

Dionex ICS-4000 Capillary HPLC System

Operator's Manual

065468 Revision 01

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Software version: Chromeleon 7.1 SR2 MUa and later

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Low Voltage/Safety Directive: 2006/95/EC

EMC Directive: 2004/108/EC

Safety Standards: EN 61010-1
UL 61010-1
CAN/CSA-C22.2 No. 61010-1

EMC Standards: EN 61326-1

EMC compliance has been evaluated by MET Laboratories, Inc. Low voltage safety compliance has been evaluated by TUV.

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CAUTION Symbol	CAUTION	VORSICHT	ATTENTION	PRECAUCION	AVVERTENZA
	Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	Elektroschock: In diesem Gerät werden Hochspannungen verwendet, die Verletzungen verursachen können. Vor Wartungsarbeiten muß das Gerät abgeschaltet und vom Netz getrennt werden. Betreiben Sie das Gerät nicht mit abgenommenem Deckel. Nehmen Sie die Schutzabdeckung von Leiterplatten nicht ab.	Choc électrique: L'instrument utilise des tensions capables d'infliger des blessures corporelles. L'instrument doit être arrêté et débranché de la source de courant avant tout intervention. Ne pas utiliser l'instrument sans son couvercle. Ne pas enlever les étuis protecteurs des cartes de circuits imprimés.	Descarga eléctrica: Este instrumento utiliza altas tensiones, capaces de producir lesiones personales. Antes de dar servicio de mantenimiento al instrumento, éste deberá apagarse y desconectarse de la línea de alimentación eléctrica. No opere el instrumento sin sus cubiertas exteriores quitadas. No remueva las cubiertas protectoras de las tarjetas de circuito impreso.	Shock da folgorazione. L'apparecchio è alimentato da corrente ad alta tensione che può provocare lesioni fisiche. Prima di effettuare qualsiasi intervento di manutenzione occorre spegnere ed isolare l'apparecchio dalla linea elettrica. Non attivare lo strumento senza lo schermo superiore. Non togliere i coperchi a protezione dalle schede di circuito stampato (PCB).
	Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	Chemikalien: Dieses Gerät kann gefährliche Chemikalien enthalten. Tragen Sie Schutzhandschuhe beim Umgang mit toxischen, karzinogenen, mutagenen oder ätzenden/reizenden Chemikalien. Entsorgen Sie verbrauchtes Öl entsprechend den Vorschriften in den vorgeschriebenen Behältern.	Chimique: Des produits chimiques dangereux peuvent se trouver dans l'instrument. Portez des gants pour manipuler tous produits chimiques toxiques, cancérigènes, mutagènes, ou corrosifs/irritants. Utiliser des récipients et des procédures homologuées pour se débarrasser des déchets d'huile.	Química: El instrumento puede contener productos químicos peligrosos. Utilice guantes al manejar productos químicos tóxicos, carcinógenos, mutágenos o corrosivos/irritantes. Utilice recipientes y procedimientos aprobados para deshacerse del aceite usado.	Prodotti chimici. Possibile presenza di sostanze chimiche pericolose nell'apparecchio. Indossare dei guanti per maneggiare prodotti chimici tossici, cancerogeni, mutageni, o corrosivi/irritanti. Utilizzare contenitori aprovo e seguire la procedura indicata per lo smaltimento dei residui di olio.
	Heat: Before servicing the instrument, allow any heated components to cool.	Hitze: Warten Sie erhitzte Komponenten erst nachdem diese sich abgekühlt haben.	Haute Temperature: Permettre aux composants chauffés de refroidir avant tout intervention.	Altas temperaturas: Permita que los componentes se enfríen, ante de efectuar servicio de mantenimiento.	Calore. Attendere che i componenti riscaldati si raffreddino prima di effettuare l'intervento di manutenzione.
	Fire: Use care when operating the system in the presence of flammable gases.	Feuer: Beachten Sie die einschlägigen Vorsichtsmaßnahmen, wenn Sie das System in Gegenwart von entzündbaren Gasen betreiben.	Incendie: Agir avec précaution lors de l'utilisation du système en présence de gaz inflammables.	Fuego: Tenga cuidado al operar el sistema en presencia de gases inflamables.	Incendio. Adottare le dovute precauzioni quando si usa il sistema in presenza di gas infiammabili.
	Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	Verletzungsgefahr der Augen: Verspritzte Chemikalien oder kleine Partikel können Augenverletzungen verursachen. Tragen Sie beim Umgang mit Chemikalien oder bei der Wartung des Gerätes eine Schutzbrille.	Danger pour les yeux: Des projections chimiques, liquides, ou solides peuvent être dangereuses pour les yeux. Porter des lunettes de protection lors de toute manipulation de produit chimique ou pour toute intervention sur l'instrument.	Peligro par los ojos: Las salicaduras de productos químicos o partículas que saltan bruscamente pueden causar lesiones en los ojos. Utilice anteojos protectores al manipular productos químicos o al darle servicio de mantenimiento al instrumento.	Pericolo per la vista. Gli schizzi di prodotti chimici o delle particelle presenti nell'aria potrebbero causare danni alla vista. Indossare occhiali protettivi quando si maneggiano prodotti chimici o si effettuano interventi di manutenzione sull'apparecchio.
	General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual. When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific Sunnyvale Products.	Allgemeine Gefahr: Es besteht eine weitere Gefahr, die nicht in den vorstehenden Kategorien beschrieben ist. Dieses Symbol wird im Handbuch außerdem dazu verwendet, um den Benutzer auf Anweisungen hinzuweisen. Wenn Sie sich über die Sicherheit eines Verfahrens im unklaren sind, setzen Sie sich, bevor Sie fortfahren, mit Ihrer lokalen technischen Unterstützungsorganisation für Thermo Fisher Scientific Sunnyvale Produkte in Verbindung.	Danger général: Indique la présence d'un risque n'appartenant pas aux catégories citées plus haut. Ce symbole figure également sur l'instrument pour renvoyer l'utilisateur aux instructions du présent manuel. Si la sûreté d'une procédure est incertaine, avant de continuer, contacter le plus proche Service Clientèle pour les produits de Thermo Fisher Scientific Sunnyvale.	Peligro general: Significa que existe un peligro no incluido en las categorías anteriores. Este símbolo también se utiliza en el instrumento par referir al usuario a las instrucciones contenidas en este manual. Cuando la certidumbre acerca de un procedimiento sea dudosa, antes de proseguir, pongase en contacto con la Oficina de Asistencia Técnica local para los productos de Thermo Fisher Scientific Sunnyvale.	Pericolo generico. Pericolo non compreso tra le precedenti categorie. Questo simbolo è utilizzato inoltre sull'apparecchio per segnalare all'utente di consultare le istruzioni descritte nel presente manuale. Quando è in dubbio la misura di sicurezza per una procedura, prima di continuare, si prega di mettersi in contatto con il Servizio di Assistenza Tecnica locale per i prodotti di Thermo Fisher Scientific Sunnyvale.

CAUTION Symbol	CAUTION	危険警告	危險警告
	Electric Shock: This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	電撃: この計測器は高電圧を使用し、人体に危害を与える可能性があります。保守・修理は、必ず作業を停止し、電源を切ってから実施して下さい。上部カバーを外したままで計測器を使用しないで下さい。プリント配線板の保護カバーは外さないで下さい。	電撃: 儀器設備使用會造成人身傷害的高伏電壓。在維修之前，必須先關儀器設備並切除電源。務必要在頂蓋蓋上的情況下操作儀器。請勿拆除PCB保護蓋。
	Chemical: This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	化学物質: 危険な化学物質が計測器中に存在している可能性があります。毒性、発がん性、突然変異性、腐食・刺激性などのある薬品を取り扱う際は、手袋を着用して下さい。廃油の処分には、規定の容器と手順を使用して下さい。	化学品: 儀器設備中可能存在有危險性的化學物品。接觸毒性致癌、誘變或腐蝕/刺激性化學品時，請配帶手套。處置廢油時，請使用經過許可的容器和程序。
	Heat: Before servicing the instrument, allow any heated components to cool.	熱: 熱くなった部品は冷えるのを待ってから保守・修理を行って下さい。	高温: 請先等高温零件冷卻之後再進行維修。
	Fire: Use care when operating the system in the presence of flammable gases.	火災: 可燃性のガスが存在する場所でシステムを操作する場合は、充分な注意を払って下さい。	火災: 在有易燃氣體的場地操作該系統時，請務必小心謹慎。
	Eye Hazard: Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	眼に対する危険: 化学物質や微粒子が飛散して眼を傷つける危険性があります。化学物質の取り扱い、あるいは計測器の保守・修理に際しては防護眼鏡を着用して下さい。	眼睛傷害危險: 飛濺の化學品或顆粒可能造成眼睛傷害。處理化學品或維修儀器設備時請佩戴安全眼鏡。
	General Hazard: A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.	一般的な危険: この標識は上記以外のタイプの危険が存在することを示します。また、計測器にこの標識がついている場合は、本マニュアル中の指示を参照して下さい。	一般性危險: 説明未包括在上述類別中的其他危險。此外，儀器設備上使用這個標誌，以指示用戶本使用手冊中的說明。
	When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific Sunnyvale Products.	安全を確保する手順がよくわからない時は、作業を一時中止し、お近くのサーモエレクトロンサンローゼプロダクトのテクニカルサポートセンターにご連絡ください。	如對安全程序有疑問，請在操作之前與當地的菲尼根技術服務中心聯繫。

Contents

	Preface	xv
	Related Documentation	xv
	Safety and Regulatory Information	xv
	Safety and Special Notices	xv
	Safety Symbols	xvi
	Deionized Water Requirements for IC	xvii
	Contacting Us	xvii
Chapter 1	Introduction	1
	Introduction to Ion Chromatography (IC)	1
	Introduction to the Dionex ICS-4000 Capillary HPIC System	3
	Optional Features	4
	System Control	4
Chapter 2	Functional Description	5
	System Flow Schematics	7
	Touch Screen	10
	Chromleon	10
	Panels	11
	Software Control Modes	12
	Eluent Reservoirs	13
	Eluent Degasser	14
	Eluent Valve	14
	Pump	15
	Fast Ramping	15
	Pump Components	16
	Piston Seal Wash System (Optional)	17
	Eluent Generator (EG)	17
	Eluent Generator Cartridge (EGC)	18
	Continuously Regenerated Trap Column (CR-TC)	18
	Temperature Control Zones	19

Dionex IC Cube	19
EG Degas Cartridge	20
Injection Valve	20
Column Heater and Column Cartridge.	22
Suppressor Cartridge or Suppressor Bypass Cartridge	22
CRD Cartridge or CRD Bypass Cartridge.	23
Dionex IC Cube Flow Schematics.	23
Detectors.	25
Dionex ICS-4000 Conductivity Detector (CD)	26
Dionex ICS-4000 Electrochemical Detector (ED).	26
Dionex ICS-4000 Charge Detector (QD)	29
Rear Panel Features	30
Gas Regulator and Low-Pressure Valve Mounting Locations.	30
Tubing Chase and Waste Lines	31
Power Switch, Fuse Holder, and Power Receptacle	31
USB Status LED, and USB Connectors	31
I/O Connectors (TTL, Analog Outputs, Power, and Relay)	32
Low-Pressure Valve Connectors.	32
PWM Drive Connector.	33
Vacuum Port.	33
Drain Port.	33
Pump and Valve Options	33
Peristaltic Pump	33
High-Pressure Valves.	33
Low-Pressure Valves	34
Chapter 3 Operation	35
Starting Up the System (Overview)	36
Setting Up the Reservoir	36
Setting Up the Piston Seal Wash System.	39
Turning On the System Power	39
Connecting to Chromeleon.	39
Priming the Pump.	41
Setting Operating Parameters and Equilibrating the System	43
Setting Operating Parameters	43
Equilibrating the System and Verifying Operational Readiness	43
Preparing Samples	44
Collecting and Storing Samples.	44
Pretreating Samples	45
Diluting Samples.	45
Loading Samples Into the Sample Loop or Concentrator	46
Performing Sample Analyses	47
System Shutdown	48
Consumables Storage.	48
ED Cell Storage.	48

