PRODUCT MANUAL

IonPac® AS24
IonPac® AG24

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PRODUCT MANUAL

for the

IONPAC® AS24 Column

(2 x 250 MM, P/N 064153)

IONPAC® AG24 Column

(2 x 50 MM, P/N 064151)

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SECTION 1 - INTRODUCTION

The IonPac® AS24 2-mm Analytical and AG24 2mm Guard Columns are hydroxide selective anion exchange columns designed for separation of several environmental ions including haloacetic acids (HAA) and bromate in drinking water (See Dionex Application Note 217). The IonPac AS24 column is a low bleed column designed to be used for ion chromatography separations coupled with Mass Spectrometry (IC/MS), or (IC/MS/MS). It is a high capacity column, which allows relatively large injection volumes. Water samples can be directly injected into an ion chromatography system coupled to a mass spectrometer for sensitive detection. The use of IonPac AS24 for IC-MS and IC-MS/MS eliminates the need for sample pretreatment and preconcentration typically needed to eliminate sample matrix effects on analyte peak efficiency and resolution such as in HAA determination or other environmental ions in relatively high matrix samples.

The high capacity and compatibility of this column with mass spectroscopy makes it useful with Reagent Free Ion Chromatography (RFIC™) systems for several environmental ions. The AS24 is compatible with pH 0-14 eluents and samples. The AS24 can be used with any suppressible ionic eluent that does not exceed the capacity of the suppressor. The IonPac AS24 has nominal efficiency of at least 4,000 plates/column for Thiosulfate using Quality Assurance test conditions. The maximum operating pressure should be less than 3,000 psi (20.68 MPa).

Table 1
IonPac AS24 Operating Parameters

Column	Typical Back Pressure at 30 C psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS24 2-mm Analytical	< 2,000 (13.8)	0.3	0.5
AG24 2-mm Guard	< 300 (2.07)	0.3	0.5
AS24 2-mm Column Set	< 2,300 (15.87)	0.3	0.5

SECTION 2 - ION CHROMATOGRAPHY SYSTEMS

A 2-mm Ion Chromatography system setup is used. A microbore (2-mm) gradient pump is 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 at flow rates less than 0.5 mL/min, Dionex recommends a microbore (1/16) piston isocratic pump or a microbore gradient pump

See Appendix B, "Configuration" for specific recommended settings and parts including pumps, eluent flow rate, Self-Regenerating Suppressor® (SRS®), injection loop, system void volume, detectors, and tubing back pressure.

SECTION 3 - INSTALLATION

3.1. System Requirements

3.1.1. System Requirements for 2-mm Operation

3.1.2. System Void Volume

Since the AS24 is a 2-mm column, it is particularly important to minimize system void volume. For best performance, all of the tubing installed between the injection valve and conductivity detector should be PEEK tubing 0.005" ID (P/N 044221). PEEK tubing 0.010" ID (P/N 042260) may be used but peak efficiency will be compromised. Minimize the lengths of all connecting tubing and remove all unnecessary switching valves and couplers.

3.2. The Injection Loop

A 2.5 - 100 µL injection loop is required for IC-MS or IC-MS/MS determination of haloacetic acids using the IonPac AS24 2-mm column analytical system. Generally, you should not inject more than 12.5 nanomoles of any one analyte onto a 2-mm analytical column. Injecting a larger number of moles from 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 AS24 2-mm requires a microbore IC (Ion Chromatograph) system configuration.

3.3. The IonPac AG24 Guard Column

An IonPac AG24 Guard Column is normally used with the IonPac AS24 Analytical Column. Retention times will increase by approximately 2.5% 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. Replacing the AG24 Guard Column at the first sign of peak efficiency loss or decreased retention time will prolong the life of the AS24 Analytical Column.

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

The Eluent Generator (EG) with EGC II KOH cartridge and a CR-ATC Continuously Regenerated Trap Column (P/N 060477) should be installed at the EGC eluent outlet. This is required for removal of trace level anionic contaminants from the carrier deionized water. See the "Product Manual for CR-ATC (Document No. 031910)" for instructions.

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

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

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

3.5. Eluent Storage

IonPac AS24 columns are designed to be used with hydroxide eluent systems. Storage under a helium atmosphere ensures contamination free operation and proper pump performance. Nitrogen can be used if eluents do not contain solvents.

MS or MS/MS analysis requires post-column addition of acetonitrile. It is preferred that the acetonitrile be stored in glass bottles instead of plastic.

3.6. Anion Self-Regenerating Suppressor Requirements

An Anion Self-Regenerating Suppressor should be used whether suppressed conductivity or MS detection is used. 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® modes of operation. Solvent containing eluents should be used in the AutoSuppression® External Water Mode. For MS detection the suppressor must be used in the External Water Mode, since eluent recycle is not possible.

For detailed information on the operation of the Anion Self-Regenerating Suppressor, see the "Product Manual for the Anion Self-Regenerating Suppressor ASRS ULTRA II (Document No. 031367)."

3.7. Solvent Addition prior to ESI-MS detection

The AS24 is well suited for IC-MS and IC-MS/MS applications. Examples shown in this manual are generated using the MDS/Sciex API 2000 triple quadrupole mass spectrometer. In a typical IC-MS or IC-MS/MS configuration, the conductivity cell outlet is connected to the MS-electrospray inlet through a tee (P/N 063143). The tee is used to mix acetonitrile, pumped by an auxiliary pump with the analytical flow, before MS analysis. Post-column addition of acetonitrile improves the quality of electrospray and greatly enhances sensitivity.

3.8. Using the Eluent Generator with AS24

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

SECTION 4 - OPERATION

4.1. General Operating Conditions

Sample Volume:	2.5 µL Loop + 0.8 µL Injection valve dead volume
Column:	AS24 2-mm Analytical Column + AG24 2-mm Guard Column
Eluent:	55 mm KOH
Eluent Source:	EGCII KOH Cartridge with CR-ATC
Eluent Flow Rate:	0.30 mL/min
Temperature:	15 ºC
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA II (2-mm) or ASRS MS (2mm), External water mode
Expected Background Conductivity:	< 2 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

4.2. IonPac AS24 Operation Precautions

4.2.1. Filter and Degas Eluents

If you suspect particulates in your DI water supply, then it is recommended that you filter the eluents before connecting to the system. Degassing eluents will ensure optimum performance of the pump.

4.2.2. Filter Samples

Particulates in the sample can clog the bed support. Filtering samples can avoid this problem.

4.2.3. Eluent and Sample pH

AS24 can handle a wide range of samples or eluents, pH (0-14). However, it is always recommended to avoid switching eluents from one extreme to another. When in doubt about the pH compatibility of the column add a DI water step when switching eluents from one extreme to another.

4.2.4. Maximum Flow Rate

The average AS24 analytical and guard column operating flow rate is 0.30 mL/min. However, the AS24 can handle flow rates up to 0.50 mL/min. Running higher than the recommended flow rate for long periods of time can cause head space to the inlet of the column.

4.2.5. Maximum Operating Pressure

The average AS24 analytical and guard column operating pressure is below 2,300 psi at 15 ºC. However, the column can handle up to a maximum of 3,000 psi (20.7 MPa) of pressure. Exposing the column above 3,000 psi can cause head space to the inlet of the column.

4.3. Chemical Purity Requirements

Obtaining reliable, consistent, and accurate results requires eluents that are free of ionic impurities. Chemicals, solvents, and deionized water used to prepare eluents must be of the highest purity available. Low trace impurities and low particle levels in eluents also help to protect your ion exchange columns and system components. Dionex cannot guarantee proper column performance when the quality of the chemicals, solvents, and water used to prepare eluents has been compromised.

4.3.1. Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label.

