Errata

Product Manual for Dionex IonPac™ AS10 and AG10 Columns
034519-08

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

Part	Old Part Number in this manual	Updated Part Number to use for new orders
PROD,COL,IP,ATC-3,4X35MM	*059661*	*079932*

PRODUCT MANUAL

for

IonPac® AG10
IonPac® AS10

Now sold under the
Thermo Scientific brand

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DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

IONPAC® AG10 GUARD COLUMN

(4 x 50 mm, P/N 043119)
(2 x 50 mm, P/N 043124)

IONPAC® AS10 ANALYTICAL COLUMN

(4 x 250 mm, P/N 043118)
(2 x 250 mm, P/N 043123)

Document No. 034519
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SECTION 1 - INTRODUCTION TO IONPAC AS10/AG10 CHROMATOGRAPHY

The IonPac® AS10 Analytical Column and the AG10 Guard Column are designed for the analysis of inorganic anions and organic acids. The AS10 is compatible with pH 0-14 eluents and eluents containing organic solvents from 0 - 100% in concentration. The AS10 can be used with any suppressible ionic eluent that does not exceed the capacity of the Anion Self-Regenerating Suppressor (ASRS-ULTRA). The IonPac AS10 has nominal efficiency for chloride using standard operating conditions of at least 36,000 plates/meter.

Table 1
IonPac AS10/AG10 Packing Specifications

Column	Particle Diameter µm	Substrate[a] X-linking %	Latex Diameter nm	Latex[b] X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
AS10 4 x 250 mm	8.5	55	65	5	170	Alkanol quaternary ammonium	Low
AG10 4 x 50 mm	8.5	55	65	5	34	Alkanol quaternary ammonium	Low
AC10 4 x 50 mm	13.0	55	160	5	4	Alkanol quaternary ammonium	Low
AS10 2 x 250 mm	8.5	55	65	5	42.5	Alkanol quaternary ammonium	Low
AG10 2 x 50 mm	8.5	55	65	5	8.5	Alkanol quaternary ammonium	Low
AC10 2 x 50 mm	13.0	55	160	5	0.8	Alkanol quaternary ammonium	Low

[a] AS10, AG10: macroporous (2,000 Å) divinylbenzene/ethylvinylbenzene polymer
AC10: microporous divinylbenzene/ethylvinylbenzene polymer
[b] microporous polyvinylbenzylammonium polymer cross-linked with divinylbenzene

Table 2
AS10/AG10 Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS10 4-mm Analytical	≤ 2,150 (14.82)	1.0	2.0
AG10 4-mm Guard	≤ 700 (4.82)	1.0	2.0
AC10 4-mm Concentrator	≤ 300 (2.07)	1.0	3.0
AS10 + AG10 + AC10 4-mm columns	**≤ 3,150 (21.71)**	**1.0**	**2.0**
AS10 2-mm Analytical	≤ 2,150 (14.82)	0.25	0.5
AG10 2-mm Guard	≤ 600 (4.14)	0.25	0.5
AC10 2-mm Concentrator	≤ 300 (2.07)	0.25	1.25
AS10 + AG10 + AC10 2-mm columns	**≤ 3,050 (21.03)**	**0.25**	**0.5**

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

CONFIGURATION	2-mm	4-mm
Eluent Flow Rate	0.25 mL/min	1.0 mL/min
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 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 tubing with long lengths or IDs greater than 0.005".	Minimize dead volumes. Switching valves, couplers can be used. Use the GM-2, GM-3, GM-5 or recommended gradient mixers.
Pumps	Use the GP40, GS50, GP50, IP20, or IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer. The older GPM-2 has a large delay volume. Gradient applications in this manual can not be reproduced without method modifications.	Use the GP40, GS50, GP50, IP20, or IP25 in Standard-Bore Configuration. Use the GM-5 Gradient Mixer 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 (6-mm, 7.5 µL, P/N 046423) VDM-2 Cell (3-mm, 2.0 µL, P/N 043120) CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Replace the TS-1 with the TS-2 (P/N 043117) on the CDM-2 or the CDM-3. The TS-2 has been optimized for 2-mm operation. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 50-75 psi back pressure.	AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) VDM-2 Cell (6-mm, 10 µL) P/N 043113 CD20, CD25, CD25A, ED40, ED50, or ED50A Conductivity Cell with DS3 P/N 044130 or with shield P/N 044132 CDM-2/CDM-3 Cell P/N 042770 Either the TS-1 with the TS-2 can be used with the CDM-2 or the CDM-3. Do not use the TS-2 or the TS-1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A. DIONEX Back Pressure Regulator 75 psi rating (P/N 039760, 046480) or Tubing (see Table 3) Ensure 50-75 psi back pressure.

Table 3
Tubing Back Pressures

Tubing ID in	H_20 Back Pressure 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 AS10 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 AS10 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. Ensure that the proper injection valve is used (see Section 2, “Comparison Ion Chromatography Systems” and Section 3.4, “The Injection Loop”). If you need assistance in properly configuring your system contact the nearest DIONEX Office (see, “DIONEX Worldwide Offices”).

