Configuring the Dionex Integrion HPIC System for Fast Anion Determinations Using Prepared Eluents

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Technical Note 174

Key Words
Standard Bore, Microbore, Drinking Water, Integrion, IonPac AS22 column, IonPac AS22-Fast-4µm column

Goal
Provide instructions for installing a fast anion application on the Thermo Scientific™ Dionex™ Integrion™ HPIC™ system.

Introduction
The latest advancement in integrated ion chromatography (IC) instrumentation, the Thermo Scientific Dionex Integrion HPIC system, can operate continuously up to 5000 psi for both 4 mm and 2 mm i.d. column formats with automated eluent generation and 6000 psi with manual eluent preparation. These higher pressures allow the analyst to take full advantage of the high efficiency and fast separations offered by smaller particle size separation columns.

The Dionex Integrion HPIC system features the following:

- Compact fully integrated system design
- Separate compartments for pump, column heater with injection valve, and detector with suppressor to provide faster column oven equilibration
- Thermo Scientific™ Dionex™ IC PEEK Viper™ fittings that replace standard fitting connections in specified positions and minimize void volume problems to improve chromatography and ensure accurate reporting
- Components that are tracked by consumables device tracking technologies for GMP compliance tracking and to assure installation of compatible devices
- Independent tablet control for convenient continuous monitoring, independent manual control, and access to the instrument manual and troubleshooting guides
- New Thermo Scientific™ Dionex™ Chromeleon™ 7 Chromatography Data System (CDS) software features that provide easy instrument configuration, monitoring of consumable devices, and integrated video instructions for conditioning columns, suppressors, and other electrolytic devices

In this technical note, we provide installation recommendations to set up a Dionex Integrion HPIC system configured for analytical flow rates (0.10–2.0 mL/min) with manually prepared eluents. Results that highlight some of the instrument, column, and suppressor features will be shown.

Equipment
- Thermo Scientific Dionex Integrion HPIC system including the following:
  - Optional CD Conductivity Detector
  - Optional Column Oven Temperature Control
  - Optional Tablet Control
  - Optional Consumables Device Tracking Capability
- Thermo Scientific™ Dionex™ AS-DV Autosampler (with 5 mL vial adapter (P/N 068907) or with 0.5 mL vial adapters (P/N 068908)

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Software

Chromeleon CM 7.2 SR4 CDS software was used.

Table 1 lists the consumable products needed for the Dionex Integrion HPIC system configured for suppressed conductivity detection.

Table 1. Consumables list for the Dionex Integrion HPIC System.

Product Name	High-Pressure Device	Part Number
Dionex IC PEEK Viper Fitting Tubing Assembly Kits	Thermo Scientific Dionex IC PEEK Viper Fitting Kit for Dionex Integrion HPIC System with Conductivity Detector. Includes one each of P/Ns: 088805, 088806, 088808, 088810	088796
Dionex IC PEEK Viper Fitting Tubing Assemblies, Included in Kit, P/N 088796	Guard column to separator column: 0.007 × 4 in (102 mm)	088805
	Injection valve, Port C (Port 2) to guard column: 0.007 × 5.5 in (140 mm)	088806
	Separator column out to Suppressor Eluent In: 0.007 × 7.0 in (178 mm)	088808
	Suppressor Eluent Out to CD In: 0.007 × 9.0 in (229 mm)	088810
Dionex AS-DV Autosampler Vials: 5 mL	5 mL PolyVials with plain caps: box of 250	038008
	5 mL PolyVials with filter caps: box of 250	038141
	Vial holders for 5 mL vials	068947
Dionex AS-DV Autosampler Vials: 0.5 mL	0.5 mL PolyVials with plain caps: box of 250	038010
	0.5 mL PolyVials with filter caps: box of 250	038142
	Vial holders for 0.5 mL vials	068948
Thermo Scientific™ Dionex™ AERS™ 500 Carbonate Suppressor, 4 mm	For applications shown in Figures 9A, 9B, and 10: Suppressor for 4 mm and 5 mm columns, using carbonate eluents and recycle mode	085029
Thermo Scientific™ Dionex™ IonPac™ AG22-Fast-4µm column, 4 mm	For applications shown in Figures 9B and 10: Fast anion guard column, 4 × 30 mm	088487
Dionex IonPac AS22-Fast-4µm column, 4 mm	For applications shown in Figures 9B and 10: Fast anion separation column, 4 × 150 mm	088486
Dionex AERS 500 Carbonate Suppressor, 2 mm	For application shown in Figure 11: Suppressor for 2 mm and 3 mm columns, using carbonate eluents and recycle mode	085028
Dionex IonPac AG22-Fast-4µm column, 2 mm	For application shown in Figure 11: Fast anion guard column, 2 × 30 mm	088489
Dionex IonPac AS22-Fast-4µm column, 2 mm	For application shown in Figure 11: Fast anion separation column, 2 × 150 mm	088488
Thermo Scientific™ Nalgene™ Syringe Filter	Syringe filters, 25 mm, PES membrane, 0.2 µm. This type is compatible with IC analysis.	Thermo Scientific 7252520 / Fisher Scientific 09-740-113