4.3.2. Deionized Water

The deionized water used to prepare eluents should be a "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

Acetonitrile should be added postcolumn after the conductivity detection to enhance ESI-MS and ESI-MS/MS response. 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. Several manufacturers are currently making ultra high purity solvents that are compatible for HPLC and spectrophotometric applications. These ultra high purity solvents will usually ensure that your MS signal is not affected by ionic impurities. At Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

NOTE

The Anion Self-Regenerating Suppressor must be operated in the "Autosuppression External Water Mode" when using IC-MS or IC-MS/MS system.

4.4. Eluent Preparation

Using an eluent generator is recommended, however if an EG is not available hydroxide eluents can be prepared as described below.

4.4.1. Sodium Hydroxide Eluent Concentration

4.4.1.1. Weight Method

When formulating eluents from 50% sodium hydroxide, Dionex recommends weighing out the required amount of 50% sodium hydroxide. Use Fisher Grade 50% sodium hydroxide. Do not use pellets.

Example: To make 1 L of 55 mM NaOH use 4.4 g of 50% sodium hydroxide.

$$\textbf{For 55 mM:} \quad \frac{0.055 \text{ mole/L} \times 40.00 \text{ g/mole}}{50\%} = \textbf{4.4 g diluted to 1 L}$$

4.4.1.2. 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:

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

* This density applies to 50% NaOH. If the concentration of the NaOH solution is significantly different from 50%, the "Weight Method" calculation should be used instead.

4.4.2. Sodium Hydroxide Eluents

To make the AS24 eluents, dilute 50% (w/w) NaOH in DI water. The DI Water must be degassed-deionized (DI) water having a specific resistance of 18.2 megohm-cm. Create 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% NaOH and DI water.

Table 2
Dilution of 50% (w/w) NaOH to Make
1000 grams of Standard AS24 Eluents

50% (w/w) NaOH g (mL)	Concentration of NaOH Eluent (mM)
0.40 (0.26)	5
4.40 (2.88)	55
8.00 (5.25)	100
160.00 (104.6)	2 M

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" on optimized Ion Chromatographs. Different systems will differ slightly in performance due to slight differences in column sets, system void volumes, liquid sweep-out times of different components, and laboratory temperatures.

The IonPac AS24 is designed for the determination of haloacetic acids, using gradient hydroxide eluent delivered with an Eluent Generator. In any type of gradient elution system it is important to use eluents that produce a minimum shift in baseline conductivity during the run, as well as a fast equilibration time from one run to the next. The best choice for an eluent is sodium or potassium hydroxide as they are converted to water in the suppressor. 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 can begin at 1.0 mM NaOH and end at 80 mM NaOH, with a resulting total baseline change of 1 to 2 µS.

Ensure that adequate equilibration time is allowed between runs. If downward shift in baseline is observed during the isocratic section of the chromatogram, increase the equilibration time.

You can increase the sensitivity of your system by using sample concentration techniques. However, none has been used for the test chromatograms.

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

5.2. Production Test Chromatograms

Isocratic elution of inorganic anions on the IonPac AS24 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 IonPac AS24 Analytical Column should always be used with the IonPac AG24 Guard Column. To guarantee that all IonPac AS24 Analytical Columns meet high quality and reproducible performance specification standards, all columns undergo the following production control test.

Sample Volume:	2.5 µL Loop + 0.8 µL Injection valve dead volume
Column:	See chromatogram
Eluent Source:	EGC II KOH Cartridge with CR-ATC
Eluent:	55 mM KOH
Flow Rate:	0.30 mL/min
Temperature:	15 °C
Detection:	Suppressed Conductivity
Suppressor:	Anion Self-Regenerating Suppressor (ASRS®-ULTRA II 2-mm) AutoSuppression® Recycle Mode
Applied Current:	41 mA
Expected Background Conductivity:	< 3 µS
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Sulfate	10.0
3.	Chloride	10.0
4.	Thiosulfate	20.0
5.	Bromide	20.0
6.	Nitrate	20.0

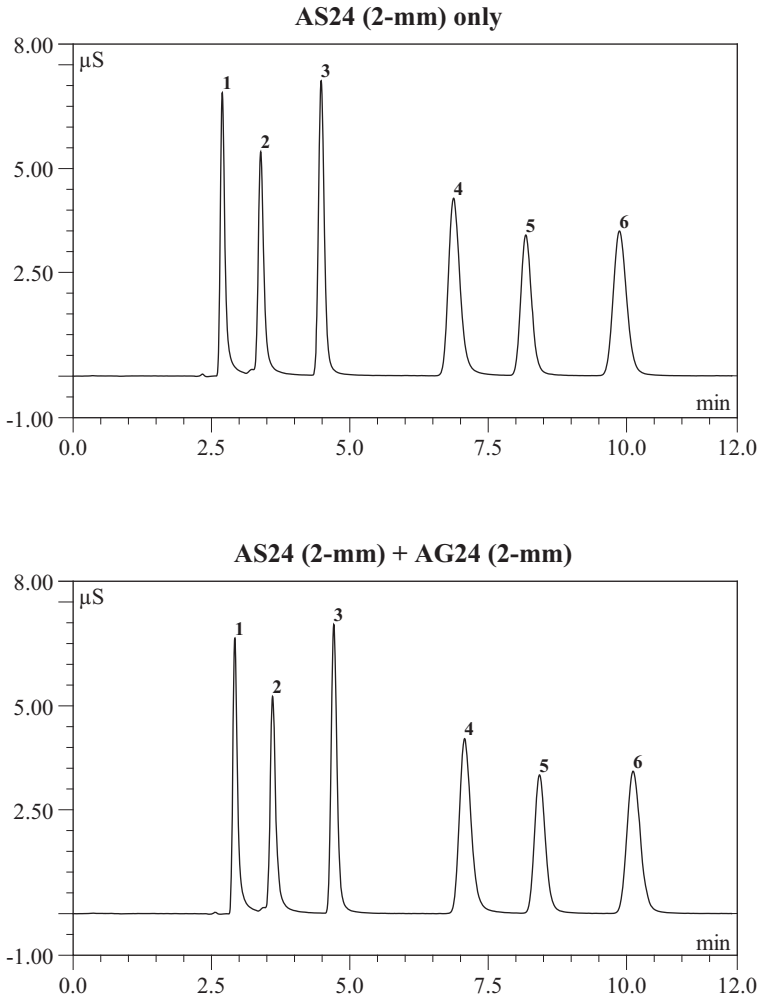


Figure 1
IonPac AS24 Production Test Chromatograms

5.3. Effect of Temperature on the AS24 Selectivity

The following chromatograms demonstrate the effect of temperature on the AS24 selectivity when using constant eluent concentration.

NOTE: There is a greater effect on the divalent anions (sulfate and thiosulfate) compared to monovalent (fluoride, chloride, bromide and nitrate) anions when the temperature is changed from 30 °C to 15 °C.