3.2 THE ANION TRAP COLUMN

When performing an anion exchange application that involves a hydroxide gradient, an IonPac Anion Trap Column (ATC-3, (4-mm) P/N 059660 or ATC-3 (2-mm), P/N 059661) should be installed in place of the high pressure Gradient Mixer between the Gradient Pump Module (GPM-2) or the Advanced Gradient Pump (AGP) or the GP40/GP50/GS50 and the injection valve. The ATC-3 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).

3.3 THE SAMPLE CONCENTRATOR

The IonPac AC10 Anion Concentrator Column is designed primarily for high purity water analysis. The function of the AC10 is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the AC10 leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the AC10 to the analytical chemist is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

For detailed information on the installation and use of the IonPac AC10 Anion Concentrator Column see the IonPac AC10 Anion Concentrator Column Product Manual (Document No. 034529).

3.4 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

3.4.1 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. DIONEX recommends that a 2.5 µL injection loop be used to avoid overloading the AS10 2-mm Analytical Column. Generally, you should not inject more than 2.5 nanomoles (25 - 50 ppm) of any one analyte onto a 2-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. The AS10 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 (see Section 2, "Comparison of 2-mm and 4-mm Ion Chromatography Systems").

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

For most applications on a 4-mm analytical system, a 10 - 50 µL injection loop will be sufficient. DIONEX recommends that a 10 µL injection loop be used to avoid overloading the AS10 4-mm Analytical Column. Generally, do not inject more than 10 nanomoles (100 - 200 ppm) of any one analyte onto the 4-mm analytical column. Injecting larger volumes of samples can result in overloading the column which can affect the detection linearity. This phenomenon will be more prevalent at higher concentrations of the analytes of interest.

3.5 THE IONPAC AG10 GUARD COLUMN

An IonPac AG10 Guard Column is normally used with the IonPac AS10 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 AG10 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS10 Analytical Column.

3.6 ELUENT STORAGE

IonPac AS10 columns are designed to be used with sodium hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance (nitrogen can be used if eluents do not contain solvents).

3.7 ANION SELF-REGENERATING SUPPRESSOR 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.**

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

If you are installing an IonPac AS10 4-mm Analytical Column, use an ASRS-ULTRA (4-mm, P/N 053946).
If you are installing an IonPac AS10 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 (4-mm) and the ASRS-ULTRA (2-mm)."

3.8 ANION MICROMEMBRANE SUPPRESSOR REQUIREMENTS

An Anion Micro Membrane Suppressor (AMMS® III) may be used instead of an ASRS-ULTRA (4-mm) for applications that require suppressed conductivity detection. Use an AMMS III (P/N 056750) with the IonPac AS10 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.

3.10 USING AUTOREGEN WITH THE ASRS-ULTRA OR THE AMMS III IN THE CHEMICAL SUPPRESSION MODE

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

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

SECTION 4 - OPERATION

4.1 GENERAL OPERATING CONDITIONS

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:	2-mm: AS10 2-mm Analytical Column + AG10 2-mm Guard Column 4-mm: AS10 4-mm Analytical Column + AG10 4-mm Guard Column
Eluent:	80 mM NaOH
Eluent Flow Rate:	2-mm: 0.25 mL/min 4-mm: 1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA, (4-mm or 2-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor (AMMS III) (4-mm or 2-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 8 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2 IONPAC AS10 OPERATION PRECAUTIONS

CAUTION
Filter and Degas Eluents
Filter Samples
Eluent pH between 0 and 14
Sample pH between 0 and 14
2.0 mL/min Maximum Flow Rate for 4-mm Columns
0.5 mL/min Maximum Flow Rate for 2-mm Columns

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 particulate 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 AS10 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 ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Currently at DIONEX, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima Solvents by Fisher Scientific.

When using a solvent in an ionic eluent, column 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 AS10 columns is 3,500 psi (24.12 MPa).

The AS10 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 AS10 Columns

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

CAUTION
The Anion Self-Regenerating Anion Suppressor (ASRS-ULTRA) must be operated in the AutoSuppression External Water Mode when using eluents containing solvents.

4.4 Making Eluents

4.4.1 Making Sodium Hydroxide Eluents

The sodium hydroxide eluents that can be used with the IonPac AS10 columns will readily absorb carbon dioxide, producing carbonate. Precautions must be taken during eluent preparation to minimize contamination with carbon dioxide from the air. These precautions, if taken, ensure smooth, reproducible ramps, with 1 to 5 µS total change in background conductivity.

The eluents can be prepared either volumetrically using a syringe or by weighing. Using a syringe is more effective in preventing carbonate contamination while preparing the eluents. If you decide to use the weighing method, pipette, do not pour, the 50% sodium hydroxide into the weighing dish. Minimize the time that the solution is exposed to air.

4.4.2 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.

WARNING

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

4.5 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."

4.6 SAMPLE CONCENTRATION

Detection limits can be enhanced through the concentration of relatively pure samples on the IonPac AC10 Anion Concentrator Column (4-mm, P/N 043133 or 2-mm, P/N 043134). The concentrator column is used in lieu of the sample loop. Pump the sample onto the AC10 in the **OPPOSITE** direction of the eluent flow. See DIONEX Technical Note #8, "The Use of Concentrator Columns"

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 answers. During the concentration step, it is possible 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 for high sensitivity work with IonPac AS10 columns see Document No. 034529, "Product Manual for the AC10 Anion Concentrator Columns."

