Thermo Scientific

Dionex IonPac AS26

Column Product Manual

P/N: 065444-03 June 2013

Thermo SCIENTIFIC

Product Manual

for

Thermo Scientific Dionex IonPac™ AG26 Guard Column

4 x 50 mm (P/N 076021)
2 x 50 mm (P/N 076023)

Thermo Scientific Dionex IonPac AG26 Capillary Guard Column

0.4 x 50 mm (P/N: 076019)

Thermo Scientific Dionex IonPac AS26 Analytical Column

4 x 250 mm (P/N 076020)
2 x 250 mm (P/N 076022)

Thermo Scientific Dionex IonPac AS26 Capillary Column

0.4 x 250 mm (P/N 076018)

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SECTION 1 – INTRODUCTION

The Thermo Scientific Dionex IonPac AS26 Analytical Column is a high capacity, hydroxide selective anion exchange column specially designed for the separation of 9 haloacetic acids (HAAs) including monochloro acetic acid (MCAA), dichloro acetic acid (DCAA), trichloro acetic acid (TCAA), monobromo acetic acid (MBAA), dibromo acetic acid (DBAA), tribromo acetic acid (TBAA), bromochloro acetic acid (BCAA), dibromochloro acetic acid (DBCAA) and dichlorobromo acetic acid (DCBAA) in drinking water matrices. This column is also suitable for the analysis of inorganic anions and oxyhalides, such as chlorite, bromate and chlorate. The Dionex IonPac AS26 column is available in 0.4 x 250 mm, 2 x 250 mm and 4 x 250 mm formats thus supporting a range of operating flow rates from 0.010 to 2.0 ml/min.

The Dionex IonPac AS26 column has a capacity of 250 µeq/column (4 x 250 mm). The high capacity of this column allows for direct large loop injections of complex samples without overloading the column. The Dionex IonPac AS26 column is also compatible with pH 0 – 14 eluents and eluents containing organic solvents.

The Dionex IonPac AS26 Capillary Column (0.4 x 250 mm) is packed with the same material as the equivalent standard bore version (producing the same performance as a 4 mm column), but requires only one-hundredth (1/100) the eluent flow rate. The capillary format has the advantage of less eluent consumption and waste generation, thus resulting in reduced operational costs.

Table 1
Dionex IonPac AS26/AG26 Packing Specification

Column	Particle Diameter µm	Substrate X-linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
Dionex IonPac AS26* 4 x 250 mm	7.5	55	250	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG26** 4 x 50 mm	11	55	6	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS26* 2 x 250 mm	7.5	55	62.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG26** 2 x 50mm	11	55	1.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AS26* 0.4 x 250 mm	7.5	55	2.5	Alkanol quaternary ammonium	Ultralow
Dionex IonPac AG26** 0.4 x 250 mm	11	55	0.06	Alkanol quaternary ammonium	Ultralow

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

Table 2
Dionex IonPac AS26/AG26 Operating Parameters

Column	Typical Back Pressure psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
Dionex IonPac AS26 4-mm Analytical	< 1,800 (12.41)	1.0	2.0
Dionex IonPac AG26 4-mm Guard	< 300 (2.07)	1.0	2.0
Dionex IonPac AS26 and AG26 4-mm columns	< 2,100 (14.48)	1.0	2.0
Dionex IonPac AS26 2-mm Analytical	< 1,800 (12.41)	0.25	0.5
Dionex IonPac AG26 2-mm Guard	< 300 (2.07)	0.25	0.5
Dionex IonPac AS26 and AG26 2-mm columns	< 2,100 (14.48)	0.25	0.5
Dionex IonPac AS26 0.4-mm Capillary	< 1,800 (12.41)	0.010	0.020
Dionex IonPac AG26 0.4-mm Capillary Guard	< 300 (2.07)	0.010	0.020
Dionex IonPac AS26 and AG26 0.4-mm Capillary and Capillary Guard	< 2,100 (14.48)	0.010	0.020

For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

SECTION 2 – ION CHROMATOGRAPHY SYSTEMS

The proper configuration of an Ion Chromatography System (ICS) in 2-mm or 4-mm format is based on the ratio of the 2-mm to 4-mm column cross-sectional area (a factor of 1/4). The selected format will affect the type of pump recommended. A gradient pump is designed to blend and pump isocratic, linear, or gradient mixtures of up to four mobile phase components at precisely controlled flow rates. An isocratic pump is for applications not requiring gradient and multi-eluent proportioning capabilities. Both are offered in either standard bore or microbore options.

- For an ICS in 2-mm format, a microbore isocratic pump, standard bore isocratic pump, microbore gradient pump, or standard bore gradient pump is recommended.
- For an ICS in 4-mm format, a standard bore isocratic pump or standard bore gradient pump is recommended.
- For an ICS in 0.4 mm format, a Capillary IC system such as the Thermo Scientific Dionex ICS-5000 system is recommended.

See Appendix B, "Configuration" for specific recommended settings and parts including pumps, eluent flow rate, Thermo Scientific Dionex Self-Regenerating Suppressor (SRS), Thermo Scientific Dionex MicroMembrane Suppressor (MMS), Thermo Scientific Dionex Capillary Electrolytic Suppressor (CES), injection loop, system void volume, detectors, and tubing back pressure.

NOTE ***Do not operate suppressors over 40 °C. If application requires a higher temperature, place the suppressor outside of the chromatographic oven. Use of a Thermo Scientific Dionex EG with a Thermo Scientific Dionex EGC III KOH (P/N 074532) cartridge for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***

SECTION 3 – INSTALLATION

3.1. System Requirements

3.1.1. System Requirements for 0.4 mm Operation

The Dionex IonPac AS26 0.4 mm Capillary Guard and Capillary Column are designed to be run on a capillary ion chromatograph system equipped with suppressed conductivity detection. It is recommended to run the capillary column only on the Thermo Scientific Dionex ICS-5000 capillary system for best performance. Refer to section 3.1.5 for temperature requirement for haloacetic acid analysis.

3.1.2. System Requirements for 2-mm Operation

The Dionex IonPac AS26 2-mm Guard and Analytical Columns are designed to run on Thermo Scientific Dionex Ion Chromatographs equipped with suppressed conductivity detection. Isocratic analyses at flow rates of 0.5 mL/min can be performed on a pump with standard (1/8" pistons) pump heads. For isocratic analyses at flow rates below 0.5 mL/min and gradient analyses, a microbore pump (1/16" pistons) is recommended. Refer to section 3.1.5 for temperature requirement for haloacetic acid analysis.

3.1.3. System Requirements for 4-mm Operation

The Dionex IonPac AS26 4-mm Guard and Analytical Columns are designed to run on any Dionex Ion Chromatograph equipped with suppressed conductivity detection. Gradient methods and methods requiring solvent containing eluents should be performed on a system having a pump with a standard pump heads (1/8" pistons). Isocratic analysis can also be performed on a pump with standard bore pump heads (1/8" pistons). Refer to section 3.1.5 for temperature requirement for haloacetic acid analysis.

3.1.4. System Requirement for two-dimensional Matrix-Elimination Ion Chromatography

The application of the two-dimensional Matrix-Elimination Ion Chromatography for the haloacetic acid analysis can greatly enhance the detection limit. It is recommended that a hybrid Dionex ICS 5000 system with a 4 mm format in the 1st dimension and a capillary format in the 2nd dimension. Refer to section 3.1.5 for temperature requirement for haloacetic acid analysis.

3.1.5. Temperature Control Requirement for Haloacetic Acid Analysis

Monobromoacetic acid (MBAA), chlorodibromoacetic acid (CDBAA) and tribromoacetic acid (TBAA) degrade readily at high pH. The reaction is temperature dependent. To minimize the sample degradation, the separation is performed at subambient temperature, specifically at 15^{o}C. It is recommended to run the analysis on the Dionex ICS-5000 system. A refrigerated autosampler capable of maintaining samples at a temperature of less than or equal to 10^{o}C is also recommended.

3.1.6. System Void Volume

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

3.2. The Sample Concentrator

The Thermo Scientific Dionex Trace Anion Concentrator Low Pressure Column (Dionex TAC-LP1, P/N 046026), the Thermo Scientific Dionex Trace Anion Concentrator Ultra Low Pressure Column (Dionex TAC-ULP1, P/N 061400), the Thermo Scientific Dionex Ultra Trace Anion Concentrator Low Pressure Column (Dionex UTAC-LP1, P/N 063079) or (Dionex UTACLP2, P/N 072779), the Thermo Scientific Dionex Ultra Trace Anion Concentrator Ultra Low Pressure Column (Dionex UTAC-ULP1, P/N 063475) or (Dionex UTAC-ULP2, P/N 072780), the Thermo Scientific Dionex Ultra Trace Anion Concentrator Extremely Low Pressure Column (Dionex UTAC-XLP1, P/N 063459) or (Dionex UTAC-XLP2, P/N 072781), or the Dionex IonPac AG26 Guard Column can be used for trace anion concentration work with the 2 mm and 4 mm Dionex IonPac AS26 columns. For trace anion concentration work with the 0.4 mm Dionex IonPac AS26 column, use the Thermo Scientific Dionex IonSwift MAC-100 or Dionex IonPac AG26 Capillary Guard Column. The function of a concentrator column in these applications is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process "concentrates" the desired analyte species onto the concentrator column, lowering detection limits by 2-5 orders of magnitude. The concentrator column is used in lieu of the sample loop. For 2D-IC applications with the capillary column in the second dimension, use the Thermo Scientific Dionex IonSwift MAC-200 Concentrator Column.

Pump the sample onto the concentrator column in the OPPOSITE direction of the eluent flow. When using concentration techniques; do not overload the concentrator column by concentrating an excessive amount of sample. Concentrating an excessive amount of sample can result in inaccurate results being obtained. It is possible during the concentration step for the polyvalent anions such as phosphate and sulfate to elute the weakly retained anions such as fluoride and acetate off the concentrator column. For more detailed information on sample concentration techniques for high sensitivity work and a detailed discussion of anion concentration techniques refer to:

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

Dionex IonPac Trace Anion Concentrator Column, Dionex TAC-2 (P/N 043101), is not optimized for use with hydroxide eluents and should not be used for concentrator work with the Dionex IonPac AS26.

CAUTION

3.3. The Injection Loop

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

For most applications on a 2-mm analytical system, a 2 - 15 µL injection loop is sufficient. Generally, do 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 Dionex IonPac AS26 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.

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, do not inject more than 50 nanomoles of any one analyte onto the 4-mm analytical column. Injecting larger number of moles of a sample can result in overloading the column which can affect the detection linearity. For low concentrations of analytes, larger injection loops can be used to increase sensitivity.

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

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

3.4. The Dionex IonPac AG26 Guard Column

A Dionex IonPac AG26 Guard Column is normally used with the Dionex IonPac AS26 Analytical Column. Retention times will increase by approximately 4% 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 Dionex IonPac AG26 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the Dionex IonPac AS26 Analytical Column.

3.5. Thermo Scientific Dionex CR-ATC Trap Column Installation with Dionex EGC

For Dionex IonPac AS26 applications using the Dionex EGC KOH cartridge, a Dionex CR-ATC Continuously Regenerated Trap Column (P/N 060477 or 072078) should be installed at the Dionex EGC eluent outlet to remove trace level anionic contaminants from the carrier deionized water. See the Dionex CR-TC Product Manual (Document No. 031910) for instructions. As an alternative, the Thermo Scientific Dionex ATC-HC Trap Column (P/N 059604) should be installed between the pump outlet and the inlet of the Dionex EluGen Cartridge in the Module to remove anionic contaminants from the carrier deionized water. See the Dionex ATC-HC Product Manual (Document No. 032697) for instructions.

