Configuring a High-Pressure Integrated Capillary IC System

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Key Words

HPIC, setup, installation, capillary IC, ICS-4000

Goal

Demonstrate the installation and setup of Dionex ICS-4000 HPIC system with suppressed conductivity detection.

Introduction

The Thermo Scientific[™] Dionex[™] ICS-4000 HPIC[™] system is a single channel compact, integrated capillary IC with modular detector options designed to meet high resolution and fast analysis needs. The system can be configured with conductivity, electrochemical, or charge detectors for different applications. The Dionex ICS-4000 HPIC system can operate at system pressures up to 5000 psi, which forms a platform to use small particle size columns for sample analysis. At a typical flow rate (0.010 mL/min), the consumption of water is 15 mL/day, which reduces both waste generation and the overall cost of ownership.

Table 1. Consumables list for the Dionex ICS-4000 system.

Product Name	Type, Capillary	Dionex Part Number	
Therma Calantifia™ Dianov™ IC Cuba™	with 4-port valve	072000	
Thermo Scientific Dionex TC Cube	with 6-port valve	078841	
Dionex EG Degas HP cartridge	High pressure EG degas cartridge, up to 5000 psi	AAA-074459	
Dionex HP fittings (blue)	Bolts / Ferrules	074449 / 074373	
	Package of 100, polystyrene vials, caps, blue septa	074228	
Dionex AS-AP autosampler vials	Package of 100, Septa for trace ion (ppb to ppt) applications	074927	
	Package of 100, polypropylene vials	079812+	
Anions			
Thermo Scientific [™] Dionex [™] IonPac [™] ATC-500	2 mm trap column between pump and EGC cartridge	079018	
Black PEEK [™] (Vitrex PLC) Tubing	Tubing from pump to ATC-500 trap	078497	
Thermo Scientific Dionex EGC-KOH (Capillary)	Anion Eluent Generator cartridge	072076	
Thermo Scientific Dionex CR-ATC	Anion Electrolytic trap column	072078	
Thermo Scientific Dionex CRD 200	Anion Carbonate Removal Device	072054	
Thermo Scientific [™] Dionex [™] ACES [™] 300	Anion Suppressor	072052	
Cations			
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	
Thermo Scientific [™] Dionex [™] CCES [™] 300	Cation Suppressor	072053	

Equipment

- Dionex ICS-4000 HPIC Integrated IC system
- Thermo Scientific Dionex AS-AP Autosampler
- Thermo Scientific[™] Dionex[™] Chromeleon[™] Chromatography Data System 7 (CDS)
- Chromeleon Version 7.1 with SR2 MUa or later.

Table 1 lists the consumable products for use with the Dionex ICS-4000 system.



High-Pressure Dionex ICS-4000 HPIC System



Figure 1. Flow diagram for the Dionex ICS-4000 HPIC system (not to scale).

Instrument Setup and Installation

Configuring the Dionex ICS-4000 in Chromeleon Install the Dionex IC Cube and CD detector into the Dionex ICS-4000 system while the instrument is poweredoff. Then connect the power cables and turn on the system. To configure the system, start the Chromeleon Services Manager program, and then create a new Instrument. Add Dionex AS-AP autosampler and Dionex ICS-4000 to the Instrument. Each component configuration has additional steps to complete as shown in Table 2.

Dionex CRD 200ª	- Columns
Dionex CES Suppressor	Dionex EG Degas
	QDb
CD	Dionex QD Cell ^b

^a CRD Bypass for cation determinations ^b Optional QD Detector

Figure 2. Dionex IC Cube with CD and QD detectors installed.