	Operating the Eluent Generator (EG)	49
	Selecting an Eluent Concentration	49
	Entering the Eluent Concentration	50
	Operating the Dionex ICS-4000 Electrochemical Detector	50
	ED Cell Operating Precautions	51
	Electrochemical Detection Modes	53
	Waveforms	56
	Collecting and Storing Amperometry Data	57
	Using Rear Panel Inputs and Outputs	57
	Connecting the I/O Connectors	58
	Assigning TTL Input Functions	60
	Controlling Relay and TTL Outputs	63
	Selecting Analog Output Settings	64
	Controlling a Peristaltic Pump or Other Accessory with the PWM Drive	66
Chapter 4	Touch Screen Operation	69
	Overview of Touch Screen Operation	69
	Using the Touch Screen	70
	Using the Touch Screen with Chromeleon	71
	Controlling the Pump	72
	Controlling the Eluent Generator	74
	Controlling the Suppressor	75
	Controlling the Injection Valve	76
	Controlling Temperature Zones	76
	Selecting Detector Operating Parameters	77
	Selecting CD Operating Parameters	77
	Selecting QD Operating Parameters	79
	Selecting ED Operating Parameters	80
	Viewing a Plot of the Detector Output	82
	Setting Analog Output Options	82
	Controlling TTLs and Relays	84
	Controlling Optional Valves	85
	Controlling Accessories	86
	Selecting Setup Options	87
	Viewing System Information	88
	Opening Calibration Screens	88
Chapter 5	Maintenance	89
	Daily Maintenance	89
	Weekly Maintenance	89
	Periodic Maintenance	89
	Annual Maintenance	90
Chapter 6	Troubleshooting	91
	Error Messages	92

Troubleshooting Error Messages	93
No Communication with Chromeleon	105
Liquid Leaks/Leak Alarm.	106
Low System Backpressure	107
High System Backpressure.	108
Pump Does Not Start	108
Pump Stops.	108
Degasser Low Vacuum	109
Degas Vacuum Pump Does Not Run	109
EG Stops Operation	109
Noisy Baseline	110
High Background	112
Low Detector Output	113
No Peaks	114
Tailing Peaks.	114
Poor Retention Time Reproducibility	115
Peak Retention Times Are Too Early	116
Peak Retention Times Are Too Late	117
ED Cell Troubleshooting	117
ED Cell pH Readout Always 7.0.	117
Cannot Set ED Cell pH Readout to 7.0	117
Shift in ED Cell pH Readout	118
No ED Cell pH Readout or Intermittent Readout	118
Shift in Ag/AgCl Reference Potential.	119
Chapter 7 Service	121
Replacing Tubing and Fittings	122
Fitting and Tube Connection Guidelines	123
Ferrule Fitting Guidelines	123
Capillary Tube Fitting Guidelines	123
Cutting Non-Capillary Tubing	124
Servicing Pump Components	124
Replacing Pump Check Valves	124
Replacing Pump Piston Seals.	127
Replacing a Pump Piston.	132
Replacing the Seal on the Pump Priming Valve Knob.	134
Servicing Eluent Generator Components.	136
Replacing an EGC.	136
Replacing a CR-TC.	144
Servicing High-Pressure (Injection) Valves	146
Rebuilding a High-Pressure Valve	146
Replacing a High-Pressure Valve Pod	147
Replacing a Dionex IC Cube Cartridge.	148
Replacing Capillary Columns	149

Servicing Dionex ICS-4000 Conductivity and Charge Detectors	153
Calibrating a CD Cell	154
Replacing a CD	156
Replacing a Dionex QDC 300 (Capillary) (QDC)	157
Replacing a QD	158
Servicing a Dionex ICS-4000 Electrochemical Detector	159
Replacing an ED	159
Disconnecting an ED Cell	161
Replacing a Disposable Working Electrode Gasket	161
Replacing a Conventional Working Electrode Gasket	162
Polishing a Conventional Working Electrode	164
Replacing a pH-Ag/AgCl Reference Electrode	166
Replacing a PdH Reference Electrode	171
Changing the Main Power Fuses	175
Appendix A Specifications	177
Appendix B Reordering Information	181
Index	185

Contents

Preface

This manual describes the functional features of the Thermo Scientific Dionex™ ICS-4000 Capillary HPIC System and provides instructions for system start up, shut down, operation, maintenance, troubleshooting, and service.

Related Documentation

In addition to this guide, the following related documents are provided on the Thermo Scientific Reference Library DVD (P/N 053891).

- *Dionex ICS-4000 Capillary HPIC System Installation Instructions* (Document No. 065469)
- *Dionex AS-AP Autosampler Operator's Manual* (Document No. 065361)
- *Dionex AS-DV Autosampler Operator's Manual* (Document No. 065259)
- Manuals for consumable products (columns, suppressors, CR-TC, EGC, CRD)

Safety and Regulatory Information

The Thermo Scientific Dionex ICS-4000 Capillary HPIC System was manufactured by Thermo Fisher Scientific at the following location: 527 Lakeside Drive, Sunnyvale, CA 94088-3603 U.S.A. The Dionex ICS-4000 is designed for IC (ion chromatography) applications and should not be used for any other purpose. Operation of a Dionex ICS-4000 in a manner not specified by Thermo Fisher Scientific may result in personal injury.

If there is a question regarding appropriate usage, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:



CAUTION Highlights hazards to humans, property, or the environment. Each CAUTION notice is accompanied by an appropriate CAUTION symbol.







IMPORTANT Highlights information necessary to prevent damage to the system or software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

Note Highlights information of general interest.

Tip Highlights helpful information that can make a task easier.

Safety Symbols

These symbols appear on the Dionex ICS-4000 or on labels affixed to the system:

	Alternating current
	Primary protective conductor terminal
	Secondary protective conductor terminal
	Power supply is on
	Power supply is off
	Indicates a potential hazard. Refer to this operator's manual for an explanation of the hazard and how to proceed.

Deionized Water Requirements for IC

For eluent generation or when manually preparing eluent and regenerant, use ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [Table 1](#).

Table 1. ASTM filtered, Type I deionized water specifications for ion chromatography

Contaminant	Specification
Ions–Resistivity	>18.0 (megohm-cm)
Organics–TOC	<10 ppb
Iron/Transition Metals*	<1 ppb
Pyrogens	<0.03 (Eu/mL)
Particulates > 0.2 µm	<1 (units/mL)
Colloids–Silica	<10 ppb
Bacteria	<1 (cfu/mL)

* Iron/transition metal content not specified for ASTM Type I water

Contacting Us

❖ For Technical Support for Dionex products

In the U.S. and Canada, call 1-800-346-6390.

Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

❖ For additional contact information

Go to www.thermoscientific.com/Dionex

Preface

Introduction

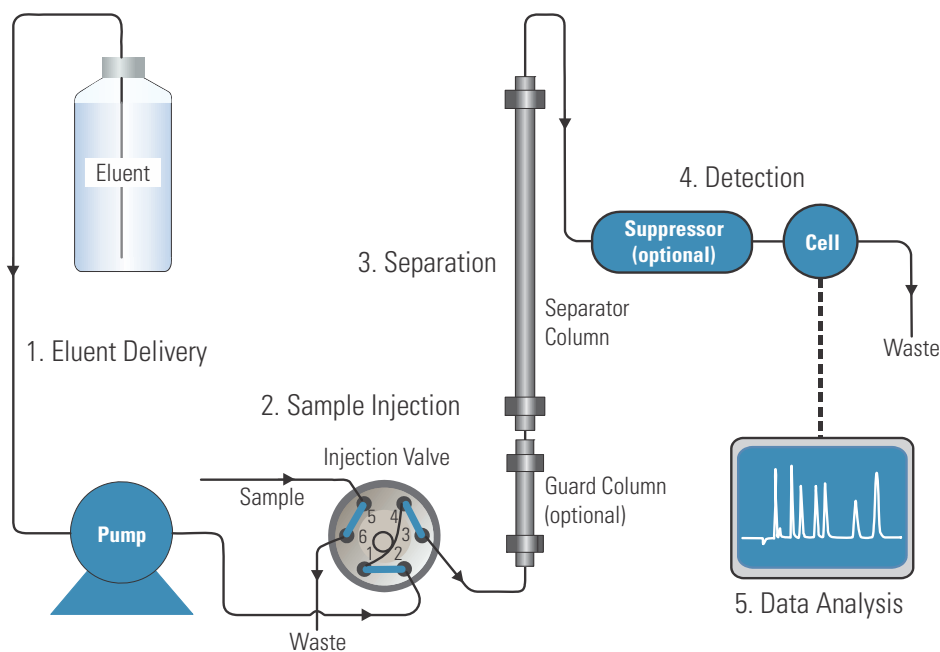
Introduction to Ion Chromatography (IC)

An ion chromatography system like the Dionex ICS-4000 Capillary HPIC System typically consists of a liquid eluent, a high-pressure pump, a sample injector, an analytical column, a detector cell, and a data collection system.

Before running a sample, the ion chromatography system is calibrated using a standard solution. By comparing the data obtained from a sample to that obtained from the known standard, sample ions can be identified and quantitated. The data collection system (typically a computer running chromatography software) produces a *chromatogram* (a plot of the detector output vs. time). The chromatography software converts each peak in the chromatogram to a sample concentration.

IC analysis consists of the five stages shown in [Figure 1](#).

Figure 1. Ion analysis process



1. Eluent Delivery

- *Eluent* (a liquid that helps to separate the sample ions) carries the sample through the ion chromatography system. The Dionex ICS-4000 includes an eluent generator, which generates eluent online from deionized water.
- When the Dionex ICS-4000 is controlled from the front panel, only isocratic eluent delivery is possible. In isocratic delivery, the eluent composition and concentration remain constant throughout the run. When the Dionex ICS-4000 is controlled by a Thermo Scientific Dionex Chromeleon™ Chromatography Data System (the data collection system for the Dionex ICS-4000), gradient eluent delivery is possible. In gradient delivery, the eluent concentration changes over time.

2. Sample Injection

- The liquid sample is loaded into the injection valve either manually or by an automated sampler. When triggered, the sample is injected into the eluent stream.
- The pump pushes the eluent and sample through the analytical column (a chemically-inert tube packed with a polymeric resin). If installed, a guard column removes contaminants that might foul the analytical column.

3. Separation

- As the eluent and sample are pumped through the analytical column, the sample ions are separated. In the Dionex ICS-4000, the mode of separation is called ion exchange. This is based on the premise that different sample ions migrate through the IC column at different rates, depending upon their interactions with the ion exchange sites.
- In conductivity detection applications, the eluent and sample ions flow through a suppressor after they leave the column. The suppressor selectively enhances detection of the sample ions while suppressing the conductivity of the eluent.