If the lower capacity Thermo Scientific Dionex ATC-3 Trap Column (P/N 059660 and 059661) is used, it should be installed between the gradient pump and the injection valve to remove anionic contaminants from the eluent. The Dionex ATC-3 column is used when performing sodium hydroxide gradient anion exchange applications using hand-prepared bottled eluents. See the Dionex ATC-3 Product Manual (Document No. 032697) for instructions.

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

3.6. Eluent Storage

The Dionex IonPac AS26 column is designed to be used with hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance. Nitrogen can be used if eluents do not contain solvents.

3.7. Thermo Scientific Dionex Anion Self-Regenerating Suppressor Requirements

A Dionex 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 Thermo Scientific Dionex ASRS 300 modes of operation.

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

CAUTION

For Dionex IonPac AS26 4-mm Analytical Column, use the Dionex ASRS 300 4-mm (P/N 061561).
For Dionex IonPac AS26 2-mm Analytical Column, use the Dionex ASRS 300 2-mm (P/N 061562).
For Dionex IonPac AS26 0.4-mm Capillary Column, use the Dionex Thermo Scientific Dionex ACES 300 0.4-mm (P/N 072052).

For detailed information on the operation of the Dionex Anion Self-Regenerating Suppressor 300, see the Product Manual for the Dionex ASRS 300 (Document No. 031956).
For detailed information on the operation of the Thermo Scientific Dionex Anion Capillary Electrolytic Suppressor 300, see the Product Manual for the Dionex ACES 300 (Document No. 065386).

3.8. Thermo Scientific Dionex Anion MicroMembrane Suppressor Requirements

A Dionex Anion MicroMembrane Suppressor (Dionex AMMS 300) may be used instead of a Dionex ASRS 300 4-mm for applications that require suppressed conductivity detection. Use a Dionex AMMS 300 4-mm (P/N 064558) with the Dionex IonPac AS26 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 Dionex AMMS 300 2-mm (P/N 064559).

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

3.9. Using Thermo Scientific Dionex Displacement Chemical Regeneration (DCR) in the Chemical Suppression Mode

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

Use proper safety precautions in handling acids and bases.

SAFETY

3.10. Detector Requirements

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

3.11. Using the Dionex EGC-KOH with the Dionex IonPac AS26

The Dionex IonPac AS26 column is recommended for use with Thermo Scientific Dionex ICS-2100, or Thermo Scientific Dionex ICS-5000 IC Systems equipped with a Dionex Eluent Generator. The Dionex Eluent Generator is used to automatically produce potassium hydroxide gradients from deionized water. The Dionex IonPac AS26 can be used with older Dionex IC Systems equipped with an Eluent Generator or a Thermo Scientific Dionex RFC-30 Reagent Free Controller. Please refer to the Dionex EG40 manual, Document No. 031373, for information on the operation of the Dionex EG40. Please refer to the Thermo Scientific Dionex EG50 Product Manual, Document No. 031908, for information on the operation of the Dionex EG50.

3.12. Detector Requirements

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

3.13. Installation of the Capillary Column

1. Before installing the new separator column, cut off the column label and slide it into the holder on the front of the cartridge (see Figure 6).
2. For reference, Figure 1 shows the column cartridge after installation of both a capillary guard column and a capillary separator column. Figure 2 shows the column cartridge after installation of only a capillary separator column.

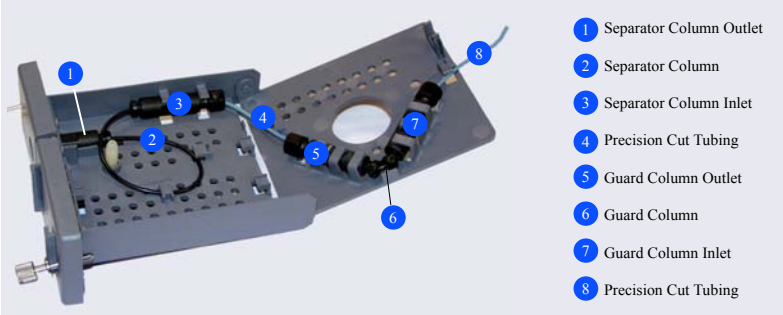


Figure 1
Separator and Guard Columns Installed in Column Cartridge

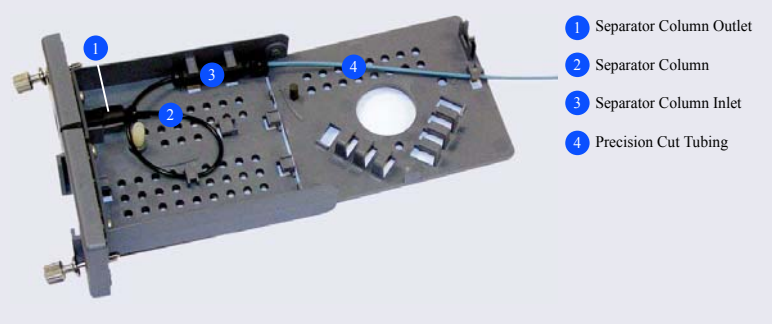


Figure 2
Separator Column Only Installed in Column Cartridge

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

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

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

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

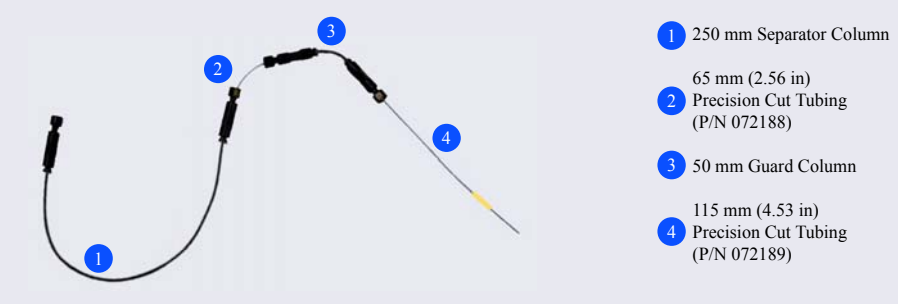


Figure 3
Tubing Connections for 250-mm Separator Column and 50-mm Guard Column

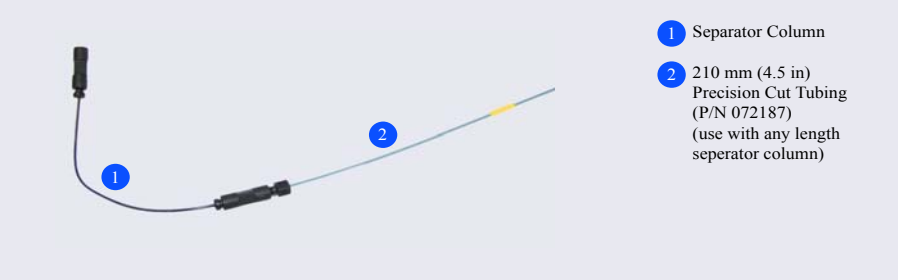


Figure 4
Tubing Connections for Separator Column Only

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

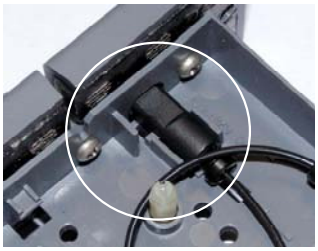


Figure 5
Column Outlet Fitting Installed in Column Cartridge

7. Coil the separator column tubing inside the cartridge as shown in Figure 1 or Figure 2. Secure the column tubing and the inlet fitting in the clips on the column cartridge.
8. Secure the inlet and outlet fittings on the guard column (if used) in the column clips on the lid of the column cartridge.
9. Route the guard column inlet tubing (if used) or the separator column inlet tubing through the clip on the top edge of the column cartridge lid.
10. Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see Figure 6).

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

Figure 6
Column Cartridge Closed

SECTION 4 – OPERATION

4.1. General Operating Conditions

Sample Volume:	2-mm:	2.5 µL Loop + 0.8 µL Injection valve dead volume
	4-mm:	10 µL Loop + 0.8 µL Injection valve dead volume
	0.4-mm:	0.4 µL Loop
Column:	2-mm:	Dionex IonPac AS26 2-mm Analytical Column + Dionex IonPac AG26 2-mm Guard Column
	4-mm:	Dionex IonPac AS26 4-mm Analytical Column + Dionex IonPac AG26 4-mm Guard Column
	0.4-mm:	Dionex IonPac AS26 0.4-mm Capillary Column + Dionex IonPac AG26 0.4-mm Capillary Guard Column
Eluent:	55 mM KOH	
Eluent Source:	Dionex EGC III KOH	
Eluent Flow Rate:	2-mm:	0.30 mL/min
	4-mm:	1.20 mL/min
	0.4-mm:	0.012 mL/min
Temperature:	15°C	
Dionex SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2-mm or 4-mm) Dionex Anion Capillary Electrolytic Suppressor, Dionex ACES 300 (0.4-mm) AutoSuppression Recycle Mode	
or Dionex MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS 300 (2-mm or 4-mm)	
Dionex MMS Regenerant:	50 mN H_2SO_4	
Expected Background Conductivity:	< 1 µS	
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate	
Short-term Storage Solution (< 1 week):	Eluent	

4.2. Dionex IonPac AS26 Operation Precautions

CAUTION

Operate below 3,000 psi (20.68 MPa).
Filter and Degas Eluents.
Filter Samples.
Eluent pH between 0 and 14.
Sample pH between 0 and 14.
0.020 mL/min maximum flow rate for 0.4-mm columns.
0.5 mL/min maximum flow rate for 2-mm columns.
2.0 mL/min maximum flow rate for 4-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 particle levels in eluents also help to protect your ion exchange columns and system components. Thermo Scientific 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 Dionex IonPac AS26 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 Thermo Scientific, we have obtained consistent results using 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 maximum back pressure limit for the Dionex IonPac AS26 columns is 3,000 psi (20.68 MPa).

The Dionex IonPac AS26 can withstand common HPLC solvents in a concentration range of 0-100%. Solvents and water should be premixed in concentrations which allow proper mixing by the gradient pump and to minimize outgassing. Ensure that all of the inorganic chemicals are soluble in the highest solvent concentration to be used during the analysis.

Table 4
HPLC Solvents for Use with Dionex IonPac AS26 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 Dionex ASRS 300 and the Dionex ACES 300 must be operated in the AutoSuppression External Water Mode when using eluents containing solvents. Do not use > 40% solvent on the Dionex ASRS 300 and the Dionex ACES 300 in the electrolytic mode (power on).***

4.4. Making Eluents that Contain Solvents

Remember to mix on a volume to volume basis when mixing solvents with water. 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 v/v eluent is obtained. Premixing water with solvent will minimize the possibility of out gassing.

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

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

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

SAFETY ***Never add the acetonitrile directly to the basic carbonate or hydroxide eluent Solutions.***

4.5. Eluent Preparation

4.5.1. Sodium Hydroxide Eluent Concentration

4.5.1.1. Weight Method

When formulating eluents from 50% sodium hydroxide, it is recommended to weigh out the required amount of 50% sodium hydroxide. Use Fisher Grade 50% sodium hydroxide. Do not use pellets.

