

Configuring High-Pressure IC Systems for Analytical Flow Rates

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Key Words

Standard bore, microbore, Dionex ICS-5000+, HPIC

Goal

Provide instructions to install a high-pressure application using standard bore or microbore columns on a Dionex ICS-5000+ HPIC system.

Introduction

The latest advancement in ion chromatography (IC) instrumentation, the high-pressure Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ system, can operate continuously at 5000 psi for both analytical and capillary scale formats. The Dionex ICS-5000+ HPIC system can therefore fully take advantage of the high-efficiency separations offered by 4 µm particle size columns. When combined with the advantages and ease of use of a Reagent-Free™ IC (RFIC™) system, this system permits excellent enhanced resolution of closely eluting peaks with excellent reproducibility, thereby yielding greater quantification accuracy and consistently reliable results. In this technical note, we provide recommendations for installing and configuring a Dionex ICS-5000+ HPIC system for analytical flow rates (0.10–2.0 mL/min).

Equipment

- High-pressure Thermo Scientific Dionex ICS-5000+ HPIC system
 - ICS-5000+ SP/DP Pump module
 - ICS-5000+ EG Eluent Generator module with high-pressure degas module
 - ICS-5000+ DC Detector/Chromatography module
 - AS-AP Autosampler
- Thermo Scientific Dionex Chromeleon™ Chromatography Data system (CDS)
 - CM 6.80 DU11e build 3320 or CM 7.1 SR2 build 1460 or later
 - Pump firmware FW 6.0.0 or later
 - EG firmware: 2.0.0 or later

Table 1 lists the consumable products needed for the Dionex ICS-5000+ HPIC system using analytical flow rates.

Table 1. Consumables list for the Dionex ICS-5000+ HPIC System.

| Product Name | High Pressure Device | Dionex Part Number |
|--------------------------------------|--|--------------------|
| Dionex HP Degas Module | Degas module | 075522 |
| Dionex AS-AP Autosampler Vials | Polystyrene 10 mL vials, caps, red septa, package of 100 | 055058 |
| | Polystyrene 10 mL vials, caps, blue septa, package of 100 | 074228 |
| | Septa for trace ion (ppb to ppt) applications, for 10 mL vials, package of 100 | 074927 |
| | Polypropylene 1.5 mL vials, package of 100 | 079812 |
| Anions | | |
| Thermo Scientific Dionex EGC 500 KOH | Eluent Generator cartridge | 075778 |
| Thermo Scientific Dionex CR-ATC 500 | Electrolytic trap column | 075550 |
| Thermo Scientific Dionex ASRS™ 300 | Suppressor for 2 mm and 3 mm columns | 064555 |
| | Suppressor for 4 mm and 5 mm columns | 064554 |
| Cations | | |
| Dionex EGC 500 MSA | Eluent Generator cartridge | 075779 |
| Dionex CR-CTC 500 | Electrolytic trap column | 075551 |
| Thermo Scientific Dionex CSRS 300 | Suppressor for 2 mm and 3 mm columns | 064557 |
| | Suppressor for 4 and 5 mm columns | 064556 |
| High-Pressure Fittings (blue) | High Pressure Bolts / Ferrules | 074449 / 074373 |

Instrument Setup and Installation

The Dionex ICS-5000⁺ Reagent-Free HPIC System is a high-pressure version of the Dionex ICS-5000 IC system. The two systems are not interchangeable. The Dionex ICS-5000⁺ HPIC system and the EGC 500 KOH cartridge and CR-TC 500 consumable products are redesigned for high-pressure conditions at analytical flow rates. The high-pressure degas module is no longer plumbed with regenerant lines. The regenerant solution only flows from the CD detector to the suppressor, the CR-TC, and then to waste.