4. Detection

- In conductivity detection, a conductivity cell measures the electrical conductance of the sample ions as they emerge from the suppressor. In electrochemical detection, an amperometric cell measures the current resulting from the oxidation or reduction of the sample ions as they emerge from the column. In charge detections, the cell measures current as analytes are transferred across an ion-exchange membrane. For each detection type, the cell produces a signal based on the measurements and transmits the signal to a data collection system.

5. Data Analysis

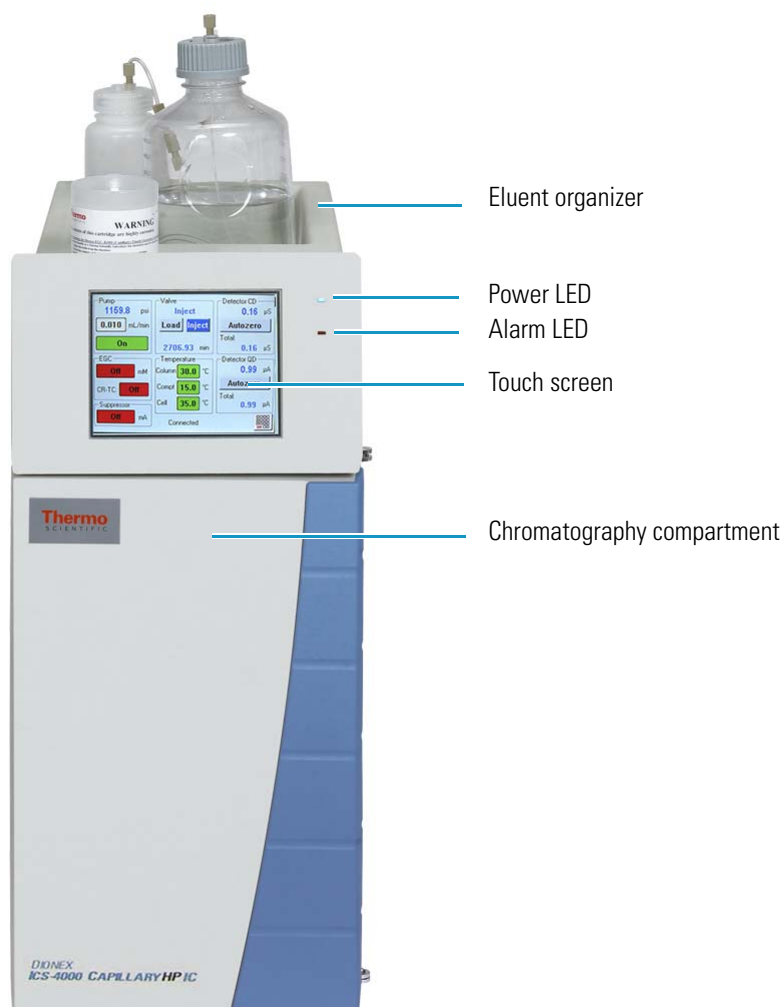
- The data collection system identifies the ions based on retention time, and quantifies each analyte by integrating the peak area or peak height. The data is quantitated by comparing the sample peaks in a chromatogram to those produced from a standard solution. The results are displayed as a chromatogram and the concentrations of ionic analytes can be automatically determined and tabulated.

Introduction to the Dionex ICS-4000 Capillary HPIC System

The Dionex ICS-4000 is an integrated HPIC (high-pressure ion chromatography) system designed for running capillary IC applications. The system includes a pump, an eluent generator (EG), and a temperature-controlled chromatography compartment. The chromatography compartment houses an injection valve, a detector, a suppressor (optional), a column heater, a guard column (optional), and an analytical column. Three types of detectors are available for the Dionex ICS-4000: a Thermo Scientific Dionex ICS-4000 Conductivity Detector (CD), a Thermo Scientific Dionex ICS-4000 Charge Detector (QD), and a Thermo Scientific Dionex ICS-4000 Electrochemical Detector (ED).

A touch screen display provides system status and basic control functions. The screen lifts up for access to the Thermo Scientific Dionex EGC (eluent generator cartridge) and Thermo Scientific Dionex Continuously Regenerated Trap Column (CR-TC). The eluent organizer on top of the Dionex ICS-4000 holds two 1-liter or 2-liter reservoirs.

Figure 2. Dionex ICS-4000 Capillary HPIC System



Optional Features

- Piston seal wash system
- Peristaltic pump for delivering external water or chemical regenerant
- Power supply for controlling additional electrolytic devices (for example, an electrolytic water polisher)
- Control for up to two high-pressure valves (6-port or 10-port) for sample preparation applications
- Control for up to two solenoid (low-pressure) valves

System Control

The Dionex ICS-4000 system can be controlled two ways:

- With the front panel LCD touch screen. The touch screen is used for instrument control. It can display a real-time plot of detector output, but does not store data or provide data processing functions. An analog output on the rear panel can be connected to a separate data acquisition device.
- With Thermo Scientific Dionex Chromeleon 7 Chromatography Data System or Thermo Scientific Dionex Chromeleon 6.8 Chromatography Data System. Chromeleon 7 and Chromeleon 6.8 provide complete instrument control, data acquisition, and data processing functions.

For communication between the system and Chromeleon, the Dionex ICS-4000 is connected to a USB (Universal Serial Bus) port on the computer or a USB hub.

Functional Description

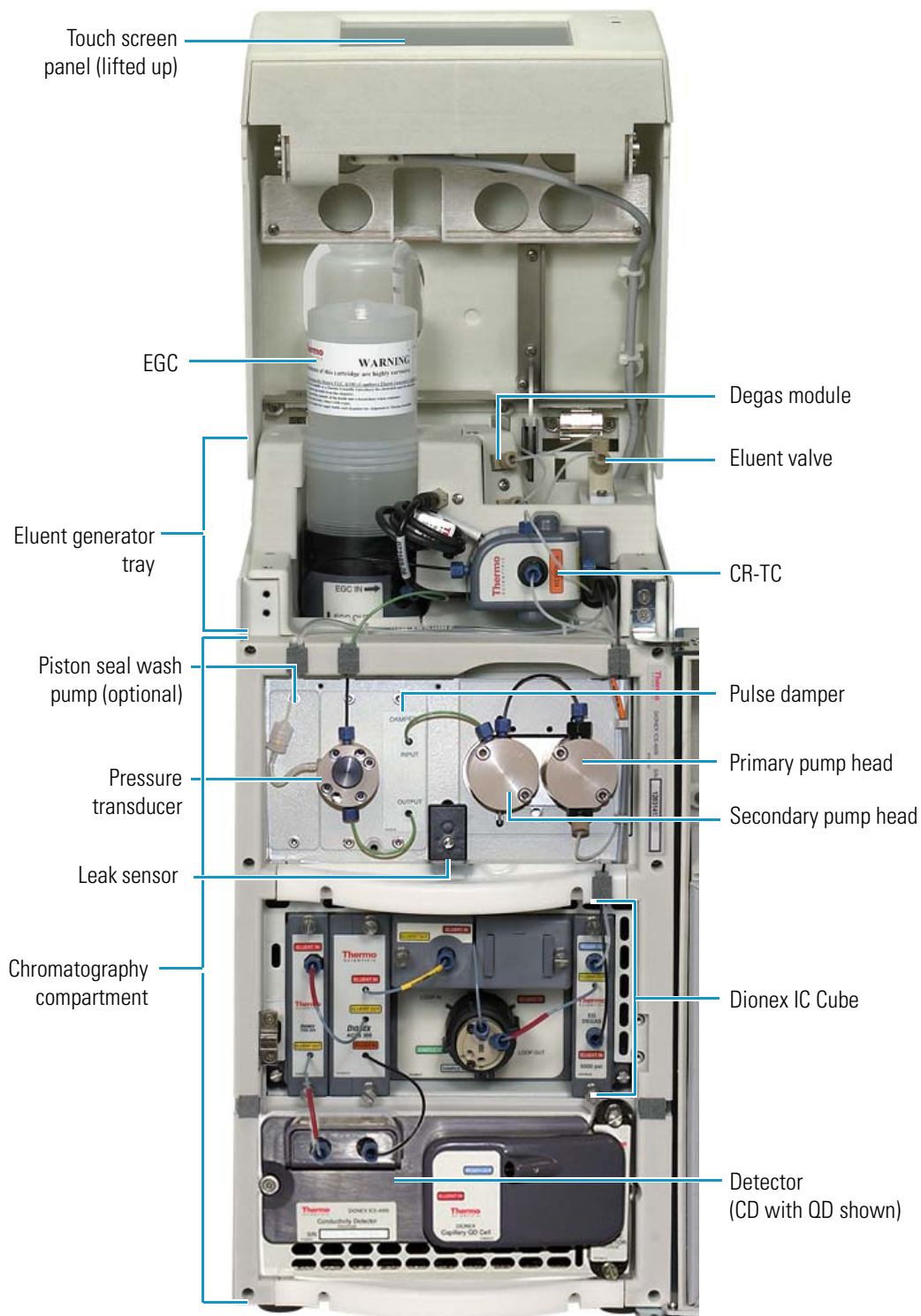
This chapter describes the principal components of the Dionex ICS-4000 Capillary HPIC System and their functions.

Contents

- System Flow Schematics
- Touch Screen
- Chromeleon
- Eluent Reservoirs
- Eluent Degasser
- Eluent Valve
- Pump
- Eluent Generator (EG)
- Temperature Control Zones
- Dionex IC Cube
- Detectors
- Rear Panel Features
- Pump and Valve Options

Figure 3 illustrates the principal components of the system.