Column:	IonPac® AS24, AG24, 2 mm
Eluent Source:	EGC II KOH Cartridge
Eluent:	35 mM KOH
Flow Rate:	0.30 mL/min
Temperature:	See Chromatogram
Detection:	Suppressed Conductivity
Suppressor:	Anion Self-Regenerating Suppressor (ASRS®-ULTRA II 2-mm) AutoSuppression® Recycle Mode
Applied Current:	26 mA
Injection Volume:	2.5 µL
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Chloride	10.0
3.	Sulfate	10.0
4.	Bromide	20.0
5.	Nitrate	20.0
6.	Thiosulfate	20.0

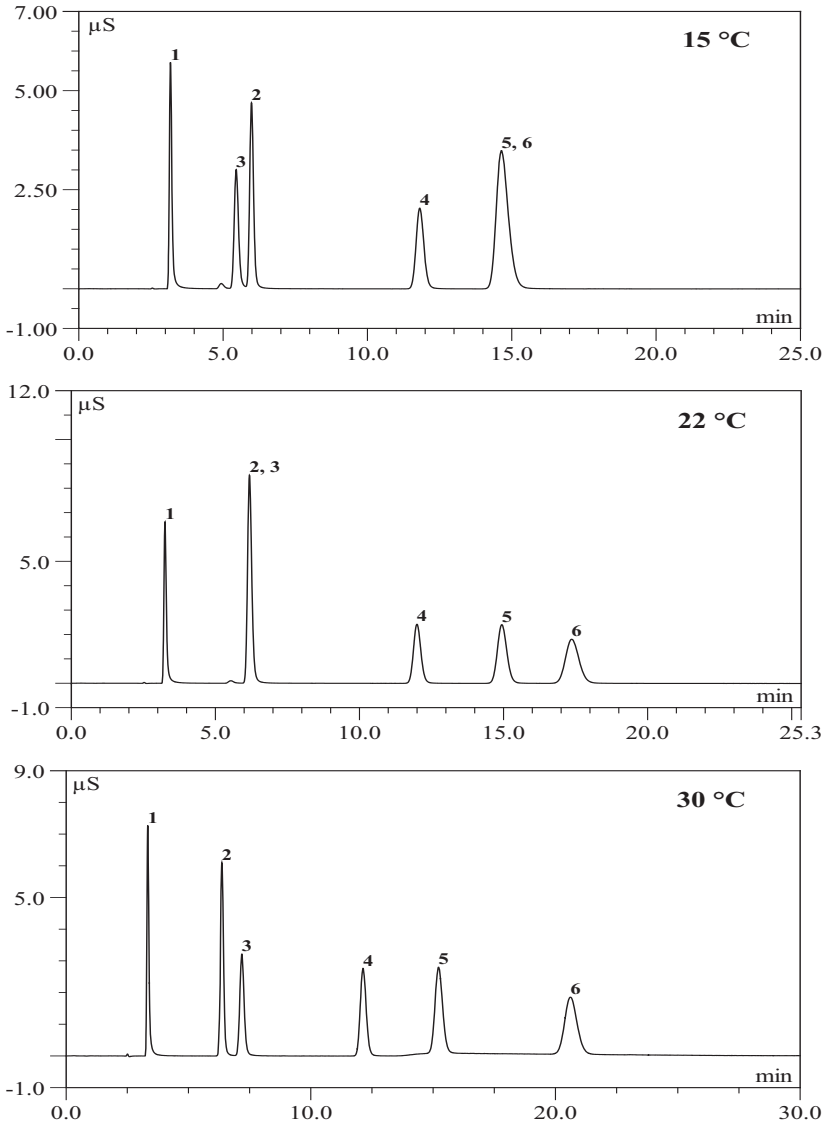


Figure 2
Effect of Temperature on the AS24 Selectivity

5.4. Separations of Various Inorganic and Organic Anions on AS24 Column

The following chromatogram demonstrates the separations of various inorganic and organic anions using the AS24 column.

Column:	IonPac® AS24, AG24, 2 mm
Eluent Source:	EGC II KOH Cartridge with CR-ATC
Eluent:	5 mM to15 mM KOH from 0 to 10 minutes, 15 mM to 30 mM KOH from 10 to 20 minutes; 30 to 60 mM KOH from 20 to 38 minutes
Flow Rate:	0.30 mL/min
Temperature:	15 °C
Detection:	Suppressed Conductivity
Suppressor:	Anion Self-Regenerating Suppressor (ASRS®-ULTRA II 2-mm) AutoSuppression® Recycle Mode
Applied Current:	45 mA
Injection Volume:	2.5 µL
Long-term Storage Solution (> 1 week):	100 mM Sodium Borate
Short-term Storage Solution (< 1 week):	Eluent

	Analyte	mg/L (ppm)
1.	Fluoride	5.0
2.	Acetate	15.0
3.	Formate	15.0
4.	Chlorite	15.0
5.	Bromate	15.0
6.	Chloride	15.0
7.	Trifloroacetate	15.0
8.	Nitrite	15.0
9.	Sulfate	15.0
10.	Chlorate	25.0
11.	Bromide	25.0
12.	Oxalate	25.0
13.	Nitrate	25.0
14.	Phosphate	40.0
15.	Arsenate	40.0
16.	Thiosulfate	40.0
17.	Chromate	40.0
18.	Citrate	40.0
19.	Isocitrate	40.0

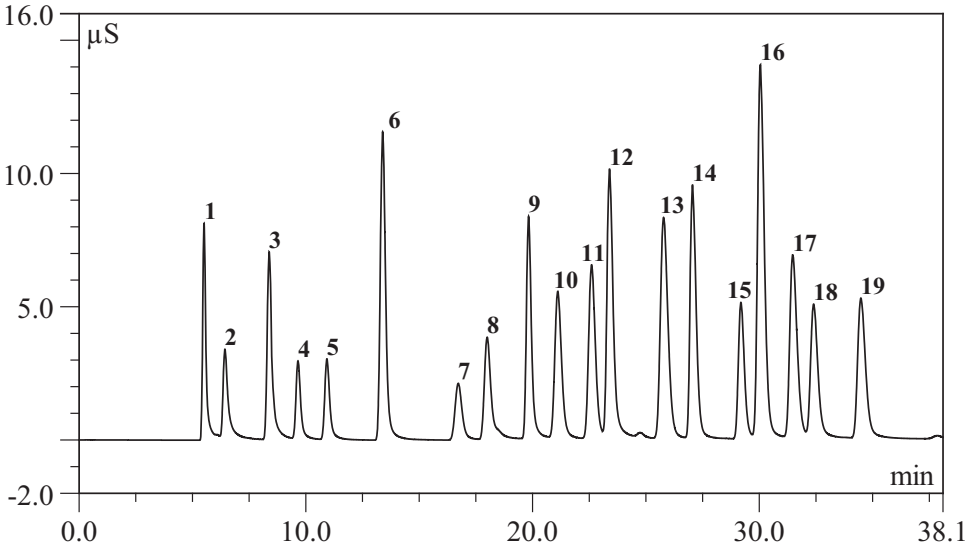


Figure 3
Separations of Various Inorganic and Organic Anions on AS24 Column

5.5. Drinking Water: A) Unfortified and B) Fortified with 0.5 ppb Bromate

The following chromatogram demonstrates the use of a Two-Dimensional Ion Chromatography System to determine trace concentrations of Bromate in drinking water samples. For further information on this application, refer to Application Note 187.

Second-Dimension Conditions

Column:	IonPac AG24, AS24, 2 mm		
Eluent Source:	EGC II KOH Cartridge		
Eluent:	10 mM KOH from 0 to 24 min,		
	65 mM from 24.1 to 35 min		
Flow Rate:	0.25 mL/min		
Suppressor:	Anion Self-Regenerating Suppressor (ASRS®-ULTRA II 2 mm)		
	AutoSuppression® External Water Mode		
Current:	41 mA		
Loop:	1000 µL (1st dimension)		
Conc. Column:	TAC-ULP1 (5 x 35 mm)		
Temperature:	30 °C		
Sample:	A) Unfortified DW		
	B) Fortified DW		
		A	**B**
Peak:	1. Bromate	1.19	1.69 mg/L