SECTION 5 - EXAMPLE APPLICATIONS

Before attempting any of the following example applications, take the time to ensure that your system is properly configured. It is very important that applications run on 2-mm columns utilize the proper pump configuration (see Section 2, "Comparison of 2-mm and 4-mm Ion Chromatography Systems") and have minimal system void volume.

Ensure that all of the eluents have been made from high purity reagents and deionized water. All water used in the preparation of eluents should be degassed, deionized water. See Section 4.4.1, "Degassing Deionized Water," for information on how to degass water for eluents. See Section 4.3, "Chemical Purity Requirements," for chemical purity requirements

To ensure reproducible retention times of analytes when doing gradient chromatography, it is important to install an Anion Trap Column in the system (see Section 3.2, "The Anion Trap Column").

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.

After running synthetic standards to calibrate your system, you may find that real sample matrices foul your columns. For this reason it is always advisable to use a guard column to protect the analytical column. If column performance deteriorates and it is determined that the guard and analytical columns have been fouled, refer to the column cleanup protocols in, "Column Care."

If your sample matrices are relatively low in ionic concentration, you may be able to increase the sensitivity of your system by using sample concentration techniques (see Section 3.3, "The Sample Concentrator").

Finally, when formulating eluents with 50% sodium hydroxide, 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 80 mM NaOH use 4.18 mL of 50% sodium hydroxide:
(as used in Section 5.2, "Production Test Chromatogram")

$$\textbf{80 mM} = \frac{\textbf{0.08 mole x 40.01 (g/mole)}}{\text{L}} = \frac{\textbf{1.53 (g/mL) x v (mL) x 50\%}}{\text{L}}$$

$$v = \frac{(0.08 \times 40.01)}{(1.53 \times 0.50)} = 4.18 \text{ mL}$$

5.1 Eluent Preparation

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 AS10 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 bottle or pipette the required aliquot from the top of the solution where sodium carbonate may have formed. replace the 50% NaOH reagent when sodium carbonate precipitate forms and the reagent becomes cloudy.

Table 6
Dilution of 50% (w/w) NaOH to Make Standard AS10 Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
4.0 (2.6)	50
6.4 (4.2)	80
6.8 (4.4)	85
8.0 (5.2)	100
16.0 (10.4)	200

Potassium Borate Eluents

Eluent: 3.5 mM $K_2B_4O_7$

Dissolve 1.07 g of $K_2B_4O_7 \cdot 4H_2O$ in approximately 500 mL of degassed, deionized water (having a specific resistance of 18.2 megohm-cm) in a 1,000 mL volumetric flask. After the $K_2B_4O_7$ has dissolved in the water, dilute the solution to a final volume of 1,000 mL using degassed, deionized water. Avoid the introduction of carbon dioxide from the air into the solution.

5.2 PRODUCTION TEST CHROMATOGRAM

Separation and elution of anions on the IonPac AS10 Analytical Column has been optimized utilizing a sodium hydroxide eluent. By using this eluent, monovalent and divalent anions can be isocratically separated and quantified in a single injection. To guarantee that all IonPac AS10 Analytical Columns meet high quality and reproducible performance specifications standards, all 2-mm and 4-mm analytical columns undergo the following production control test.

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:	80 mM NaOH
Eluent Flow Rate:	2-mm: 0.25 mL/min 4-mm: 1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA(4-mm or 2-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm or 2-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 8 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

