefzel Tubing | Using 0.010" ID PEEK Tubing | Using 0.005" ID PEEK Tubing |
|---|---|---|---|---|
| DIONEX BF2 Valve (8 µL Internal Volume) (10 cm Loop) | 15.2 | 10.5 | 13.1 | 9.2 |
| DIONEX MicroInject Valve (10.5 µL Internal Volume) (14 cm Loop) | 20.5 | 14.0 | 17.6 | 12.2 |
| Rheodyne Microinjection Valve Model 9126 (0.8 µL Internal Volume) (10 cm Loop) | 8.0 | 3.3 | 5.9 | 2.0 |

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

For most applications on a 2-mm analytical system, a 2 - 15 µL injection loop is sufficient. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2-mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. The AS17 2-mm requires a microbore HPLC system configuration. Install an injection loop one-fourth or less (<15 µL) of the loop volume used with a 4-mm analytical system (Section 2, “Comparison of 2-mm and 4-mm Ion Chromatography Systems”).

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

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop is sufficient. Generally, you should not inject more than 50 nanomoles of any one analyte onto the 4-mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity. For typical low to sub ppb samples, you can inject up to 2-4 mL.

## 3.4 The IonPac AG17 Guard Column

An IonPac AG17 Guard Column is normally used with the IonPac AS17 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 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS17 Analytical Column.

## 3.5 The Anion Trap Column, ATC-3

**Gradient applications require an IonPac Anion Trap Column.** Order the ATC-3 (P/N 059660) for 4-mm systems or the ATC-3 (2-mm) (P/N 059661) for 2-mm systems. It should be installed in place of the high pressure gradient mixer between the gradient pump and the injection valve. The ATC is filled with high capacity anion exchange resin which helps to minimize the baseline shift caused by increasing anionic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis.

To install the ATC-3 (4-mm) or ATC-3 (2-mm), complete the following steps:

**A. Remove the Gradient Mixer** installed between the gradient pump pressure transducer and the injection valve.

**B. Connect the gradient pump directly to the ATC-3**. Connect a waste line to the ATC-3 outlet and direct the line to a waste container.

**C. Flush the ATC-3 with 100 mL of 2.0 M NaOH through the 4-mm ATC-3 Column or 50 mL for the 2-mm ATC-3 Column.**

**D. Pump 20 mL of eluent through the 4-mm ATC-3 or 10 mL for the 2-mm ATC-3 Column**.

**E. Reconnect the ATC-3 after flushing it with eluent.** Connect the ATC-3 to the eluent line that is connected to the injection valve.

The background conductivity of your system should be between 1.5 µS and 2.5 µS when 0.75 mM NaOH is being pumped through the chromatographic system. The baseline shift should be no greater than 5 µS during a gradient eluent concentration ramp from 0 to 80 mM NaOH. If the baseline shifts are greater than 5 µS, the ATC-3 should be cleaned using steps B - E above.

**At the end of each operating day**, the ATC-3 should be flushed to remove any impurities that may have accumulated on it.

Under normal operating conditions, the ATC-3 column should be regenerated at the end of each operational day to remove any contaminants that may have collected on it, including carbonate. The daily regeneration of the ATC-3 column ensures that the IC system is systematically equilibrated for the most reproducible determinations of those anions being eluted by the weak eluents.

See the conditioning procedure above for regeneration of ATC-3 columns. For detailed information refer to the ATC-3 Product Manual (Document No. 032697).

## 3.6 Eluent Storage

IonPac AS17 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 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 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 modes of operation.

**NOTE**

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

If you are installing an IonPac AS17 4-mm Analytical Column, use an ASRS-ULTRA (4-mm, P/N 053946).
If you are installing an IonPac AS17 2-mm Analytical Column, use an ASRS-ULTRA (2-mm, P/N 053947).

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, the ASRS-ULTRA."

## 3.8 Anion MicroMembrane Suppressor Requirements

An Anion MicroMembrane Suppressor (AMMS III) may be used instead of an ASRS-ULTRA (4-mm) for applications that require suppressed conductivity detection. Use an AMMS III 4-mm (P/N 056750) with the IonPac AS17 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 in the Chemical Suppression Mode or the AMMS

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 EG40 with AS17

Please refer to the EG40 manual, Document No. 031373, for information on the operation of the EG40 and the modules available for it.

# SECTION 4 - OPERATION

## 4.1 General Operating Conditions

| | |
|---|---|
| Sample Volume: | 2-mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume<br>4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | 2-mm: AS17 2-mm Analytical Column + AG17 2-mm Guard Column<br>4-mm: AS17 4-mm Analytical Column + AG17 4-mm Guard Column |
| Eluent: | 15 mM NaOH |
| Eluent Flow Rate: | 2-mm: 0.25 mL/min<br>4-mm: 1.0 mL/min |
| Temperature: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (2-mm or 4-mm)<br>AutoSuppression Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 2 µS |
| Storage Solution: | Eluent |

## 4.2 IonPac AS17 Operation Precautions

**CAUTIONS**
**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 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 columns is 4,000 psi (27.57 MPa).

The AS17 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 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) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents. Do not use > 40% solvent on the ASRS-ULTRA 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.**

**CAUTION**

**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")

For 15 mM: $\frac{0.015 \text{ mole/L} \times 40.01 \text{ g/mole}}{50\%}$ = 1.20 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")

For 15 mM: $\frac{0.015 \text{ mole/L} \times 40.01 \text{ g/mole}}{50\% \times 1.53 \text{ g/mL}}$ = 0.78 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 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 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 (ASRS-ULTRA), 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 is designed for the determination of inorganic anions in less than ten minutes using a hydroxide gradient delivered with an EG40 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 µS 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 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<br>4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | See Chromatogram |
| Eluent: | 15 mM NaOH |
| Eluent Flow Rate: | 0.25 mL/min (2-mm), 1.0 mL/min (4-mm) |
| Temperature: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (2-mm or 4-mm)<br>AutoSuppression® Recycle Mode (300 mA) |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (2-mm or 4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 2 µS |
| Storage Solution: | 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 |