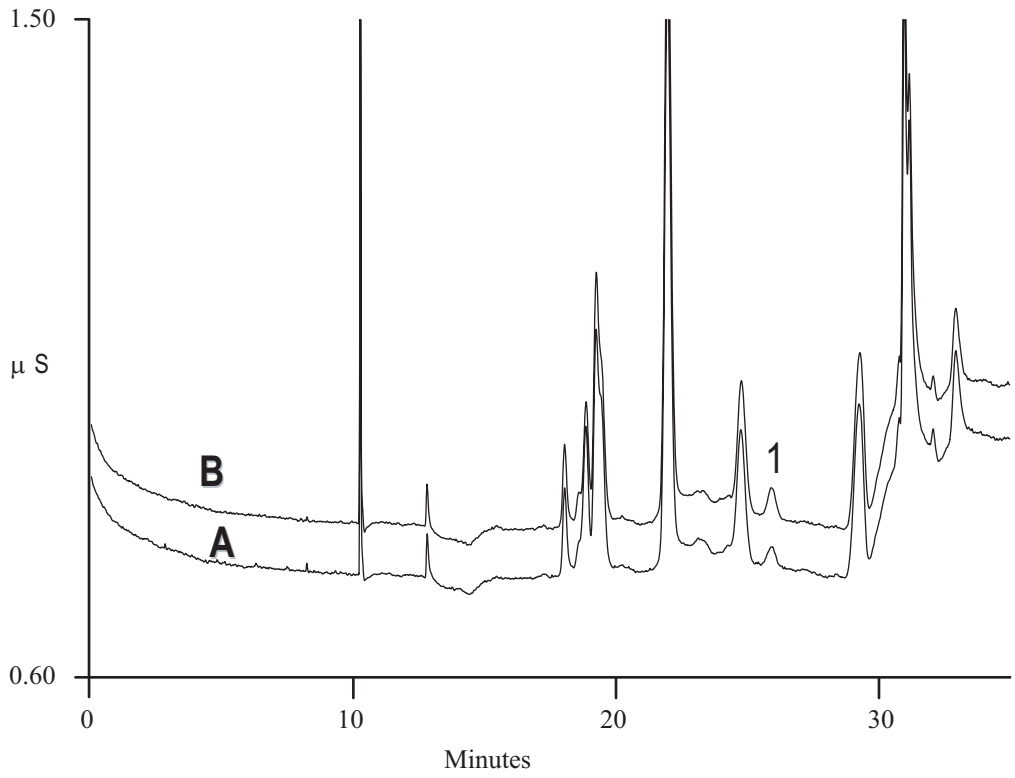


Figure 4
Drinking Water: A) Unfortified and B) Fortified with 0.5 ppb Bromate

5.6. Separation and Detection of 5 Haloacetic Acids using IC-MS

The Following Chromatogram demonstrates the separation of 5 Haloacetic Acids using IC-MS

Column:	IonPac® AS24, AG24, 2-mm
Eluent Source:	EGC II KOH Cartridge
Eluent:	KOH gradient
Flow rate:	0.3 mL/min
Suppressor:	Anion Self-Regenerating Suppressor (ASRS®-ULTRA II 2 mm) AutoSuppression® External Water Mode
Postcolumn solvent:	Acetonitrile, 0.2 mL/min
Column temp.:	15 °C
Injection vol.:	100 µL
Detector:	MSQ™ Plus, -ESI, 400 ^{o}C, 3 kV

Peaks: all 100 µg/L	**SIM m/z**	**cone voltage**
1. Chloroacetate	93	35
2. Bromoacetate	137	30
3. ^{13}C-Bromoacetate ISTD	139	35
4. Dichloroacetate	127	30
5. Dibromoacetate	216.8	40
6. Trichloroacetate	161	30

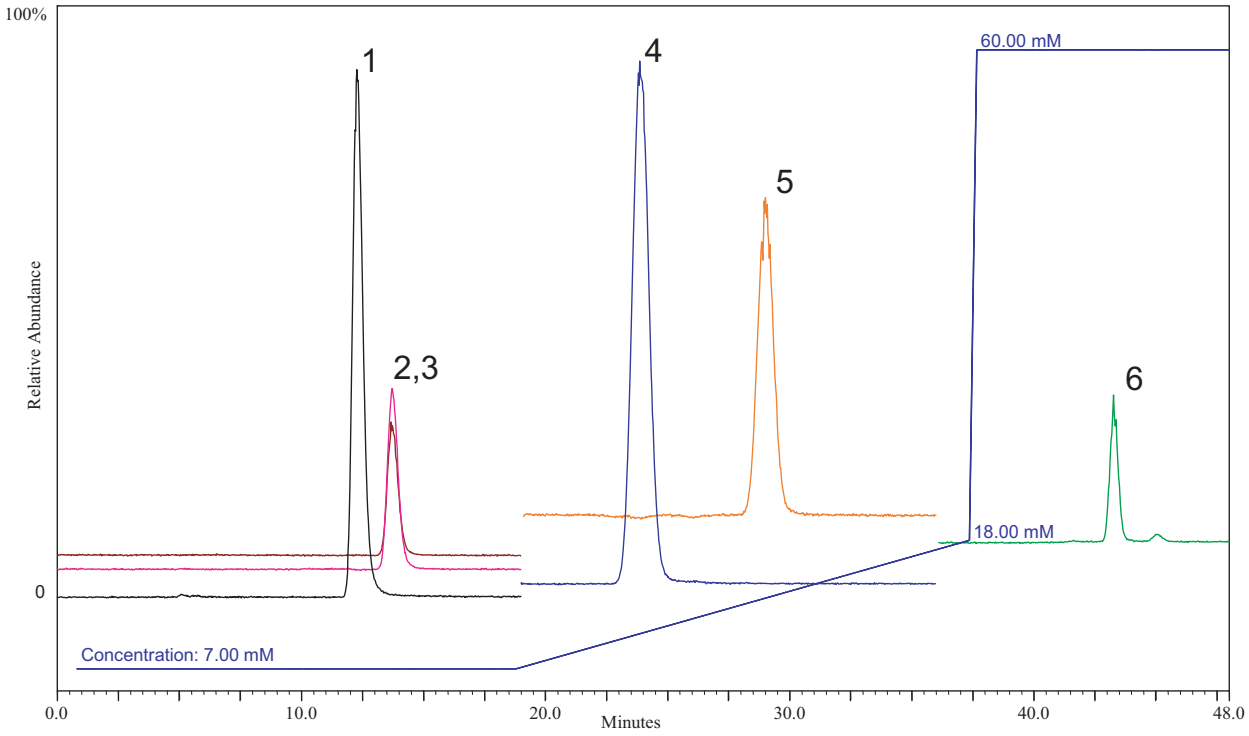


Figure 5
Separation and Detection of 5 Haloacetic Acids using IC-MS

5.7. Separation and Detection of 9 Haloacetic Acids using IC-MS/MS (See Dionex Application Note 217)

5.7.1. Equipment

Ion Chromatograph:
Dionex ICS-3000 composed of:

- DP Pump module (optional, if SP pump module is used an auxiliary pump is needed for postcolumn addition of acetonitrile such as AXP-MS pump)
- DC Detector/Chromatography module
- EG Eluent Generator module
- AS Autosampler
- Mixing tee (Upchurch, U-400) Dionex P/N: 063143
- Triple Quadrupole Mass Spectrometer:
- MDS Sciex API 2000™ Operated in MRM mode or equivalent

Software:

- Dionex Chromeleon® DCMS Link
- MDS Sciex Analyst

5.8. Consumables

- Source of nitrogen gas, 60 psi regulated to API 2000
- Source of Zero air 100 psi regulated to API 2000
- IonPac AS24, 2-mm I.D.
- IonPac AG24, 2-mm I.D. for fouling matrices
- ASRS® ULTRA II, 2-mm, P/N 053947 or ASRS MS 2-mm P/N 063008
- EGC-KOH Cartridge, P/N 053921
- CR-ATC Continuously regenerated Anion Trap Column P/N 060477

5.9. Experimental Conditions

Column:	IonPac AG24, AS24 (2 mm)
Suppressor:	ASRS ULTRA II, 2-mm, or ASRS MS 2-mm, external water, 60 mA
Eluent Source:	EG40 or EG50 or ICS-3000 EG module EGC-KOH
Flow Rate:	0.3 mL/min
Temperature:	15 °C
Injection Volume:	80-100 µL (see Figures)
Detection:	1. Conductivity 2. MS/MS MDS/Sciex API 2000
Run time:	56 min
Expected System Backpressure:	2100-2400 psi
Expected Background Conductance:	<2 µS