Component	Tab	Action	
	General	Select model S/N numbers and down load appropriate firmware	
Dionex ICS-4000	Electrolytics	Check EluentGenerator, CR_TC, and Suppressor	
	Inject Valve	Check autosampler as Inject Device	
	Thermo Controls	Check Column_TC, and Compartment_TC	
	Sharing	Select Instrument (if more than one instrument is present)	
	Segments / Pump Link	Select 10 mL PolyVials or 1.5 mL vials for "Red", "Blue", and "Green"	
Dionex AS-AP autosampler	Options	Select "Push" for injection mode, select (100, 250, 1000 μL) syringe size, enter 0.4 μL loop size, select "1200" (μL) buffer loop size	
	Relays	Remove check marks	
	Inputs	Remove check marks	

Tahle '	2	System	configuration	for Dioney	105-4000	HPIC system
Table .	Ζ.	Svstern	connuuration	IOI DIOLIEX	163-4000	TIPIC System.

Plumbing the Capillary System

Install Dionex EG, Dionex CR-TC, Dionex EG degas, Dionex CRD 200 (Capillary) (CRD Bypass for cation applications), and CES suppressor according to Figures 1 and 2. To achieve the best chromatography with capillary IC, it is important to minimize void volumes in all connections by using factory precision cut tubing, high pressure connectors and fittings (colored blue), and seating the ferrule > 2 mm above the end of the tubing. These tips are thoroughly discussed in "TN 113: Practical Guidance for Capillary IC".¹ Extra care should be used to prevent trapping air in all consumables or tubing by observing a steady flow before installing the next device in line.

To plumb this IC, first connect the pump eluent line to the eluent bottle containing deionized water degassed by vacuum filtration. Prime the pump until water is flowing out of the pump waste line at a steady rate. Hydrate the Dionex EGC, Dionex CR-TC, and CES according to product manuals and the operator's manual.^{2–5} Install the column cartridge containing separation and guard columns into the Dionex IC Cube according to installation manual instructions. Turn on Dionex EGC and the suppressor when liquid is flowing through the device. Set the eluent concentration, suppressor current, column temperature, compartment temperature, and cell heater according to the application's needs. Allow the system to equilibrate until a stable baseline is observed.

For anion applications install the Dionex IonPac ATC-500 trap column from the pump with black PEEK tubing (P/N 078497) and the Dionex EGC KOH cartridge but temporarily do not connect the free end of the tubing to the Dionex EGC KOH cartridge. To flush the trap column, first initiate the priming function on the pump (1 mL/min), point the Dionex IonPac ATC-500 column upward, and flush for 30 min to allow air to escape. After 30 min, turn off the the pump prime, and connect the tubing to the Dionex EGC-KOH cartridge.

Plumbing the Dionex AS-AP Autosampler

During shipment, the needle of the Dionex AS-AP autosampler may become misaligned. Check needle alignment and realign if necessary by following the commands to realign the needle to the Wash Port and Injection Port. (Section B.12 in the Operator's Manual) respectively.⁵ Fill up the syringe and buffer wash line with wash water by priming the syringe. Select 5000 µL volume for buffer line rinse to speed up the process. Then calibrate the transfer line volume by following the prompts of TLV Calibration icon. The volume will be recorded automatically. For more information review Section 5.9 in the Dionex AS-AP Operator's Manual.⁵

Creating an Instrument Method

To create a new instrument method using Chromeleon 7 software, select Create, Instrument Method, and specific Instrument. Table 3 describes additional general conditions needed to create an Instrument Method using conductivity detection. Specific conditions for an application will be described in each individual application document.

Table 3. Additional conditions to create an instrument method.

Page Title	Mode	Mode	Action
Injection Mode	Injection Mode	PushCap	Select PushCap on pull down menu.
		Accept Recommended Conditions	Click on box.
General Settings	General Settings	Accept Recommended Conditions	Click on box.
	Inject Wash Property	AfterInj	Select AfterInj on pull down menu
Temperature Options	Column	Separation temperature	Enter value from application in box.
	Compartment	Temperature needed for Dionex CES suppressor	Enter 15 (°C) in box.
	Туре	Dionex ACES 300 for anions or Dionex CCES 300 for cations	Select from pull down menu.
Suppressor Options		Concentration (calculator for Dionex CES current)	Enter in eluent concentration in the box next to the eluent type.

Conclusion

This technical note provides recommendations to setup a capillary IC application on a Dionex ICS-4000 HPIC system using Chromeleon 7.1 software.

References

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

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