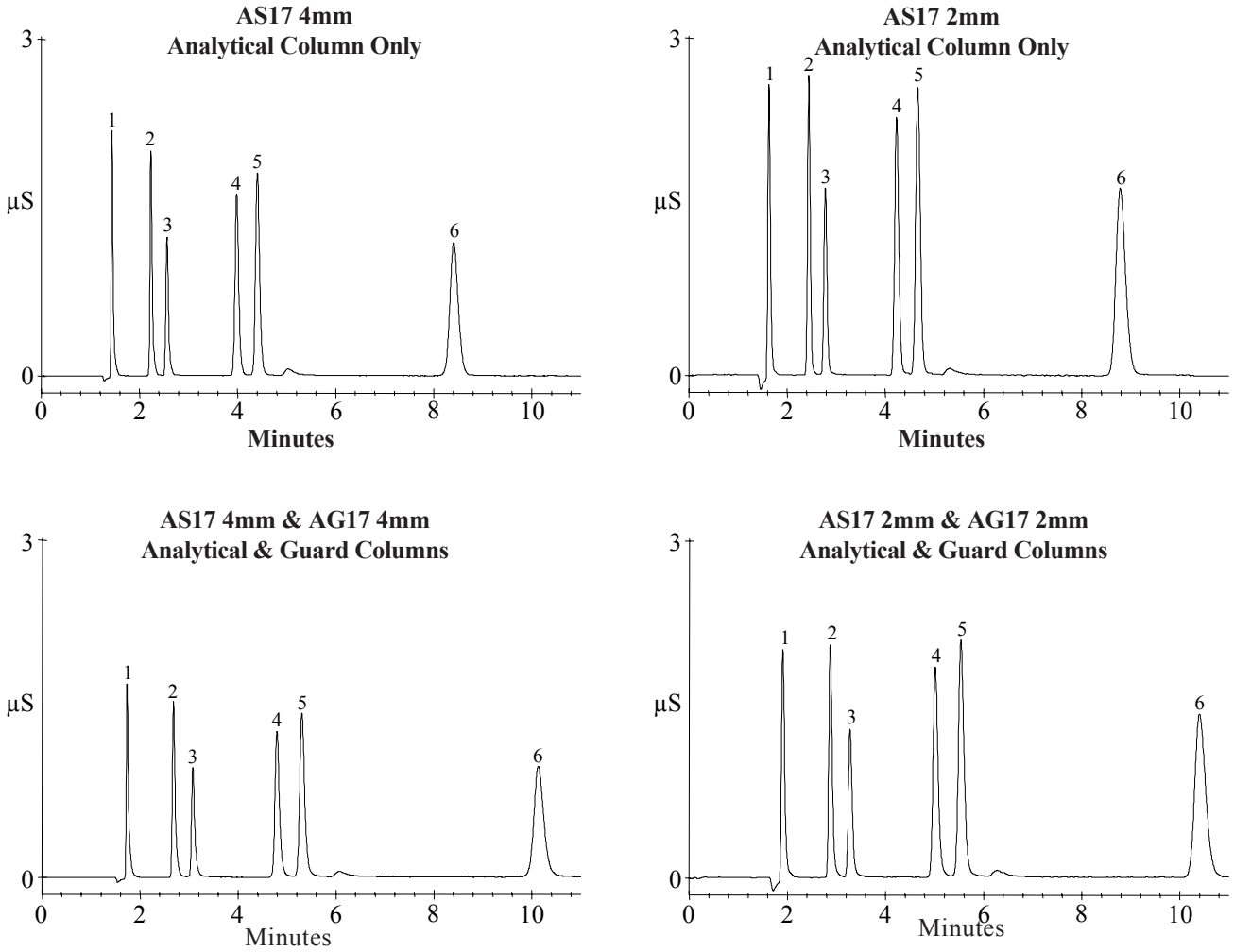



**Figure 1**
**IonPac AS17 Production Test Chromatograms**

## 5.3 Production Test Chromatograms at Room Temperature

The quality assurance test for the IonPac AS17 analytical column has been optimized at 30°C. However, the column can be operated at room temperature. Notice at room temperature (22°C), the divalent ion (sulfate) has a shorter retention time with 15 mM NaOH. 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 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac AG17, AS17 4-mm |
| Eluent: | 15 mM NaOH |
| Eluent Flow Rate: | 1.0 mL/min |
| Temperature: | See Chromatogram |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode (300 mA) |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 2 µS |
| Storage Solution: | 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 |

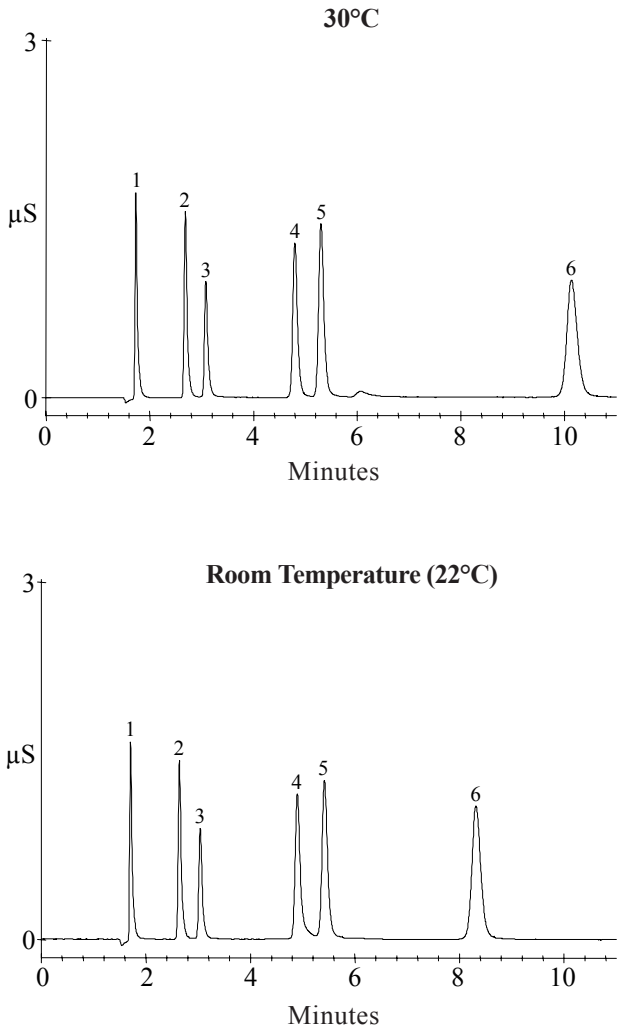



**Figure 2**
**IonPac AS17 Production Test Chromatograms at Room Temperature**

## 5.4 Analysis of Common Inorganic Anions Using EG40 Eluent Delivery System

The IonPac AS17 analytical column in combination with the AG17 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 EG40 Eluent Generator. The following chromatograms illustrate the analysis of common inorganic anions using the EG40 eluent delivery system at 30°C and at room temperature (22°C). Notice at room temperature, in order to achieve similar separation and retention times, a lower eluent concentration is required.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source: | EG40 |
| Eluent: | See Table |
| Eluent Flow Rate: | 2.0 mL/min (4-mm) |
| Temperature: | See Chromatogram |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | 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 |

**EG40 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 | |

**EG40 gradient for room temperature (22°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** | 15 | |
| **9.0** | 40 | |

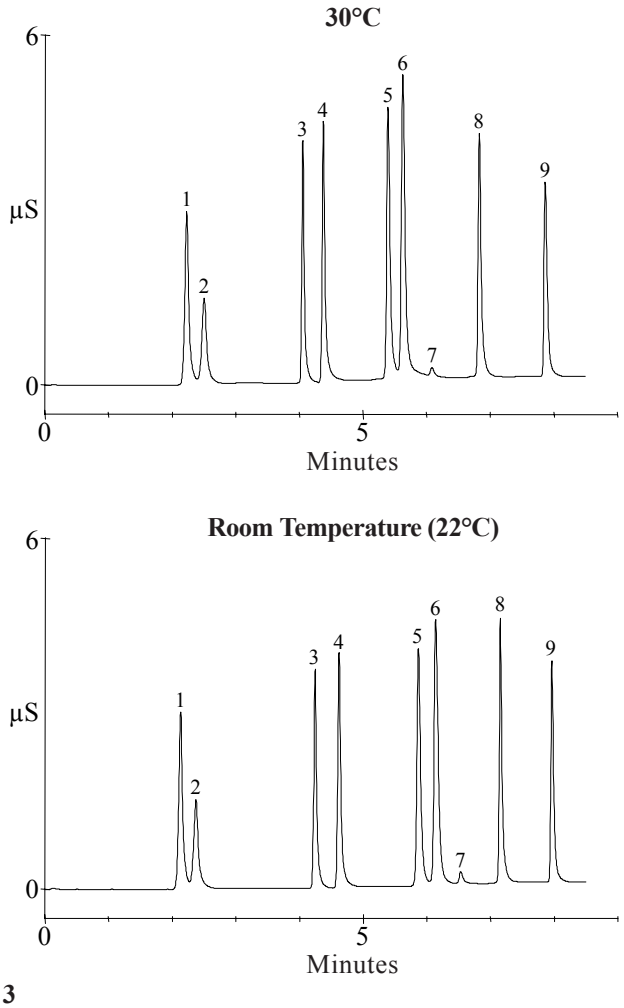



**Figure 3**
**Analysis of Common Inorganic Anions using EG40 Eluent Delivery System**

## 5.5 Gradient Analysis of Common Inorganic Anions at Room Temperature

The following chromatograms compare the use of the EG40 eluent delivery system vs. the bottle eluent delivery system for the determination of common inorganic anions at room temperature. Notice that with the bottle eluent delivery system, longer equilibration time is required before injection.