API 2000 MS/MS operating conditions for haloacetic acids

- Curtain gas: 20
- Collision gas: 5
- Temperature: 475 °C
- Ion Source Gas 1 (GS1): 75
- Ion Source Gas 2 (GS2): 85

Table 3
API 2000 Instrument Parameters for Measured Haloacetic Acids

Analyte	MRM Transitions	DP (volts)	FP (volts)	EP (volts)	CE (volts)	CXP (volts)
Chloroacetic acid	93/35	-20	-300	-10	-14	-6
Dichloroacetic acid	127/83	-11	-350	-7	-12	-14
Bromoacetic acid	136.8/78.8	-11	-350	-7	-12	-14
Trichloroacetic acid	161/117	-6	-290	-4	-6	-8
Bromochloroacetic acid	171/79	-16	-300	-6	-28	-8
Dibromoacetic acid	215/79	-11	-340	-4.5	-12	-10
Tribromoacetic acid	250.6/79	-11	-350	-5	-32	-12
Bromodichloroacetic acid	79/79	-12	-300	-1.5	-6	-14
Chlorodibromoacetic acid	207/79	-11	-310	-5	-20	-6

All nine haloacetic acids addressed in EPA methods are measured with negative polarity using the API 2000 in the MRM mode. The table shows the MRM transitions, declustering potential (DP), focusing potential (FP), entrance potential (EP), collision energy (CE) and cell exit potential (CXP) for the nine measured haloacetic acids. These values are obtained by manual optimization of the instrument. The collision gas was set at a value of 5 which corresponds to vacuum 2.5 e-5 torr. Application Note 217 contains conditions for the application using 5 mass spectrometers.

5.10. System Setup

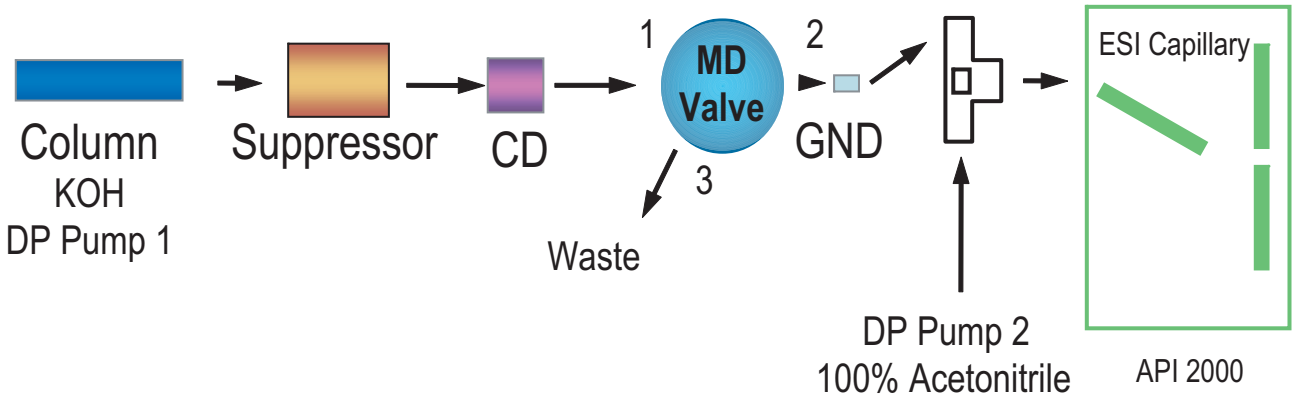


Figure 6
Flow diagram for IC-MS and IC-MS/MS Analysis

The setup described in this section provides a general guidance to the required connections for optimal performance. For information about individual modules, refer to the installation guides and manuals available for these modules. The ion chromatograph is configured for microbore operation. Red peak tubing (0.005" ID) should be used for all chromatographic connections, except the connections after the suppressor which should be black PEEK tubing (0.010"). The CR-ATC is installed between the pump and the eluent generator as described in the CR-ATC manual. The eluent generator and cartridge are purged and operated according to the eluent generator installation guide for low-flow rate operation. The backpressure through the system should not be less 2100 psi for optimal performance of the eluent generator. See EG40 or EG50 manual (P/N 031373 or 031908) for complete instructions. The column and suppressor are plumbed in the normal configuration. Referring to the Figure 6 above, the column outlet is connected to the suppressor. For MS detection, the suppressor is operated in the external water mode. The air pressure provided to the external water bottle is normally about 10-15 psi. Chemical regeneration of the suppressor is not recommended because higher background signal is seen in the MS.

The eluent out is connected to the conductivity cell inlet. The cell outlet is connected to a 6 port valve that is used for matrix diversion.

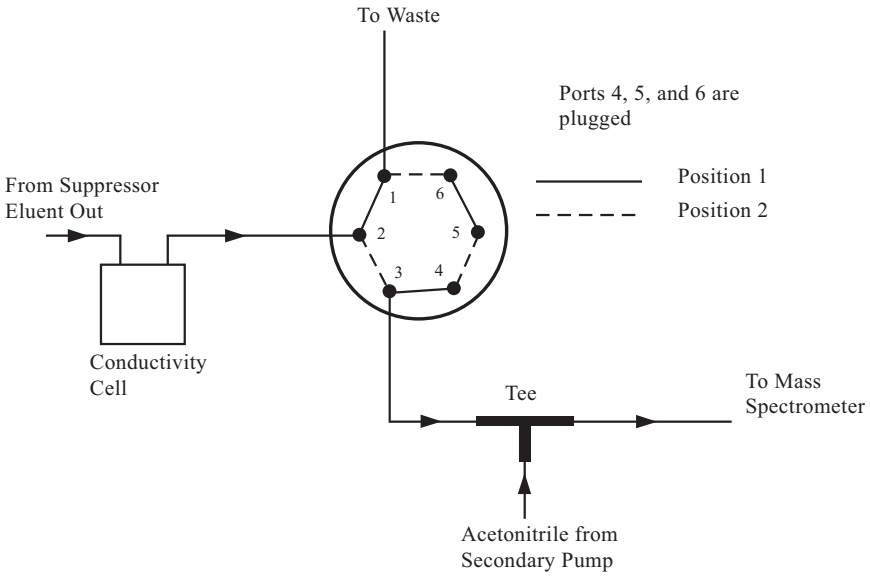


Figure 7
Connections from Ion Chromatograph to Mass Spectrometer through a Matrix Diversion Valve

Figure 7 shows the connections for the matrix diversion valve. When analytes are eluting the six port valve is set to the position 2 where the analytical flow from the conductivity cell is directed to the Mass Spectrometer for detection. At times when the matrix ions such as; sulfate, chloride, nitrate and carbonate are eluting, the valve is switched to position 1 where the appropriate connections are made to direct the flow to waste. Matrix diversion is necessary to prevent contamination of the mass spectrometer with matrix ions that are present at high concentration in samples. Poor sensitivity and poor recoveries will result if matrix ions that are typically present at ppm (mg/L) in samples are not diverted to waste. The mass spectrometer will require physical cleaning of the source followed by several hours of pumping down to regain pristine performance.
The IC should not be connected to the MS until the background conductivity is below 3 µS.

If an ICS 3000 is used any of the 6 or 10 port valves in the automation manager (AM) module can be used for matrix diversion.

Acetonitrile is added to the analytical flow to improve MS response. If an ICS 3000 with a DP pump module is used the second pump can be used for pumping acetonirile, otherwise, a second pump is needed for this purpose. An AXP-MS (P/N: 60684) auxiliary pump can be used. The optimum flow of acetonitrile is 0.2-0.3 mL/min. Higher flow rates should be avoided to prevent excessive backpressure on the suppressor.

Grounding Connection: A grounding adaptor is needed when the liquid line connection from the IC is made directly to the mass spectrometer electrospray probe. However, if the connection is made through the adapter present on the API 2000 or the MSQ Plus, there is no need for another ground.