Chromatographic Conditions	
Columns	Thermo Scientific™ Dionex™ IonPac™ AG22 guard and Dionex IonPac AS22 separation columns (the column format is shown in the chromatograms)
Eluent	4.5 mM sodium carbonate, 1.4 mM sodium bicarbonate
Flow Rate	See chromatograms
Column Temperature	30 °C
Injection Volume	10 or 2.5 µL, see chromatograms
Detection	Suppressed conductivity, Dionex AERS 500 Carbonate Suppressor, 4 mm for 4 mm i.d. columns or 2 mm for 2 mm i.d. columns, recycle mode
Run Time	See chromatograms
Background Conductance	~ 20 µS
Noise	< 1 nS
System Backpressure	Described in the Results section

Reagents and Standards

- 18 MΩ-cm resistivity degassed deionized (DI) water
- Thermo Scientific™ Dionex™ Combined Seven Anion Standard II, NIST traceable (P/N 057590)
- Thermo Scientific™ Dionex™ AS22 Carbonate/ Bicarbonate Concentrate (P/N 063965)

Eluent Preparation

Dilute the Dionex AS22 Concentrate 1:100 with DI water. Alternatively, dissolve 476 ± 1 mg anhydrous sodium carbonate (Na_2CO_3) and 84.0 ± 1 mg of sodium bicarbonate ($NaHCO_3$) in a 1 L volumetric flask containing approximately 500 mL DI water. Dilute to 1 L mark with DI water, cap, and mix thoroughly by inverting the flask several times.

Instrument Set Up and Installation

The Dionex Integrion HPIC system is configured with a CD Conductivity Detector with the intention to use manually prepared eluents, optional column oven temperature control, and is optional consumables device tracking enabled. This system can also be controlled manually by the programmed optional Dionex Integrion HPIC system tablet. This system can operate at pressures up to 6000 psi without eluent generation.

To set up this application, connect the Dionex AS-DV autosampler and the Dionex Integrion HPIC system according to Figure 1. Note that the injection valve is plumbed through different ports than previous Dionex IC systems.

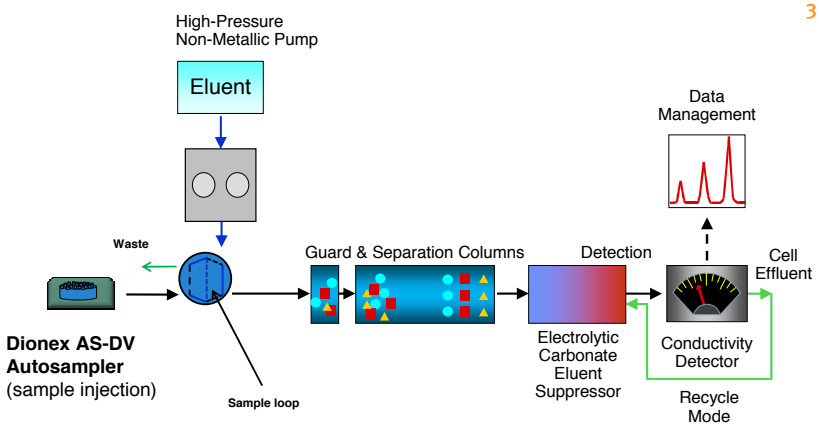


Figure 1. Flow diagram for the Dionex Integrion HPIC System.