| | |
|---|---|
| Trap Column: | Bottle Eluent System, ATC-3 located after pump<br>EG40 Eluent System, ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | See table |
| Eluent: | See table |
| Eluent Flow Rate: | 2.0 mL/min (4-mm) |
| Temperature: | Room temperature (22°C) |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | 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 |

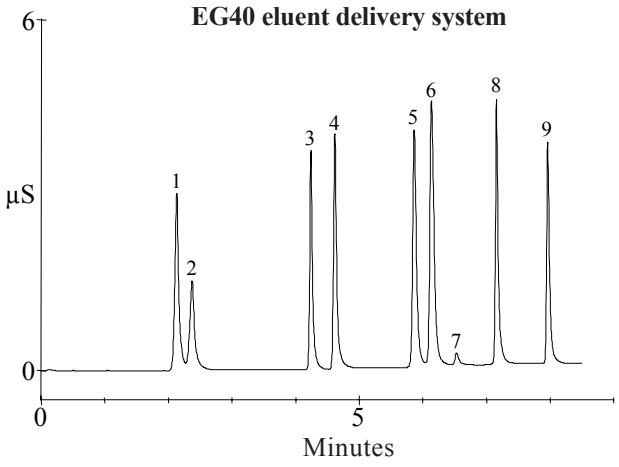
**Eluent:** Deionized water
Offset volume = 0.0 µL

| 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 | End gradient |

**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 |



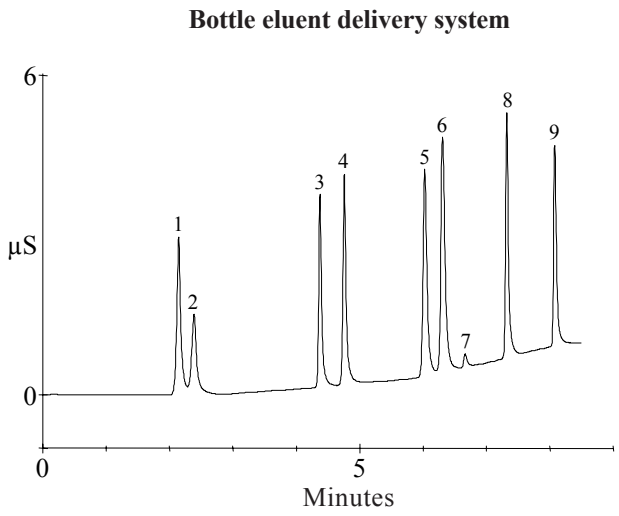




**Figure 4**
**Analysis of Common Inorganic Anions at Room Temperature**

## 5.6 Determination of Inorganic Anions, Oxyhalides, and Organic Acids Using EG40 Eluent Delivery System

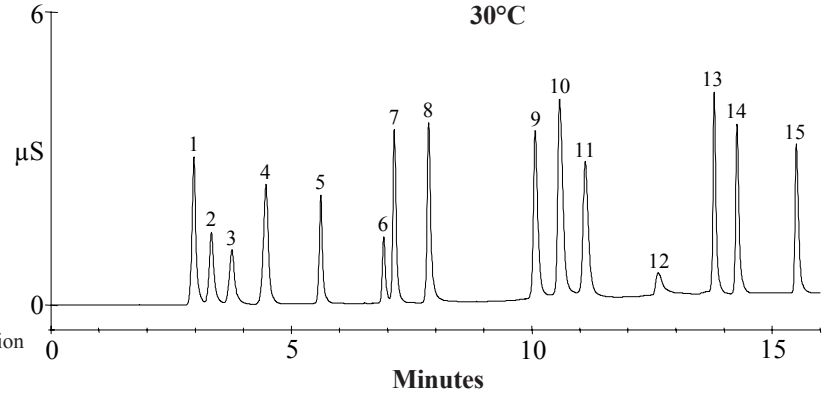
Figure 5 illustrates the separation of a large number of inorganic anions, oxyhalides, and organic acids on the IonPac AS17 using a hydroxide gradient delivered with the EG40 Eluent Generator. The following chromatograms demonstrate the effect of temperature on the separation. Notice at 30°C, the resolution of fluoride/acetate and bromate/chloride is improved.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 10 µL |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | EG40 |
| Eluent: | See Table |
| Eluent Flow Rate: | 1.5 mL/min (4-mm) |
| Temperature: | See Chromatogram |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

| | Analyte | mg/L(ppm) |
|---|---|---|
| 1. | Fluoride | 2.0 |
| 2. | Acetate | 5.0 |
| 3. | Propionate | 5.0 |
| 4. | Formate | 5.0 |
| 5. | Chlorite | 5.0 |
| 6. | Bromate | 5.0 |
| 7. | Chloride | 3.0 |
| 8. | Nitrite | 5.0 |
| 9. | Bromide | 10.0 |
| 10. | Nitrate | 10.0 |
| 11. | Chlorate | 10.0 |
| 12. | Carbonate | 20.0 |
| 13. | Sulfate | 5.0 |
| 14. | Oxalate | 5.0 |
| 15. | Phosphate | 10.0 |

**EG40 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 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 |




**EG40 gradient for room temperature (22°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** | 10.0 | |
| **18.0** | 35.0 | End gradient |

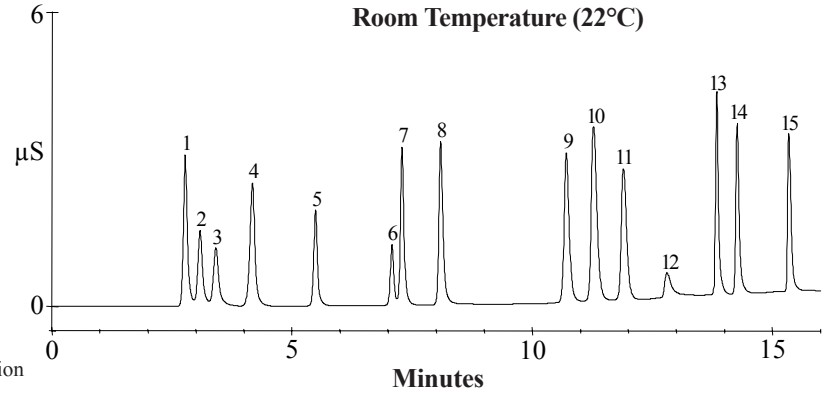



**Figure 5**
**Determination of Inorganic Anions, Oxyhalides, and Organic Acids using EG40 Eluent Delivery System**

## 5.7 Determination of Inorganic Anions, Oxyhalides, and Organic Acids at Room Temperature

The following chromatograms compare the EG40 eluent delivery system with the bottle eluent delivery system at room temperature (22°C) for the determination of inorganic anions, oxyhalides, and organic acids. Notice, due to the large system void volume with the bottle eluent system, a longer equilibration time is required before injection.

