Configuring the Dionex Integrion HPIC System for High-Pressure Reagent-Free Ion Chromatography

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Key Words

Standard Bore, Fast IC, IonPac AS19-4µm column, IonPac AS18-4µm column, IonPac AS19 column, Reagent-Free IC

Goal

Provide installation instructions to install a Reagent-Free™ IC application using standard bore columns on the Thermo Scientific™ Dionex™ Integrion™ HPIC™ system.

Introduction

The latest advancement in ion chromatography (IC) instrumentation, the high-pressure Thermo Scientific™ Dionex™ Integrion™ HPIC™ system, can operate continuously up to 5000 psi for both 4 mm and 2 mm i.d. column formats when using automated eluent generation. The Dionex Integrion HPIC system can therefore take advantage of the high-efficiency separations offered by smaller-particle-size separation columns. When combined with the advantages and ease-of-use of Reagent-Free™ IC (RFIC™), this system provides enhanced resolution of closely eluting peaks with excellent reproducibility, thereby yielding greater quantification accuracy and consistently reliable results.

The Dionex Integrion HPIC system, configured for Reagent-Free IC with suppressed conductivity detection, includes many of the recent advancements in IC instrument technology, including high-pressure capabilities, Reagent-Free IC (RFIC) consumables for inline eluent generation, column heater control, and new features designed to make the system easier to use:

- Compact, fully integrated system design
- Easy access to eluent generator cartridge and electrolytic trap column
- Separate compartments for pump, column heater with injection valve, and detector with suppressor to provide faster column oven equilibration and to extend temperature control to suppressors
- Thermo Scientific[™] Dionex[™] IC PEEK Viper[™] fittings replace standard fitting connections in specified positions to minimize void volume problems, improving chromatography and ensuring accurate reporting



- Components are tracked by consumables device tracking technologies for GMP compliance tracking and to assure installation of compatible devices
- Independent tablet control for convenient, continuous chromatography 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 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 for a Dionex Integrion HPIC system configured for analytical flow rates (0.10–2.0 mL/min) using electrolytically generated eluents. Results will highlight some of the instrument, column, and suppressor features.



Equipment

- Thermo Scientific Dionex Integrion HPIC high-pressure IC system, including:
 - Eluent Generation Capabilities
 - CD Conductivity Detector
 - Column Oven Temperature Control
 - Detector-Suppressor Compartment Temperature Control
 - Tablet Control
- Thermo Scientific™ Dionex™ AS-AP Autosampler with 10 mL vial trays

Software

Thermo Scientific Dionex Chromeleon CM 7.2 SR4 CDS software.

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

Table 1. Consumables for the Dionex Integrion HPIC system.