Example: To make 1 L of 55 mM NaOH use 4.40 g of 50% sodium hydroxide:

$$\textbf{For 55 mM:} \quad \frac{\textbf{0.055 mole/L x 40.00 g/mole}}{\textbf{50\%}} = \textbf{4.40 g diluted to 1 L}$$

4.5.1.2. Volume Method

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

$g = dvr$

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

Example: To make 1 L of 55 mM NaOH use 2.88 mL of 50% sodium hydroxide:

$$\textbf{For 55 mM:} \quad \frac{\textbf{0.055 mole/L x 40.00 g/mole}}{\textbf{50\% x 1.53 g/mL}} = \textbf{2.88 mL diluted to 1 L}$$

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

4.5.1.3. Sodium Hydroxide Eluents

Dilute the amount of 50% (w/w) NaOH (in water) specified in Table 5, "Dilution of 50% (w/w) NaOH to make standard Dionex IonPac AS26 eluents" with degassed, deionized water (with a specific resistance of 18.2 megohm-cm) to a final volume of 1,000 mL using a volumetric flask. Avoid the introduction of carbon dioxide from the air into the aliquot of 50% (w/w) NaOH or the deionized water being used to make the eluent. Do not shake the 50% (w/w) NaOH or pipette the required aliquot from the top of the solution where sodium carbonate may have formed.

Table 5
Dilution of 50% (w/w) NaOH to Make Standard Dionex IonPac AS26 Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
2.8 (1.83)	35
4.4 (2.89)	55
8.00 (5.25)	100
160.00 (104.6)	2 M

4.6. Regenerant Preparation for the Dionex AMMS 300

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

SECTION 5 – EXAMPLE APPLICATIONS

5.1. Recommendations for Optimum System Performance

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

The Dionex IonPac AS26 is designed for the determination of haloacetic acid anions and inorganic anions using eluent delivered with a Dionex EGC KOH cartridge. Resolution of specific analytes can be further optimized if necessary by using gradient elution and adjusting the oven temperature. 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 potassium hydroxide is converted to water in the suppressor, it is the best choice for an eluent. As long as the capacity of the suppressor is not exceeded, the eluent hydroxide concentration has little effect on background conductivity. For example, a gradient run could begin at 1.0 mM KOH and end at 80 mM KOH, with a resulting total baseline change of 1 to 2 µS.

5.2. Effect of Temperature on Analyte Stability

Monobromoacetic acid (MBAA), chlorodibromoacetic acid (CDBAA) and tribromoacetic acid (TBAA) degrade readily at high pH. The reaction is temperature dependent. To minimize the sample degradation, the separation is performed at subambient temperature, specifically at 15°C. A refrigerated autosampler capable of maintaining samples at a temperature of less than or equal to 10°C is also recommended.

5.2.1. Downward Shift in Baseline

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.

5.2.2. Increase the Sensitivity

You can increase the sensitivity of your system by using sample concentration techniques (see Section 3).

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

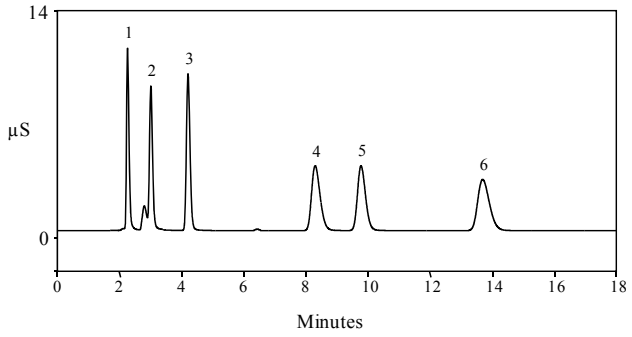
5.3. Isocratic Elution of Inorganic Anions using the Dionex IonPac AS26 Column with and without the Dionex IonPac AG26 Guard Column

Isocratic elution of inorganic anions on the Dionex IonPac AS26 Analytical Column has been optimized utilizing a hydroxide eluent. By using this eluent, mono- and divalent anions can be isocratically separated and quantitated in a single injection. The Dionex IonPac AS26 Analytical Column should always be used with the Dionex IonPac AG26 Guard Column. Note that the Dionex IonPac AG26 Guard is packed with a microporous resin of proportionally lower capacity and contributes approximately 4 % increase in retention times when a guard column is placed in-line prior to the analytical column. Refer to section 3.1.5 for temperature requirement for haloacetic acid analysis.

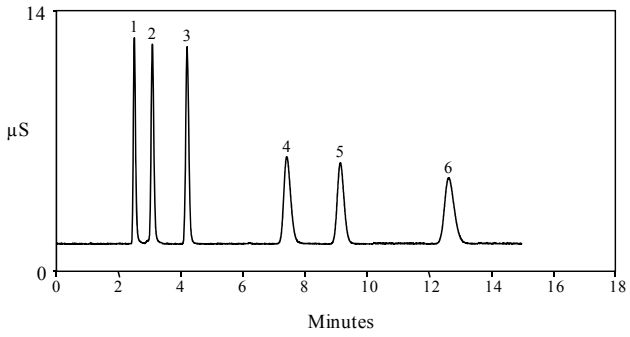
Sample Volume:	2-mm: 2.5 µL Loop + 0.8 µL Injection valve dead volume 4-mm: 10 µL Loop + 0.8 µL Injection valve dead volume
Column:	See chromatogram
Eluent:	55 mM KOH
Eluent Flow Rate:	0.30 mL/min (2-mm), 1.2 mL/min (4-mm)
Operating Temperature:	15°C
Dionex SRS Suppressor:	Dionex Anion Self-Regenerating Suppressor, Dionex ASRS 300 (2-mm or 4-mm) AutoSuppression Recycle Mode
or Dionex MMS Suppressor:	Dionex Anion MicroMembrane Suppressor, Dionex AMMS 300 (2-mm or 4-mm)
Dionex MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	< 3 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

Peaks	mg/L
1. Fluoride	5
2. Sulfate	10
3. Chloride	10
4. Thiosulfate	20
5. Bromide	20
6. Nitrate	20

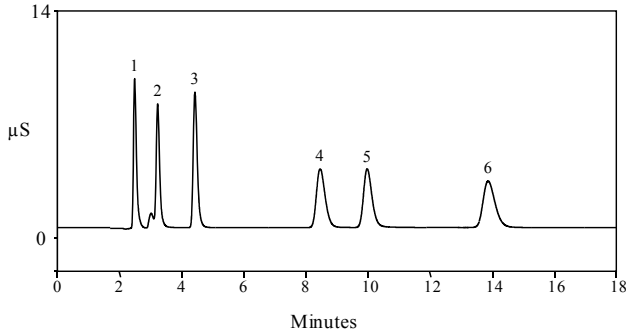
Dionex IonPac AS26 4-mm Only



Dionex IonPac AS26 2-mm Only



Dionex IonPac AS26 4-mm and AG26 4-mm



Dionex IonPac AS26 2-mm and AG26 2-mm

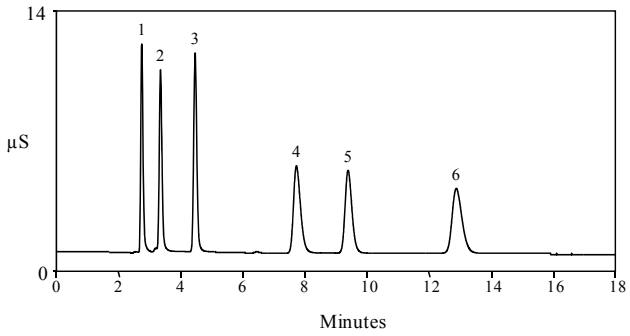


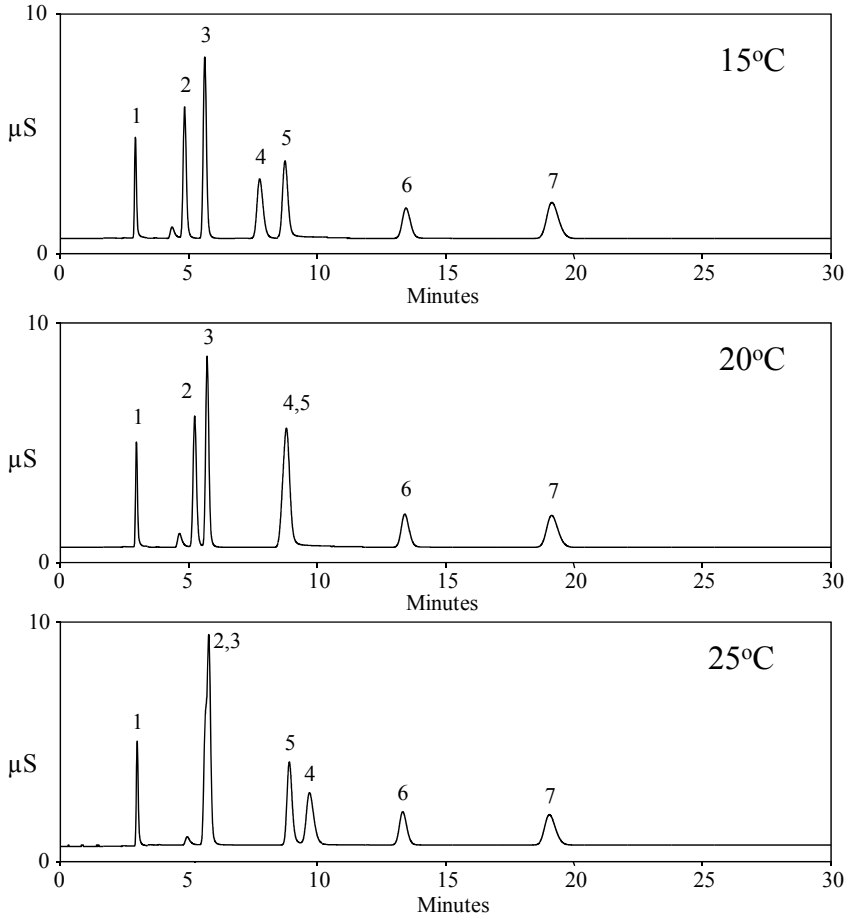
Figure 7
Production Test Chromatograms shown with and without the Guard Column

5.4. Effect of Temperature on Selectivity

The following chromatograms show the effect of temperature on the selectivity of Dionex IonPac AS26 column. At higher temperatures (20 °C and 25 °C) the resolution of sulfate and chloride is decreased and the phosphate and nitrite pair reverse order. Because of this sensitivity to temperature changes, it is important to verify the oven temperature and to adjust it, if needed, to obtain the optimal resolution.

For haloacetic analysis, the effect of temperature on analyte stability should also be considered. Monobromoacetic acid (MBAA), chlorodibromoacetic acid (CDBAA) and tribromoacetic acid (TBAA) degrade readily at high pH. The reaction is temperature dependent. To minimize the sample degradation, the separation is performed at sub-ambient temperature, specifically at 15 °C.