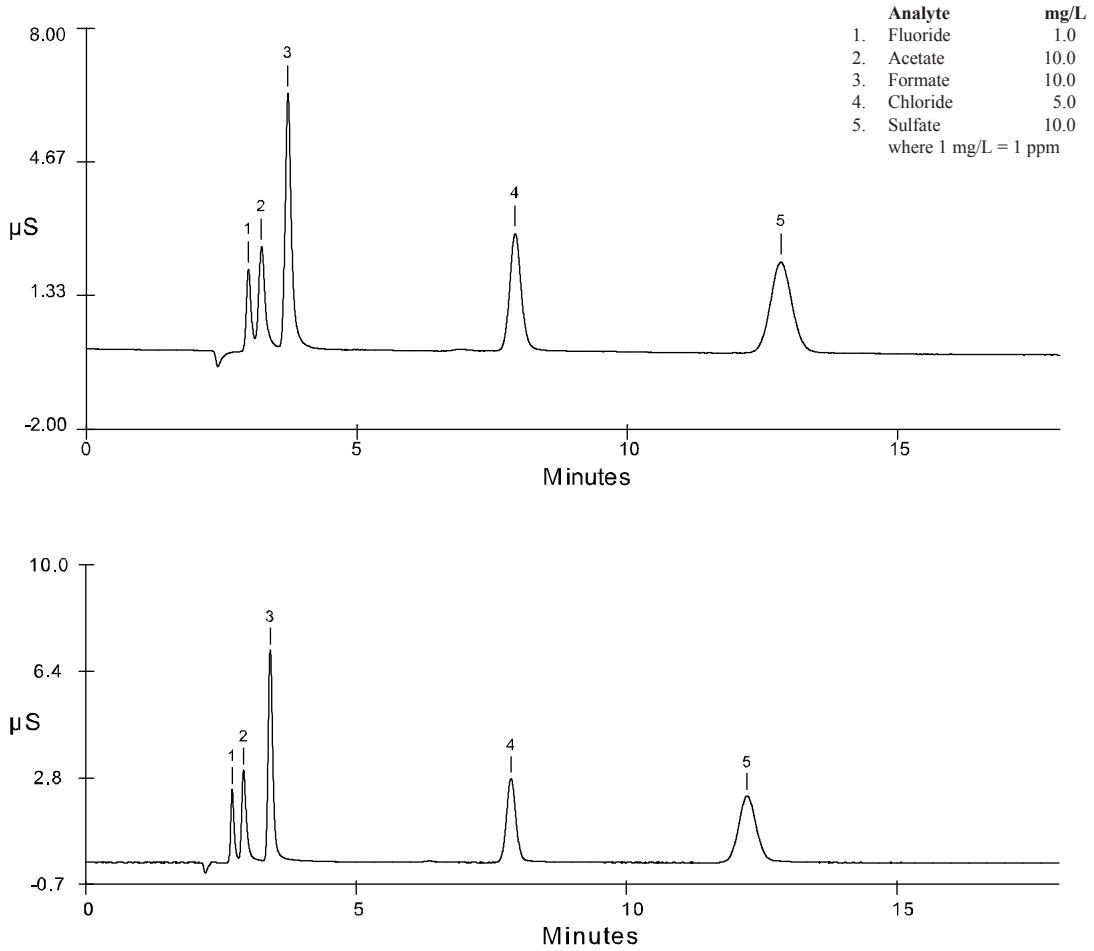
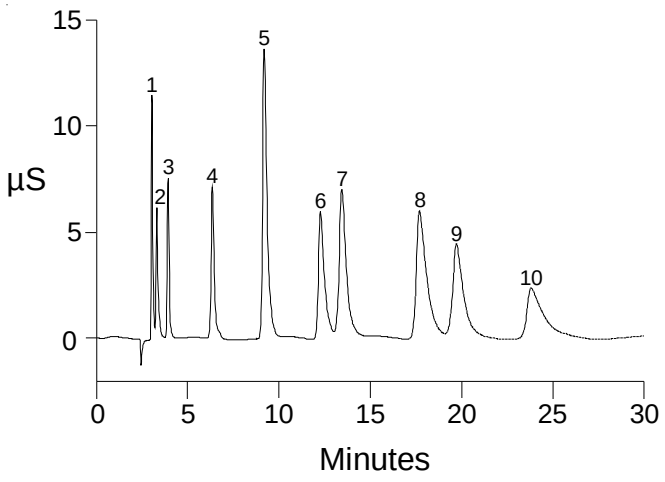


Figure 1
IonPac AS10 Test Chromatogram

5.3 ISOCRATIC ELUTION OF INORGANIC ANIONS AND ORGANIC ACIDS

Separation and elution of anions on the IonPac AS10 Analytical Column has been optimized utilizing a sodium hydroxide eluent. By using this eluent, mono-, di- and trivalent anions can be isocratically separated and quantified in a single injection.

Sample Volume:	20 µL
Column:	IonPac AS10 4-mm Analytical
Eluent:	100 mM NaOH
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor -ULTRA(4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 8 µS



	Analyte	mg/L
1.	Fluoride	0.5
2.	Acetate	23.5
3.	Formate	5.5
4.	Selenite	23.5
5.	Chloride	9.5
6.	Nitrite	23.5
7.	Sulfate	16.0
8.	Oxalate	23.5
9.	Selenate	23.5
10.	Arsenate	47

where 1 mg/L = 1 ppm

Figure 2
Isocratic Elution of Inorganic and Organic Anions

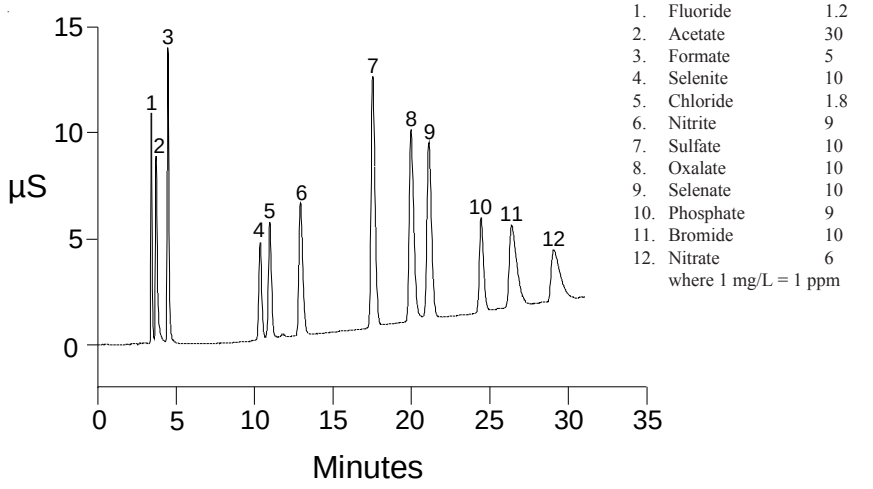
5.4 GRADIENT ELUTION OF INORGANIC ANIONS AND ORGANIC ACIDS

The isocratic separation and elution of anions on the IonPac AS10 Analytical Column has been further optimized by utilizing a sodium hydroxide gradient elution program. By using this gradient elution program, additional anions of interest can be separated and quantified.