5.11. Gradient Calculation for Separation of Haloacetic Acids

Haloacetic acids are separated using a hydroxide gradient. The separation should be done at a temperature of 15 °C to ensure stability and retention time of the analytes during separation. Since this column was developed for this separation at 15 °C this parameter is not optional for user selection. Also as the column capacity changes with use the retention times will usually shorten so the matrix diversion windows will need to be adjusted. The simplest method is to make an injection of 100 mg/L chloride and sulfate and note the start and stop times for these matrix ions. Set the divert valve times accordingly and make a trial run so that the analyte peaks are shown and the matrix peaks are not, as in Figure 9. A sample program in Chromeleon is provided in 5.12. Exact times will vary based on column capacity.

5.12. Example Chromatogram

The AS24 provides good separation of the common inorganic anions, plus monochloroacetic acid, monobromoacetic acid, dichloroacetic acid, chlorobromoacetic acid, dibromoacetic acid, trichloroacetic acid, dichlorobromoacetic acid, chlorodibromoacetic acid, tribromoacetic acid and bromate using potassium hydroxide eluent and mass spectrometric detection.

The separation of all nine haloacetic acid is shown in Figure 8.

Column:	IonPac® AS24, 250 x 2-mm I.D. plus AG24, 50 x 2-mm I.D.
Eluent:	KOH gradient (see below)
Suppressor:	ASRS® 300, 2-mm, external water
Suppressor Current:	65 mA
Analytical Flow rate:	0.3 mL/min
Postcolumn solvent:	Acetonitrile/DI water, 90/10 v/v, degassed
Postcolumn flow rate:	0.2 mL/min
Column temp.:	15 °C
Injection volume:	100 µL
Detector:	API 2000, MRM mode or equivalent
MRM conditions:	see Dionex Tech Note 217 for conditions using 5 models of mass spectrometers

Timed events	**KOH conc (mM)**	
-7.0	7	
0.0	7	
17.0		Matrix diversion valve to waste
18.0	7	
22.0		Matrix diversion valve to mass spectrometer
33.0		Matrix diversion valve to waste
36.5	18	
36.6	60	
41.0		Matrix diversion valve to mass spectrometer
52.0	60	
55.0	7	

Peaks	**Conc. (µg/L)**
1- Chloroacetic acid	3
2- Bromoacetic acid	2
3- Dichloroacetic acid	3
4- Bromochloroacetic acid	2
5- Dibromoacetic acid	1
6- Trichloroacetic acid	1
7- Bromodichloroacetic acid	2
8- Chlorodibromoacetic acid	5
9- Tribromoacetic acid	10

Figure 8 shows the separation of nine haloacetic acids in D.I. water by IC-MS/MS.

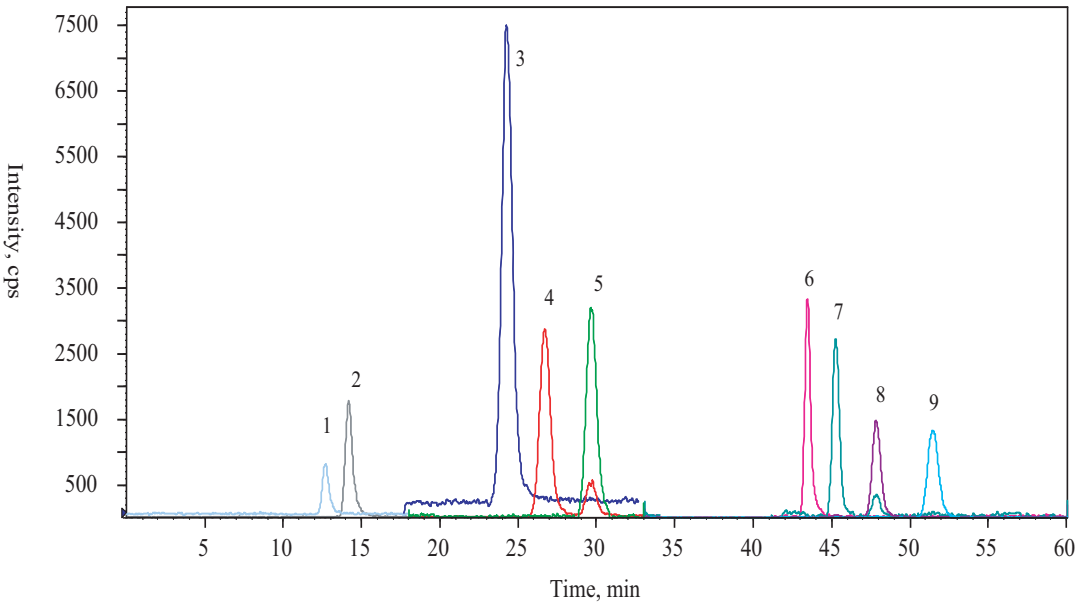


Figure 8
Analysis of Nine Haloacetic Acid in Deionized Water Using Hydroxide Gradient

Figure 9 shows the same separation in a simulated matrix composed of 250 mg/L chloride, 250 mg/L sulfate, 150 mg/L bicarbonate, 20 mg/L nitrate and 100 mg/L ammonium chloride. The baseline drop in both Figure 8 and Figure 9 between 17 min and 22 min as well as between 33 min and 41 min is due to diverting the analytical flow to waste. Diversion of flow to waste between 17 min and 22 min sends chloride to waste. The flow between 33 min and 41 sends carbonate, sulfate and nitrate to waste.

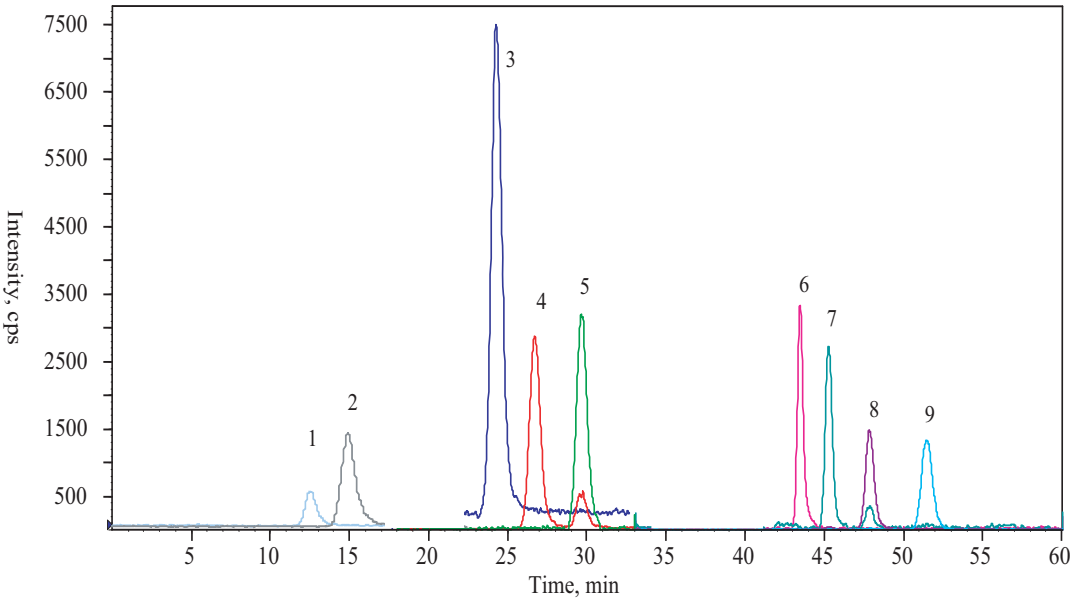


Figure 9
Analysis of Nine Haloacetic Acid in Simulated Water Matrix Using Hydroxide Gradient