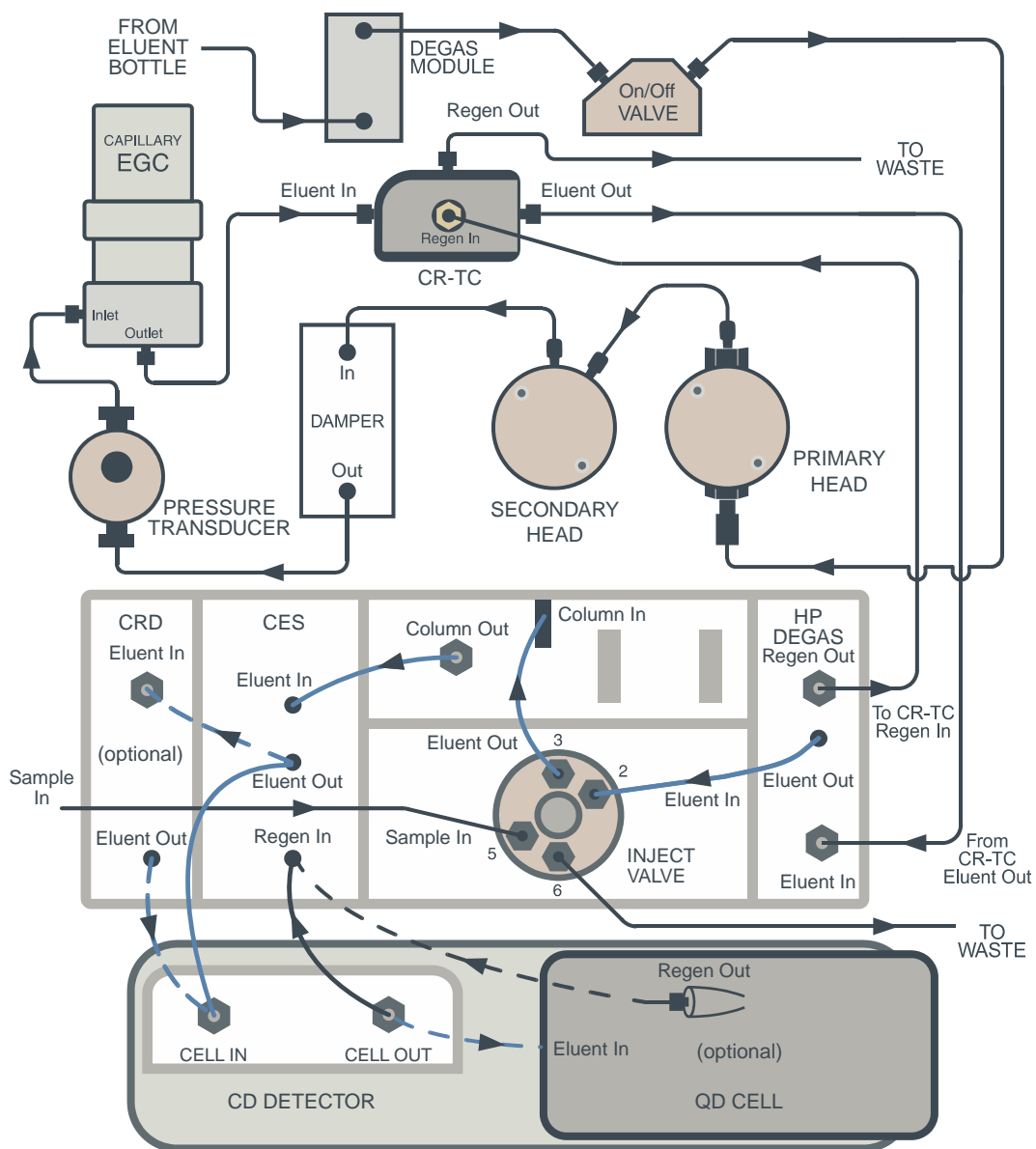
Figure 3. Dionex ICS-4000 components



System Flow Schematics

Figure 4 illustrates the system flow for a conductivity detection application using suppression in recycle mode. Figure 5 on page 9 illustrates the system flow for an electrochemical detection application.

Figure 4. System flow schematic for conductivity detection with optional charge detection

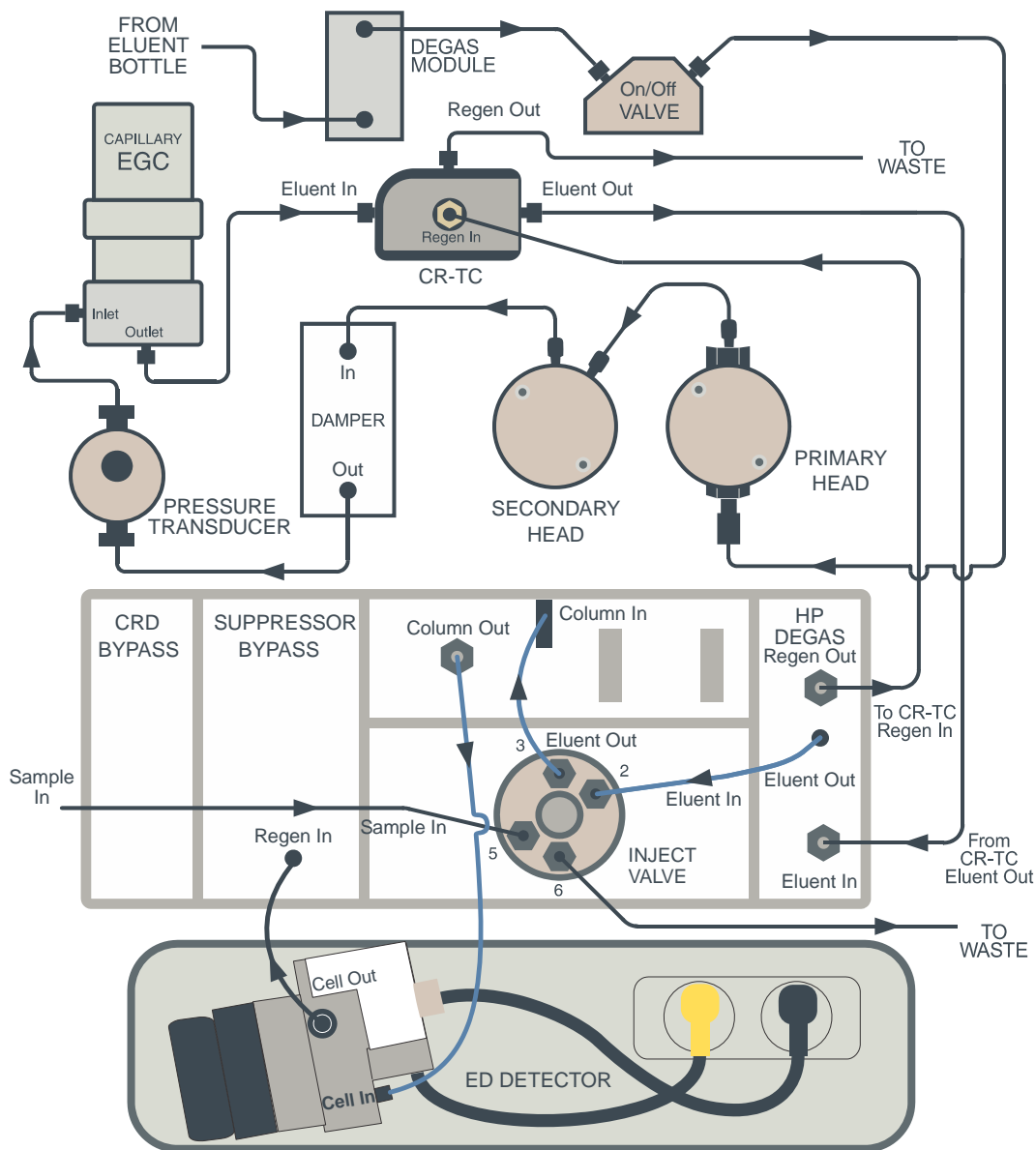


- Deionized water from the reservoir flows first through the eluent degas assembly and then through the eluent valve to the pump. The water is then pushed through a pulse damper, which smooths minor pressure variations from the pump to minimize baseline noise. From there, the water flows through the pressure transducer, which measures the system pressure.

- Deionized water from the pump enters the EGC, which generates the eluent. Eluent exits the EGC and passes through the CR-TC (which traps ionic contaminants). Eluent is then pumped through the EG degas cartridge and into the injection valve.
- After sample is loaded into the sample loop and the injection valve is toggled to the Inject position, eluent passes through the loop.
- The eluent/sample mixture is pumped through the guard column (if installed) and analytical column, and then through the suppressor.
- From the suppressor, the mixture flows through a CRD (if installed) to the cell, where the analytes are detected. A digital signal is sent to Chromeleon software. The CRD is an option for anion applications only.
- The mixture flows out of the cell and is recycled back into the suppressor, where it is used as the water source for the regenerant chamber. Regenerant flow is routed through the CRD (if installed), through the EG degas cartridge, through the CR-TC, and then out to waste.

Note If a Dionex ICS-4000 Charge Detector is installed, the output from the conductivity detector is directed to the charge detector before being recycled back into the suppressor regen inlet.

Figure 5. System flow schematic for electrochemical detection



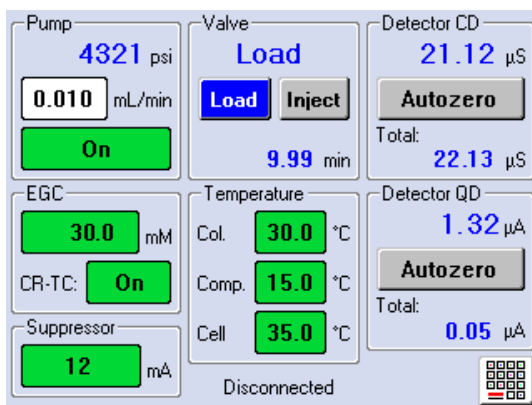
- Deionized water from the reservoir flows first through the eluent degas assembly and then through the eluent valve to the pump. The water is then pushed through a pulse damper, which smooths minor pressure variations from the pump to minimize baseline noise. From there, the water flows through the pressure transducer, which measures the system pressure.
- Deionized water from the pump enters the EGC, which generates the eluent. Eluent exits the EGC and passes through the CR-TC (which traps ionic contaminants). Eluent is then pumped through the EG degas cartridge and into the injection valve.

- After sample is loaded into the sample loop and the injection valve is toggled to the Inject position, eluent passes through the loop.
- The eluent/sample mixture is pumped through the column and then to the cell, where the analytes are detected. A digital signal is sent to Chromeleon software.
- The mixture flows out of the cell through the EG degas cartridge, through the CR-TC, and then out to waste.

Touch Screen

The LCD touch screen provides direct control of Dionex ICS-4000 operating functions. When you select an operating command or parameter from the touch screen, the command or parameter is executed immediately. The Home screen (see [Figure 6](#)) provides controls for the most commonly used operating functions. From the Home screen, you can also go to a series of screens that provide access to all other locally controlled operating functions.

Figure 6. Home screen example



To select an operating command or parameter, touch and release the control (button, box, arrow, etc.) for the command or parameter. When you lift your finger, the action is taken.

Note When the Dionex ICS-4000 is connected to Chromeleon, most touch screen controls are disabled. For full touch screen operation, disconnect the Dionex ICS-4000 from Chromeleon.

For details about using the touch screen, see [“Touch Screen Operation”](#) on [page 69](#).

Chromeleon

The Dionex ICS-4000 system can be controlled using the Chromeleon 7 or Chromeleon 6.8 Chromatography Data Management System.

Panels

In Chromeleon 7, an ePanel Set provides centralized system control. You can use the ePanel Set to view system status information and issue commands for controlling the system. In Chromeleon 6.8, these functions are available on a panel tabset. In both the ePanel Set and the panel tabset, individual tabs provide access to detailed status and control functions for each system component (pump, chromatography compartment, suppressor, detector, EG). [Figure 7](#) shows the Chromeleon 7 ePanel Set and [Figure 8](#) shows the Chromeleon 6.8 panel tabset.