Sample Volume:	10 µL
Trap Column:	IonPac ATC-3
Column:	IonPac AS10 4-mm Analytical
Eluent:	E1: 50 mM NaOH
	E2: 200 mM NaOH
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA(4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected System Pressure:	2,500 psi (17.23 MPa)
Expected Background Conductivity:	≤ 8 µS

Gradient Conditions

Time	%E1	%E2	Comments
0.0	100	0	Inject, Begin Gradient
31.0	38	62	End Gradient



	Analyte	mg/L
1.	Fluoride	1.2
2.	Acetate	30
3.	Formate	5
4.	Selenite	10
5.	Chloride	1.8
6.	Nitrite	9
7.	Sulfate	10
8.	Oxalate	10
9.	Selenate	10
10.	Phosphate	9
11.	Bromide	10
12.	Nitrate	6

where 1 mg/L = 1 ppm

Figure 3
Gradient Elution of Inorganic and Organic Anions

5.5 Trace Anions in 1.2% Boric Acid

Detection of trace level anions in 1.2% boric acid requires a short matrix elimination step after concentration. During the concentration step, the boric acid sample is pumped through the IonPac AC10 Trace Anion Concentrator Column. If this concentrated sample is injected immediately after concentration, a very large boric acid peak is produced due to the boric acid (200 mL) that fills the interstitial volume the AC10. However, if a small volume (2.5 mL) of ultrapure water is pumped through the AC10 immediately after concentration and before injection, the volume of boric acid in the interstitial volume of the AC10 is removed. This reduces quantification interference from a large boric acid peak with chloride and other early eluting anions.

Concentrator Column:	IonPac AC10 Concentrator Column
AC10 Injection Treatment:	2.5 mL Deionized Water
Volume Concentrated:	10 mL
Column:	IonPac AS10 4-mm Analytical
Eluent:	85 mM NaOH
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA(4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	≤ 8 µS

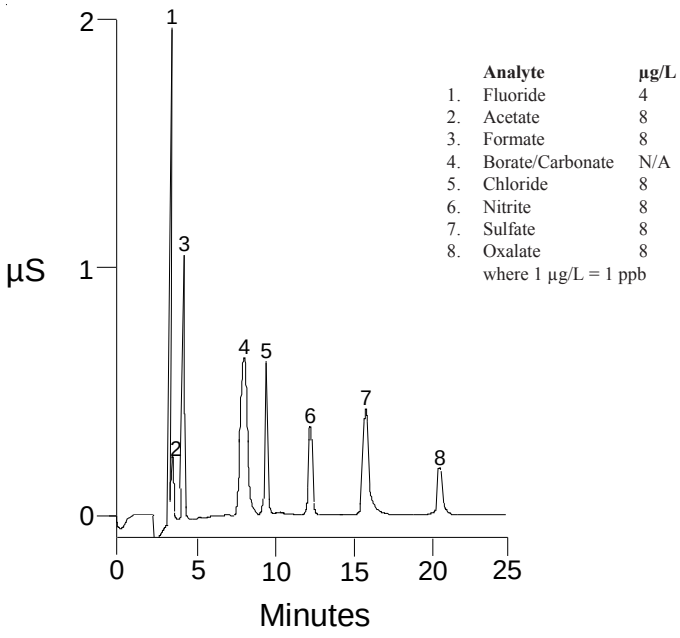


Figure 4
Trace Anions in 1.2% Boric Acid

5.6 Trace Anions in 20 mg/L Morpholine

In previous methodologies, the response factors for different matrices found in the power industry varied significantly. The IonPac AS10 Analytical Columns allow for instrument calibration using synthetic standards dissolved in deionized water and reliable subsequent analyses of a wide range of sample matrices. Similar chromatograms are obtained in ultrapure water and sample matrices containing up to 10 mg/L ammonium and 20 mg/L morpholine. Identical chromatograms can be obtained for sample matrices from 1 to 20 mg/L of morpholine. The detection of trace level anions in morpholine has been optimized using a sodium hydroxide eluent. This eluent allows the isocratic separation and quantification of mono-, di-, and trivalent anions in a single injection.