Table 1. Consumables for the Di	onex integriori in te system.	
Product Name	High-Pressure Device	Part Number
Dionex IC PEEK Viper Fitting Tubing Assembly Kits	Dionex IC PEEK Viper fitting assembly kit for the Integrion HPIC system using conductivity detection: Includes one each of P/Ns: 088805 through 088811	088798
Dionex IC PEEK Viper Fitting Tubing Assemblies, Included in Kit, P/N 088798	Guard outlet to separator column: 0.007 × 4.0 in (102 mm)	088805
	Injection Valve, Port C (Port 2) to guard column: 0.007×5.5 in (140 mm)	088806
	EGC Eluent Out to CR-TC Eluent In: 0.007 × 6.5 in (165 mm)	088807
	Separator 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
	CR-TC Eluent Out to Degasser In: 0.007 × 9.5 in (241 mm)	088811
Dionex AS-AP Autosampler Vials	Package of 100, polystyrene vials, caps, blue septa, 10 mL	074228
Thermo Scientific™ Dionex™ EGC™ 500 KOH Eluent Generator Cartridge	Eluent generator cartridge recommended for anion applications using 4 µm particle resin columns	075778
Thermo Scientific™ Dionex™ CR-ATC™ 600 Electrolytic Trap Column	Continuously regenerated trap column used with Dionex EGC KOH 500 cartridge	088662
HP EG Degasser Module	Degasser installed after Dionex CR-TC trap column and before the Injection Valve. Used with eluent generation	075522
Thermo Scientific™ Dionex™ AERS™ 500 Suppressor	Suppressor for 4 mm and 5 mm columns, using recycle mode	082540
Thermo Scientific™ Dionex™ IonPac™ AG18-4μm Column	For the applications shown in Figures 9 (Chromatogram B), 10, and 11: Anion guard column, $4\times30~\text{mm}$	076035
Dionex IonPac AS18-4µm Column	For the applications shown in Figures 9 (Chromatogram B), 10, and 11: Anion separation column, $4\times150~\text{mm}$	076034
Dionex IonPac AG19-4µm Column	For the application shown in Figure 12: Anion guard column, $4\times50~\text{mm}$	083221
Dionex IonPac AS19-4µm Column	For the application shown in Figure 12: Anion separation column, $4\times250~\text{mm}$	083217
Thermo Scientific™ Nalgene™ Syringe Filter	Syringe filters, 25 mm, PES membrane, 0.2 µm. This syringe filter is compatible with IC analysis.	Thermo Scientific 7252520 / Fisher Scientific 09-740-113

Chromatographic Conditions Columns Dionex IonPac columns, 4 mm i.d. Eluent See chromatograms **Eluent Source** Dionex EGC 500 KOH eluent cartridge, Dionex CR-ATC 600 and high-pressure degas module Flow Rate See chromatograms Column Temperature 30 °C Injection Volume See chromatograms Detection/Suppressor 15 °C Compartment Detection Suppressed conductivity, Dionex AERS 500 suppressor, 4 mm, recycle mode Run time See chromatograms **Background Conductance** $< 1 \mu S$ Noise 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)

Instrument Setup and Installation

The Dionex Integrion HPIC system is configured with a CD Conductivity Detector with the intention to use eluent generation and temperature control of the column oven and detector-suppressor compartment. This system has consumables device tracking through cable and radio frequency, optional manual controls by the Dionex Integrion HPIC system tablet, and can operate at pressures up to 5000 psi with eluent generation.

To set up this system, connect the Dionex AS-AP 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.

Dionex Integrion HPIC System Fresh 18 MΩ-cm Resistivity Deionized Water Dionex EGC S00 cartridge Dionex ERS 500 Suppressor Vent line Dionex ERS 500 Suppressor Columns

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

Connect the USB cables from the IC system to the 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 Dionex 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-AP 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.

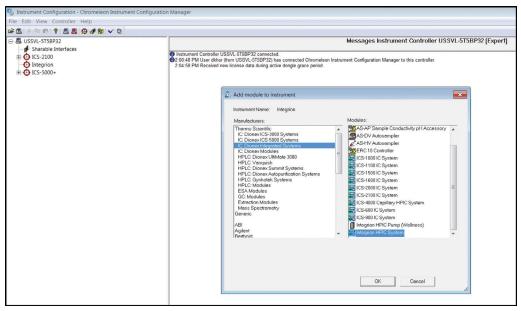


Figure 2. Creating a configuration.

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

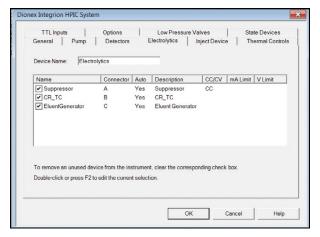


Figure 3. Automatic Detection of Electrolytic Devices.

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 a USB address to link the module to the configuration. Select the Devices tab and click on the Pressure Signal(s) box (Figure 4).