Figure 7. Example Chromeleon 7 ePanel Set

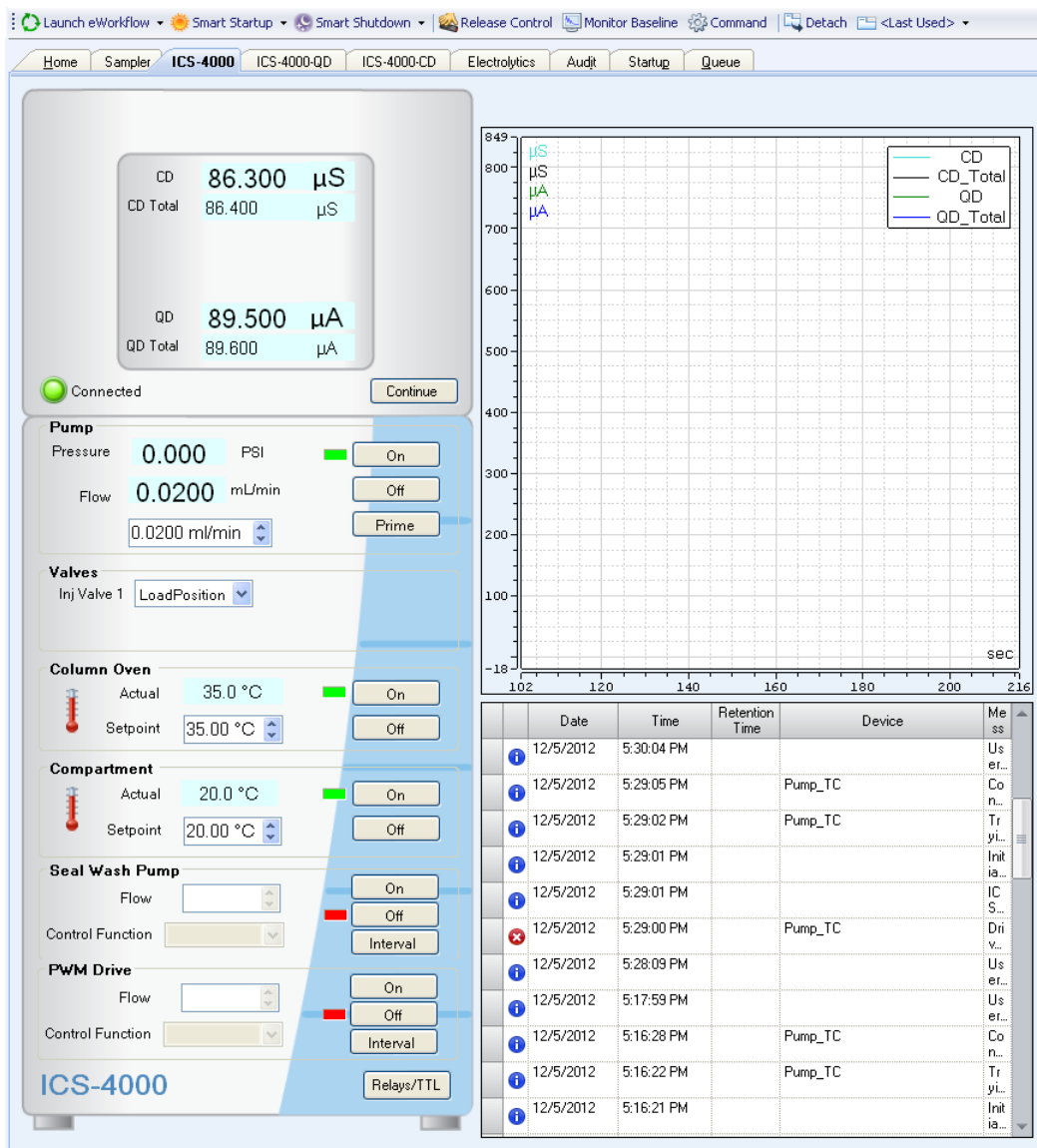
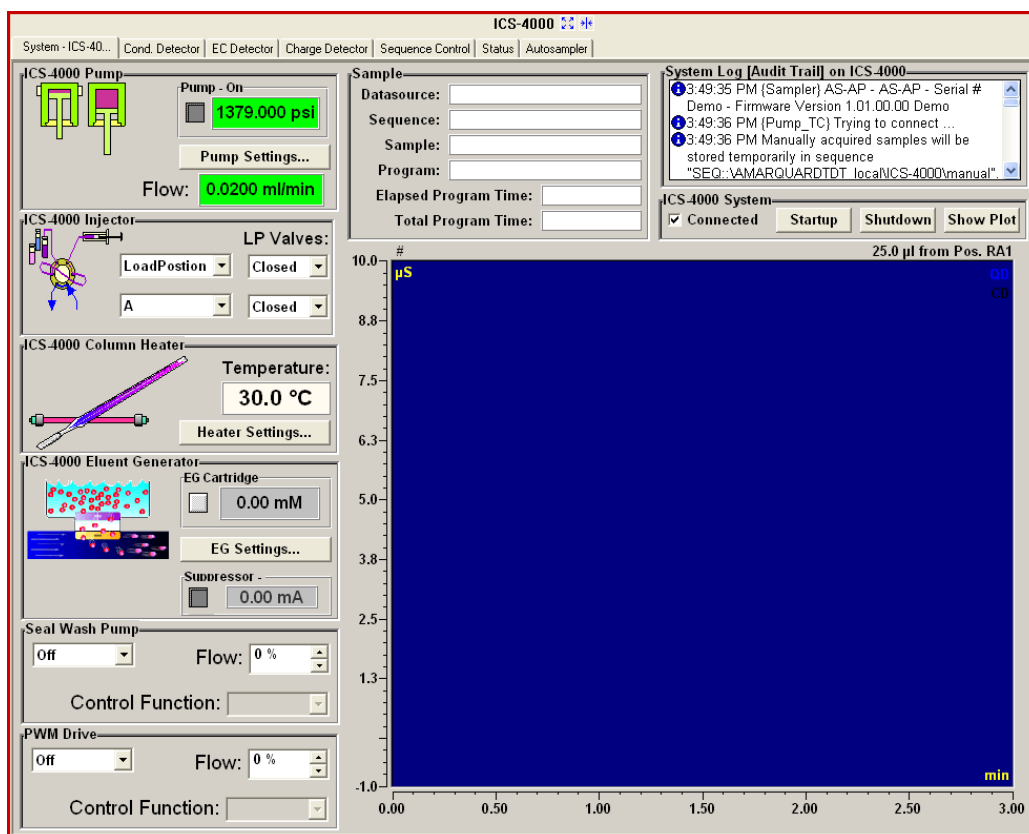


Figure 8. Example Chromeleon 6.8 panel tabset



For instructions about how to connect to the ePanel Set or panel tabset, see “Connecting to Chromeleon” on page 39.

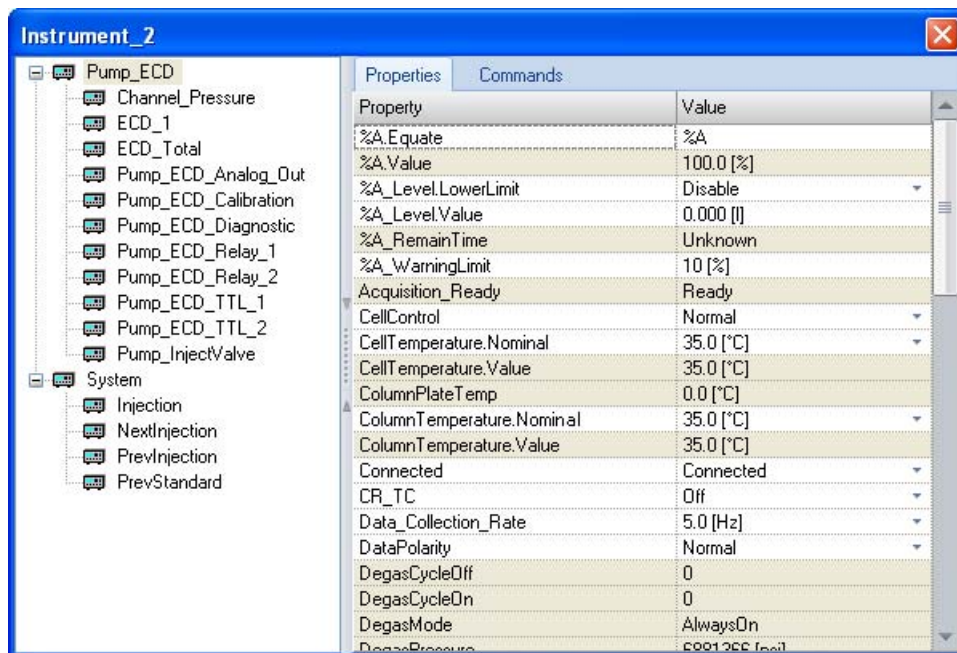
Software Control Modes

Two modes of software control are available: direct control and automated control.

- With direct control, you use the controls (buttons, sliders, and so forth) on the panels to issue commands and enter operating parameters. Direct control commands and parameter settings are executed as soon as they are entered.

If the function that you want to perform is not available on the panels, press the **F8** key to open the Chromeleon 7 Command window (see Figure 9) or the Chromeleon 6.8 Commands dialog box. From there, you can access all commands available for the system.

Figure 9. Chromeleon 7 Command window



- With automated control, you create a list of control commands to be executed in chronological order. For more information about automated control, see “Performing Sample Analyses” on page 47.

Eluent Reservoirs

The eluent organizer on top of the Dionex ICS-4000 holds two 1-liter or 2-liter reservoirs. The following reservoirs are available for use with the Dionex ICS-4000:

- 1-liter polypropylene reservoir (P/N 063291)
- 2-liter polypropylene reservoir (P/N 062510)
- 1-liter polystyrene reservoir (P/N 075313)



CAUTION Do not use the plastic reservoirs for offline vacuum degassing of eluents. The reservoirs were not designed for this purpose.



MISE EN GARDE N'utilisez pas le réservoir en plastique pour le dégazage à vide hors ligne d'éluants. Le réservoir n'a pas été conçu à cette fin.



VORSICHT Verwenden Sie keine Plastikbehälter zum Offline Vakkum-Entgasen von Eluenten. Die Behälter sind dafür nicht ausgelegt.

Eluent Degasser

A built-in vacuum degasser provides continuous, online eluent degassing. Liquid from the eluent reservoir is directed to the degas module (see [Figure 3](#)) before it enters the system flow path. Eluent quality significantly affects pump performance, and vacuum degassing eluents is one way to ensure high eluent quality. Degassing helps prevent bubbles (caused by eluent outgassing) from forming in the pump heads and detector cell. Degassing eluents is especially important when combining aqueous and nonaqueous components (for example, water and acetonitrile).

By default, eluent degassing is enabled when the system power is turned on. The degasser vacuum pump turns on and off as needed to maintain a vacuum. Allow about 10 minutes for equilibration before starting a run. Under some circumstances (for example, when investigating a leak), vacuum degassing may need to be disabled. To disable vacuum degassing from Chromeleon, follow the steps below. You can also disable vacuum degassing from the touch screen (see [“To enable and disable vacuum degassing”](#) on [page 73](#)).

❖ To disable vacuum degassing from Chromeleon

1. Open the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset (see [“Connecting to Chromeleon”](#) on [page 39](#)).
2. Press the **F8** key.
3. Select the pump device.
4. Locate the **DegasStatus** property and select **Off**.
5. To turn on the degasser again, select **On**.

Eluent Valve

The eluent valve controls the flow from the eluent reservoir. The valve opens automatically when the pump is started and closes when the pump is stopped. The valve can also be opened and closed manually from the Chromeleon panel or the touch screen Pump screen (see [Figure 44](#)). This lets you perform service procedures on pump components without eluent leaks occurring.

Pump

The dual-stepper motor pump provides precise and accurate capillary flow performance with the power to support high-pressure, high-efficiency HPIC columns and separations. The variable-speed, serial dual-piston, digital cam design delivers consistent flow rates and quiet detector baselines. User-selectable pressure limits automatically stop pump flow in the event of leaks, flow restrictions, or a depleted eluent reservoir.