SECTION 6 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help solve operating problems that may arise while using IonPac AS24 columns. For more information on problems that originate with the Ion Chromatograph (IC) or the suppressor, refer to the Troubleshooting Guide in the appropriate product manual. For additional help contact the "Dionex North America Technical Call Center" at 1-800-DIONEX-0 (1-800-346-6390), or the nearest Dionex Office (see, "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

Table 4
IonPac AS24 / AG24 Troubleshooting Summary

Observation	Cause	Action
High Back Pressure	Unknown	Isolate Blocked Component
	Plugged Column Bed Supports	Replace Bed Supports, Filter Eluents, and Filter Samples
	Other System Components	Unplug, Replace
High Background Conductivity	Contaminated Eluents	Remake Eluents
	Contaminated Trap Column	Clean Trap Column
	Contaminated ASRS	Clean Suppressor
	Contaminated Hardware	Clean Component
	Contaminated Regenerant Water	Replace Water
	Flow Rate Regenerant Water Too Low	Check Flow Rate
Poor Resolution	Poor Efficiency Due to Large System Void Volumes	Replumb System
	Column Headspace	Replace Column
Short Retention Times	Flow Rate Too fast	Recalibrate Pump
	Conc. Incorrect Eluents	Remake Eluents
	Column Contamination	Clean Column
Poor Front End	Conc. Incorrect Eluents	Remake Eluents
Resolution	Column Overloading	Reduce Sample Size
	Sluggish Injection Valve	Service Valve
	Large System Void Volumes	Replumb System
Spurious Peaks	Sample Contaminated	Pretreat Samples
	Sluggish Injection Valve	Service Valve

6.1. High Back Pressure

6.1.1. Finding the Source of High System Pressure

Total system pressure for the IonPac AG24 (2-mm) Guard Column plus the AS24 (2-mm) Analytical Column, when using the test chromatogram conditions, should be equal or less than 2,300 psi at 15 °C. If the system pressure is significantly higher it is advisable to determine the cause of the high system pressure. The system should be operated with a High-Pressure In-Line Filter (P/N 044105) which is positioned between the Gradient Pump pressure transducer and the injection valve. Make sure you have one in place and that it is not contaminated. The maximum flow rate is 0.50 mL/min and the maximum pressure is 3,000 psi.

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: plugged tubing or tubing with a collapsed wall, 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;

1. Disconnect the pump eluent line from the injection valve
2. Turn the pump on.
3. Watch the pressure; it should not exceed 50 psi.
4. Continue adding system components (injection valve, column(s), suppressor and detector) one by one while monitoring the system pressure.
5. The pressure should increase up to a maximum when the Guard and Analytical columns are connected (see Table 4, "Typical AS24 Operating Back Pressures").

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

Table 5
Typical IonPac AS24 / AG24 Operating Back Pressures

Column	Typical Back Pressure at 30 °C psi (MPa)	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
AS24 2-mm Analytical	< 2,000 (13.8)	0.3	0.5
AG24 2-mm Guard	< 300 (2.07)	0.3	0.5
AS24 2-mm Column Set	< 2,300 (15.87)	0.3	0.5

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, and 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 another 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. Make sure that the end of the column tube is clean and free of any particulate matter.
g. Place a new bed support assembly into the end fitting. seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting.

Table 6
IonPac AS24 2-mm Columns

Product	IonPac AS24 2-mm Columns (P/N)
Bed Support Assembly	044689
End Fitting	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.***

h. Screw the end fitting back onto the column.
i. Tighten the screw finger tight, then an additional 1/4 turn (25 in. lb.). Tighten further only if leaks are observed.
i. Reconnect the column to the system and resume operation.

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

6.1.3. Filter Eluent

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

6.1.4. Filter Samples

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

6.2. High Background or Noise

The background conductivity level for the standard eluent system is shown below.

Table 7
Standard System Eluent Background Conductivity

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

6.2.1. Preparation of Eluents

a. Ensure the eluents and the regenerant (if used) were made correctly.
b. Ensure the eluents were made from chemicals with the recommended purity.
c. Ensure the deionized water, used to prepare the reagents, had a specific resistance of 18.2 megohm-cm.

6.2.2. A Contaminated Trap Column

High background may be caused by contamination of the ATC-HC or ATC-3 with carbonate or other anions from the eluent. Clean the ATC-HC or 4-mm ATC-3 with 100 mL of 2.0 M NaOH or 50 mL for the 2-mm ATC-3. Rinse the ATC-HC or 4-mm ATC-3 immediately with 20 mL of eluent or 10 mL of eluent for the 2-mm ATC-3 into a beaker prior to use.

6.2.3. A Contaminated CR-ATC Column

For EG50 or EG40 operation, use a CR-ATC Trap Column. Install a CR-ATC Anion Trap Column (P/N 060477) if using an Eluent Generator with EGC II KOH cartridge. If the CR-ATC becomes contaminated please refer to the "CR-ATC Product Manual (Document No. 031910) Section 6."

6.2.4. A Contaminated Guard or Analytical Column

Remove the IonPac AG24 Guard and AS24 Analytical Columns from the system. Install a backpressure coil that generates approximately 1,500 psi and continue to pump eluent. If the background conductivity decreases the column(s) is (are) the cause of the high background conductivity. To eliminate downtime, clean or replace the AS24 at the first sign of column performance degradation as compared to the original test chromatogram. Clean the column(s) as instructed in, "Column Cleanup" (See "Column Care").

6.2.5. Contaminated Hardware

To eliminate hardware as the source of the high background conductivity, bypass the columns and the suppressor. Install a backpressure coil that generates approximately 1,500 psi and continue to pump eluent. Pump deionized water with a specific resistance of 18.2 megohm-cm through the system. The background conductivity should be less than 2 µS. If it is not, then check the detector/conductivity cell calibration by injecting deionized water directly into it. See the appropriate manual for details.

6.2.6. A Contaminated ASRS ULTRA II or ASRS-MS Suppressor

If the above items have been checked and the problem persists, the Anion Self-Regenerating Suppressor (ASRS ULTRA II or ASRS-MS) is probably causing the problem. For details on ASRS operations refer to the "Product Manual for ASRS ULTRA II" (Document No. 031367).

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 Auto Suppression External Water mode
c. Check the eluent flow rate.
d. Replace the Regenerant water

6.2.7. A Contaminated Regenerant Water

If the suppressor is used with external water mode, especially in IC-MS or IC-MS/MS applications, contaminated water can affect the system background and elevate conductivity and background.

6.3. Poor Peak Resolution

Poor peak resolution can be due to any or all of the following factors.

6.3.1. Loss of Column Efficiency

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

b. Symmetric Inefficient Peaks: Extra-column effects can result in sample band dispersion making the peaks' elution less efficient. Make sure you are using PEEK tubing with an ID of no greater than 0.005" before the conductivity cell or no greater than 0.010" between the conductivity cell and the MS to make all eluent liquid line connections. Cut the tubing lengths as short as possible. Check for leaks.

6.3.2. Poor Resolution Due to Shortened Retention Times

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

a. Check the flow rate: See if the eluent flow rate is equivalent to the flow rate specified by the analytical protocol. Measure the eluent flow rate after the column using a stopwatch and graduated cylinder.

b. Check to see if the eluent compositions and concentrations are correct: An eluent that is too concentrated will cause the peaks to elute faster. Prepare fresh eluent. If you are using a gradient pump to proportion the eluent, components from two or three different eluent reservoirs, the resulting eluent composition may not be accurate enough for the application. Use one reservoir containing the correct eluent composition to see if this is the problem. This may be a problem when one of the proportioned eluents is less than 5%.

c. Column contamination can lead to a loss of column capacity: This is because all of the anion exchange sites will no longer be available for the sample ions. For example, polyvalent anions from the sample or metals may concentrate on the column. Refer to, "Column Cleanup" (see "Column Care"), for recommended column cleanup procedures. Possible sources of column contamination are impurities in chemicals and in the deionized water used for eluents or components of the sample matrix. Be especially careful to make sure that the recommended chemicals are used. The deionized water should have a specific resistance of 18.2 megohm-cm.

d. Diluting the eluent will improve peak resolution, but will also increase the analytes' retention times. If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution, or if the resulting increase in retention times is unacceptable, clean the column (see, "Column Cleanup" in "Column Care").