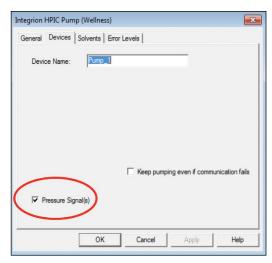
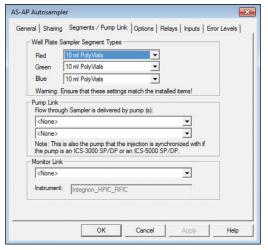


Figure 4. Adding the Integrion HPIC Pump Wellness Module to the Instrument Configuration.

Add the Dionex AS-AP Autosampler to the Configuration

Add a new module by selecting the Dionex AS-AP autosampler. Right-click on the module and select the USB address. On the Segments/Pump Link tab, select the appropriate vial trays for each color zone. On the Options tab, select *Push*, installed syringe size, 1.2 mL for buffer size, and enter the sample loop volume (Figure 5). Save the configuration, select check the configuration, and then close the Chromeleon Instrument Configuration program.



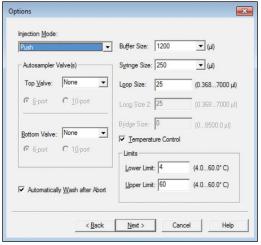


Figure 5. Configuring the AS-AP Autosampler.

As shown in Table 2, many of the devices are automatically detected providing a fast, easy configuration.

Table 2. Summary of the System Configuration for a Dionex Integrion HPIC system.

Tab	Action	Result
Integrion HPIC Module	'	
General	Link to USB address	(Figure 2)
Pump	-	Flow rate and pressure limits are displayed
Detectors	-	Automatically detected
Electrolytics	-	Automatically detects Dionex eluent generator cartridges and Dionex CR-TC trap columns (Figure 3)
Inject Device	-	Automatically detected
Thermal Controls	-	Automatically detects thermal control options for column, detector, and suppressor
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 Dionex AS-AP Auto	sampler	
Add Module	Link to USB address	-
Sharing	-	Only if more than one instrument is detected. If this option is present, select Instrument
Segments / Pump Link	-	Select 10 mL polystyrene vials or 1.5 mL vials for "Red", "Blue", and "Green" (Figure 5)
Options	-	Select Push, select syringe size, select 1.2 mL buffer line, enter the loop size (Figure 5)
Finish Instrument Conf	iguration	
Check Configuration	Select Controller	(On the top banner above the icons)
	Select Check Configuration	Checks the configuration and reports errors.
Save	Select File, Save Installation	Saves configuration
Close	Close Instrument Configuration file	Closes configuration program

Plumbing the 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 DI water previously degassed (vacuum filtration and ultrasonic agitation). Prime the pump by opening the priming knob ½ turn and press 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 Operator's Manual¹ by selecting "?" on the tablet.

IC PEEK Viper Fittings

Tip: To achieve the best chromatography, it is important to gently tighten IC PEEK Viper fittings to finger-tight and to use the fitting assemblies in the following locations (Figure 6):

- Dionex EGC 500 KOH eluent generator cartridge
 Eluent Out to Eluent In on Dionex CR-ATC 600 trap column
- Dionex CR-ATC 600 trap column *Eluent Out* to *Eluent In* on the Dionex Degas Module
- Injection Valve "Column" port to the guard column
- Between the guard and separation columns
- Separation column to *Eluent In* on the Dionex AERS 500 Suppressor
- Dionex AERS 500 suppressor *Eluent Out* to *Eluent In* on CD Conductivity Cell.

IC PEEK Viper fittings (Figure 6) minimize void volume in critical chromatography components, such as the columns and suppressor. IC PEEK Viper 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.