| | |
|---|---|
| Trap Column: | Bottle Eluent System, ATC-3 located after pump<br>EG40 Eluent System, ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | See table |
| Eluent: | See table |
| Eluent Flow Rate: | 1.5 mL/min (4-mm) |
| Temperature: | Room temperature (22°C) |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

| | Analyte | mg/L(ppm) |
|---|---|---|
| 1. | Fluoride | 2.0 |
| 2. | Acetate | 5.0 |
| 3. | Propionate | 5.0 |
| 4. | Formate | 5.0 |
| 5. | Chlorite | 5.0 |
| 6. | Bromate | 5.0 |
| 7. | Chloride | 3.0 |
| 8. | Nitrite | 5.0 |
| 9. | Bromide | 10.0 |
| 10. | Nitrate | 10.0 |
| 11. | Chlorate | 10.0 |
| 12. | Carbonate | 20.0 |
| 13. | Sulfate | 5.0 |
| 14. | Oxalate | 5.0 |
| 15. | Phosphate | 10.0 |

**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 |

**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 5 min |
| **5.0** | 20 | 80 | 0 | |
| **Analysis** | | | | |
| **5.1** | 20 | 80 | 0 | Start isocratic analysis |
| **5.5** | 20 | 80 | 0 | Inject valve to load position |
| **7.0** | 20 | 80 | 0 | Begin gradient analysis |
| **14.0** | 0 | 90 | 10 | |
| **18.0** | 0 | 65 | 35 | End gradient |

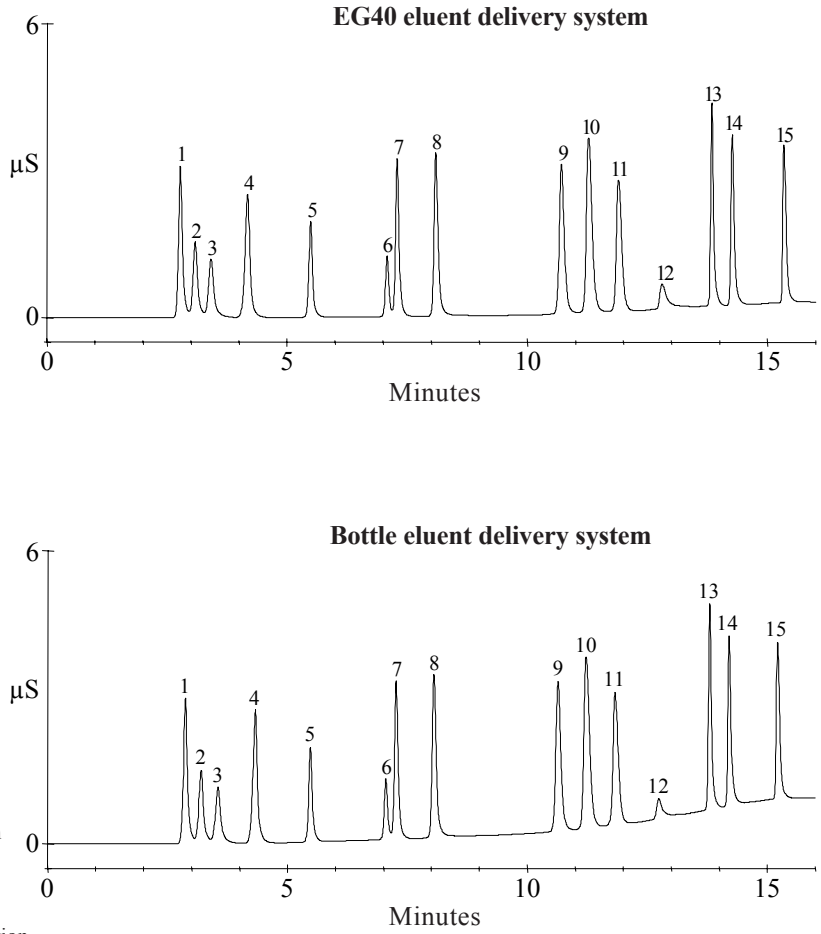



**Figure 6**
**Determination of Inorganic Anions, Oxyhalides, and Organic Acids at Room Temperature**

## 5.8 Analysis of a Simulated Drinking Water Sample Using AS17

The following chromatogram illustrates the analysis of a simulated drinking water sample using the AS17 column with a KOH eluent.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac AG17, AS17 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 (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

**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 |

| | Analyte | mg/L(ppm) |
|---|---|---|
| 1. | Fluoride | 0.2 |
| 2. | Chlorite | 0.2 |
| 3. | Bromate | 0.2 |
| 4. | Chloride | 40.0 |
| 5. | Nitrite | 0.2 |
| 6. | Bromide | 0.2 |
| 7. | Nitrate | 10.0 |
| 8. | Chlorate | 0.2 |
| 9. | Carbonate | 150.0 |
| 10. | Sulfate | 40.0 |
| 11. | Phosphate | 0.4 |

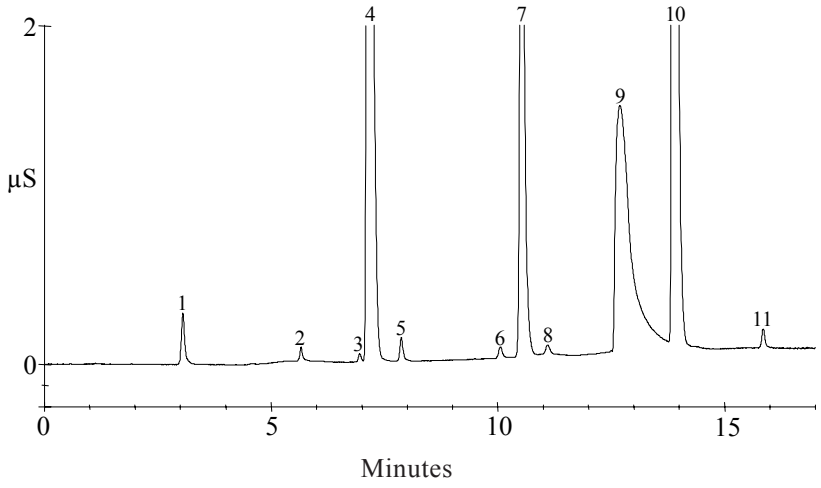



**Figure 7**
**Analysis of Simulated Drinking Water Sample**

## 5.9 Analysis of Municipal and Bottled Drinking Water Samples Using AS17

The following chromatograms illustrates the analysis of a municipal drinking water sample and a bottled drinking water sample using the AS17 column with a KOH eluent.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac AG17, AS17 4-mm |
| Eluent: | See table |
| Eluent Flow Rate: | 2 mL/min (4-mm) |
| Temperature: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

**Analyte**
1. Fluoride
2. Chlorite
3. Chloride
4. Nitrite
5. Bromide
6. Nitrate
7. Chlorate
8. Carbonate
9. Sulfate
10. Oxalate
11. Phosphate