Connect the USB cables from the Dionex Integrion HPIC system to the Dionex AS-DV autosampler and to the computer. Connect the power cables and turn on the IC instrument and the autosampler.

Configuring the Modules in the Chromeleon CDS Software

To configure the IC system, first start the Chromeleon Instrument Controller program and then select the link, *Configure Instruments*, to open the Chromeleon Instrument Configuration Manager. Right-click on computer name, select *Add an Instrument*, and enter an appropriate name (for example: Integrion_EPA300_1). Add the following modules to this instrument configuration: Integrion HPIC system, Integrion HPIC Pump Wellness, and Dionex AS-DV Autosampler.

Integrion HPIC System Module

Select *Add a Module, IC: Dionex Integrated Modules*, and *Integrion HPIC System* (Figure 2). The configuration for each module is summarized at the end of this section in Table 2.

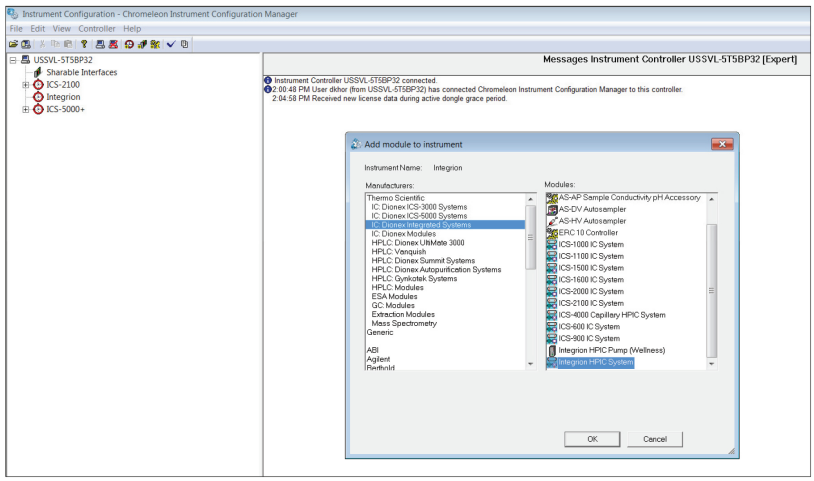


Figure 2. Creating a configuration.

A window with multiple tabs will automatically open up (Figure 3). Select the Model Serial No. on the General tab. The Chromeleon CDS software will automatically detect all electrolytic devices, detectors (Figure 3), Pump Degasser, and Seal Wash pump (not shown).

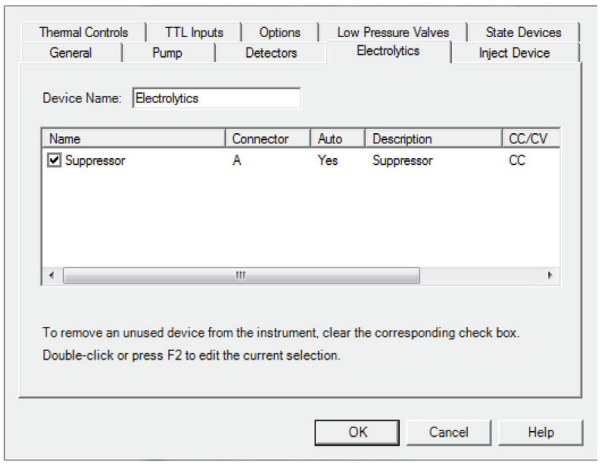


Figure 3. Automatic Detection of Electrolytic Devices.

