

# Configuring High-Pressure Capillary IC on the Modular IC System

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## Key Words

HPIC, setup, installation, capillary IC, Dionex ICS-5000, Dionex ICS-5000+

## Goal

Demonstrate the installation and setup of high-pressure capillary IC on a modular IC system.

## Introduction

High-pressure capillary ion chromatography is the latest advancement in ion chromatography instrumentation. Typically with a reagent-free RFIC system, the system pressure is limited to < 3000 psi because of the limitations of materials in the accessories. Now with the upgrade to high pressure, the Thermo Scientific™ Dionex™ ICS-5000+ Capillary HPIC™ system can operate continuously at system pressures up to 5000 psi. This advance in technology allows increased flow rates with all the same advantages as standard pressure capillary IC; faster analysis, low consumption of water (30 to 40 mL/d of water) and low waste generation. In capillary IC, the system can remain turned on with minimal use of resources, i.e., capillary IC is always on and ready for analysis. These advantages result in:

- Faster analysis, less calibration and equilibration
- Greater ease-of-use
- Longer eluent generator cartridge life
- Lower cost of ownership

Additionally, with the increase in mass sensitivity, comparable results are achieved as with a standard bore system using a sample injection of only 0.4 µL.

## Equipment

Dionex ICS-5000+ HPIC high pressure modular IC system\*

- SP/DP Pump capillary IC module
- EG Eluent Generator module with high pressure degas module
- DC Detector/Chromatography module
- Thermo Scientific™ Dionex™ IC Cube™
- Thermo Scientific Dionex AS-AP Autosampler

\* The Dionex ICS-5000 capillary IC systems can also be used. Capillary systems shipped prior to February 2011 may need to be upgraded to operate at 5000 psi.

- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS)
- CM 6.80 SR10 or CM 7.1 or later



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

Product Name	Type, Capillary	Dionex Part Number
Dionex IC Cube™	with 4-port valve	072000
	with 6-port valve	078841
EG Degas HP cartridge	High pressure EG degas cartridge, up to 5000 psi	AAA-074459
Dionex HP fittings (blue)	Bolts / Ferrules*	074449/074373
Dionex AS-AP autosampler vials	Package of 100, polystyrene vials, caps, blue septa	074228
	Package of 100, Septa for trace ion (ppb to ppt) applications	074927
	Package of 100, polypropylene vials	079812*
<b>Anions</b>		
Thermo Scientific™ Dionex™ EGC-KOH (Capillary)	Anion Eluent Generator cartridge	072076
Thermo Scientific™ Dionex™ CR-ATC (Capillary)	Anion Electrolytic trap column	072078
Thermo Scientific™ Dionex™ CRD 200 (Capillary)	Carbonate Removal Device cartridge	072054
Thermo Scientific™ Dionex™ ACES™ 300	Anion Suppressor cartridge	072052
<b>Cations</b>		
Dionex EGC-MSA (Capillary)	Cation Eluent Generator cartridge	072077
Dionex CR-CTC (Capillary)	Cation Electrolytic trap column	072079
Dionex CRD Bypass cartridge	Bypass (needed for flow path)	072055
Dionex CCES™ 300	Cation Suppressor cartridge	072053

\* Previous P/N was 061696.

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

### Instrument Setup and Installation

#### Configuring the Modules Using Chromeleon Software

High-pressure capillary IC was developed on the Dionex ICS-5000 system platform, therefore the configuration is

the same for both instruments: the Dionex ICS-5000 capillary system and ICS-5000+ capillary HPIC system. To set up high-pressure capillary applications on either instrument, connect the Dionex AS-AP Autosampler, DC, and high pressure DP and EG modules according to Figure 1.