**EG40 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 | End gradient |

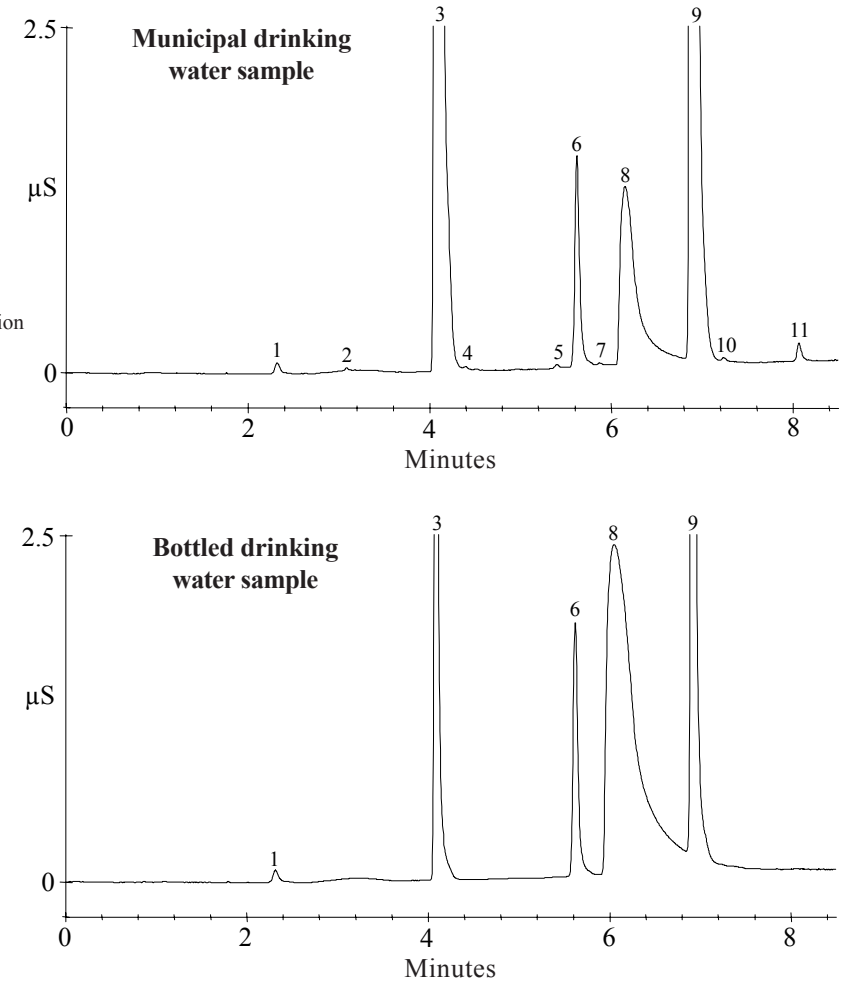



**Figure 8**
**Analysis of Municipal and Bottled Drinking Water Sample**

## 5.10 Determination of Sulfur Species in a Simulated Industrial Waste Water Sample

Figure 9 illustrates the separation of sulfur species, organic acids, and inorganic anions found in a simulated industrial waste water sample on the IonPac AS17 using a hydroxide gradient delivered with the EG40 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: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | EG40 |
| Eluent: | See Table |
| Eluent Flow Rate: | 2.0 mL/min (4-mm) |
| Temperature: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

**EG40 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 | End gradient |

| | Analyte | mg/L (ppm) |
|---|---|---|
| 1. | Acetate | 2.0 |
| 2. | Glycolate | 2.0 |
| 3. | Formate | 2.0 |
| 4. | Chloride | 20.0 |
| 5. | Nitrate | 1.0 |
| 6. | Carbonate | 50.0 |
| 7. | Sulfite | 4.0 |
| 8. | Sulfate | 40.0 |
| 9. | Thiosulfate | 12.0 |

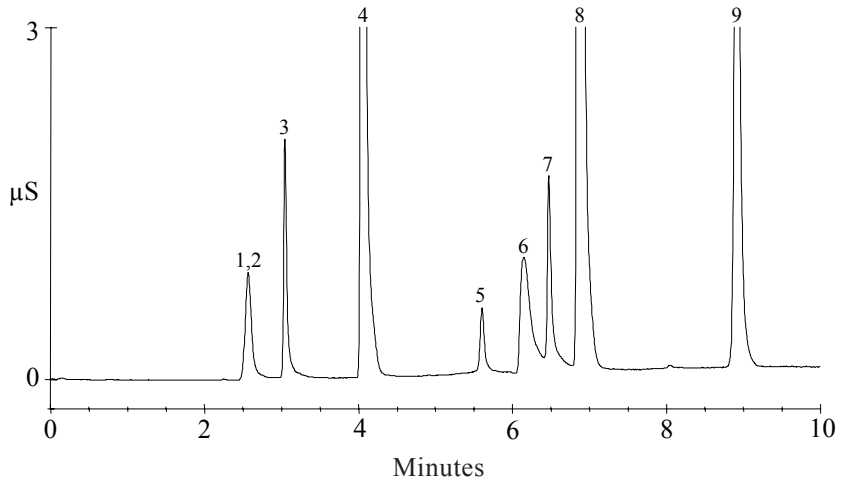



**Figure 9**
**Determination of Sulfite Species in a Simulated Industrial Waste Water Sample**

## 5.11 Separation of Anionic Additives in Toothpaste

The excellent retention of fluoride on the IonPac AS17 makes it ideal for the determination of fluoride and monofluorophosphate in dental care products. Figure 10 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 column.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 10 µL |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | EG40 |
| Eluent: | See Table |
| Eluent Flow Rate: | 1.5 mL/min (4-mm) |
| Temperature: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

| | Analyte | Standard Conc. mg/L(ppm) |
|---|---|---|
| 1. | Fluoride | 1.0 |
| 2. | Acetate | 1.0 |
| 3 | Formate | 1.0 |
| 4. | Chloride | 1.0 |
| 5. | Nitrite | 1.0 |
| 6. | Nitrate | 1.0 |
| 7. | Benzoate | 5.0 |
| 8. | Carbonate | 20.0 |
| 9. | Monoflurophosphate | 10.0 |
| 10. | Sulfate | 5.0 |
| 11. | Oxalate | 5.0 |
| 12. | Phosphate | 5.0 |

**EG40 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 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 |

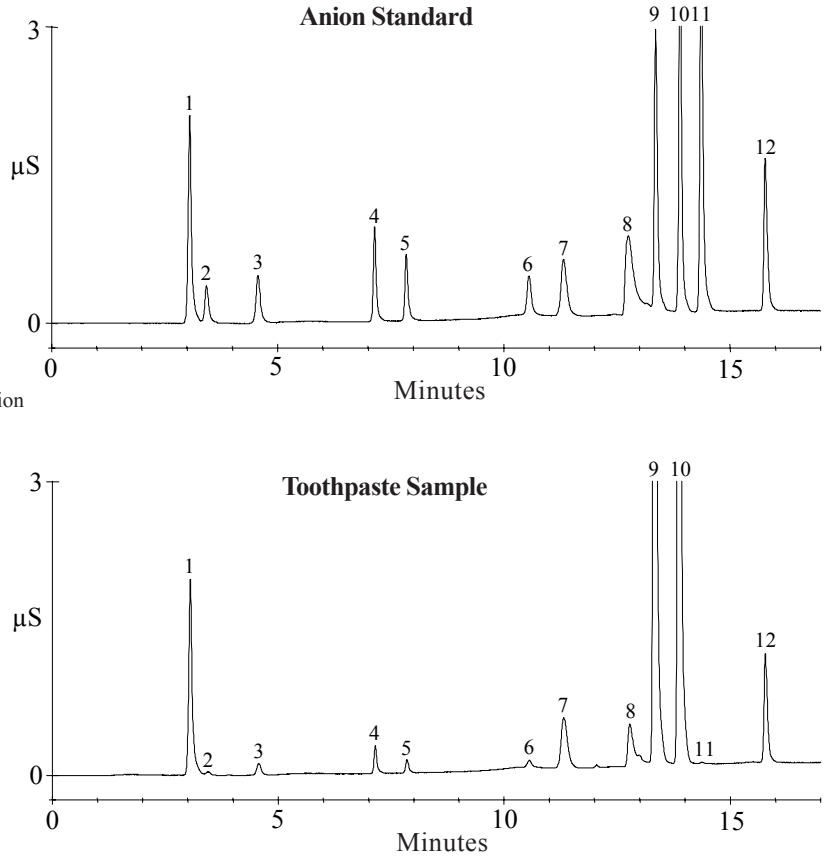



**Figure 10**
**Separation of Anionic Additives in toothpaste**

## 5.12 IonPac AS17 Selectivity for Inorganic and Organic Anions

Figure 11 demonstrates the IonPac AS17 selectivity for a variety of inorganic and organic anions using a 35 minute hydroxide gradient.