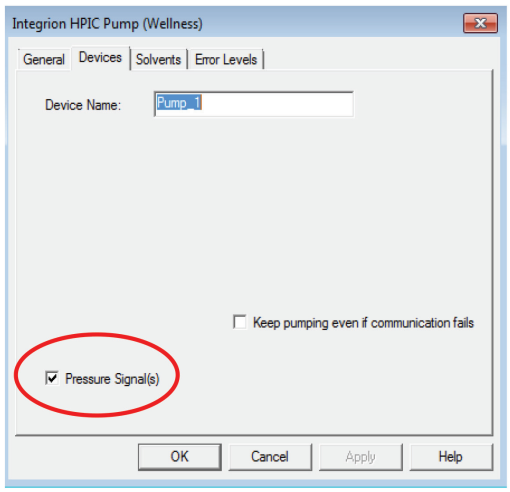


Figure 4. Adding the Integrion HPIC Pump Wellness Module to the Instrument Configuration.

Integrion HPIC Pump Wellness Module

To add pressure monitoring capabilities, it is necessary to add another module: right-click and select *Add a Module, IC: Dionex Integrated Modules, Integrion HPIC Pump Wellness* module and then select the USB address to link the module to the configuration. Select the Devices tab and click on the Pressure Signal(s) box (Figure 4).

Add a Dionex AS-DV Autosampler to the Configuration

Add a new module by selecting the Dionex AS-DV autosampler. Right-click on the module and select the USB address. Leave all parameters with the default options.

Table 2. System configuration for Dionex Integrion HPIC System.

Tab	Action	Result
Integrion HPIC Module		
General	Link to USB address	-
Pump	-	Flow rate and pressure limitations are displayed
Detectors	-	Automatically detected
Electrolytics	-	Automatically detects suppressor (Figure 3)
Inject Device	-	Automatically detected
Thermal Controls	-	Automatically detects thermal control options for column. Thermal control for detector and suppressor are not available for this model.
High-Pressure Valves	-	Automatically detected
Low-Pressure Valves	-	Automatically detected
Options	-	Automatically detects Pump Degasser and Seal Wash pump
Pump Wellness Module		
Devices	Check Pressure Signal(s) box	Activates pressure monitoring feature (Figure 4)
Add Autosampler		
Add Dionex AS-DV Autosampler Module	Link to USB address	Leave all settings on default

Plumbing the High-Pressure Dionex Integrion HPIC System

First loosen the waste lines, including the metal-wrapped waste line, in the back of the instrument and direct the free ends to a waste container. To plumb the system, first connect the pump eluent line to the eluent bottle containing 4.5 mM sodium carbonate/1.4 mM sodium bicarbonate eluent. Prime the pump by opening the priming knob ¼ turn and pressing the priming button. Prime the pump until no bubbles are visible and water is flowing at a steady rate out of the pump waste line. Close priming knob to finger-tight. For more information, review the Dionex Integrion HPIC System Operations Manual[1] by selecting "?" on the tablet.

IC PEEK Viper fittings

Tip: To achieve the best chromatography, it is important to gently tighten the IC PEEK Viper fittings to finger-tight and to use the fitting assemblies in the following locations (Figure 5):

- Injection Valve — "Column" port to the guard column
- Between the guard and separation columns
- Separation column to *Eluent In* on the Dionex AERS 500 Carbonate Suppressor
- Dionex AERS 500 Carbonate Suppressor — *Eluent Out* to *Eluent In* on CD Conductivity Cell

IC PEEK Viper fittings (Figure 5) minimize void volume in critical chromatography components, such as the columns and suppressor. These fittings are also recommended for use in consumable devices, such as the eluent generator cartridge and trap column to minimize installation issues. The tubing is standard for both standard bore and microbore column applications.

The tubing length is specified for each connection (see Table 1). The recommended practice is to tighten the IC PEEK Viper fittings to finger-tight and then, if leaking is observed, an additional 1/16 to 1/8 turn clockwise. Caution: Using a wrench or any other tool to tighten the IC PEEK Viper fittings may permanently damage the fitting.

Figure 5. Dionex IC PEEK Viper Fittings.

Important: Do not remove consumables tracking device tags from the columns or consumable devices. These tags are required for monitoring functionality.

Conditioning Suppressor and Columns

To hydrate the Dionex ERS 500 Suppressor, follow the QuickStart Instructions received with the suppressor or those in the suppressor product manual.[2] Wait for 20 min for the suppressor to fully hydrate before installing the suppressor in the Detector compartment. Install the black PEEK (0.010 in i.d. tubing) backpressure loop from the slotted compartment next to the CD detector (exerting an additional ~40 psi) between the CD outlet and the suppressor *Regen In* port.