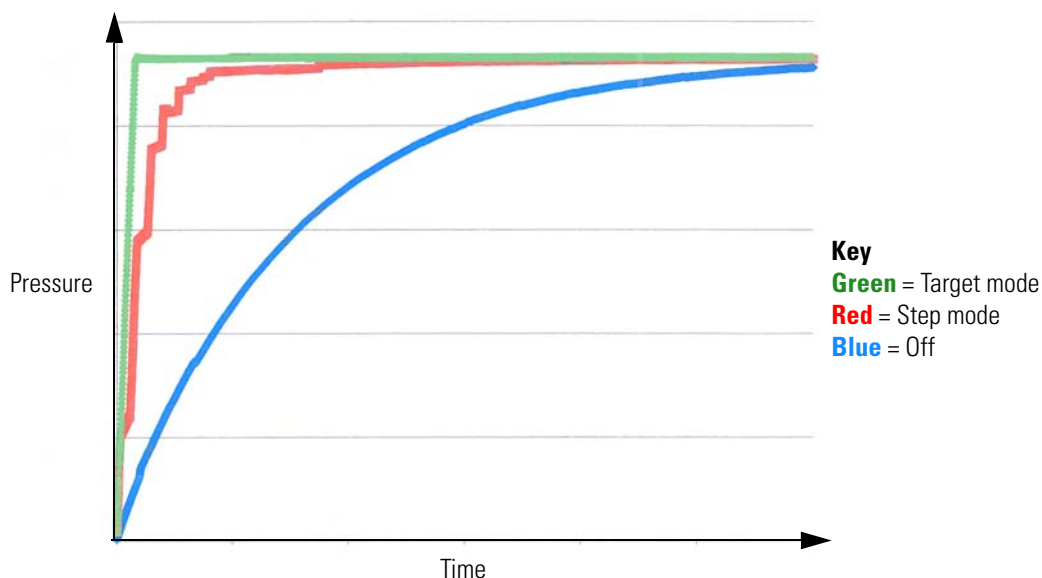
Fast Ramping

Fast ramping refers to the automatic quick ramp-up of pressure to the system operating pressure when the pump is first turned on. This is achieved by temporarily increasing the pump flow rate to 0.100 mL/min. When fast ramping is complete, the flow rate automatically returns to the set flow rate. Two methods of fast ramping are available, or the function can be turned off.

- If **Target** mode is selected, the pressure ramps up quickly to the selected target pressure.
- If **Step** mode is selected, the pressure increases quickly to 3 MPa (500 psi). This is followed by a series of smaller step increases, each consisting of a small fast ramp up followed by a short period of regular operation. When the pressure stabilizes at the system operating pressure, fast ramping stops.
- If **Off** is selected, the pressure increases at a slower rate, determined by the set flow rate.

Figure 10 compares how quickly the pressure increases in a system, for each of the options above.

Figure 10. Fast ramping mode comparison



Flow rate changes that occur during fast ramping are internal. The set flow rate displayed in Chromeleon or on the touch screen does not change during fast ramping.

The fast ramping mode can be selected in Chromeleon or on the Pump touch screen (see [“Controlling the Pump”](#) on page 72).

Pump Components

Pump flow components are chemically inert, made with high-quality PEEK heads and fittings, inert polymer seals, sapphire pistons, and sapphire check valve components.

Pump Heads

The two pump heads on the Dionex ICS-4000 pump are connected in series. Eluent passes through the primary head and secondary head in succession.

The primary pump head delivers eluent at the selected flow rate, while simultaneously filling the secondary pump head. The secondary pump head serves as a reservoir and delivers eluent while the primary head carries out the refill stroke.

Pulse Damper

Flow output from the secondary pump head continues to the pulse damper, which smooths minor pressure variations. From there, flow is directed to the pressure transducer.

The pulse damper is equipped with a heater that maintains a constant temperature. The default temperature setting is 37 °C. If ambient temperature in the laboratory rises above 30 °C, the heater temperature can be adjusted (see [“To control the pulse damper heater”](#) on page 87).

Pressure Transducer

The Dionex ICS-4000 instrument control firmware uses system pressure measurements from the pressure transducer to minimize pressure deviations during overlap of primary and secondary piston strokes. In addition, system pressure is monitored for high- and low-pressure alarms. From the pressure transducer, flow is directed to the eluent generator and then to the remainder of the chromatography system.

Leak Sensor

If liquid collects in the drip tray below the pump components, the leak sensor reports the leak and an error message is displayed on the touch screen and in the Chromeleon audit trail. In addition, the Alarm LED on the front panel (see [Figure 2](#)) lights.

Piston Seal Wash System (Optional)

The optional piston seal wash system rinses the piston seal on each pump head. This prolongs seal lifetime by preventing eluent crystallization on the piston surfaces when using manually-prepared eluent.

The piston seal wash system consists of a seal wash pump, a reservoir containing wash solution, and the connecting tubing. The wash solution is usually ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “Deionized Water Requirements for IC” on page xvii.

A Seal Wash Kit (P/N 078434) is available that provides the parts for adding a piston seal wash system to the Dionex ICS-4000.

Eluent Generator (EG)

The EG electrolytically generates high purity acid or base eluents online from deionized water. The EG consists of the following components:

- A high-precision programmable current source (power supply)
- A disposable eluent generator cartridge (Dionex EGC) that contains an electrolyte concentrate solution, appropriate for the type of eluent being generated
- A Continuously Regenerated Trap Column (CR-TC) to remove any extraneous contaminants from the deionized water source. The Dionex CR-TC is electrolytically-regenerated, which allows it to operate for extended periods without chemical regeneration
- An EG degas cartridge (capillary) that removes electrolysis gases from the generated eluent

The EG power supply is built into the Dionex ICS-4000. The EGC, CR-TC, and EG degas cartridge must be ordered separately.

The EGC and CR-TC are installed on the top tray of the Dionex ICS-4000. To access these components, lift up the touch screen panel (see [Figure 3](#)). The EG degas cartridge is installed inside the chromatography compartment in the Thermo Scientific Dionex IC Cube™ module. For details about the Dionex IC Cube, see [page 19](#).

Eluent Generator Cartridge (EGC)

Table 1 lists the versions of eluent generator cartridges available for use with the EG. Each EGC contains an electrolyte concentrate solution appropriate for the particular eluent generation.

Table 1. Eluent generator cartridge versions

EGC Version	Part Number	Function
EGC KOH (Capillary)	072076	Generates potassium hydroxide eluent for anion exchange separations
EGC MSA (Capillary)	072077	Generates methanesulfonic acid eluent for cation exchange separations

For more information, refer to the EGC manual, included on the Thermo Scientific Reference Library DVD (P/N 053891).

You can select the concentration of eluent to be generated from either Chromeleon or from the touch screen Home screen. For details, see “[Operating the Eluent Generator \(EG\)](#)” on [page 49](#)

Continuously Regenerated Trap Column (CR-TC)

The CR-TC is a high pressure, electrolytically-regenerated trap column that removes anionic or cationic contaminants in the eluent or deionized water. The following versions of the CR-TC can be used with the Dionex ICS-4000 EG:

Table 2. CR-TC versions

CR-TC Version	Part Number
Continuously Regenerated Anion Trap Column (Capillary); CR-ATC (Capillary)	072078
Continuously Regenerated Cation Trap Column (Capillary); CR-CTC (Capillary)	072079

For more information, refer to the CR-TC manual, included on the Thermo Scientific Reference Library DVD (P/N 053891).

Note The Thermo Scientific Dionex IonPac® ATC-HC Trap Column (P/N 059604) or IonPac CTC-1 Trap Column (P/N 040192) may be used with the Dionex ICS-4000 EG. However, both IonPac trap columns require off-line chemical regeneration. Contact Thermo Fisher Scientific for more information.

Temperature Control Zones

The Dionex ICS-4000 provides the following temperature control zones:

- Column heater
- Chromatography compartment
- Heated conductivity cell
- Pump pulse damper heater

The temperatures achieved for a particular zone can vary from the control range, depending on the ambient temperature (see [Table 3](#)).

Table 3. Temperatures achieved for each of the temperature zones

Temperature Zone	Control Range	Achieved Temperatures (Based on Ambient)
Column heater	15 to 80 °C	Minimum temperature \geq (chromatography compartment temperature + 5 °C)
Chromatography compartment	10 to 40 °C	<ul style="list-style-type: none"> • If the conductivity cell and column heater are not on: Minimum temperature \geq (ambient – 20 °C) • If the conductivity cell or column heater is set to 60 °C or above: Minimum temperature \geq (ambient – 17 °C) <p>To achieve the minimum compartment temperatures listed above, set the compartment fan speed to high (see page 87).</p>
Conductivity cell	15 to 60 °C	Minimum temperature \geq (chromatography compartment temperature + 5 °C)
Pulse damper heater	15 to 40 °C	Minimum temperature \geq (chromatography compartment temperature + 5 °C)

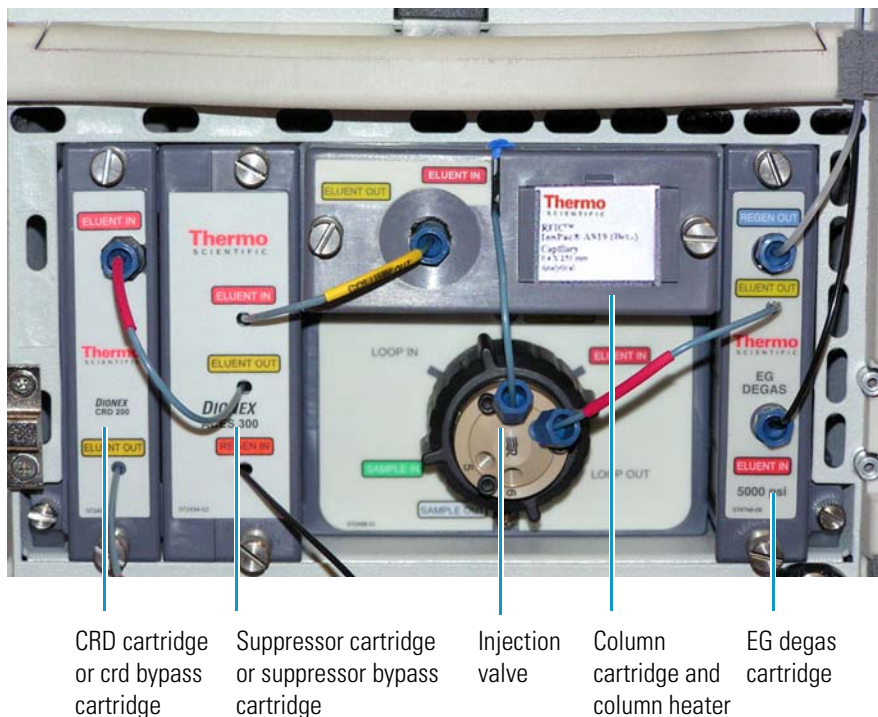
Minimum temperatures are also affected by the ambient humidity. If the humidity is high, the minimum temperature achieved for a temperature zone is not as low as that achieved in a low ambient humidity environment.

Dionex IC Cube

The Dionex IC Cube is installed inside the chromatography compartment of the Dionex ICS-4000 (see [Figure 3](#)). The Dionex IC Cube houses an injection valve, a column heater, and the consumable components (columns, EG degasser, suppressor, CRD) required for running capillary IC applications (see [Figure 11](#)). The consumables are installed in removable cartridges that include the plumbing required to connect the component to other system components.

Note If the Dionex IC Cube does not include a CRD or suppressor, bypass cartridges must be installed. Bypass cartridges provide the internal plumbing connections required for eluent and regenerant flow through Dionex IC Cube components.

Figure 11. Dionex IC Cube features (example with CRD and suppressor cartridges installed)



EG Degas Cartridge

The EG degas (capillary) cartridge contains a tubing assembly that purges the electrolysis gas from eluent generated by the Dionex ICS-4000. After passing through the degas tubing, the eluent is directed to the injection valve and then to the guard column (if installed) and analytical column.

The EG degas cartridge also contains plumbing connections that route the regenerant flow out of the Dionex IC Cube to the regenerant inlet port of the CR-TC.