Sample Volume:	2.5 µL
Column:	Dionex IonPac AS26 Analytical, 2-mm
Eluent:	35 mM KOH
Eluent Flow Rate:	0.3 mL/min
Operating Temperature:	A) 15°C B) 20°C C) 25°C
Detection:	Suppressed Conductivity, Dionex ASRS 300 2-mm AutoSuppression, recycle Mode



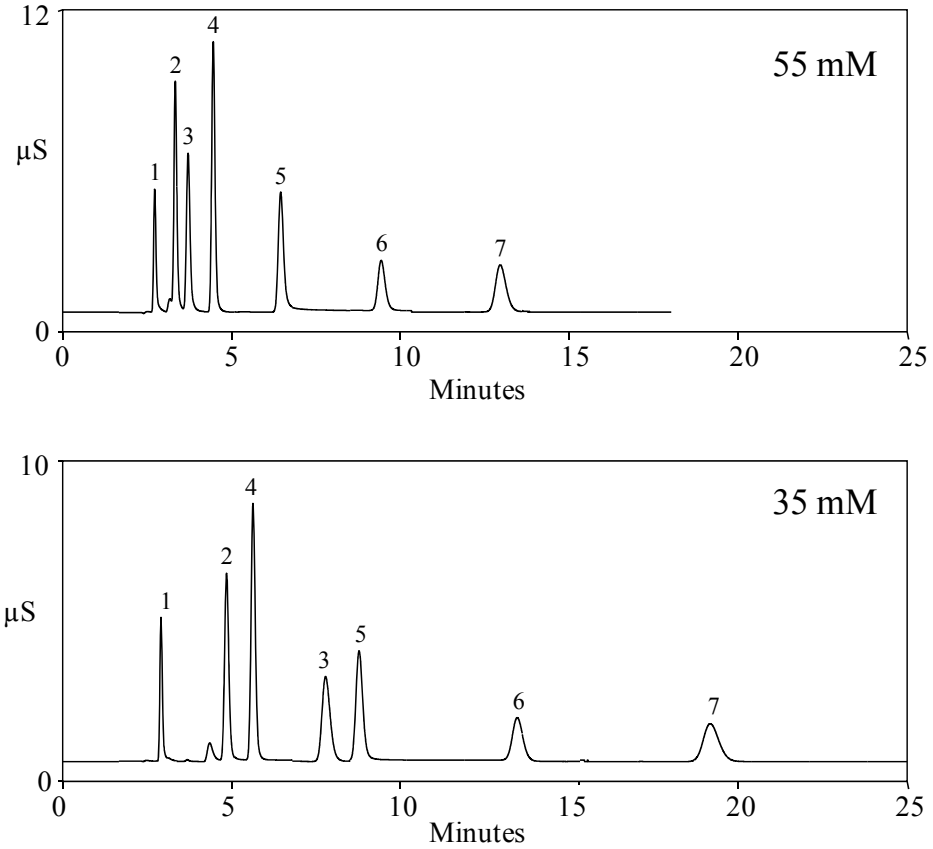
Peaks		mg/L
1.	Fluoride	2
2.	Sulfate	15
3.	Chloride	3
4.	Phosphate	15
5.	Nitrite	10
6.	Bromide	10
7.	Nitrate	10

Figure 8
Effect of Temperature on selectivity

5.5. Effect of Eluent Concentration on Selectivity

The following chromatograms show the effect of eluent concentration on the selectivity of Dionex IonPac AS26 column. At the lower eluent concentration of 35 mM, the phosphate and chloride elute in reverse order compared to the elution order when using the standard eluent concentration of 55 mM.

Sample Volume:	2.5 µL
Column:	Dionex IonPac AS26 Analytical, 2-mm
Eluent:	A: 55 mM KOH
	B: 35 mM KOH
Eluent Flow Rate:	0.3 mL/min
Operating Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex ASRS 300 (2-mm)
	AutoSuppression, recycle Mode



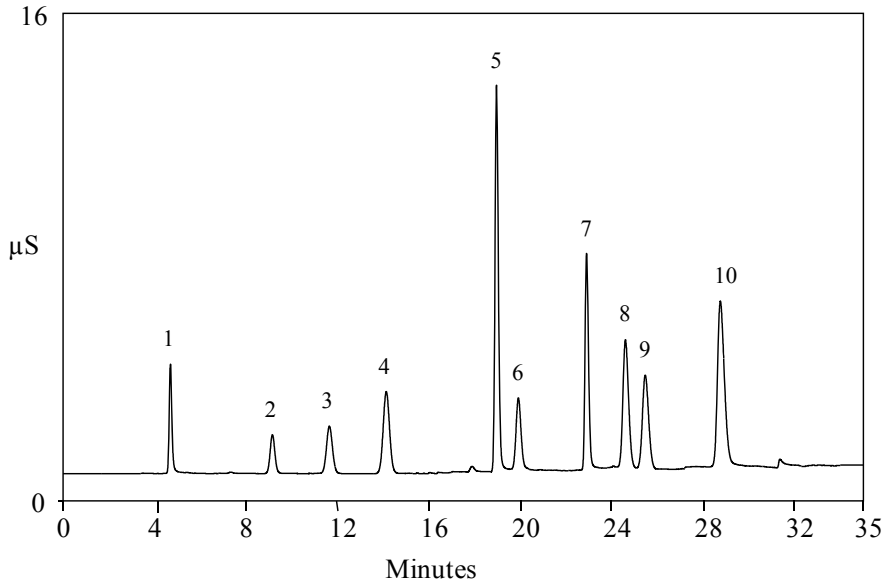
Peaks		mg/L
1.	Fluoride	2
2.	Sulfate	15
3.	Phosphate	15
4.	Chloride	3
5.	Nitrite	10
6.	Bromide	10
7.	Nitrate	10

Figure 9
Effect of Concentration on Selectivity

5.6. Separation of Oxyhalides and Inorganic Anions Using Hydroxide Gradient

The following chromatogram demonstrates the separation of oxyhalides and inorganic anions using a hydroxide gradient. As illustrated in Figure 10, a simple hydroxide gradient will resolve chlorite from bromate and easily separate the common inorganic anions.

Injection volume:	2.5 µL
Column:	Dionex IonPac AG26/AS26 2-mm
Eluent:	KOH: 12 mM from 0 to 10 min and 12 to 65 mM from 10 to 25 minutes
Eluent Source:	Dionex EGC III KOH Cartridge with Dionex CR-ATC
Flow Rate:	0.25 mL/min
Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex ASRS 300, 2-mm AutoSuppression, recycle mode



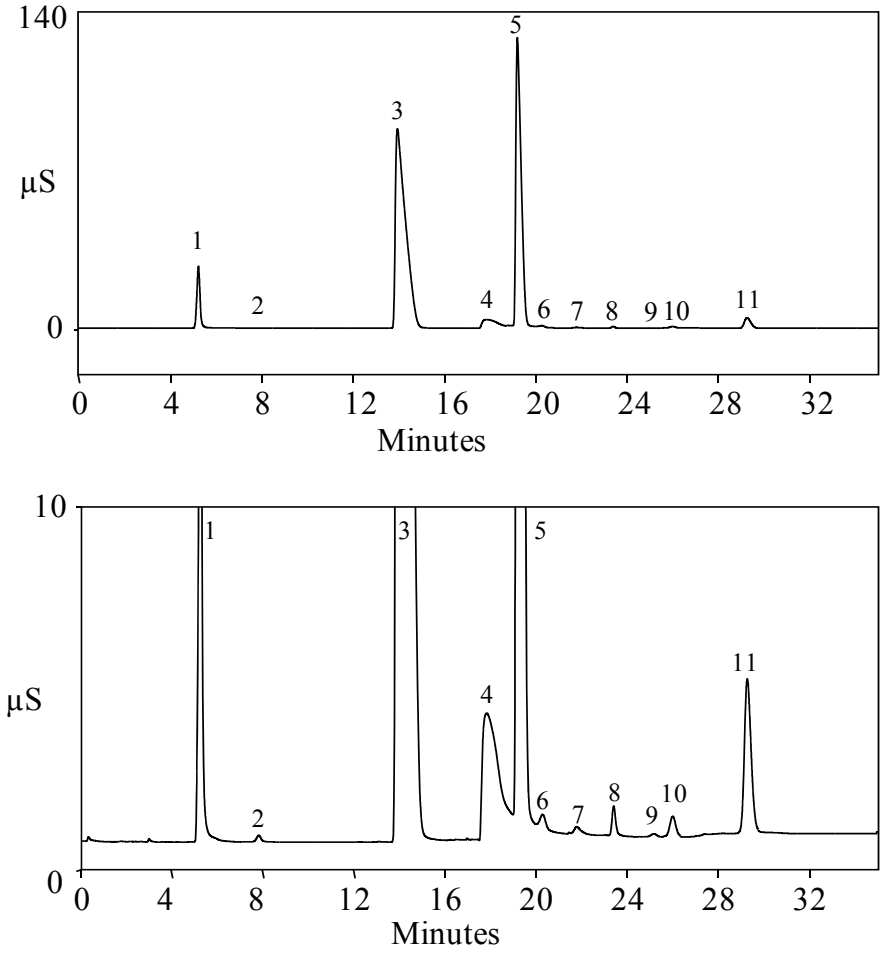
Peaks		mg/L
1.	Fluoride	3
2.	Chlorite	10
3.	Bromate	20
4.	Chloride	6
5.	Sulfate	30
6.	Nitrite	15
7.	Phosphate	40
8.	Bromide	25
9.	Chlorate	25
10.	Nitrate	25

Figure 10
Separation of Oxyhalides and the Inorganic Anions Using Hydroxide Gradient

5.7. Determination of Oxyhalides and Inorganic Anions in Drinking Water Samples Using Gradient Elution

The following chromatograms show the analysis of a drinking water sample using the Dionex IonPac AS26 column and a 100-µL injection volume.

Sample Volume:	100 µL
Column:	Dionex IonPac AG26/AS26 2-mm
Eluent :	KOH: 12 mM from 0 to 10 min and 12 to 65 mM from 10 to 25 minutes
Eluent Source:	Dionex EGC III KOH Cartridge with Dionex CR-ATC
Eluent Flow Rate:	0.25 mL/min
Operating Temperature:	15 °C
Detection:	Suppressed Conductivity, Dionex ASRS 300 (2-mm) AutoSuppression, recycle mode, 41 mA
Sample:	Drinking water



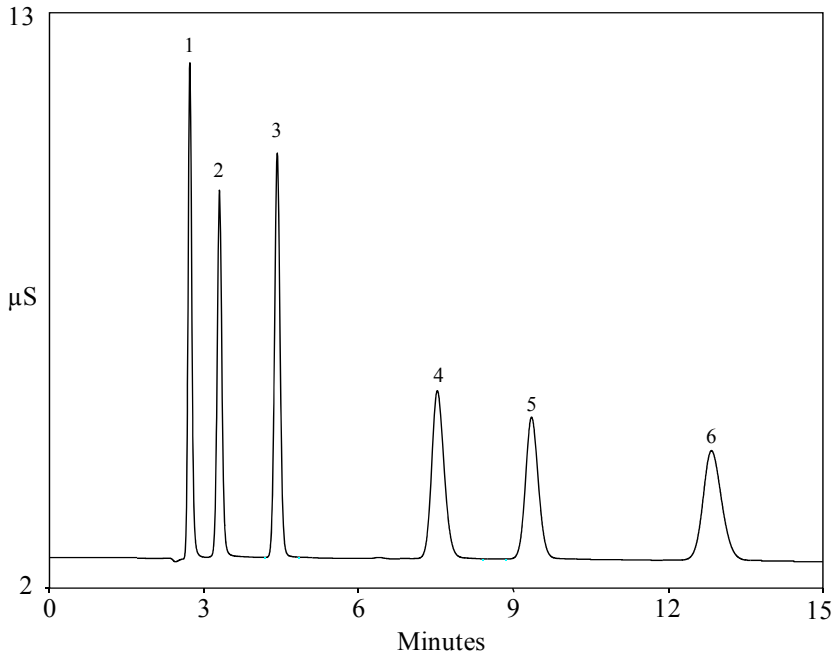
Peaks	mg/L
1. Fluoride	0.67
2. Formate	---
3. Chloride	11.6
4. Carbonate	---
5. Sulfate	12.1
6. Nitrite	0.03
7. Oxalate	---
8. Phosphate	0.12
9. Bromide	0.015
10. Chlorate	---
11. Nitrate	0.68

Figure 11
Determination of Oxyhalides and Inorganic Anions in Drinking Water Samples Using Gradient Elution

5.8. Analysis of Anions Using the Dionex IonPac AS26 Capillary Column

Below is the separation of 6 anions using the Dionex IonPac AS26 capillary column. An operating temperature of 15°C is used to ensure reproducible resolution and retention.