Condition the columns for 30 min according to the instructions from the *Consumables, Install Column* section in the Chromeleon CDS software (**Figure 6**). The general practice is use the eluent and flow rate conditions listed in the QAR report while directing the eluent exiting the column to a waste container.[3] Complete the installation according to the **Figure 1** flow diagram.

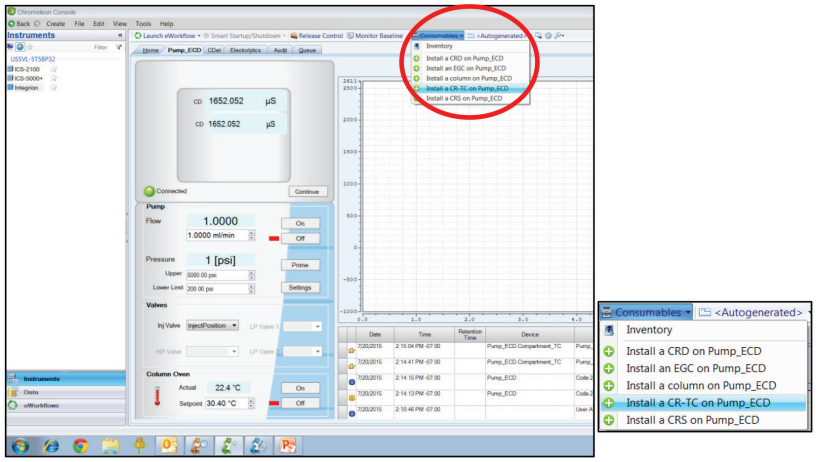


Figure 6. Consumables online installation instructions.

Installing the Dionex AS-DV Autosampler

Install the autosampler transfer line into Port S in the injection valve. For more information review the Thermo Scientific Dionex AS-DV Operator's Manual.[4]

Starting the Dionex Integrion HPIC System

To start the system, turn on the pump and immediately turn on the Dionex ERS 500 Carbonate Suppressor when liquid is flowing through the device. Set the eluent concentration and suppressor current, compartment oven, and cell temperatures as shown in the Chromatographic Conditions section. Allow the system to equilibrate for 30 min.

Creating an Instrument Method

To create a new instrument method using the Chromeleon Wizard, select Create, Instrument Method, and select an Instrument. Enter the values from the Chromatographic Conditions section and those in Table 3 for the Dionex AS-DV autosampler (See also Figure 7). Save the instrument method.

Table 3. Additional conditions to create a program.

Page Title	Mode	Action
Preparation Options for AS-DV	Option 1: Full injection of sample vial	Edit Mode, Advanced, DeliverSample, Command: Full
	Option 2: Injection of set volume (10 µL as example)	Edit Mode, Advanced, DeliverSample, Command: 10 µL
	Option 3: Injection based on flush volume	Edit Mode, Basic
	-	To estimate flush volume: Enter loop volume (µL) in Total Volume Delivered Estimator. Will add in delay volume *10x flush factor for a total volume of 1350 µL

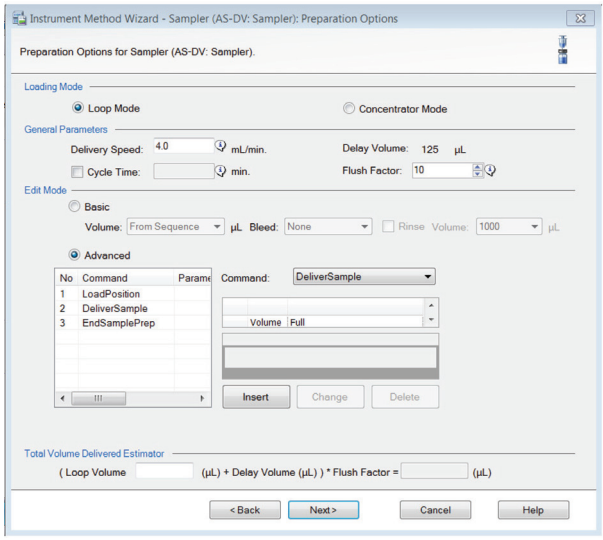


Figure 7. Creating an instrument program when using the Dionex AS-DV autosampler (Option 1 is shown).