Configuring the Modules in Chromeleon

To setup this application, connect the Dionex AS-AP Autosampler and the Dionex ICS-5000⁺ HPIC system modules according to Figure 1. Then connect the power cables and turn-on all of the modules. Configure the system by starting Chromeleon server configuration (Chromeleon 6.8) or Chromeleon Services Manager (Chromeleon 7) program, and then create a new timebase (Chromeleon 6.8) or new instrument (Chromeleon 7.1). Add each module (Dionex ICS-5000⁺ SP/DP, EG, DC) and the Dionex AS-AP autosampler module to the new instrument configuration by selecting the module address. Each module configuration has additional steps to complete the configuration shown in Table 2.

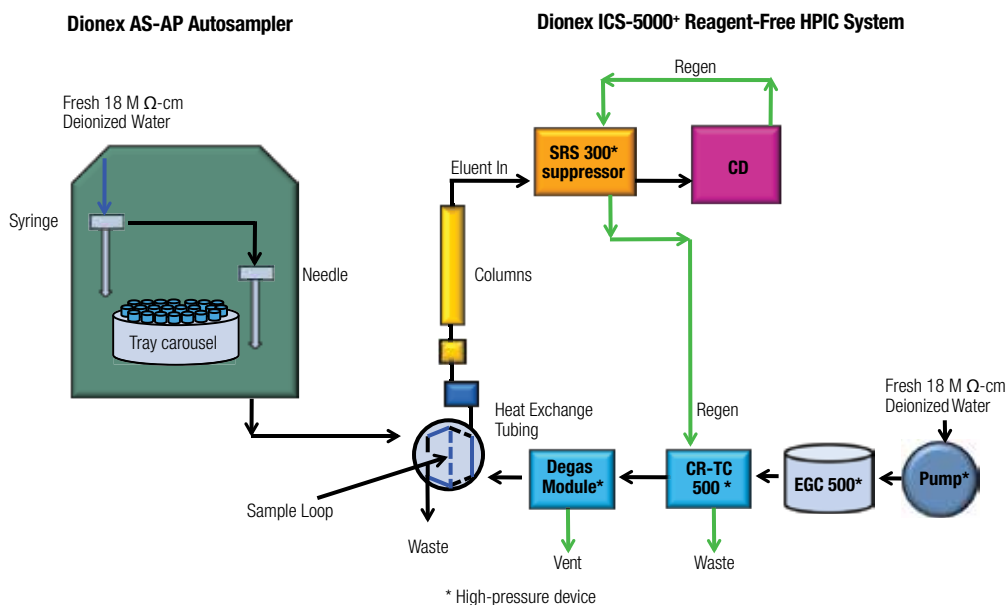


Figure 1. Flow diagram for the high-pressure Dionex ICS-5000⁺ Reagent-Free HPIC system.

Table 2. System configuration for Dionex ICS-5000+ HPIC System.

| Module | Tab | Action |
|-------------------|----------------------|---|
| DP | Device | Link Pump to Instrument. |
| EG | Cartridges | Link to Instrument, check EGC-1 for one cartridge, link to Pump_1 |
| DC | General | Select instrument |
| | Detectors | Select CDet1, double-click on CDet1, Link to Pump_1, Check CD_1 and CD_1_total signal boxes |
| | Thermal Controls | Check Compartment_TC, and Column_TC |
| | Suppressors | Double click Suppressor1, Link to Pump_1 |
| | High Pressure Valves | Double click InjectValve_1, Select Controlled by AS |
| | Low Pressure Valves | Remove check marks |
| AS-AP Autosampler | Sharing | Only if more than one instrument is detected. If this option is present, select Instrument |
| | Segments / Pump Link | Select 10 mL PolyVials or 1.5 ml vials for "Red", "Blue", and "Green" |
| | Options | Select Push, select syringe size (125, 250 or 1000 μ L*), enter the loop size |
| | Relays | Remove check marks |
| | Inputs | Remove check marks |

* The 125, 250 or 1000 μ L syringe sizes are suitable for a 25 μ L injection volume.

Plumbing the High-Pressure Dionex ICS-5000+ Reagent-Free HPIC System

Tip: To achieve the best chromatography with high-pressure IC, it is important to use high-pressure connectors and ferrules (colored blue) for all connections prior to the suppressor. The high-pressure Dionex ICS-5000+ HPIC system is designed to operate from 2000 to 5000 psi for Reagent-Free IC applications. The system can operate at pressures up to 6000 psi for applications using prepared eluents.