| | |
|---|---|
| Trap Column: | ATC-3 (2), 1 located after pump;<br>1 located at eluent outlet of EG40 degas module and before injector |
| Sample Volume: | 10 µL |
| Column: | IonPac® AG17, AS17 4-mm |
| Eluent Source | EG40 |
| Eluent: | See Table |
| Eluent Flow Rate: | 1.0 mL/min (4-mm) |
| Temperature: | 30°C |
| Suppressor*: | Anion Self-Regenerating Suppressor-ULTRA (4-mm)<br>AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | Eluent |

**EG40 gradient for 30°C**

**Eluent:** Deionized water

Offset volume = 0.0 µL

| Time (min) | Eluent Conc. (mM) | Comments |
|---|---|---|
| **Equilibration** | | |
| **0** | 0.3 | 0.3 mM KOH for 5 min |
| **5.0** | 0.3 | |
| **Analysis** | | |
| **5.1** | 0.3 | Start isocratic analysis |
| **6.0** | 0.3 | Inject valve to load position |
| **12.0** | 0.3 | Begin gradient analysis |
| **25.0** | 15.0 | |
| **35.0** | 60.0 | End gradient |

| | Analyte | mg/L(ppm) |
|---|---|---|
| 1. | Quinate | 5 |
| 2. | Fluoride | 1 |
| 3. | Lactate | 5 |
| 4. | Iodate | 5 |
| 5. | Acetate | 5 |
| 6. | Glycolate | 5 |
| 7. | Propionate | 5 |
| 8. | Formate | 5 |
| 9. | Butyrate | 5 |
| 10. | Methylsulfonate | 5 |
| 11. | Pyruvate | 5 |
| 12. | Chlorite | 5 |
| 13. | Valerate | 5 |
| 14. | Bromate | 5 |
| 15. | Chloride | 2 |
| 16. | Nitrite | 5 |
| 17. | Trifluoroacetate | 5 |
| 18. | Azide | 5 |
| 19. | Bromide | 5 |
| 20. | Nitrate | 5 |

| | Analyte | mg/L(ppm) |
|---|---|---|
| 21. | Chlorate | 5 |
| 22. | Glutarate | 5 |
| 23. | Carbonate | 10 |
| 24. | Succinate | 5 |
| 25. | Malate | 5 |
| 26. | Malonate | 5 |
| 27. | Tartrate | 5 |
| 28. | Maleate | 5 |
| 29. | Sulfate | 5 |
| 30. | Oxalate | 5 |
| 31. | Fumarate | 5 |
| 32. | Tungstate | 5 |
| 33. | Phosphate | 5 |
| 34. | Phthalate | 5 |
| 35. | Chromate | 5 |
| 36. | Citrate | 10 |
| 37. | Isocitrate | 10 |
| 38. | cis-Aconitate | 10 |
| 39. | trans-Aconitate | |

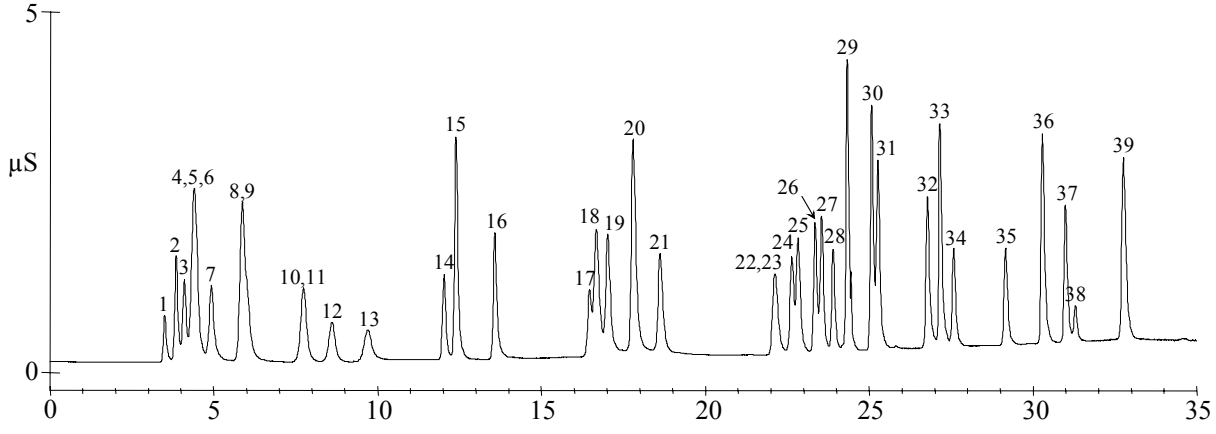



**Figure 11**
**IonPac AS17 Selectivity for Inorganic and Organic Anions**

## 5.13 Clean-up After Humic Acid Samples

Solvent compatibility of the IonPac AS17 permits the use of organic solvents to effectively remove organic contaminates from the column. An AS17 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 µL |
| Column: | IonPac AS17 4-mm |
| Eluent: | 15 mM KOH |
| Eluent Flow Rate: | 1.0 mL/min |
| Operating Temp: | 30°C |
| SRS Suppressor: | Anion Self-Regenerating Suppressor-ULTRA (4-mm) AutoSuppression® Recycle Mode |
| or MMS Suppressor: | Anion MicroMembrane Suppressor, AMMS III (4-mm) |
| MMS Regenerant: | 50 mN $H_2SO_4$ |
| Expected Background Conductivity: | < 1.0 µS |
| Storage Solution: | 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 |