Consumable Device Monitoring

Tip: An action (either approve or correct an incompatibility between devices) is required to start a sequence after installing any new consumable device.

A new feature of the Dionex Integrion HPIC system is cable and radio frequency device monitoring and tracking, which automatically detects the electrolytic devices and the columns. Review and approval of the devices is required to start the first sequence on the Dionex Integrion HPIC system and when new consumables are installed. To access this approval, select *Consumables* and then *Inventory* (Figure 8). The device monitoring shows the device history, tracking: *Part No., Size, Serial Numbers, Manufacture Lot*, installed location *(On Device)*, and *Best if Used by Date* (Figure 8, top). Additionally, the device monitoring will provide warnings if there is incompatibility with the devices installed (Figure 8, bottom left). To start the sequence, correct any errors, review the inventory, *Approve*, and *Close* the page (Figure 8, bottom right). Then select the Instrument Queue tab, and conduct a *Ready Check* on the sequence and *Start*.

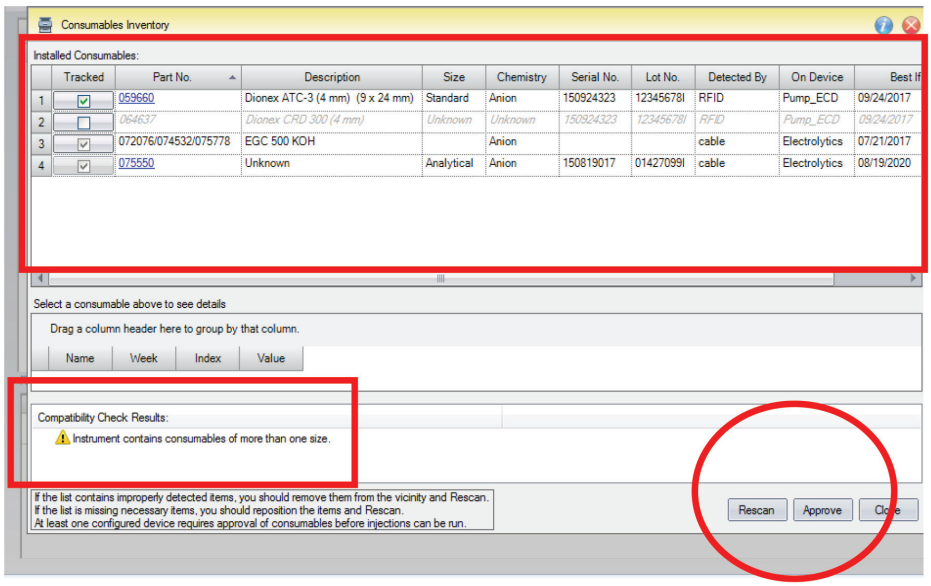


Figure 8. Consumables tracking.

Results and Discussion

The determination of inorganic anions in municipal drinking, waste, and bottled waters according to the U.S. EPA Methods 300.0 and 300.1 is the most popular and widely used IC method. The Dionex IonPac AS22 column family represents the latest innovation in columns optimized for inorganic anion separations using carbonate eluents. The Dionex IonPac AS22 column family includes:

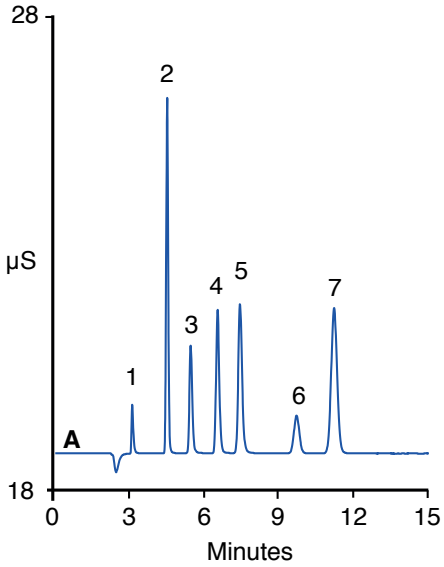
- Three formats of the 250 mm length, 6 µm super macroporous resin particle columns (0.4 mm, 2 mm, and 4 mm i.d.) when higher resolution is needed for more complex samples,
- Two formats of the Dionex IonPac AS22-Fast, 150 mm length, 6 µm super macroporous resin particle columns (2 mm, and 4 mm i.d.) for faster separations of well-defined samples,
- Three formats of the Dionex IonPac AS22-Fast-4µm 150 mm length, 4 µm super macroporous resin particle columns (0.4 mm, 2 mm, and 4 mm i.d.) combining higher resolution with faster runs.