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

Conditioning Electrolytic Devices and Columns

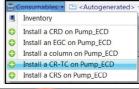
Install the Dionex EGC 500 KOH cartridge and Dionex CR-ATC 600 Continuously Regenerating Anion Trap Column in the Integrion Reservoir Tray compartment. Condition the devices according to instructions in the Chromeleon Instrument Panel, drop-down menu (Consumables, Install) (Figure 7). This information is also available in the product manuals and the Dionex Integrion HPIC System Operator's manual.¹⁻³

To hydrate the Dionex ERS 500 suppressor, follow the QuickStart Instructions received with the suppressor and also in the suppressor product manual.⁴ 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 7). The general practice is to use the eluent and flow rate conditions listed in the QAR report while directing the eluent exiting the column to a waste container.^{5,6} Complete the installation according to flow diagram in Figure 1.



Figure 6. Dionex IC PEEK Viper fittings.



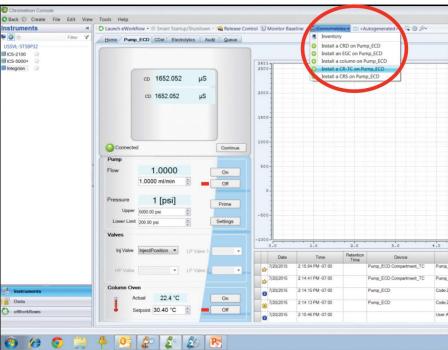


Figure 7. Consumables installation instructions on instrument panel.

Installing and Optimizing the Dionex AS-AP Autosampler

For accurate injections and precise retention times, the Dionex AS-AP autosampler needle must be aligned to the injection port. To align the autosampler needle, first select the Sampler tab on the instrument panel, and press the Alignment button. Follow the commands to align the autosampler needle to the Injection Port and Wash Port (Section B.12 in the Operator's Manual).7 Connect the autosampler syringe line to the wash container containing degassed DI water. Prime the syringe to flush out any air in the Buffer Wash line and syringe. Initially select a 5000 µL wash volume until a steady flow of water is observed at the Wash Port. Then, calibrate the transfer line volume by following the prompts on the TLV Calibration button. This volume will be recorded automatically. For more information, review Section 5.9 in the Dionex IC Series AS-AP Autosampler Operator's Manual.7

Starting the Dionex Integrion HPIC System

To start the system, turn on the pump, and immediately turn on the Dionex EGC 500 eluent generator cartridge. Turn on the Dionex CR-ATC 600 trap and Dionex AERS 500 suppressor when liquid is flowing through the devices. The system backpressure is dependent on the flow rate and type of column, but the system pressure must be above 2000 psi to support the Dionex EGC cartridges. Typically, columns with 4 µm resin particles operate above 2000 psi and, therefore, do not require backpressure tubing. However, if additional pressure is needed to achieve system pressures > 2000 psi, install yellow PEEK backpressure tubing (yellow PEEK, 0.076 mm i.d., 0.003 in i.d.) between the Dionex HP Degas device and the injection port (Pump port position).

Set the eluent concentration, column oven, compartment oven, and cell temperatures as shown in the Conditions section in the application. 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 discussed under the Dionex AS-AP section. Save the instrument method.

Consumables Device Tracking

Tip: An action to 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 consumables device monitoring and tracking. This feature 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 consumable devices 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.

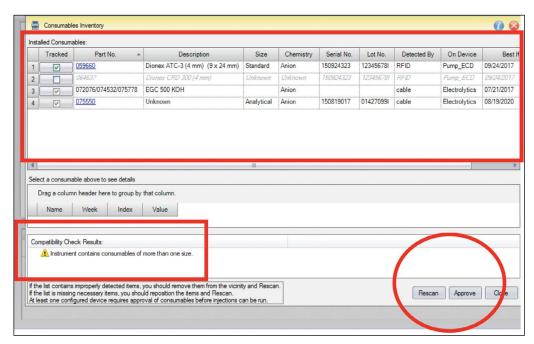


Figure 8. Consumables tracking.

Results and Discussion

The determination of inorganic anions and oxyhalides from disinfection processes in municipal drinking, waste, and bottled waters according to the U.S. EPA Methods 300.0 (A) (B) and 300.1 (A) (B) are the most popular and widely used ion chromatography methods.