If the system does not include an EG, the eluent inlet line from the EG degas cartridge is connected directly to the pump outlet. The regenerant outlet line is directed to waste.

Injection Valve

A 4-port high-pressure valve (P/N 074525) is installed in the Dionex IC Cube. The 4-port valve has a 0.4 μL internal sample loop.

A 6-port high-pressure valve can be configured by replacing the 4-port valve pod with a 6-port valve pod (P/N 075971). For instructions, see “[Replacing a High-Pressure Valve Pod](#)” on [page 147](#). You can install either an external sample loop, for example, a 10 μL PEEK™ sample loop (P/N 042949) or a concentrator column on the 6-port valve.

The injection valve has two operating positions (Load and Inject). Liquid flows through either the Load or Inject path, depending on the valve position. [Figure 12](#) shows the flow schematic for a 4-port valve. [Figure 13](#) shows the flow schematic for a 6-port valve.

- In the Load position, sample flows from the syringe or autosampler line (if installed), through the valve, and into the sample loop, where it is held until injection. Excess sample flows out to waste. Eluent flows from the pump, through the valve, and to the column, bypassing the sample loop.
- In the Inject position, eluent flows from the pump, through the sample loop, and on to the column, carrying the contents of the sample loop with it.

Figure 12. Injection valve flow schematics (4-port valve)

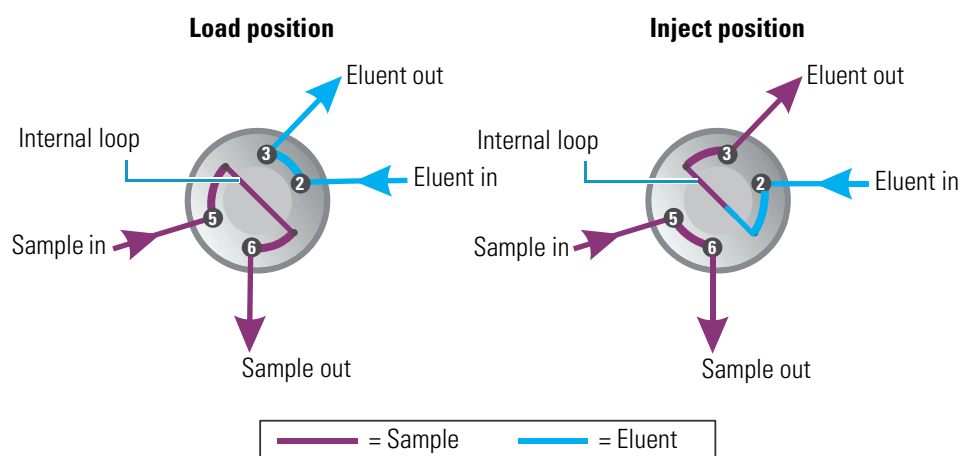
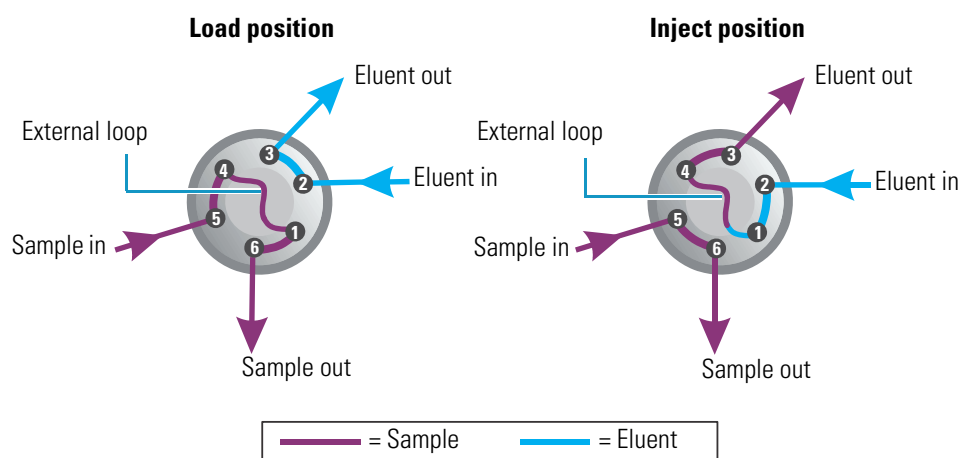


Figure 13. Injection valve flow schematics (6-port valve)



Column Heater and Column Cartridge

The column heater provides a temperature controlled compartment for the capillary guard and analytical columns. The heater temperature range is from 5 °C above the temperature of the chromatography compartment to 80 °C.

The capillary guard column (if included) and the capillary analytical column are installed in a cartridge (see Figure 14) that slides into the column heater. The column cartridge and column heater are included with the Dionex ICS-4000. The capillary columns are ordered separately. For information about how to replace columns and column tubing, see “Replacing Capillary Columns” on page 149.

Figure 14. Column cartridge



Suppressor Cartridge or Suppressor Bypass Cartridge

Two versions of capillary suppressor cartridges are available: Thermo Scientific Dionex ACES™ 300 Anion Capillary Electrolytic Suppressor cartridge (P/N 072052) and Thermo Scientific Dionex CCES™ 300 Cation Capillary Electrolytic Suppressor cartridge (P/N 072053). Each cartridge holds a suppressor and the required tubing connections for eluent and regenerant flow to the suppressor. The suppressor and tubing connections inside the cartridge are not accessible. Tubing exits the front of the cartridge to allow connection to other capillary IC components.

When the cartridge is installed in the Dionex IC Cube, pins on the rear of the cartridge connect the suppressor to its power source. For details about the suppressors, refer to the suppressor manuals, included on the Thermo Scientific Reference Library DVD (P/N 053891).

Installation of a suppressor is optional. If a suppressor is not used, a suppressor bypass cartridge (P/N 072055) must be installed. The suppressor bypass cartridge contains the plumbing connections necessary for eluent and waste flow through the system.

CRD Cartridge or CRD Bypass Cartridge

The CRD 200 (Capillary) cartridge (P/N 072054) contains a CRD 200 Carbonate Removal Device (Capillary). The CRD 200 (Capillary) removes the carbonate peak contributed by the sample in hydroxide eluent chemistries. Removal occurs immediately after suppression of the eluent, before the sample reaches the detector. The CRD 200 (Capillary) is optimized for operation at the flow rate range typically used with capillary columns.

The CRD and tubing connections inside the cartridge are not accessible. Tubing exits the front of the cartridge to allow connection to other capillary IC components.

Installation of a CRD 200 (Capillary) is an option for anion applications. If a CRD is not used, a CRD bypass cartridge (P/N 072056) must be installed. The bypass cartridge contains the plumbing connections necessary for eluent and waste flow through the system.

Dionex IC Cube Flow Schematics

The following figures show schematics of Dionex IC Cube configurations:

- Conductivity detection ([Figure 15](#))
- Conductivity detection with charge detection ([Figure 16](#))
- Electrochemical detection ([Figure 17](#)).

The numbers on the schematics indicate the flow path of eluent and regenerant through the plumbing connections.

Figure 15. Dionex IC Cube flow schematic for conductivity detection (shown with optional CRD)

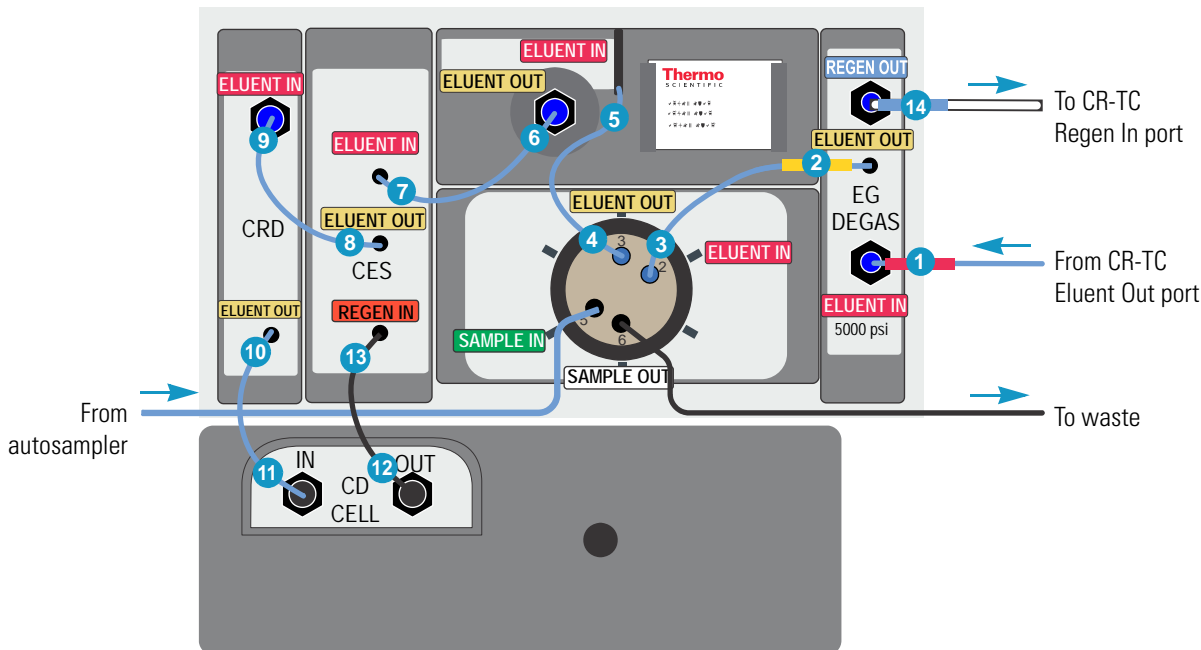
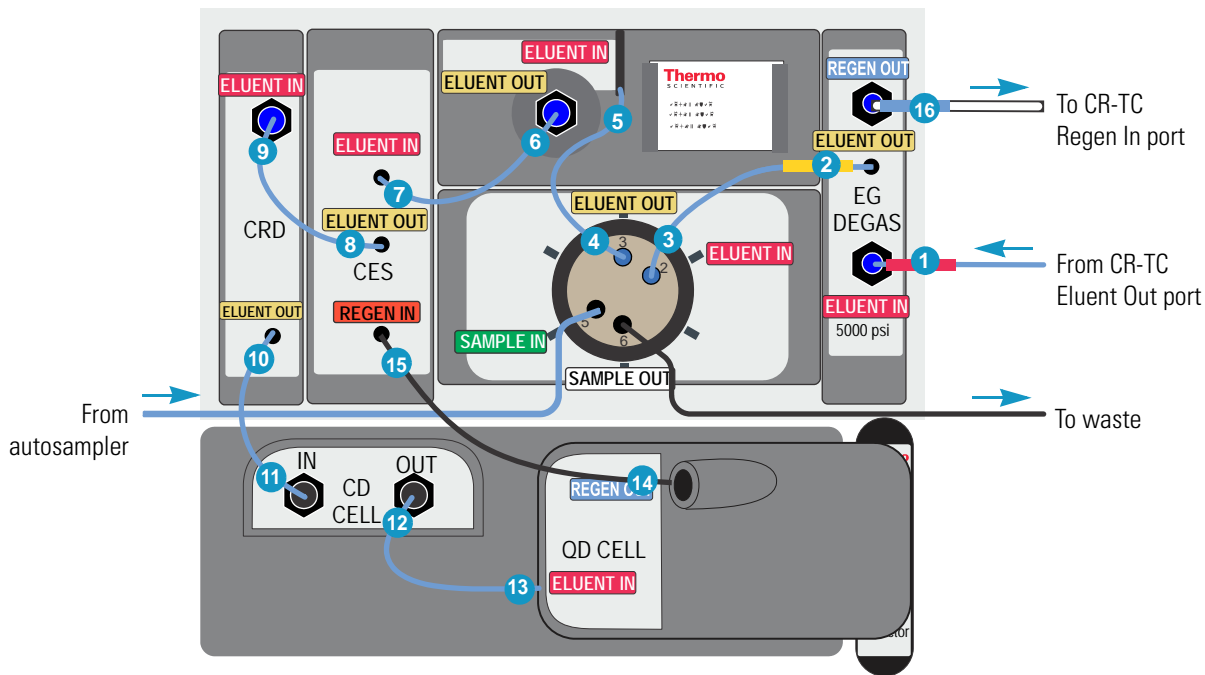
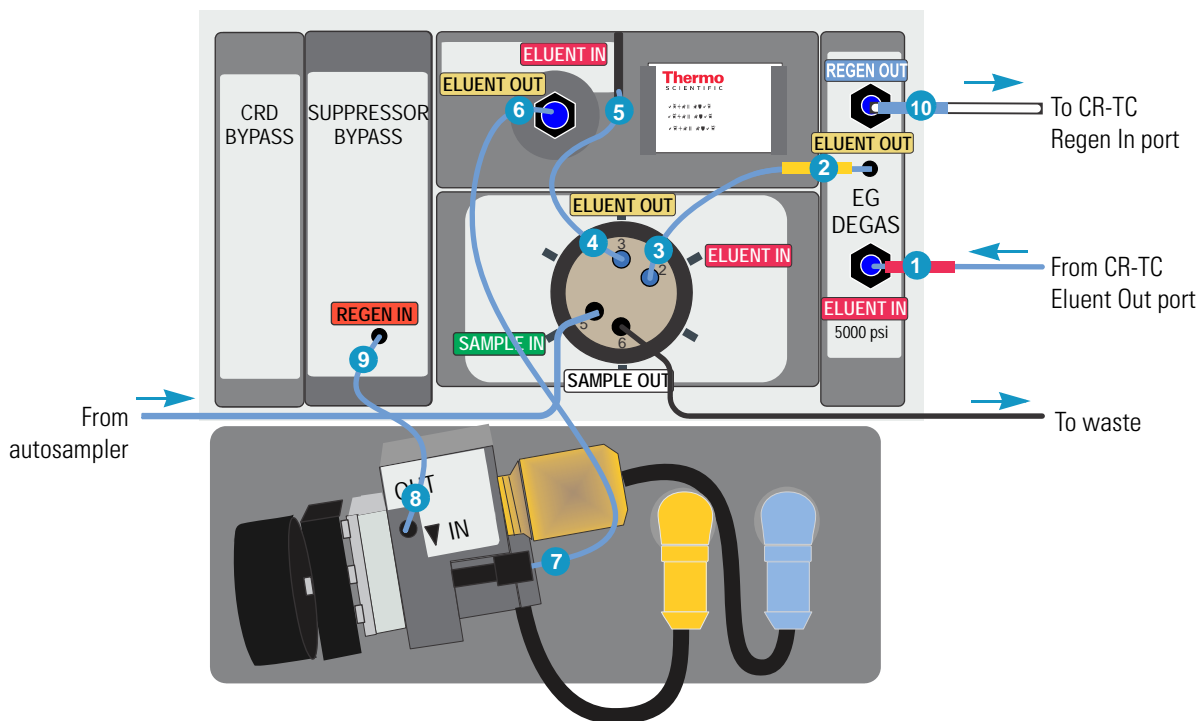