Concentrator Column:	IonPac AC10 Concentrator Column
Volume Concentrated:	10 mL
Column:	IonPac AS10 4-mm Analytical
Eluent:	85 mM NaOH
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA (4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected System Pressure:	2,000 psi (13.78 MPa)
Expected Background Conductivity:	≤ 8 µS

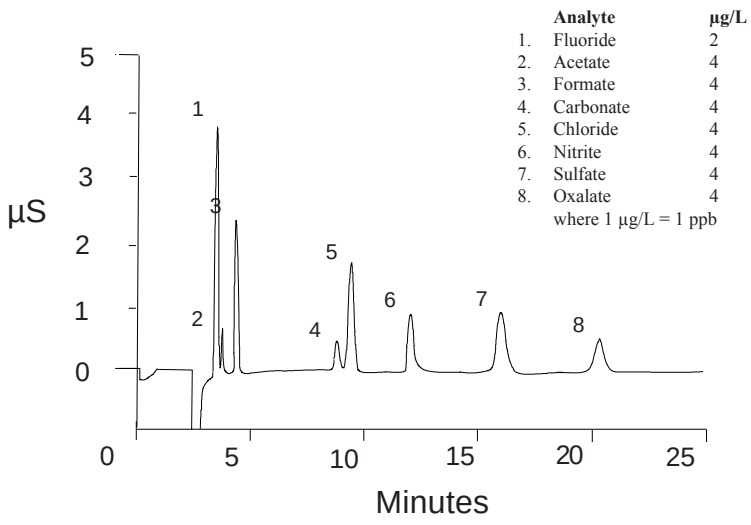


Figure 5
Trace Anions in 20 mg/L Morpholine

5.7 Trace Anions in 0.5 M Hydrochloric Acid

The IonPac AS10 2-mm Analytical Column is capable of monitoring trace level anions in concentrated solutions. Compared to the IonPac AS4A-SC 2-mm Analytical Column packing, the IonPac AS10 2-mm Analytical Column packing provides an eight fold increase in column capacity which allows for much higher sample loading. Additional eluent suppression capacity is obtained through the use of the 2-mm version of the ASRS-ULTRA. The reduced flow rates used in the 2-mm version of the IonPac AS10 allow for a maximum practical suppression capacity of 200 mM NaOH. The maximum practical suppression capacity of the 4-mm version of the IonPac AS10 Analytical Column is 150 mM NaOH. This increase in column capacity allows the analysis of trace anions in very concentrated matrices such as 0.5 M HCl.

Injection Volume:	10 µL
Column:	IonPac AS10 2-mm Analytical
Eluent:	E1: Deionized Water
	E2: 200 mM NaOH
Eluent Flow Rate:	0.25 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor -ULTRA (2-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected System Pressure:	2,000 psi (13.78 MPa)
Expected Background Conductivity:	≤ 8 µS

Gradient Conditions

Time	%E1	%E2	Comments
0.0	95	5	Inject, 20 mM NaOH
30.0	95	5	20 mM NaOH, Begin Gradient
45.0	0	100	200 mM NaOH, End Gradient

	Analyte	mg/L
1.	Fluoride	1.5
2.	Chloride	(0.5 M)
3.	Sulfate	5.0
4.	Phosphate	5.0
5.	Bromide	1.5
6.	Nitrate	3.0

where 1 mg/L = 1 ppm

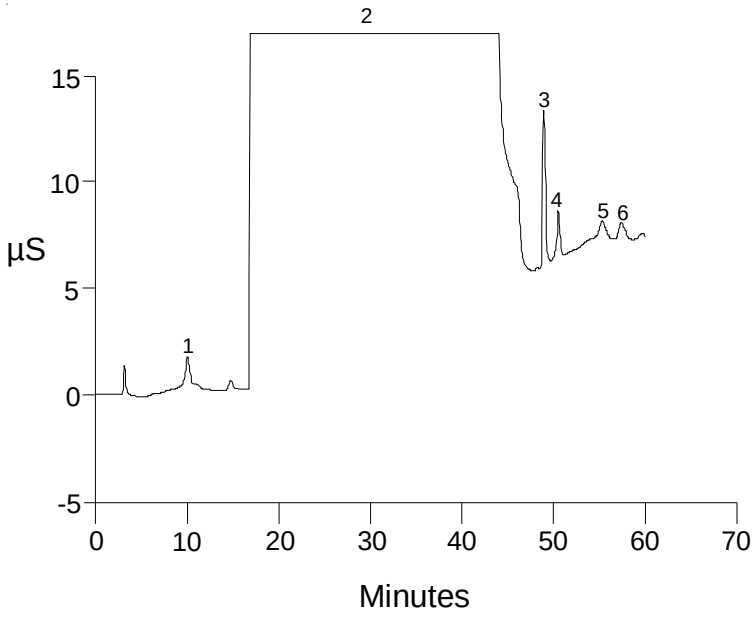


Figure 6
Trace Anions in 0.5 M HCl

5.8 Trace Anions in 5% Hydrofluoric Acid

The IonPac AC10 Concentrator Column is capable of concentrating trace level anions in hydrofluoric acid solutions. The trace anions can then be directly injected onto the analytical system from the AC10. Detection limits (MDL) of 25 to 50 µg/L (ppb) can be obtained for most trace anions in 5% hydrofluoric acid. For details on the concentration and analysis of trace anions in 5% hydrofluoric acid, see Application Note 78, "Determination of Trace Anions in Concentrated Hydrofluoric Acid."