Column: Dionex IonPac AS26 (0.4 mm x 250 mm)
Eluent Source: Dionex Capillary EGC-KOH cartridge
Eluent: 55 mM KOH
Temperature: 15 °C
Detection: Suppression conductivity, Dionex ACES 300, AutoSuppression, recycle mode



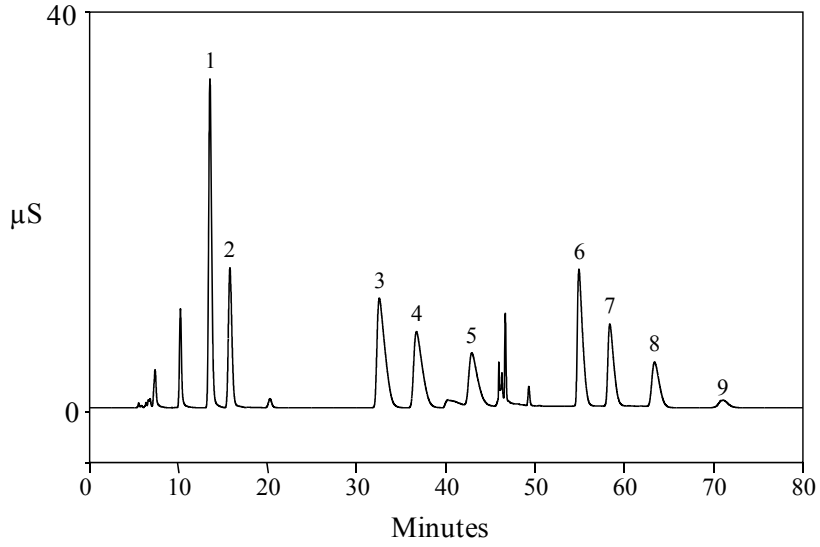
Peaks	mg/L
1. Fluoride	5
2. Sulfate	10
3. Chloride	10
4. Thiosulfate	20
5. Bromide	20
6. Nitrate	20

Figure 12
Analysis of Anions Using the Dionex IonPac AS26 Capillary Column

5.9. Gradient Separation of Haloacetic Acid Anions Using the Dionex IonPac AS26 Column

The Dionex IonPac AS26 column has been optimized for the gradient separation of 9 haloacetic acid anions including monochloro acetic acid (MCAA), dichloro acetic acid (DCAA), trichloro acetic acid (TCAA), monobromo acetic acid (MBAA), dibromo acetic acid (DBAA), tribromo acetic acid (TBAA), bromochloro acetic acid (BCAA), dibromochloro acetic acid (DBCAA) and dichlorobromo acetic acid (DCBAA) in drinking water matrices. An example of the separation is shown below. Monobromoacetic acid (MBAA), chlorodibromoacetic acid (CDBAA) and tribromoacetic acid (TBAA) degrade readily at high pH. The reaction is temperature dependent. To minimize the sample degradation, the separation is performed at sub-ambient temperature, specifically at 15 °C. The haloacetic acid anion elution is sensitive to the oven temperature, therefore, the oven temperature should be verified. If necessary, the oven temperature should be adjusted to obtain the optimal resolution. Lower detection limits for the haloacetic acids can be obtained using the 2D MEIC method described in Section 5.10.

Column:	Dionex IonPac AG26/AS26 2-mm
Eluent:	KOH: 7 mM from 0 to 12 min, 7 to 10 mM from 12 to 42 min, Step to 85 mM at 42 minutes
Eluent Source:	Dionex EGC III KOH
Flow rate:	0.25 mL/min
Inj. Volume:	250 µL
Temperature:	15 °C
Detection:	Suppressed conductivity, Dionex ASRS 300 2 mm, AutoSuppression, recycle mode, 53 mA



Peaks		µg/L
1.	MCAA	100
2.	MBAA	100
3.	DCAA	100
4.	BCAA	100
5.	DBAA	100
6.	TCAA	100
7.	BDCAA	100
8.	CDBAA	100
9.	TBAA	100

Figure 13
Gradient Separation of Haloacetic Acid Anions Using the Dionex IonPac AS26 Column

5.10. 2D MEIC Analysis of 9 Haloacetic Acid Anions Using the Dionex IonPacAS26 Capillary Column

The detection limit for the haloacetic acid anions can be further improved with the use of the two-dimensional Matrix-Elimination Ion Chromatography (2D MEIC) setup. Figure 14 shows the instrumental setup for the 2D MEIC method. A high-capacity Dionex IonPac AS24A/AG24A 4-mm column set is used in the first dimension to provide the initial separation between the haloacetic acid anions and the major matrix ions, such as chloride, sulfate and carbonate. A Thermo Scientific Dionex Carbonate Removal Device, Dionex CRD 300 4-mm unit is installed after the suppressor to remove the carbonic acid (dissolved carbon dioxide or carbonate or bicarbonate) from the samples. A Dionex CRD 300 was used in this application to maximize the removal of both carbon dioxide and ammonia. The standard set up for CRD with hydroxide chemistry is in the recycle mode of operation using the basic suppressor waste stream as the regenerant for the CRD. However, in this analysis since ammonium chloride is typically added to the drinking water samples at a final concentration of 100 ppm to remove any residual chlorine, the presence of large amount of ammonia in the suppressor waste imposes some constraints. For example the ammonia which is a gas in the basic environment can be transported across the CRD membrane into the suppressed eluent stream and can increase the suppressed effluent pH and background. This will, in-turn lower the recovery for the early eluting peaks by eluting it off the concentrator column. To eliminate this effect an independent source of base is supplied to the Dionex CRD 300 unit. This way any removed ammonia is sent to waste by the suppressed effluent and the Dionex CRD 300 unit is not exposed to ammonia. The base stream is supplied by the first channel in a two channel peristaltic pump.

After suppression, the effluent from the first dimension is directed to a diverter valve where the effluent can be either directed back to the suppressor for suppression, or loaded onto a Thermo Scientific Dionex Monolith Anion Concentrator Column, Dionex MAC-200, located in the second injection valve. Only the portions containing the analytes of interest will be focused onto the concentrator and subsequently analyzed in the second dimension with Dionex IonPac AS26/AG26 capillary column set. To minimize the carryover of highly retained species from the first dimension into the second dimension column, the concentrator column is rinsed with 100 mM NaOH solution in the concurrent direction before the loading of the next sample in the counter current direction. A Thermo Scientific Dionex AS autosampler with a diverter valve is used to deliver the base for the rinse. It is also possible to have a pressurized reservoir filled with base to supply the base rinse. The use of a capillary column in the second dimension greatly increases the detection limit and provides enhanced selectivity.

5.11. Sample Overlap

The overall run time for the two-dimensional matrix elimination ion chromatography can be reduced through the use of sample overlap. Sample overlap simply means reducing the overall analysis time by processing analysis steps in parallel. For example instead of waiting for the completion of the second dimension analysis, the first dimension analysis for the next sample can start after the focused ions from the concentrator column have been injected into the second dimension. To achieve this, the two analysis channels are configured as two separate timebases. As illustrated in Figure 14, the first timebase consists of the first analysis channel, autosampler and the diverter valve, and the second timebase consists of the second analysis channel. The timing between the two timebases is controlled through the use of the ExclusiveAccess command in the Chromeleon Data System. Once the focused ions from the concentrator column have been injected into the second dimension, the second timebase switches the concentrator column to the load position and releases the exclusive access. The first timebase will take over and start rinsing the concentrator column with base. After the rinse, the autosampler will load the next sample into the loop and start the first dimensional analysis. By overlapping some of the analysis steps in the two dimensions significant reduction in analysis time can be achieved.

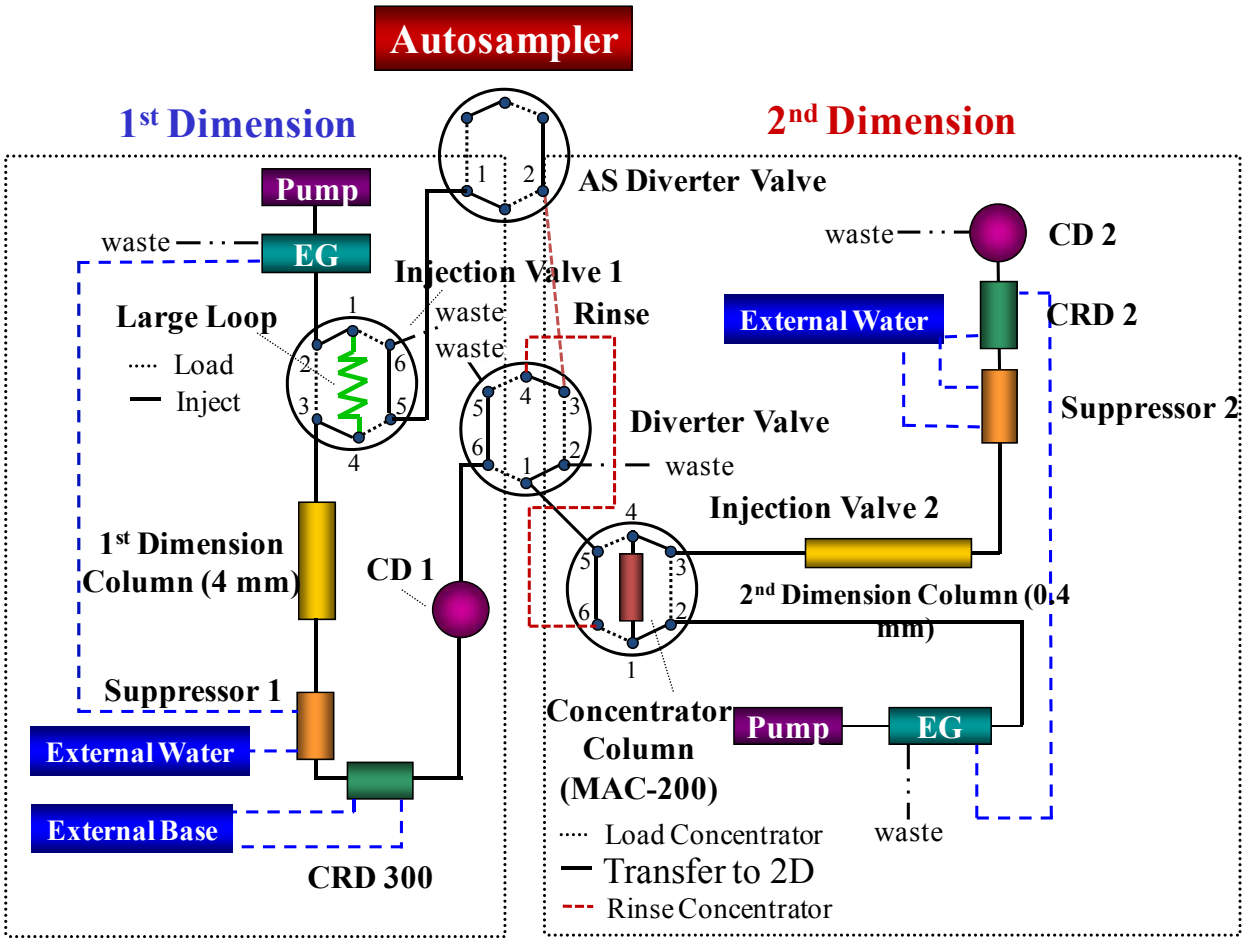


Figure 14
Instrumental Setup for the two-dimensional Matrix-Elimination Ion Chromatography