After cleaning the column, reinstall it in the system and let it equilibrate with eluent for about 30 minutes. No water wash is necessary. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment sense the contaminants should be eluted from the column. If you need assistance in solving resolution problems contact the "Dionex North America Technical Call Center" at 1-800-DIONEX-0 (1-800-346-6390), or the nearest Dionex Office (see "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

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 then check for the following possible problems:

a. Improper eluent concentration: Remake the eluent as required for your application. Ensure that the water and chemicals used are of the required purity.

b. Column overloading: 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: 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: Swap components, one at a time, in the system prior to the analytical column and test for front-end resolution after every system change. Use the shortest tubing lengths possible.

APPENDIX A - Column Care

A.1 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 AS24 column is 3,000 psi (20.7 MPa).

A.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.

A.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. You can also place the column in the refrigerator for short or long term storage.

A.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 but the following precautions should be observed.

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

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

A.4.1 Choosing the Appropriate Cleanup Solution

a. Concentrated hydroxide solutions such as a 10X concentrate of the most concentrated eluent used in the application is sufficient to remove hydrophilic contamination of low valence.

b. Concentrated acid solutions such as 1 to 3 M HCl, remove high valence hydrophilic ions by ion suppression and elution by the chloride ion.

c. Metal contamination often results in asymmetric peak shapes and/or variable analyte recoveries. For example, iron or aluminum contamination often results in tailing of sulfate and phosphate. Aluminum contamination can also result in low phosphate recoveries.

Concentrated acid solutions such as 1 to 3 M HCl remove a variety of metals. If after acid treatment, the chromatography still suggests metal contamination, treatment with chelating acids such as 0.2 M oxalic acid is recommended.

d. Organic solvents can be used alone if the contamination is nonionic and hydrophobic. The degree of nonpolar character of the solvent should be increased as the degree of hydrophobicity of the contamination within the range of acceptable solvents listed in Table 3, HPLC Solvents for Use with IonPac AS24 Column.

e. Concentrated acid solutions such as 1 to 3 M HCl can be used with compatible organic solvents to remove contamination that is ionic and hydrophobic. The acid suppresses ionization and ion exchange interactions of the contamination with the resin. The organic solvent then removes the subsequent nonionic and hydrophobic contamination. See Section D above.

   A frequently used cleanup solution is 200 mM HCl in 80% acetonitrile. This solution must be made immediately before use because the acetonitrile will decompose in the acid solution during long term storage.

f. Regardless of the cleanup solution chosen, use the following cleanup procedure in, "Column Cleanup Procedure", to clean the AS24 Analytical and Guard Columns.

A.4.2 Column Cleanup Procedure

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

b. Disconnect the ASRS ULTRA II, from the IonPac AS24 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.

c. Set the pump flow rate to 0.25 mL/min for a AS24 Analytical or Guard Column.

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

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

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

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

h. Reconnect the ASRS ULTRA II, to the AS24 Analytical Column and place the guard column in line between the injection valve and the analytical column if your system was originally configured with a guard column.

APPENDIX B - Configuration

Table 8
Configuration

SRS Suppressor	ASRS ULTRA II (2-mm) (P/N 061562)	
Injection Loop	2.5 - 100 µL	
System Void Volume	Eliminate unnecessary switching valves, couplers and the Gradient Mixer.	
Pumps	Use the DP/GS50/GP50/GP40/IP20/IP25 in Microbore Configuration with a Microbore GM-4 (2-mm) Gradient Mixer.	***The use of an EG40 (P/N 053920) or EG50 (P/N 060585) with an EGC II KOH cartridge (P/N 058900) for gradient applications is highly recommended for minimum baseline change when performing eluent step changes or gradients.***
	The GPM 2 can be used for 2-mm isocratic chromatography at flow rates of 0.5 mL/min or greater. GPM 2 cannot be used for 2-mm gradient chromatography.	
Detectors	CD20, CD25, CD25A, ED40, ED50, or ED50A	
	Conductivity Cell with DS3 P/N 044130 or Conductivity Cell with shield P/N 044132 or P/N 061830	
	CDM 2, CDM 3 Cell P/N 042770	***Do not use the TS 2 or the TS 1 with the ED40/ED50/ED50A or the CD20/CD25/CD25A.***
	CDM 2 or the CDM 3: Replace the TS 1 with the TS 2 (P/N 043117). The TS-2 has been optimized for 2-mm operation.	

Table 9
Tubing Back Pressures

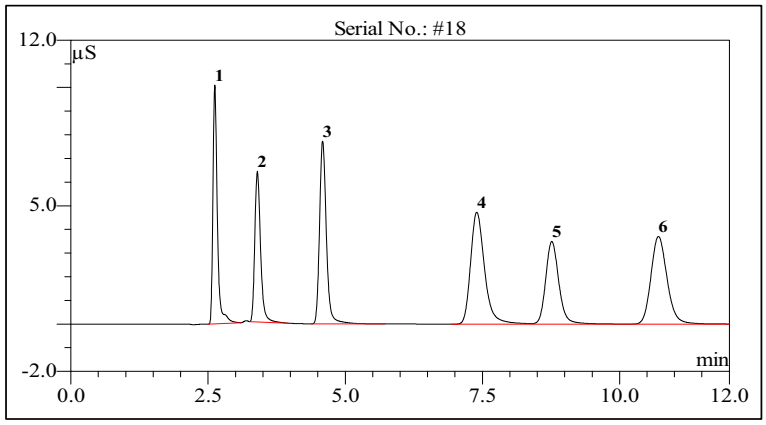
Color	Dionex P/N	ID Inches	ID cm	Volume mL/ft	Back Presure psi/ft at 1 mL/min	Back Presure psi/ft at 0.25 mL/min	Back Presure psi/cm at 1 mL/min
Green	044777	0.030	0.076	0.137	0.086	0.021	0.003
Orange	042855	0.020	0.051	0.061	0.435	0.109	0.015
Blue	049714	0.013	0.033	0.026	2.437	0.609	0.081
Black	042690	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

Quality Assurance Report 2x250 mm

IonPac® AS24
Analytical (2 x 250 mm)
Product No. 064153

Date: 07-Jun-07 09:29
Serial No. : #18
Lot No. : 06-18-149

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®-ULTRA II 2-mm) AutoSuppression® Recycle Mode
Applied Current: 41 mA
Injection Volume: 2.5 µL
Long-term Storage Solution (> 1 week): 100 mM Sodium Borate
Short-term Storage Solution (< 1 week): Eluent



No.	Peak Name	Ret.Time (min)	Asymmetry (AIA)	Resolution (EP)	Efficiency (EP)	Concentration (mg/L)
1	Fluoride	2.62	1.5	4.82	5933	5.0
2	Sulfate	3.40	1.5	5.93	5380	10.0
3	Chloride	4.59	1.3	8.49	7151	10.0
4	Thiosulfate	7.40	1.4	3.15	4388	20.0
5	Bromide	8.76	1.2	4.10	6853	20.0
6	Nitrate	10.71	1.3	n.a.	6609	20.0

QA Results:

Analyte	Parameter	Specification	Results
Thiosulfate	Efficiency	>=3600	Passed
Thiosulfate	Asymmetry	1.0-2.0	Passed
Thiosulfate	Retention Time	6.02-7.78	Passed
(Bromide-Thiosulfate)/ (Nitrate-Bromide)	Retention Time Ratio	0.64-0.86	Passed
	Pressure	<=2200	1625

Production Reference:
Datasource: QAR
Directory: Anion\AS24
Sequence: AS24_2X250MM
Sample No.: 1

6.80 Build 2212