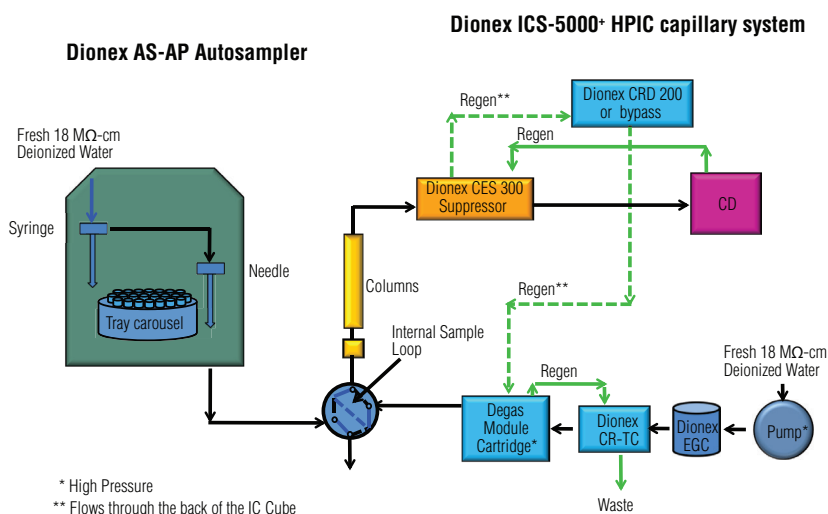


Figure 1. Flow diagram for the Dionex ICS-5000+ capillary system setup.

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
	IC Cubes	Double-click, select Instrument, Press Configure buttons next to the suppressor and valve
	Thermal Controls	Check Compartment_TC
	Suppressors**	Remove check marks if present
	High Pressure Valves**	Remove check marks if present
	Low Pressure Valves	Remove check marks
AS-AP autosampler	Sharing	Select Instrument (if more than one timebase is present)
	Segments / Pump Link	Select 10 mL PolyVials or 1.5 mL vials for "Red", "Blue", and "Green"
	Options	Select Push, select (125, 250,1000 µL) syringe size, enter 0.4 µL loop size
	Relays	Remove check marks
	Inputs	Remove check marks

\* For a dual capillary IC system, select Pump\_1. If the system is a hybrid capillary-analytical scale system, select Pump\_2.

\*\* This section is only present if the system is a hybrid.

Install the IC Cube and CD detector into the DC module while the instrument is off. Then connect the power cables and turn-on all of the modules. Configure the system by starting the 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). Add each module (Dionex ICS-5000+ DP, EG, DC) and the Dionex AS-AP autosampler module to the new instrument by selecting the module address. Each module configuration has additional steps to complete the configuration shown in Table 2.

### Plumbing the Capillary System

Tip: To achieve the best chromatography with capillary IC, it is important to minimize void volumes between connections by using precision cut tubing, high-pressure connectors and fittings (colored blue), and by seating the ferrule > 2 mm above the end of the tubing. These tips are thoroughly discussed in "TN 113 Practical Guidance for Capillary IC".<sup>1</sup> Extra care should be used to prevent introducing air into any of the consumables or tubing by observing a steady liquid flow before installing the next device in line. The high-pressure Dionex ICS-5000+ Capillary HPIC system is designed to operate continuously up to 5000 psi which results in very low noise and increased pump stability.

To plum the system, first start the Dionex Chromeleon program by connecting it to the instrument or timebase. For the Chromeleon 7 version, open the Chromeleon program, find and select the Instrument created during the previous section, and select "Instruments" which will automatically open the instrument panel. For the Chromeleon 6.8 version, open Chromeleon program, select the Panel icon, and connect to the timebase which will automatically open the instrument panel. To plum the capillary ICS-5000+ HPIC IC 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 hydrate the capillary Dionex EGC cartridges and CR-TC traps in the EG module, the degas cartridge and Capillary Electrolytic Suppressor (Thermo Scientific™ Dionex™ CES™) in the IC Cube according to product manuals and Section 3.1.8 of the installation manual, Plumbing the Capillary IC (Figure 2).<sup>2-5</sup>