5.12. 2D Analysis of 9 Haloacetic Acid Anions Using the Dionex IonPac AS26 Capillary Column

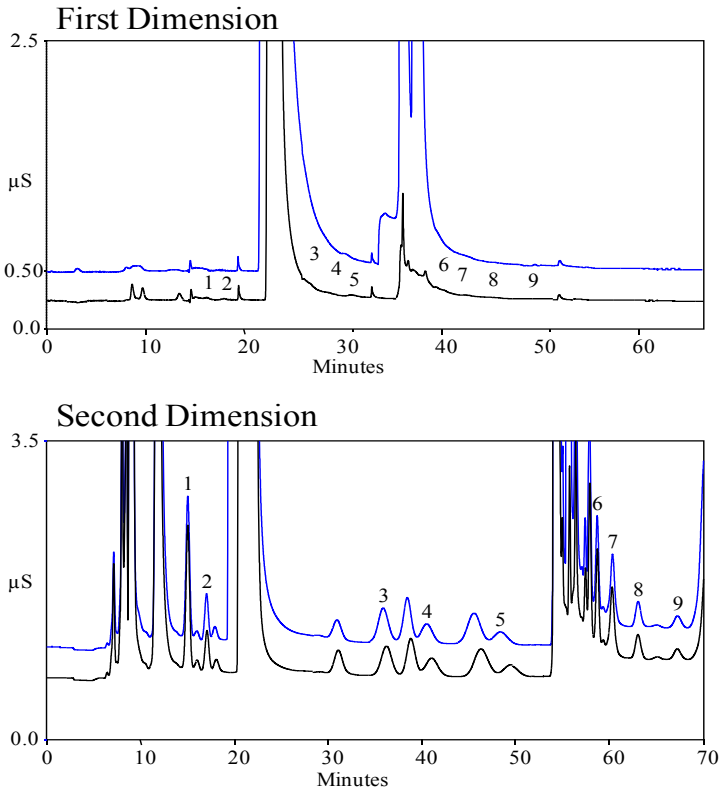
The following outlines the conditions for the separation of the 9 haloacetic acid anions with the two-dimensional matrix elimination ion chromatography using an AS24A/AG24A 4-mm column set for the first dimension and an AS26/AG26 capillary column set for the second dimension. If an AS26/AG26 2-mm column set is used, refer to section 5.9 for gradient conditions . For optimal performance, set both the DC lower compartment temperature and the IC Cube temperature to 15 °C. The DC upper compartment temperature must be reduced in order for the IC Cube to maintain 15 °C.

First Dimension:

Column:	Dionex IonPac AS24A/AG24A (4 mm)
Eluent Source:	Dionex EGC-KOH cartridge
Eluent:	KOH: 7 mM from 0 to 12 min, 7 to 18 mM from 12 to 42 min, Step to 65 mM at 42 minutes
Flow Rate:	1.0 mL/min
Temperature:	15 °C
Detection:	Suppressed conductivity, Dionex ASRS 300, AutoSuppression, recycle mode
Injection Volume:	500 µL

Second Dimension:

Column:	Dionex IonPac AS26/AG26 (0.4 mm)
Eluent Source:	Dionex Capillary EGC-KOH cartridge
Eluent:	KOH: 6 mM from 0 to 50 min, step to 160 mM at 50 min, 160 mM from 50 to 57 min, Step to 130 mM at 57 minutes
Flow Rate:	12 µL/min
IC Cube Temperature:	15 °C
Detection:	Suppressed conductivity, Dionex ACES 300, AutoSuppression, recycle mode
Concentrator:	Dionex IonSwift MAC-200
Black Trace:	100 ppm ammonium chloride
Blue Trace:	High Ionic Water (250 ppm Chloride, 250 ppm Sulfate, 150 ppm Bicarbonate, 20 ppm nitrate and 100 ppm ammonium chloride



Peaks	µg/L
1. MCAA	5
2. MBAA	5
3. DCAA	5
4. BCAA	5
5. DBAA	5
6. TCAA	5
7. BDCAA	5
8. CDBAA	5
9. TBAA	5

Figure 15

2D Analysis of 9 Haloacetic Acid Anions Using the Dionex IonPac AS26 Capillary Column

SECTION 6 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using Dionex IonPac AS26 column. 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. **For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.**

Table 6
Dionex IonPac AS26/AG26 Troubleshooting Summary

Observation	Cause	Action	Reference Section
High Back Pressure	Unknown	Isolate Blocked Component	6.1.1
	Plugged Column Bed Supports	Replace Bed Supports, Filter Eluents, and Filter Samples	6.1.2, 6.1.3, 6.1.4
	Other System Components	Unplug, Replace	Component Manual
High Background Conductivity	Contaminated Eluents	Remake Eluents	6.2, 6.2.1
	Contaminated Trap Column	Clean Trap Column	6.2.2, 6.2.3, Component Manual
	Contaminated Guard or Analyte Column	Clean Guard and Analytical Column	6.2.4
	Contaminated Dionex ASRS, ACES or AMMS	Clean Suppressor	6.2.6, Component Manual
	Contaminated Hardware	Clean Component	6.2.5, Component Manual
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System	6.3.1.2
	Column Headspace	Replace Column	6.3.1.1
Short Retention Times	Flow Rate Too fast	Recalibrate Pump	6.3.2.1
	Conc. Incorrect Eluents	Remake Eluents	6.3.2.2
	Column Contamination	Clean Column	6.3.2.3
Poor Front End	Conc. Incorrect Eluents	Remake Eluents	6.3.3.1
Resolution	Column Overloading	Reduce Sample Size	6.3.3. 2
	Sluggish Injection Valve	Service Valve	6.3.3.3, Component Manual
	Large System Void Volumes	Replumb System	6.3.3.4, Component Manual
Spurious Peaks	Sample Contaminated	Pretreat Samples	6.3.4.1
	Sluggish Injection Valve	Service Valve	6.3.4.2

6.1. High Back Pressure

6.1.1. Finding the Source of High System Pressure

Total system pressure for the Dionex IonPac AG26 (4-mm) Guard Column plus the Dionex IonPacAS26 (4-mm) Analytical Column when using the test chromatogram conditions should be equal or less than 2,100 psi. If the system pressure is higher than 2,100 psi, determine the cause of the high system pressure. The system should be operated with a Thermo Scientific Dionex High-Pressure In-Line Filter (P/N 044105) which is positioned between the gradient pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated. The maximum flow rate is 2 mL/min and the maximum pressure is 3,000 psi (20.68 MPa).

A. Ensure 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 Dionex 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 Dionex IonPac AS26/AG26 Operating Back Pressures”).

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

Table 7
Typical Dionex IonPacAS26/AG26 Operating Back Pressures

Column	Typical Back Pressure psi (MPa)	Flow Rate mL/min
Dionex IonPac AS26 4-mm Analytical	< 1800 (12.41)	1.0
Dionex IonPac AG26 4-mm Guard	< 300 (2.07)	1.0
Dionex IonPac AS26 + AG26 4-mm columns	< 2100 (14.48)	1.0
Dionex IonPac AS26 2-mm Analytical	< 1800 (12.41)	0.25
Dionex IonPac AG26 2-mm Guard	< 300 (2.07)	0.25
Dionex IonPac AS26 + AG26 2-mm columns	< 2100 (14.48)	0.25
Dionex IonPac AS26 0.4-mm Capillary	< 1800 (12.41)	0.010
Dionex IonPac AG26 0.4-mm Capillary Guard	< 300 (2.07)	0.010
Dionex IonPac AS26 + AG26 0.4-mm Columns	< 2100 (14.48)	0.010

6.1.2. Replacing Column Bed Support Assemblies

If the column inlet bed support is determined to be the cause of the high back pressure, it should be replaced. To change the inlet bed support assembly, refer to the following instructions, using one of the two spare inlet bed support assemblies included in the Ship Kit.

a) Disconnect the column from the system.

b) Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.

c) Remove the bed support.

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

e) Discard the old bed support assembly.

f) Place a new bed support assembly into the end fitting.

g) Ensure 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.

h) Use the end of the column to carefully start the bed support assembly into the end fitting.

Table 8
Product Information

Product	Dionex IonPac AS26 4-mm Columns (P/N)	Dionex IonPac AS26 2-mm Columns (P/N)	Dionex IonPac AS26 0.4-mm Columns (P/N)
Analytical Column	063148	063065	075399
Guard Column	063154	063066	075400
Bed Support Assembly	042955	044689	N/A
End Fitting	052809	043278	N/A

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

i) Screw the end fitting back onto the column. Tighten it finger tight, then an additional 1/4 turn (20 in. lb. for the 4-mm, 10 in.oz. for the 2-mm). Tighten further only if leaks are observed.

j) Reconnect the column to the system

k) Resume operation.

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

6.1.3. Filter Eluent

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

6.1.4. Filter Samples

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

6.2. High Background or Noise

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

Table 9
Background Conductivity

Eluent	Expected Background Conductivity
55 mM NaOH (Bottle Eluent)	< 3 µS
55 mM EGC III KOH	< 1.0 µS

6.2.1. Preparation of Eluents

a) Check the eluents and the regenerant (if used) are made correctly; specifically, check that eluents were made from chemicals with the recommended purity.

b) Ensure the deionized water, used to prepare the reagents, has a specific resistance of 18.2 megohm-cm.

6.2.2. A Contaminated Trap Column

High background may be caused by contamination of the Dionex ATC-HC or Dionex ATC-3 with carbonate or other anions from the eluent.

a) Clean the Dionex ATC-HC or Dionex ATC-3 (4-mm) with 100 mL of 2.0 M NaOH or 50 mL for the Dionex ATC-3 (2-mm).

b) Rinse the Dionex ATC-HC or Dionex ATC-3 (4-mm) immediately with 20 mL of eluent or 10 mL of eluent for the Dionex ATC-3 (2-mm) into a beaker prior to use.

6.2.3. Contaminated Dionex CR-ATC Column

a) Install a Dionex CR-TC Anion Trap Column (P/N 060477 or 072078) if using a Dionex Eluent Generator with Dionex EGC KOH.

b) If the Dionex CR-ATC becomes contaminated, please refer to Section 6, Clean-Up, in the Dionex CR-ATC Product Manual (Document No. 031910).