The Dionex IonPac AS18-chemistry separation columns optimized for inorganic anion determinations are among the latest innovations in hydroxide-eluent-optimized column chemistry. As such, there are many members of the Dionex IonPac AS18-chemistry family of columns:

- Three formats of the 250 mm length, 7.5 µm super macroporous resin particle columns (0.4 mm, 2 mm, and 4 mm i.d.) for higher resolutions are needed for more complex samples
- Three formats of the Dionex IonPac AS18-Fast,
 150 mm length, with the same resin as the 250 mm columns, for faster separations of well-defined samples
- Three formats of the Dionex IonPac AS18-4µm,
 150 mm length, 4 µm super macroporous resin particle columns combining higher resolution with faster separations

The Dionex IonPac AS19-chemistry high-capacity anion-exchange columns are optimized for oxyhalide anion determinations but also suitable for inorganic anion determinations. These columns are available in three formats (0.4 mm, 2 mm, and 4 mm i.d. by 250 mm) for both the 7.5 μm super macroporous resin and the 4 μm super macroporous resin particles.

In the following environmental applications, inorganic anions were determined in wastewater and surface water samples using the Dionex IonPac AS18-4 μ m columns (4 × 150 mm). Oxyhalides were determined in a bottled water sample using the 2 × 250 mm, Dionex IonPac AS19-4 μ m column.

The analyses were facilitated by the Dionex Integrion HPIC system using the eluent generation and temperature control of the both the column compartment and the detector-suppressor compartment.

In Figure 9, Chromatograms A and B compare the anion separations of a standard on the 4 µm and 7.5 µm resin particles formats, respectively of 4×150 mm (fast) Dionex IonPac AS18 columns using the same conditions. In both examples, the anions were separated using an electrolytically generated hydroxide gradient from 15 to 44 mM KOH at 1 mL/min and detected by suppressed conductivity within 9 min. As expected, the smaller-particle-size column had increased peak efficiencies, as evident in the smaller peak widths and higher peak response. The column pressure also increased as expected. In Chromatogram B, the system backpressure using the Dionex IonPac AS18-4µm column is approximately 2600 psi, more than twice (~1100) that of the same system running the Dionex IonPac AS18-Fast column (Chromatogram A). The Dionex Integrion HPIC system brings high-pressure capabilities to the Integrated systems for standard bore and microbore columns, thereby taking advantage of the 4 µm resin particle columns.

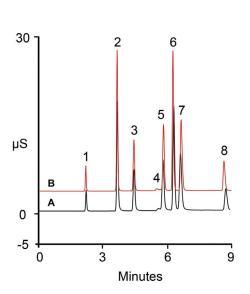


Figure 9. Comparison of Dionex IonPac AS18 columns.

Columns:	A: Dionex IonPac AG18-Fast,
	Dionex IonPac AS18-Fast
	B: Dionex IonPac AG18-4µm,
	Dionex IonPac AS18-4µm
KOH Eluent:	15-44 mM (0.2 to 6 min),
	AA malA (C to O main)

44 mM (6 to 9 min)
Eluent Source: Dionex EGC-500 KOH cartridge,

with Dionex CR-ATC 600 trap column, Dionex HP degasser

Flow Rate: 1 mL/min Inj. Volume: 10 µL Column Temp.: 35 °C Suppressor Comp.:15 °C

Peaks:

Detection: Suppressed conductivity,

Dionex AERS 500, 4 mm, 109 mA,

recycle mode

1. Fluoride 1.0 mg/L
2. Chloride 12.0
3. Nitrite 8.0
4. Carbonate -5. Bromide 9.0

6. Sulfate 10.0 7. Nitrate 9.0 8. Phosphate 6.5 Fast anion determinations were demonstrated in a five-fold diluted municipal wastewater sample (Figure 10) and in an undiluted surface water sample (Figure 11) using the Dionex IonPac AS18-4µm column. For more information on analyzing environmental water samples for common anions please see Application Update 200.8