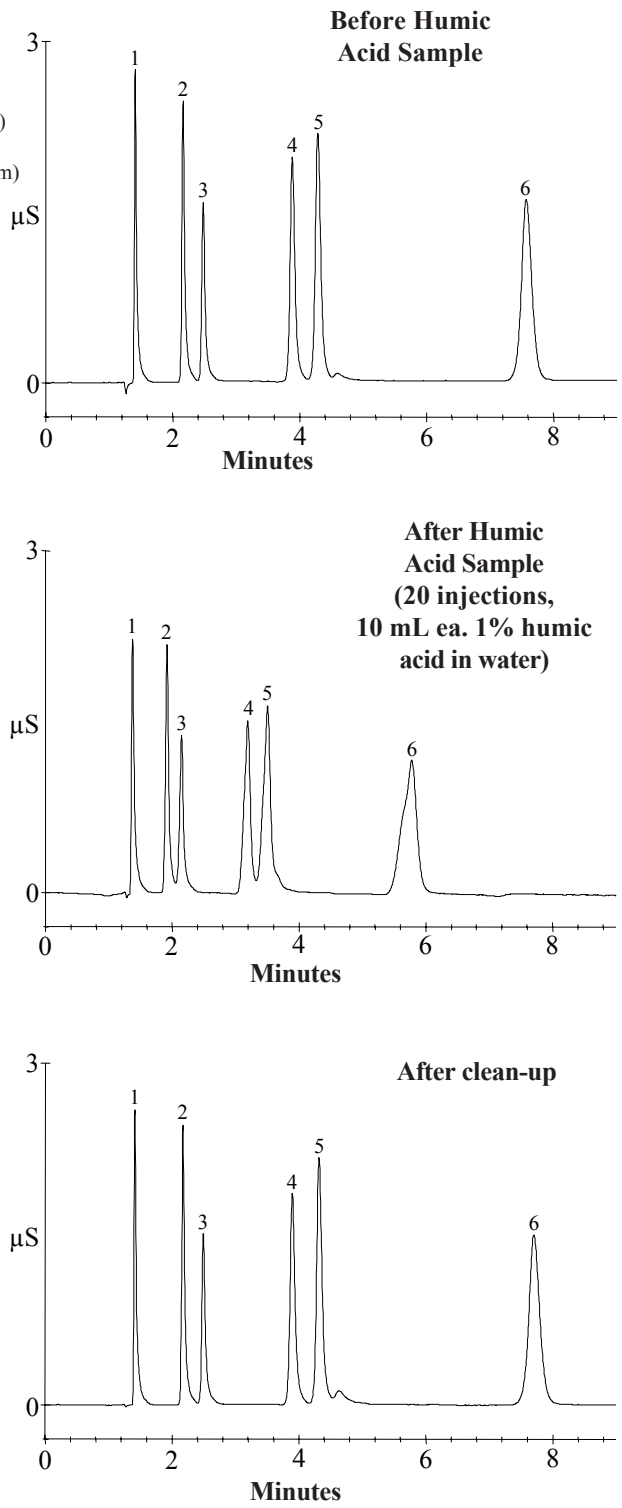



**Figure 12**
**Clean-up after Humic Acid Samples**

## SECTION 6 - TROUBLESHOOTING GUIDE

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

| Observation | Cause | Action | Reference Section |
|---|---|---|---|
| **High Back Pressure** | Unknown | Isolate Blocked Component | 6.1.1 |
| | Plugged Column<br>Bed Supports | Replace Bed Supports<br>Filter Eluents<br>Filter Samples | 6.1.2<br>6.1.3<br>6.1.4 |
| | Other System Components | Unplug, Replace | Component Manual |
| **High Background Conductivity** | Contaminated Eluents | Remake Eluents | 6.2, 6.2.1 |
| | Contaminated Trap Column | Clean Trap Column | 6.2.2, Component Manual |
| | Contaminated ASRS or AMMS | Clean Suppressor | 6.2.5, Component Manual |
| | Contaminated Hardware | Clean Component | 6.2.4, Component Manual |
| **Poor Resolution** | Poor Efficiency Due to Large System Void Volumes | Replumb System | 6.3.1.B, Component Manual |
| | Column Headspace | Replace Column | 6.3.1.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 (4-mm) Guard Column plus the AS17 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 1,000 psi. 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/AG17 Operating Back Pressures").

The Anion Self-Regenerating Suppressor-ULTRA 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/AG17 Operating Back Pressures**

| Column | Typical Back Pressure psi (MPa) | Flow Rate mL/min |
|---|---|---|
| AS17 4-mm Analytical | < 750 (5.17) | 1.0 |
| AG17 4-mm Guard | < 150 (1.03) | 1.0 |
| **AS17 + AG17 4-mm columns** | **< 900 (6.20)** | **1.0** |
| AS17 2-mm Analytical | < 750 (5.17) | 0.25 |
| AG17 2-mm Guard | < 150 (1.03) | 0.25 |
| **AS17 + AG17 2-mm columns** | **< 900 (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 4-mm Columns (P/N)** | **IonPac AS17 2-mm Columns (P/N)** |
|---|---|---|
| Analytical Column | 055682 | 055683 |
| Guard Column | 055684 | 055685 |
| 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:

| ELUENT | EXPECTED BACKGROUND CONDUCTIVITY |
|---|---|
| 15 mM NaOH | < 2 µS |
| 15 mM KOH (EG40) | < 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

**When doing gradient analysis, ensure that the Anion Trap Column, the ATC-3 (2-mm) or the ATC-3 (4-mm) has been installed correctly.** If it has not, install one as directed in Section 3.2, "The Anion Trap Column," and watch the background conductivity. If the background conductivity is now low, this means that the ATC is trapping contaminants from the eluent. The eluents probably have too many impurities (see items A - C above).

**Determine if the ATC is the source of high background conductivity.** Remove the ATC. If the background conductivity remains high, then the ATC is not the problem. If the background conductivity decreases, the ATC is the source of the high background conductivity.

**A. Disconnect either the ATC-3** (2-mm) or the ATC-3 (4-mm) from the injection valve and direct the outlet to waste.

**B. Flush the ATC-3** (4-mm) **with 100 mL of 200 mM NaOH or 50 mL of 200 mM NaOH for 2-mm ATC-3.** Use a flow rate of 0.5 mL/min on a 2-mm system or a flow rate of 2.0 mL/min on a 4-mm system.

**C. Pump 20 mL of eluent through 4-mm ATC-3 or 10 mL for the 2-mm ATC-3.**

**D. If the problem persists, replace the ATC.**

### 6.2.3 A Contaminated Guard or Analytical Column

Remove the IonPac AG17 Guard and AS17 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 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").

### 6.2.4 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.

### 6.2.5 A Contaminated Suppressor

If the above items have been checked and the problem persists, the Anion MicroMembrane Suppressor is probably causing the problem.

**A. Check the eluent flow rate.** In general, the eluent flow rate for 4-mm applications should be 1.5 mL/min. Refer to the Anion Self-Regenerating Suppressor Product Manual (Document No. 034449-02) for assistance in determining that the eluent is within suppressible limits.

**B. If the background is very high, (>1,000 µS) or the baseline noise is very high, the ASRS may have failed to suppress the eluent.** You may need to replace the ASRS-ULTRA suppressor.

C. If you are using eluents containing solvents, use the ASRS-ULTRA in external water mode and flow rate should be 7 - 10 mL/min.

**D. Check the regenerant flow rate at the REGEN OUT port of the AMMS.** For the example isocratic applications, this flow rate should be 3 - 5 mL/min.

**E. If you are using an AutoRegen Accessory with the SRS (in the Chemical Suppression Mode) or the MMS, 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 SRS or MMS.**

**2. If the background conductivity is low when freshly prepared regenerant is run through the SRS or MMS 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 (see, Figure 13).