6.2.4. A Contaminated Guard or Analytical Column

a) Remove the Dionex IonPac AG26 Guard and Dionex IonPac AS26 Analytical Columns from the system.

b) Install a back pressure coil that generates approximately 1,500 psi and continue to pump eluent.
   - If the background conductivity decreases, the column(s) is (are) the cause of the high background conductivity.

c) To eliminate downtime, clean or replace the Dionex IonPac AG26 at the first sign of column performance degradation.
   - Clean the column as instructed in, "Column Cleanup" (See Appendix B "Column Care").

6.2.5. Contaminated Hardware

Eliminate the hardware as the source of the high background conductivity.

a) Bypass the columns and the suppressor.

b) Install a back pressure coil that generates approximately 1,500 psi and continue to pump eluent.

c) Pump deionized water with a specific resistance of 18.2 megohm-cm through the system.

d) The background conductivity should be less than 2 µS. If it is not, check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

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

If the above items have been checked and the problem persists, the Dionex Anion Self-Regenerating Suppressor, the Dionex Anion Capillary Electrolytic Suppressor or the Dionex Anion MicroMembrane Suppressor is probably causing the problem. For details on Dionex Anion Self-Regenerating Suppressor operation, refer to the Dionex Anion Self-Regenerating Suppressor 300 Product Manual (Document No. 031956). For details on Dionex Anion Membrane Suppressor 300 operation, refer to the Product Manual (Document No. 031727) for assistance. For details on the Dionex Anion Capillary Electrolytic Suppressor (Dionex ACES 300) operation, refer to the product manual (Document No. 065388) for assistance.

a) Check the power level and alarms on the Dionex SRS Control.

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

c) Check the eluent flow rate.

d) If you are using a Dionex AutoRegen accessory with the Dionex ASRS in the Chemical Suppression Mode or the Dionex AMMS, prepare fresh regenerant solution.

e) Test both the suppressor and the Dionex AutoRegen Regenerant Cartridge for contamination.

   - If the background conductivity is high after preparing fresh regenerant and bypassing the Dionex AutoRegen Regenerant Cartridge, you probably need to clean or replace your Dionex ASRS, Dionex ACES or Dionex AMMS.

If the background conductivity is low when freshly prepared regenerant is run through the Dionex ASRS or Dionex AMMS without an AutoRegen accessory in-line, test the Dionex AutoRegen Regenerant Cartridge to see if it is expended.

a) Connect the freshly prepared regenerant to the Dionex AutoRegen Regenerant Cartridge.

b) Pump approximately 200 mL of regenerant through the Dionex AutoRegen Regenerant Cartridge to waste before recycling the regenerant back to the regenerant reservoir.

   - If the background conductivity is high after placing the Dionex AutoRegen accessory in-line, you probably need to replace the Dionex AutoRegen Regenerant Cartridge. Refer to the "Dionex 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

6.3.1.1. Peak Fronting:

a) 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.

b) 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.

6.3.1.2. Symmetric Inefficient Peaks:

a) Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient.

b) Ensure you are using PEEK tubing with an ID of no greater than 0.010" for 4-mm systems or greater than 0.005" for 2-mm systems.

c) Check all eluent liquid line connections between the injection valve and the detector cell inlet.

d) Cut the tubing lengths as short as possible.

e) Check for leaks.

6.3.2. Poor Resolution Due to Shortened Retention Times

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

6.3.2.1. Flow Rate:

a) Check the flow rate to see if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol.

b) Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

6.3.2.2. Compositions and Concentrations:

a) Check to see if the eluent compositions and concentrations are correct.

- An eluent that is too concentrated will cause the peaks to elute faster.

b) Prepare fresh eluent.

NOTE ***If you are using a gradient pump to proportion the eluent, or components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. This may be a problem when one of the proportioned eluents is less than 5%.***

c) Use one reservoir containing the correct eluent composition.

6.3.2.3. Column Contamination:

- 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 Appendix B "Column Care"), for recommended column cleanup procedures.

NOTE

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.

6.3.2.4. Diluting the Eluent:

- 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 Appendix B, "Column Cleanup" in "Column Care").

a) After cleaning the column, reinstall it in the system.

b) Let the column 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. For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

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:

6.3.3.1. Improper Eluent Concentration

a) Remake the eluent as required for your application.

b) Ensure that the water and chemicals used are of the required purity.

6.3.3.2. Column Overloading

a) 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.

6.3.3.3. Sluggish Operation of the Injection Valve

a) Check the air pressure.

b) Ensure there are no gas leaks.

c) Check that the port faces are not partially plugged. Refer to the valve manual for instructions.

6.3.3.4. Improperly Swept Out Volumes

a) Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change. Remember to use the shortest tubing lengths possible.

6.3.4. Spurious Peaks

6.3.4.1. Contaminated Columns

- 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.

a) Clean the column as indicated in "Column Cleanup" (see Appendix B "Column Care").

If you need assistance in determining the best way to clean strongly retained solutes in your specific sample matrix from the IonPac AS26 columns, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

NOTE

6.3.4.2. Injection Valve May Require 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).

a) Check to see that there are no restrictions in the tubing connected to the valve.

b) Check the valve port faces for blockage and replace them if necessary. Refer to the Valve Manual for troubleshooting and service procedures.

- Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantification of the peaks of interest.

6.3.5. Poor Efficiency Using Capillary Columns

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

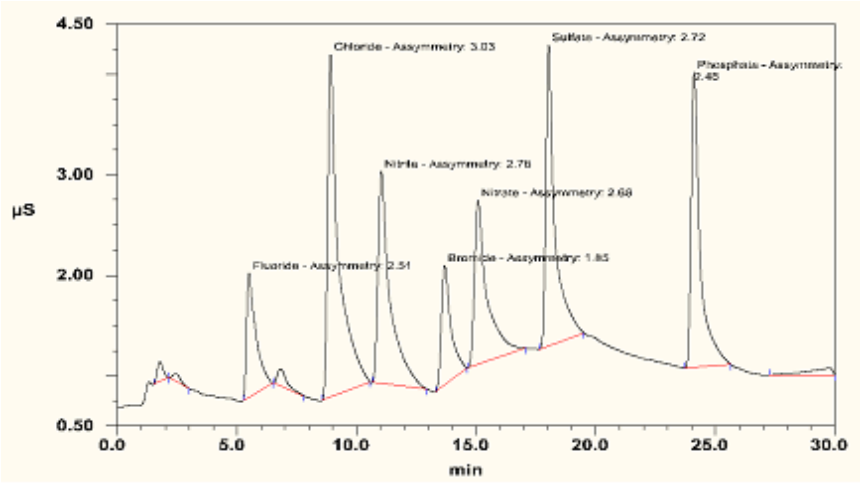


Figure 16
Tailing Peaks Caused by Incorrectly Installed Capillary Tubing Fittings

When connecting a capillary tube fitting, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing. Figure 17 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

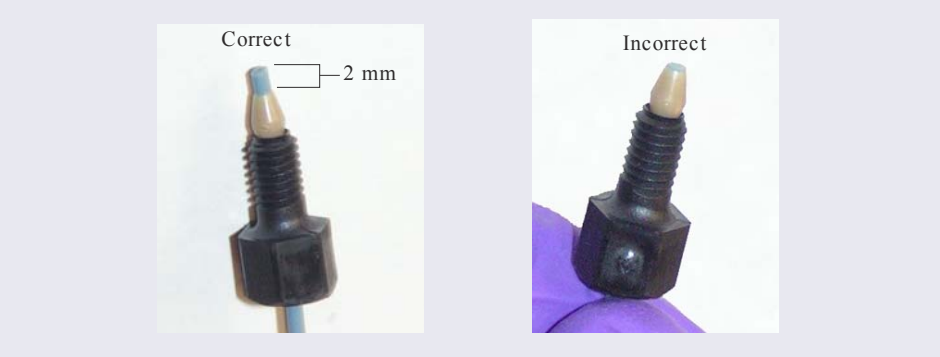


Figure 17
Correct and Incorrect Ferrule and Fitting Bolt Placement for Capillary Tubing Connections

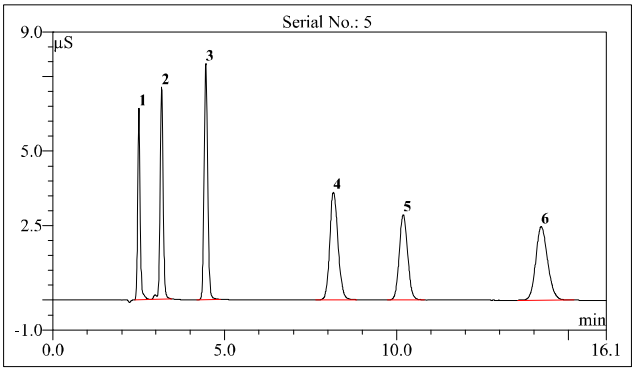
APPENDIX A - QUALITY ASSURANCE REPORTS

DIONEX IONPAC AS26 2-MM ANALYTICAL 2X250 MM

IonPac® AS26
Analytical (2 x 250 mm)
Product No. 076022

Date: 01-Aug-11 16:00
Serial No. : 000005
Lot No. : 011-19-054G

Eluent: 55 mM KOH
Eluent Source: EGC II KOH Cartridge
Flow Rate: 0.30 mL/min
Temperature: 15 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 4mm)
AutoSuppression® Recycle Mode
Applied Current: 41 mA
Injection Volume: 2.5 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.50	1.3	4.56	5841	5.0
2	Sulfate	3.16	1.2	7.40	6306	10.0
3	Chloride	4.45	1.1	11.84	8682	10.0
4	Thiosulfate	8.16	1.3	4.66	5583	20.0
5	Bromide	10.20	1.2	7.52	8676	20.0
6	Nitrate	14.21	1.3	n.a.	8062	20.0

QA Results:

Analyte	Parameter	Specification	Results
Bromide	Efficiency	>=5400	Passed
Bromide	Asymmetry	1.0-2.0	Passed
Bromide	Retention Time	8.38-11.13	Passed
(Nitrate - Bromide)/ (Bromide - Chloride)	Retention Time Ratio	0.65-0.76	Passed
	Pressure	<=2200	1725

Production Reference:
Datasource: Column
Directory: CPC\CPC_12
Sequence: RQ_AS26_2X250MM_Validation_7-20-11
Sample No.: 28

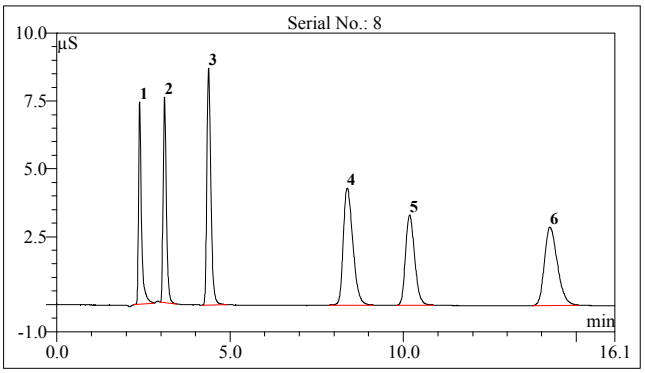
6.80 DU10c Build 2859 (179491) (Demo-Installation)