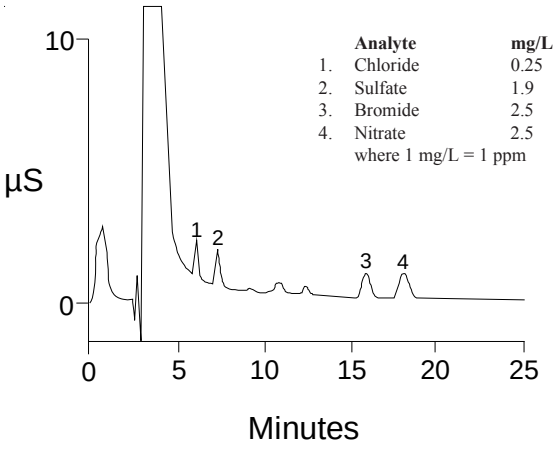
Injection Volume:	10 µL
Column:	IonPac AS10 2-mm Analytical
Eluent:	E1: Deionized Water
	E2: 400 mM NaOH
Rinsing Reagent:	70% v/v Methanol
Eluent Flow Rate:	0.25 mL/min (Gradient pump)
Rinsing Flow Rate:	1.0 mL/min (DQP sampling pump)
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA(2-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (2-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected System Pressure:	3,050 psi (21.03 MPa)
Expected Background Conductivity:	≤ 8 µS

Gradient Pump Program

Time (min)	%E1	%E2	V5*	V6*	Comments
0.0	75	25	0	0	Manual sample loop fill/AC10 rinse
2.5	75	25	1	0	Begin AC10 load/rinse
12.0 **	75	25	0	1	End AC10 load/rinse, begin analysis

*V5 = Injection Valve (Rheodyne)
V6 = Concentration Valve
where 1 = ON and 0 = OFF

**Begin data sampling at 12.0 minutes



	Analyte	mg/L
1.	Chloride	0.25
2.	Sulfate	1.9
3.	Bromide	2.5
4.	Nitrate	2.5

where 1 mg/L = 1 ppm

Figure 7
Trace Anions in 5% HF

5.9 Carboxylic Acids

Compared to the IonPac AS4A-SC (4-mm) Analytical Column, the IonPac AS10 4-mm Analytical Column provides an eight fold increase in column capacity which not only allows for the higher sample loading capacity which enhances the chromatographer's ability to separate and quantify analytes that are weakly retained on columns with lower column capacity. The following set of carboxylic acids are very weakly retained on the IonPac AS4A-SC (4-mm) Analytical Column but easily separated on the IonPac AS10 4-mm Analytical Column.

Injection Loop Volume:	10 µL
Column:	IonPac AS10 4-mm Analytical
Eluent:	3.5 mM $K_2B_4O_7$
Eluent Flow Rate:	1.0 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor-ULTRA(4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor III, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
Expected System Pressure:	2,500 psi (17.23 MPa)
Expected Background Conductivity:	≤ 5 µS

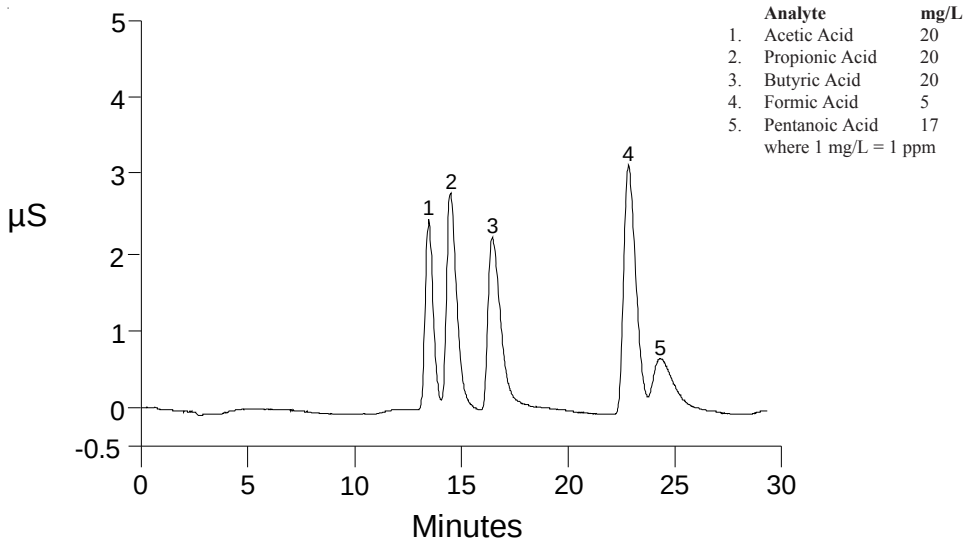


Figure 8
Carboxylic Acids

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using IonPac AS10 columns.

For more information on problems that originate with the Ion Chromatograph (IC) refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

Table 7
AS10/AG10 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports	6.1.2
	Other System Components	Disconnect, Replace	Component Manual
High Background Conductivity	Bad Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Columns	Clean Column	6.2.2, 6.2.3, Column Care
	Contaminated ASRS or AMMS	Clean Suppressor	6.2.5, Component Manual
	Contaminated Hardware	Clean Component	6.2.4, Component Manual
Poor Resolution	Unequilibrated System	Lengthen First Eluent Time before Inject	
	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.1.A, Component Manual
	Column Headspace	Replace Column	6.3.1.B
Short Retention Times	Flow Rate Too Fast	Recalibrate Pump	6.3.2.A
	Bad Eluents	Remake Eluents	6.3.2.B
Poor Front End Resolution	Column Contamination	Clean Column	6.3.2.C, 6.3.2.D, Column Care
	Bad Eluents	Remake Eluents	6.3.3.A
	Column Overloading	Reduce Sample Size	6.3.3.B, 3.4.1, 3.4.2
	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 Contamination	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 AG10 (4-mm) Guard Column plus the AS10 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,000 psi (13.78 MPa). If the system pressure is higher than 3,200 psi (22.05 MPa), 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 (0.34 MPa). Continue adding system components (injection valve, column(s), suppressor and detector) one by one, while monitoring the system pressure. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 8, "Typical AS10/AG10 Operating Back Pressures").