Three formats of Dionex IonPac AS22 anion exchange columns—a 250 mm length column with 6 µm resin particles (4 mm i.d.), and two 150 mm length columns with 4 µm resin particles, (4 and 2 mm i.d.)—were used to demonstrate the determination of anions with the new Dionex AERS 500 Carbonate Suppressor on the Dionex Integrion HPIC system. This suppressor was designed and optimized for carbonate eluents; as a result, the noise is low, < 1 nS.

An anion standard was run under the same conditions at 1.2 mL/min on two formats of 4 mm i.d. Dionex IonPac AS22 column (Figure 9).

Figure 9. Chromatogram A — the standard length Dionex IonPac AS22 column (4 × 250 mm) with 6 µm resin particles. The system backpressure was relatively low at ~ 1800 psi.

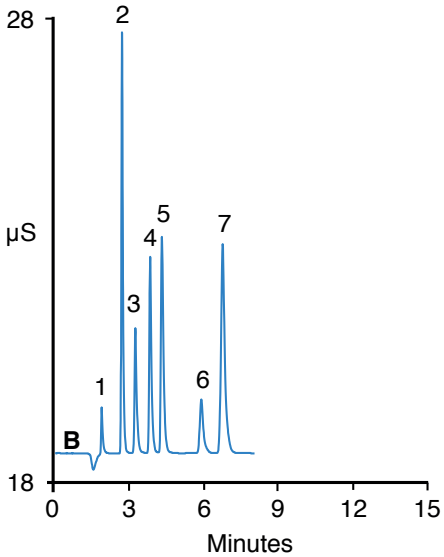
Figure 9 Chromatogram B — the fast 4 µm resin particle column, the Dionex IonPac AS22-Fast-4µm. The chromatograms demonstrate that both applications (7 min versus 12 min runs) can be run on the Dionex Integrion HPIC system. With the Dionex IonPac AS22-Fast-4µm column the system backpressure was 50% higher at ~ 2700 psi.



Column: A: Dionex IonPac AG22
Dionex IonPac AS22, 4 × 250 mm
Eluent: 4.5 mM Sodium Carbonate
1.4 mM Sodium Bicarbonate
Flow Rate: 1.2 mL/min
Inj. Volume: 10 µL
Column Temp.: 30 °C
Detection: Suppressed conductivity,
Dionex AERS 500 Carbonate, 4 mm,
41 mA, recycle mode
System
Backpressure: ~ 1800 psi

Peaks:			
1. Fluorid	1.0	mg/L
2. Chlorid	12	
3. Nitrite	8.0	
4. Bromide	9.0	
5. Sulfate	10	
6. Nitrate	9.0	
7. Phosphate	6.5	

Figure 9A. Seven anion standard.

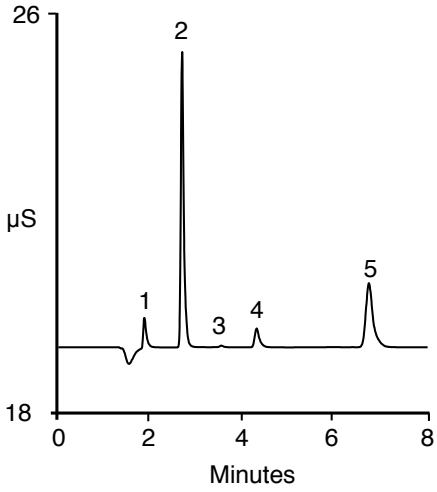


Column: B: Dionex IonPac AG22-Fast-4µm
Dionex IonPac AS22-Fast-4µm,
4 × 150 mm
Eluent: 4.5 mM Sodium Carbonate
1.4 mM Sodium Bicarbonate
Flow Rate: 1.2 mL/min
Inj. Volume: 10 µL
Column Temp.: 30 °C
Detection: Suppressed conductivity,
Dionex AERS 500 Carbonate, 4 mm,
41 mA, recycle mode
System
Backpressure: ~ 2700 psi