To plumb the system, first start the Chromeleon program by connecting it to the instrument or timebase. For Chromeleon 7.1, open the Chromeleon program, find and select the Instrument created in the previous section, and select "Instruments" which will automatically open the instrument panel. For the Chromeleon 6.8, open Chromeleon program, select the Panel icon, and connect to the timebase which will automatically open the instrument panel. To plumb the high-pressure Dionex ICS-5000+ HPIC system, first connect the pump eluent line to the eluent bottle containing deionized water previously degassed by vacuum filtration and ultrasonic agitation. Prime the pump until water is flowing out of the pump waste line at a steady rate. Install and condition the Dionex EGC 500 cartridge and Dionex CR-TC 500 Continuously Regenerating Anion Trap Column according to the product manuals and the system installation

manual.¹⁻³ Hydrate the Dionex SRS 300 suppressor (2 mm suppressor for 2 mm and 3 mm columns; 4 mm suppressor for 4 mm columns) according to the Quick Start Instructions in the Dionex SRS 300 product manual.⁴ The high-pressure degas module no longer is plumbed with regenerant lines. The regenerant solution flows only from the CD detector to suppressor, CR-TC, and then to waste. Complete the installation according to flow diagram. Use high pressure connectors (blue, Dionex P/N 074449) and ferrules (blue, Dionex P/N 074373) for all of the fittings from the pump to prior to the suppressor. Standard pressure fittings can be used for the suppressor and detector connections.

Immediately turn on both the Dionex EGC 500 cartridge and the Dionex SRS 300 suppressor when liquid is flowing through the device. The system backpressure is dependent on the flow rate and type of column, but the system must be above 2000 psi to support the Dionex EGC cartridges. Insert a backpressure tubing (yellow PEEK™, 0.076 mm i.d., 0.003 in i.d.) to increase the system backpressure above 2000 psi. Set the eluent concentration and suppressor current, and column oven, compartment oven, and cell temperatures as shown in the Conditions section in the application. Allow the system to equilibrate for 30 min. For optimum chromatography equilibrate until the total conductivity is < 1 μ S.

Table 3. Additional conditions to create a program.

| Page Title | Mode | Action |
|----------------------|---------------------------|--|
| Sampler Options | Injection Mode | PushFull |
| | Loop Overfill | 5 or 10 |
| | Injection Wash Property | After Injection |
| | Temperature | If option is available, enter 5 °C below room temperature |
| | Accept Recommended Values | Click on button |
| | Wait for Temperature | Click box |
| | Sample Prep Overlap | Click box to reduce total run times |
| DC Options | Column | Click Use box, set temperature to column temperature |
| | Compartment | Click Use box, set temperature to column temperature |
| | Cell Temperature | Set temperature to 5 °C above column temperature, but no higher than 35 °C. |
| Suppressor 1 Options | Suppressor type | Select ASRS 2 mm, ASRS 4 mm, CSRS 2 mm, or CSRS 4 mm, and enter eluent concentration |

Installing the Dionex AS-AP Autosampler

The Dionex AS-AP Autosampler needle must be aligned prior to use in the initial installation to prevent accidentally damaging the needle. To align the autosampler needle, first select the Sampler tab on the instrument panel, open the align programming by pressing the Align Tray button. Follow the commands to align the autosampler needle to the Injection Port and Wash Port (Section B.12 in the Operator's Manual).⁵ Then plumb the wash container containing degassed water to the syringe. 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.

Creating a Program

To create a new instrument program using Chromeleon Wizard, Chromeleon version 6.8, select **File, New, Program**, and select timebase. For Chromeleon 7.1, select **Create, Instrument Method**, and select **Instrument**. For both Chromeleon versions, enter the values in Table 3 and those from the Conditions section specific to the application.

Conclusion

This technical note provides recommendations to install a Dionex ICS-5000+ HPIC system configured for analytical flow rates (0.10–2.0 mL/min).

References

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