The Anion Self-Regenerating Suppressor-I 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 8
Typical AS10/AG10 Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min
AS10 4-mm Analytical	≤ 2,150 (14.82)	1.0
AG10 4-mm Guard	≤ 700 (4.82)	1.0
AC10 4-mm Concentrator	≤ 300 (2.07)	1.0
AS10 + AG10 + AC10 4-mm columns	**≤ 3,150 (21.71)**	**1.0**
AS10 2-mm Analytical	≤ 2,150 (14.82)	0.25
AG10 2-mm Guard	≤ 600 (4.14)	0.25
AC10 2-mm Concentrator	≤ 300 (2.07)	0.25
AS10 + AG10 + AC10 2-mm columns	**≤ 3,050 (21.03)**	**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. Be sure to filter DI water used for eluents before use.

A. Disconnect the column from the system.

B. Using two open end wrenches, carefully unscrew the inlet (top) column fitting.

C. 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 AS10 4-mm Columns (P/N)	IonPac AS10 2-mm Columns (P/N)
Analytical Column	043118	043123
Guard Column	043119	043124
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.

6.2 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
80 - 200 mM NaOH	5 - 8 µS

6.2.1 Preparation of Eluents

A. Make sure that the eluents and the regenerant 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 Guard or Analytical Column

Remove the IonPac AG10 Guard and AS10 Analytical Columns from the system. If the background conductivity decreases, the column(s) is (are) the cause of the high background conductivity. Clean or replace the AG10 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" in "Column Care."

6.2.3 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.

CAUTION
Sparge the DI water prior to NaOH preparation to minimize carbonate contamination.

6.2.4 Contaminated Hardware

To eliminate the hardware as the source of the high background conductivity, bypass the columns and the Anion Self-Regenerating Suppressor. 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 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-ULTRA Product Manual (Document No. 031367). For details on Anion Membrane Suppressor 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. 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.

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

6.3.2 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. Ensure that 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. If this corrects the problem, service the proportioning valve.

C. Column contamination can lead to a loss of column capacity. When contaminated, all of the anion exchange sites in the column 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" in "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. Always use a guard column to protect the analytical column.

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 Section").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminants should be eluted from the column. If you need assistance in solving resolution problems, contact the 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. Enusre that the DI water and 50% sodium hydroxide used for the eluent are free of carbonate contamination.

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.

6.3.4 Spurious Peaks

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

B. If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS10 columns, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices").

C. A baseline disturbance may occur when an injection valve is actuated. 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. Often a peak or baseline upset will be seen before the void volume when concentrating samples. This is due to pressure differences when switching from load to inject with a concentrator column in-line.

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

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 AS10 columns is 3,500 psi (24.12 MPa).

COLUMN START-UP

The column is shipped in 100 mM Sodium Borate 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 with eluents containing less than 50 mM hydroxide and less than 5% solvents to reduce the solvent content of the eluent to < 5% levels and the ionic strength of the eluent to < 50 mM levels. This will help avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

CHOOSING THE APPROPRIATE CLEANUP SOLUTION

CAUTION

Choose a cleanup solution that has a high ionic component such as 200 mM HCl/90% acetonitrile.

A. Hydrophilic ionic contamination of low valency

1. 200 mM NaOH

B. High valency hydrophilic ions by ion suppression and elution by the chloride ion

1. 200 mM HCl

C. High valency hydrophobic ions

1. 200 mM HCl in 80% acetonitrile

The acid suppresses ionization and subsequent ion exchange interactions of the ionic contamination with the resin and the organic solvent then elutes the nonionic hydrophobic contamination. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage. See also Table 5, "HPLC Solvents for Use with IonPac AS10 Columns."

D. Metal contamination

1. 0.1 M oxalic acid.

Iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

E. Regardless of the cleanup solution chosen, use the following cleanup procedure in Section 7.4.2, "Column Cleanup Procedure," to clean the AG10 and AS10

COLUMN CLEANUP PROCEDURE

A. Prepare a 500 mL solution of the appropriate cleanup solution using the guidelines in Section 7.4.1, "Choosing the Appropriate Cleanup Solution."

B. Disconnect the ASRS-ULTRA or AMMS III from the IonPac AS10 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 AS10 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for an AS10 2-mm Analytical or Guard Column.

D. Rinse the column for 15 minutes with deionized water before pumping the chosen cleanup solution over the column.

E. Pump the cleanup solution through the column for 1 to 2 hours.

F. Rinse the column for 15 minutes with deionized water before pumping eluent over the column.

G. Equilibrate the column(s) with eluent before resuming normal operation for at least 30 minutes.

H. Reconnect the ASRS-ULTRA or AMMS III to the AS10 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.