**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.

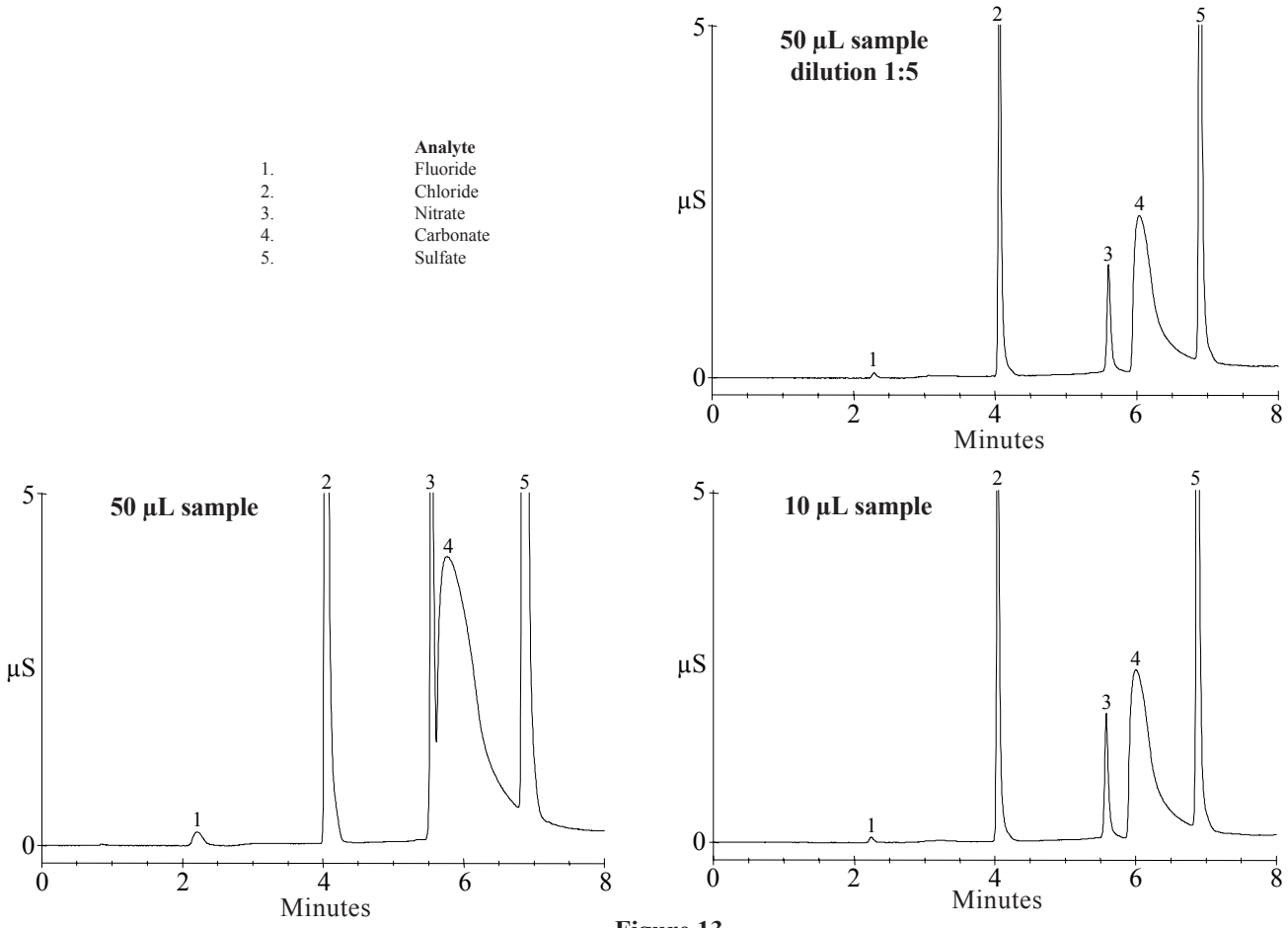



**Figure 13**
**Comparison of Diluted Sample and Smaller Injection Volume**

### 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").

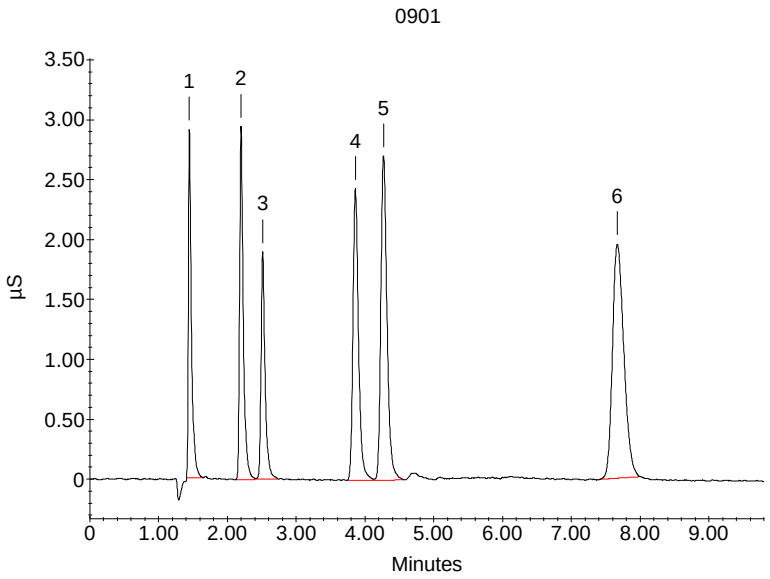
If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS17 columns, contact the North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

**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.

# IonPac® AS17
# Analytical (4 x 250 mm)
# Product No. 55682

**Serial No. : 0901** **Pressure (PSI) : 640** **Date : 9/5/02 8:08:38 AM**




**Eluent:** 15.0 mM NaOH
**Flow Rate:** 1.0 mL/min
**Detection:** Suppressed Conductivity
**ASRS®-ULTRA**
AutoSuppression® Recycle Mode

**Injection Volume:** 10 µL

**Storage Solution:** Eluent

Peak Information : Found Components

| Peak No. | Retention Time | Name | (mg/L) | Efficiency | Asymmetry (10%) | Resolution |
|---|---|---|---|---|---|---|
| 1 | 1.45 | Fluoride | 0.5 | 5697 | 2.6 | 9.16 |
| 2 | 2.20 | Chloride | 1.0 | 9532 | 2.0 | 3.30 |
| 3 | 2.51 | Nitrite | 1.0 | 9465 | 1.7 | 10.83 |
| 4 | 3.86 | Bromide | 3.0 | 11604 | 1.5 | 2.64 |
| 5 | 4.27 | Nitrate | 3.0 | 11396 | 1.5 | 14.50 |
| 6 | 7.67 | Sulfate | 3.0 | 10009 | 1.5 | n/a |

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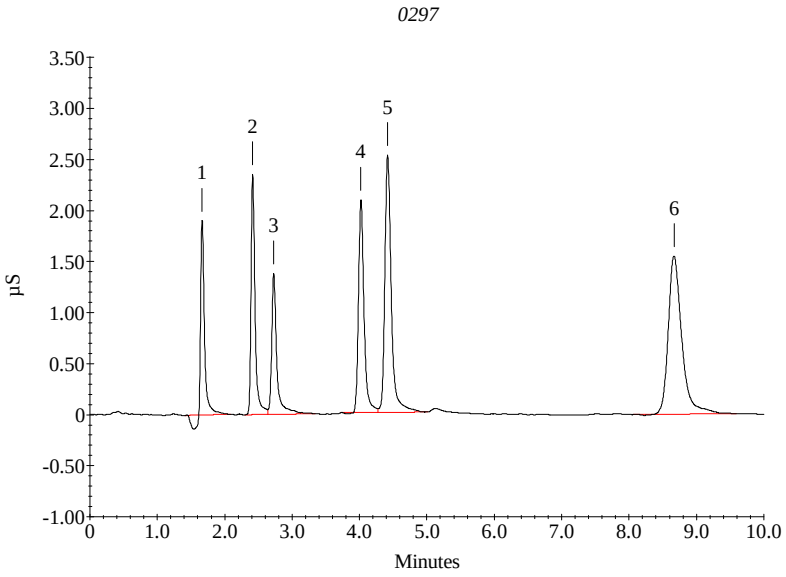
# IonPac® AS17
# Analytical (2 x 250 mm)
# Product No. 55683

**Serial No. : 0297** **Pressure (PSI) : 650** **Date : 1/11/01 11:32:11 AM**




**Eluent:** 15.0 mM NaOH
**Flow Rate:** 0.25 mL/min
**Detection:** Suppressed Conductivity
**ASRS®-ULTRA, 2-mm**
AutoSuppression® Recycle Mode

**Injection Volume:** 2.5 µL

**Storage Solution:** Eluent

Peak Information : Found Components

| Peak No. | Retention Time | Name | (mg/L) | Efficiency | Asymmetry (10%) | Resolution |
|---|---|---|---|---|---|---|
| 1 | 1.66 | Fluoride | 0.5 | 4737 | 2.0 | 7.41 |
| 2 | 2.42 | Chloride | 1.0 | 9344 | 1.5 | 2.74 |
| 3 | 2.73 | Nitrite | 1.0 | 9261 | 1.8 | 9.74 |
| 4 | 4.02 | Bromide | 3.0 | 12586 | 1.5 | 2.55 |
| 5 | 4.42 | Nitrate | 3.0 | 12192 | 1.5 | 16.72 |
| 6 | 8.67 | Sulfate | 3.0 | 9626 | 1.5 | n/a |

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# 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 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 both short-term and long-term storage, use column test eluent for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution (eluent). 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 < 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 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 and AS17.**

## 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 or AMMS** from the IonPac AS17 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 4-mm Analytical** or Guard Column or set the pump flow rate to 0.25 mL/min for an AS17 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 or AMMS** to the AS17 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.