Peaks:			
1. Fluoride	1.0	mg/L
2. Chloride	12	
3. Nitrite	8.0	
4. Bromide	9.0	
5. Sulfate	10	
6. Nitrate	9.0	
7. Phosphate	6.5	

Figure 9B. Seven anion standard.

In Figure 10, anions in a diluted, filtered swimming pool water sample were determined in 7 min at 1.2 mL/min on the 4 mm Dionex IonPac AS22-Fast-4µm column. The system backpressure was ~ 2700 psi.

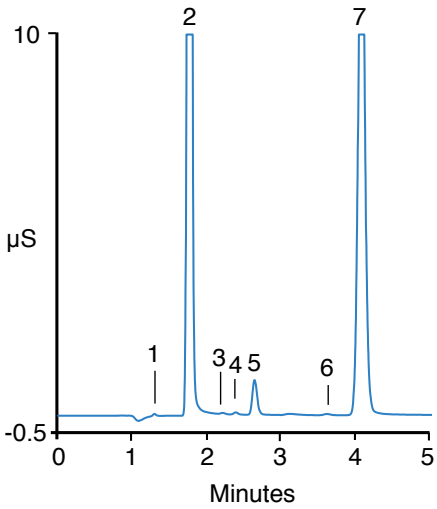


Column:	Dionex IonPac AG22-Fast-4µm Dionex IonPac AS22-Fast-4µm, 4 × 150 mm
Eluent:	4.5 mM Sodium Carbonate 1.4 mM Sodium Bicarbonate
Flow Rate:	1.2 mL/min
Inj. Volume:	10 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity, Dionex AERS 500 Carbonate, 4 mm, 41 mA, recycle mode
Sample Prep.:	4-fold dilution with DI water, filter, 0.2 µm
System Backpressure:	~ 2700 psi

Peaks:	
1. Fluoride	< 1
2. Chloride	6.5
3. Chlorate	0.012
4. Nitrate	< 1
5. Sulfate	0.3

Figure 10. Fast Determinations of anions in a pool water sample.

In Figure 11, anions in a municipal drinking water sample were determined in 5 min at 0.5 mL/min on the 2 mm, the Dionex IonPac AS22-Fast-4µm column. The flow rate is equivalent to 2.0 mL/min on the 4 mm i.d. format. The system backpressure was ~ 2600 psi.



Column:	Dionex IonPac AG22-Fast-4µm Dionex IonPac AS22-Fast-4µm, 2 × 150 mm
Eluent:	4.5 mM Sodium Carbonate 1.4 mM Sodium Bicarbonate
Flow Rate:	0.5 mL/min
Inj. Volume:	2.5 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity, Dionex AERS 500 Carbonate, 2 mm, 17 mA, recycle mode
System Backpressure:	~2600 psi

Peaks:	
1. Fluoride	< 1
2. Chloride	120
3. Nitrite	< 1
4. Bromide	< 1
5. Nitrate	1.0
6. Phosphate	
7. Sulfate	56

Figure 11. Fast determination of anions in a municipal drinking water sample.

Conclusion

The installation and set up of the Dionex Integrion HPIC system were discussed.

The analysis of environmental water samples was demonstrated using different format Dionex IonPac AS22 columns:

- The standard 4 × 250 mm length, with 6 µm resin particle particles
- The 4 × 150 mm length, with 4 µm resin particle particles
- The 2 × 150 mm length, with 4 µm resin particle particles

Seven anions were separated using manually prepared carbonate/bicarbonate eluents and detected by suppressed conductivity with the Dionex AERS 500 Carbonate suppressor designed and optimized for carbonate eluents. The system noise was low at < 1 nS.

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