DIONEX IONPAC AS26 4-MM ANALYTICAL 4X250 MM

IonPac® AS26
Analytical (4 x 250 mm)
Product No. 076020

Date: 19-Jul-11 16:29
Serial No. : 000008
Lot No. : 011-19-026

Eluent: 55 mM KOH
Eluent Source: EGC II KOH Cartridge
Flow Rate: 1.2 mL/min
Temperature: 15 °C
Detection: Suppressed Conductivity
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300 4mm)
AutoSuppression® Recycle Mode
Applied Current: 164 mA
Injection Volume: 10.0 µL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.39	2.2	4.71	4925	5.0
2	Sulfate	3.11	1.5	6.76	5323	10.0
3	Chloride	4.39	1.4	11.39	7105	10.0
4	Thiosulfate	8.39	1.6	3.72	4570	20.0
5	Bromide	10.18	1.4	7.03	7407	20.0
6	Nitrate	14.23	1.5	n.a.	6968	20.0

QA Results:

Analyte	Parameter	Specification	Results
Bromide	Efficiency	>=5400	Passed
Bromide	Asymmetry	1.0-2.0	Passed
Bromide	Retention Time	8.38-11.13	Passed
(Nitrate - Bromide) / (Bromide - Chloride)	Retention Time Ratio	0.65-0.76	Passed
	Pressure	<=2200	1335

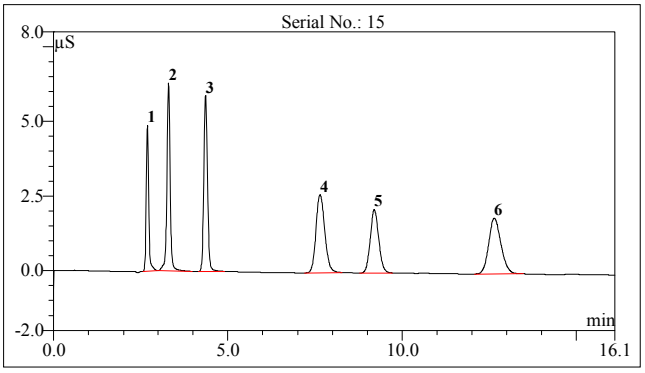
Production Reference:
Datasource: Column
Directory: CPC\CPC_11
Sequence: RQ_AS26_4X250MM_Validation_7-18-11
Sample No.: 20

DIONEX IONPAC AS26 CAPILLARY 0.4X250 MM

IonPac® AS26
Capillary (0.4 x 250 mm)
Product No. 076018

Date: 02-Aug-11 12:28
Serial No. : 000015
Lot No. : 011-19-012

Eluent: 55 mM KOH
Eluent Source: EGC-KOH (Capillary)
Flow Rate: 0.012 mL/min
Temperature: 15 °C
Detection: Suppressed Conductivity
Suppressor: Anion Capillary Electrolytic Suppressor (ACES 300)
AutoSuppression® Recycle Mode
Applied Current: 12 mA
Injection Volume: 400 nL
Storage Solution: 100 mM Sodium Tetraborate



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.69	1.2	4.10	7113	5.0
2	Sulfate	3.30	1.0	5.93	6206	10.0
3	Chloride	4.37	1.0	10.10	8057	10.0
4	Thiosulfate	7.65	1.3	3.46	4482	20.0
5	Bromide	9.20	1.2	6.33	6875	20.0
6	Nitrate	12.64	1.3	n.a.	6095	20.0

QA Results:

Analyte	Parameter	Specification	Results
Bromide	Efficiency	>=5400	Passed
Bromide	Asymmetry	1.0-2.0	Passed
Bromide	Retention Time	8.38-11.13	Passed
(Nitrate - Bromide) / (Bromide - Chloride)	Retention Time Ratio	0.65-0.76	Passed
	Pressure	<=2200	1335

Production Reference:
Datasource: Column
Directory: Capillary\Cap_Anion-2
Sequence: RQ_AS26_0p4X250MM_7-27-11
Sample No.: 37

APPENDIX B - COLUMN CARE

B.1 Recommended Operating Pressure

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for Dionex IonPac AS26 columns is 3,000 psi (20.68 MPa).

B.2 Column Start-Up

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

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

B.3 Column Storage

For short-term storage (< 1 week), use Eluent, for long-term storage (> 1 week), use 100 mM Sodium Borate for the column storage solution. Flush the column for a minimum of 10 minutes with the storage solution. Cap both ends securely, using the plugs supplied with the column.

B.4 Column Cleanup

The following column cleanup protocols have been divided into three general isocratic protocols to remove acid-soluble, base-soluble, or organic contaminants. They can be combined into one gradient protocol if desired; however, the following precautions should be observed.

WARNING

- ***Always ensure that the cleanup protocol used does not switch between eluents which may create high pressure eluent interface zones in the column.***
- ***High pressure zones can disrupt the uniformity of the packing of the column bed and irreversibly damage the performance of the column.***
- ***High pressure zones in the column can be created by pumping successive eluents through the column that are not miscible, that have eluent components in one eluent that will precipitate out in the other eluent or by using an acid eluent followed by a base eluent which may create a neutralization pressure band.***
- ***The precipitation of the salts in solvents during column rinses can result in very high pressure zones. High viscosity mixing zones can be created between two eluents having solvents with a very high energy of mixing.***

When in doubt, always include short column rinse steps to reduce the solvent content of the eluent to < 5% levels and the ionic strength of the eluent to < 50 mM levels to avoid creating high pressure zones in the column that may disrupt the uniformity of the column packing.

B.4.1 Choosing the Appropriate Cleanup Solution

Contamination	Solution
Hydrophilic Contamination of Low Valence	Concentrated hydroxide solutions such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.
High Valence Hydrophilic Ions Contamination	Concentrated acid solutions such as 1 to 3 M HCl will remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.
Metal Contamination	Metal contamination often results in asymmetric peak shapes and/or variable analyte recoveries. For example, iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.
	Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.
Nonionic and Hydrophobic Contamination	Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents.
Ionic and Hydrophobic Contamination	Concentrated acid solutions such as 1 to 3 M HCl can be used with compatible organic solvents to remove contamination that is ionic and hydrophobic. The acid suppresses ionization and ion exchange interactions of the contamination with the resin.
	A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

B.4.2 Column Cleanup Procedure

Use the following cleanup procedures to clean the Dionex IonPac AG26 and Dionex IonPac AS26.

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

b) Disconnect the Dionex ASRS 300, Dionex ACES 300 or Dionex AMMS 300 from the Dionex IonPac AS26 Analytical/Capillary Column.

c) If your system is configured with both a guard column and an analytical or capillary column, reverse the order of the guard and analytical/capillary column in the eluent flow path.

d) Double check that the eluent flows in the direction designated on each of the column labels.

CAUTION ***When cleaning an analytical column and a guard column (or capillary and capillary guard) in series, ensure that the guard column is placed after the analytical/capillary column in the eluent flow path. If not, the contaminants that have accumulated on the guard column can be eluted onto the analytical/capillary column and irreversibly damage it. If in doubt, clean each column separately.***

e) Set the pump flow rate to 1.0 mL/min for an Dionex IonPac AS26 4-mm Analytical or Guard Column or set the pump flow rate to 0.25 mL/min for a Dionex IonPac AS26 2-mm Analytical or Guard Column. Set the pump flow rate to 0.010 mL/min for a Dionex IonPac AS26 Capillary or Capillary Guard Column.

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

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

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

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

j) Reconnect the Dionex ASRS 300, Dionex ACES 300 or Dionex AMMS 300 to the Dionex IonPac AS26 Analytical or Capillary Column

k) Place the guard column in line between the injection valve and the analytical or capillary column if your system was originally configured with a guard column.

APPENDIX C - CONFIGURATION

C.1 Configuration of Ion Chromatography (IC) Systems

Table C1
Configuration of Ion Chromatography Systems

CONFIGURATION	2-mm	4-mm	0.4-mm
Eluent Flow Rate	0.25 mL/min	1.0 mL/min	0.010 mL/min
SRS Suppressor	Dionex ASRS 300 (P/N 061562)	Dionex ASRS 300 (P/N 061561)	N/A
MMS Suppressor	Dionex AMMS 300 (P/N 056751)	Dionex AMMS 300 (P/N 056750)	N/A
ACES Suppressor	N/A	N/A	Dionex ACES 300 (P/N 072052)
NOTE: ***Do not run suppressors over 40°C. If application requires a higher temperature, place suppressor outside of chromatographic oven.***			
Injection Loop	2 - 15 µL Use the Rheodyne Microinjection Valve, Model No. 9126 P/N 044697) for full loop injections <15 µL.	10 - 50 µL	0.4 µL (typical)
System Void Volume	Eliminate switching valves, couplers and the Dionex GM-3 Gradient Mixer. Use only the Dionex Microbore GM-4 (2-mm) Mixer (P/N 049135).	Minimize dead volumes. Switching valves, couplers can be used. Use the Dionex GM-2, Dionex GM-3, Dionex GM-4 or recommended gradient mixers.	Use only on an IC System equipped for capillary analysis.
Pumps	Use the Dionex DP/SP3000/DP/SP5000/GS50/ GP50/GP40/IP20/IP25 in Microbore Configuration with a Dionex Microbore GM-4 (2-mm) Gradient Mixer.	Use the Dionex DP/SP3000/DPSP5000/ GP40/GP50/IP20/IP25 in Standard-Bore Configuration.	Use only a pump designed for capillary flow rates such as the Dionex ICS-5000 capillary pump.
NOTE: ***Use of a Dionex EGC-KOH cartridge (P/N 074532 or 072076 in conjunction with a Dionex CR-ATC P/N 060477 or 072078) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***			
Chromatographic Module	A thermally controlled column oven such as the Dionex LC25,LC30,ICS-1500, 1600, 2000, 2100, 3000, 5000 DC	A thermally controlled column oven such as the Dionex LC25,LC30,ICS-1500, 1600, 2000, 2100, 3000, 5000 DC	A thermally controlled column compartment such as the Dionex ICS-5000 DC or Dionex IC-Cube.
CONFIGURATION	2-mm	4-mm	0.4-mm
Detectors	DionexCD20, CD25, CD25A, ED40, ED50 or ED50A Dionex Conductivity Cell with DS3 P/N 044130 or Dionex Conductivity Cell with Dionex P/N 061830 Dionex AD20/AD25 Cell (6-mm, 7.5 µL, P/N 046423) Ensure 30-40 psi back pressure.	Dionex CD20, CD25, CD25A, ED40, ED50 or ED50A Dionex Conductivity Cell with DS3 P/N 044130 or Dionex Conductivity Cell with Dionex P/N 061830 Dionex AD20/AD25 Cell (10-mm, 9 µL, P/N 049393) Ensure 30-40 psi back pressure.	Use only a conductivity detector designed for capillary flow rates such as the Dionex ICS-5000 Capillary CD.

Table C2
Tubing Back Pressures

Color	Part Number	I.D. inch	I.D. cm	Volume mL/ft	Back Pressure Psi/ft. at 1 mL/min	Back Pressure Psi/ft. at 0.25 mL/min	Back Pressure Psi/cm. at 1 mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	0.010	0.025	0.015	6.960	1.740	0.232
Red	044221	0.005	0.013	0.004	111.360	27.840	3.712
Yellow	049715	0.003	0.008	0.001	859.259	214.815	28.642
Light Blue	071870	0.0025	0.006	0.0009	1766.0	441.0	58.0