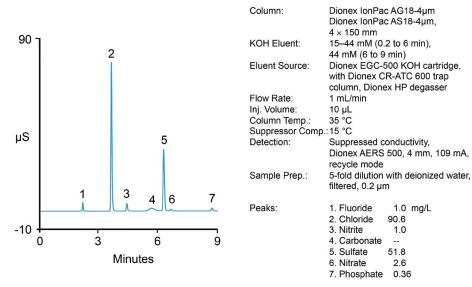


Figure 10. Anions in a municipal wastewater sample.

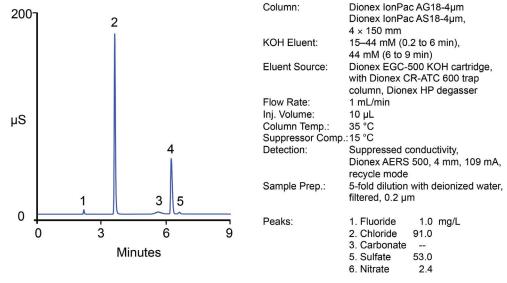
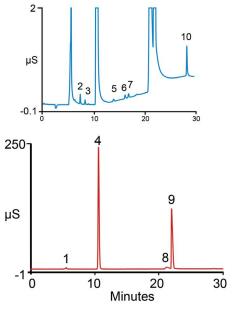


Figure 11. Surface water sample.

For oxyhalide determinations, a large volume injection is needed to achieve the ppb ($\mu g/L$) sensitivity required. In Figure 12, $\mu g/L$ concentrations of oxyhalides were determined in a bottled water sample spiked with oxyhalide standards using the high-resolution Dionex IonPac AS19-4 μ m column. The top chromatogram is rescaled to show the ppb concentrations of oxyhalides. The lower chromatogram is at full scale. The system backpressure is 3800 psi, which when using eluent generation, can only be run on an HPIC system, including a Dionex Integrion HPIC system.



Columns: Dionex IonPac AG19-4um

Dionex IonPac AS19-4µm, 4 × 250 mm KOH Eluent: 10 mM KOH (0-10 min), 10-45 mM (10-25 min), 45 mM (25-30 min)

Dionex EGC-500 KOH cartridge, with Dionex CR-ATC 600 trap column, Dionex HP degasser

Flow Rate: 1 mL/min Ini. Volume: 250 uL

Column Temp.: 30 °C Suppressor Comp.:30 °C

Eluent Source:

Detection: Suppressed conductivity. Dionex AERS 500, 4 mm,

112 mA, recycle mode

Peaks: 1. Fluoride µg/L 20 2. Chlorite

3. Bromate 25 4. Chloride --

5. Nitrite 6. Chlorate 20

7. Bromide 20 8 Carbonate

9. Sulfate 10. Phosphate

Figure 12. Bottled water sample with added oxyhalides.

Conclusion

The installation and setup of the Dionex Integrion HPIC system configured for eluent generation and suppressed conductivity detection were discussed.

The analysis of environmental water samples for inorganic anions and oxyhalides was demonstrated using electrolytically generated hydroxide eluents on different column chemistries and formats:

- The fast 4×150 mm Dionex IonPac AS18-Fast column with 7.5 µm resin particles
- The fast 4×150 mm Dionex IonPac AS18-4µm column with 4 µm resin particles
- The high-capacity 2×250 mm Dionex IonPac AS19-4µm column with 4 µm resin particles

The Dionex Integrion HPIC system, as shown in this configuration, has eluent generation, temperature control, easy access to consumables, high-pressure capability, consumables tracking, and low void volume IC PEEK Viper fittings. Together these features provide a robust easy-to-use system while taking advantage of new technology advancements in IC consumable products.

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