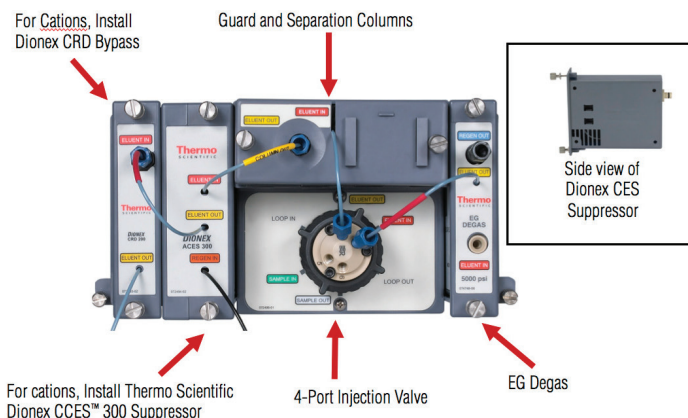


Figure 2. The Dionex IC Cube cartridge.

Table 3. Additional conditions to create a program.

Page Title	Mode	Action
Sampler Options	Injection Mode	PushCap
	Capillary Overfill	50 (times)
	Injection Wash Property	After Injection
	Temperature	30 °C
	Accept Recommended Values	Click on button
	Wait for Temperature	Click box
	Sample Prep Overlap	Click box to reduce total run times
DC Options	Column	Don't click Use box, this temperature is for the bottom section of the DC
Suppressor1 Options	Suppressor type	Select CCES for cations or ACES for anions, enter concentration
IC Cube Options	IC Cube 1 Heater	Click on Use box, set temperature to 30 °C for the separation temperature, click on Wait/Ready box

For cations, install the CRD Bypass cartridge in the Dionex IC Cube position normally occupied by the Dionex CRD 200 cartridge. Then install the guard and separation columns in the column cartridge and install the column cartridge into the Dionex IC Cube according to installation manual instructions. Immediately turn on both the Dionex EGC cartridges and the Dionex CES suppressor when liquid is flowing through the device. Set the eluent concentration, suppressor current, set the temperatures for the compartment, cell heater, and Dionex IC Cube heater according to the Conditions section. Allow the system to equilibrate until the total conductivity is < 1 µS. If retention time reproducibility is > 0.1 %RSD, increase the pump flow rate to 2x the standard flow rate for 1 h and periodically rotate the injection valve, then re-equilibrate at the standard conditions.

### Plumbing the Dionex AS-AP Autosampler

When the Dionex AS-AP autosampler is first installed, the needle must be aligned prior to use 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 Wash Port, and Injection Port (Section B.12 in the Operator's Manual).<sup>6</sup> Then plumb the wash container containing degassed water to the syringe. Prime the syringe to flush any air in Buffer Wash line and syringe. Initially select 5000 µL 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 AS-AP Operator's Manual.<sup>6</sup>

### Creating a Program

To create a new instrument program using Chromeleon version 6.8, select File, New, Program, and select timebase. For Chromeleon version 7, select Create, Instrument Method, and select Instrument. For both Chromeleon versions, open the Program Wizard; enter the conditions shown in the Conditions section and additional commands in Table 3 after program prompts.

There are four temperature controls in the Dionex ICS-5000 and ICS-5000+ DC modules: Column, Compartment, IC Cube Heater, and Cell. The Column temperature controls the temperature of the lower chamber of the DC module which is not needed for routine capillary IC applications. The Compartment temperature, which controls the temperature of the upper chamber of the DC module, should be set at 15 °C to maintain the optimum operating temperature of the Dionex CES suppressor. Use the Dionex IC Cube heater temperature to set the separation temperature for the columns. The Cell temperature controls the temperature of the CD cell.

## Conclusion

This technical note provides recommendations to install a capillary IC application on a Dionex ICS-5000+ HPIC system configured for capillary flow rates (0.01–0.03 mL/min).

## References

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6. Thermo Fisher Scientific. *Dionex AS-AP Operator's Manual*. Dionex Doc No. 065361-07, Sunnyvale, CA, 2012.

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