Figure 16. Dionex IC Cube flow schematic for conductivity detection with charge detection



For the complete system flow path for conductivity detection and conductivity detection with charge detection, see Figure 4.

Figure 17. Dionex IC Cube flow schematic for electrochemical detection



For the complete system flow path for electrochemical detections see [Figure 5](#).

Detectors

Either a Dionex ICS-4000 Conductivity Detector (CD) (P/N 075120) or a Dionex ICS-4000 Electrochemical Detector (ED) (P/N 075121) can be installed in the chromatography compartment (below the Dionex IC Cube). If a conductivity detector is installed, a Dionex ICS-4000 Charge Detector (QD) (P/N 078449) can be used.

For details about a detector, see:

- “Dionex ICS-4000 Conductivity Detector (CD)” on page 26
- “Dionex ICS-4000 Electrochemical Detector (ED)” on page 26
- “Dionex ICS-4000 Charge Detector (QD)” on page 29

To accommodate different applications, the type of detector installed in the system can be changed at any time. For detector installation instructions, see:

- “Replacing a CD” on page 156
- “Replacing a QD” on page 158
- “Replacing an ED” on page 159

Dionex ICS-4000 Conductivity Detector (CD)

The CD consists of a heated conductivity cell and the electronics required for collecting the conductivity data and sending it to the computer and the analog output.

The flow-through conductivity cell measures the electrical conductance of analyte ions as they pass through the cell. Two passivated 316 stainless steel electrodes are permanently sealed into the PEEK cell body. The cell design provides efficient sweep-out, low volume (<1 μL), and low dispersion. Temperature control and compensation help ensure good peak reproducibility and baseline stability.

CD Temperature Control

Temperature directly affects the conductivity of a solution. For example, laboratory heating and air conditioning systems can cause a regular slow cycling in the baseline. This, in turn, can affect the reproducibility of an analysis. The higher the conductivity, the more pronounced the effect. To reduce the effect of temperature variation, the Dionex ICS-4000 provides temperature control of both the chromatography compartment and the cell. A heater inside the cell regulates the cell temperature. The cell heater temperature range is from a low of 5 °C above the temperature of the chromatography compartment to a high of 60 °C.

CD Temperature Compensation

Built-in temperature compensation helps minimize changes in the baseline or in peak heights if the operating temperature is different from the temperature at which the cell was calibrated. The default temperature compensation is 1.7% per °C. This can be reset to between 0% and 3.0% per °C, depending on the eluent. If you notice that the baseline shifts up when the temperature increases, the compensation factor is too low and should be reset to a higher value. You can set the compensation factor in Chromeleon or on the Detector touch screen (see [page 79](#)).

Dionex ICS-4000 Electrochemical Detector (ED)

The ED consists of an amperometric detection cell and the detector electronics required to collect data and send it to the computer and the analog output.

The ED can perform the following electrochemical detection modes:

- DC amperometry
- Integrated amperometry—including pulsed amperometric detection (PAD) and integrated pulsed amperometric detection (IPAD)

ED Cell

The ED cell is a miniature flow-through amperometric detection cell with a titanium cell body (the counter electrode), a reference electrode, and a working electrode. The reference electrode can be either a combination pH-Ag/AgCl reference electrode or a PdH reference electrode. Several disposable and conventional (nondisposable) working electrode types are available. The type of working electrode used depends on the application. Four conventional (nondisposable) working electrode types are available: gold, platinum, silver, and glassy carbon. Five disposable working electrode types are available: gold (on either a polyester or a polytetrafluoroethylene (PTFE) substrate), silver, platinum, and carbon.

The ED cell is a thin-layer design. Eluent flows in a thin channel parallel to the surface of a flat disk electrode. The resulting smooth flow minimizes noise. The low volume (<0.2 μL) of the channel allows operation with capillary columns. The cell design minimizes the electrical resistance between the working electrode and the counter electrode by positioning the counter electrode (the cell body) directly across the thin-layer channel from the working electrode. This results in a wide linear dynamic range.

ED Cell Solvent Compatibility

The ED cell can be used with common reversed-phase solvents such as methanol and acetonitrile. If a disposable working electrode on polyester substrate is used, the percentage of methanol should not exceed 30% and the percentage of acetonitrile should not exceed 10%. In addition, prolonged exposure (more than 8 hours) of disposable gold electrodes on polyester substrate to eluents containing hydroxide concentrations greater than 100 mM is not recommended. Shorter rinse periods of 10 to 20 minutes (for example, the carbonate removal step during monosaccharide and disaccharide chromatography) at high hydroxide concentrations do not affect the electrode performance. If sustained highly alkaline eluent conditions are required, use a disposable gold electrode on a PTFE substrate or a conventional gold electrode. Refer to the disposable electrode manual (Document No. 065040) for additional eluent compatibility information.

Because conventional working electrode blocks are made of Kel-F® and use a gasket made of Ultem®, there is no restriction on the concentration of organic solvents that can be used with them (providing the solvent is compatible with PEEK tubing).

There is also no restriction on the use of organic solvents when disposable gold electrodes on PTFE substrate and PTFE gaskets are used.

Combination pH–Ag/AgCl Reference Electrode

The pH-Ag/AgCl reference electrode is a standard combination pH electrode containing a glass membrane pH half-cell and a Ag/AgCl half-cell. The combination pH electrode monitors eluent pH. The Ag/AgCl half-cell is typically used as the cell reference electrode. To minimize changes in the baseline, the combination pH–Ag/AgCl electrode can be used as the reference electrode during a pH gradient.

IMPORTANT Do not allow the pH-Ag/AgCl reference electrode to dry out. Make sure that eluent is being pumped continuously through the cell. If the cell will not be used for a short time (less than 2 days), disconnect the tubing from the inlet and outlet fittings and install fitting plugs. For longer shutdowns, remove the electrode from the cell and store it in its storage cap filled with saturated KCl solution. For details, see [“ED Cell Storage”](#) on page 48.

pH Dependence

The potentials at which many redox reactions take place on metallic electrodes are pH-dependent, with the potential shifting -0.059 V per pH unit. This is especially true for metal oxide formation, oxidation, and oxidative desorption. Since the reference potential of the combination pH-Ag/AgCl electrode also shifts by -0.059 V per pH unit, pH-dependent potential shifts at the working electrode are canceled.

Correcting for pH Dependence

At an eluent pH of 7, the reference potential of the entire electrode is the same as that of the Ag/AgCl half-cell. As the eluent pH is increased, the pH half-cell potential decreases approximately 0.059 V per pH unit. For example, at an eluent pH of 12, the reference potential of the pH half-cell would be -0.295 V relative to the Ag/AgCl half-cell. Therefore, at pH 12, the potentials applied to the working electrode must be raised approximately 0.3 V when switching from the “Ag” reference to the “pH” reference.

In acidic eluents, the reference potential of the pH half-cell is positive with respect to the Ag/AgCl half-cell, and all applied potentials must be decreased by 0.059 V per pH unit when switching from the “Ag” reference to the “pH” reference.

Monitoring the Dionex ICS-4000 Electrochemical Detector Cell pH Readout

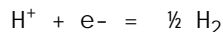
Monitoring the pH readout of a solution with a known composition lets you detect any reference potential shift that may occur over time. This allows you to determine when the pH-Ag/AgCl reference electrode needs regenerating or replacing, thus improving the reproducibility of your analyses. For instructions on how to monitor the pH readout, see [“ED Cell Operating Precautions”](#) on page 51.

Note Monitor the pH when the pH-Ag/AgCl reference electrode is used in the Ag mode as well as in the pH mode.

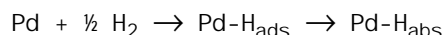
Palladium Hydrogen (PdH) Reference Electrode

The PdH reference electrode consists of palladium and platinum electrodes immersed in an aqueous solution. With a potential applied between the two electrodes, palladium is connected as a cathode and platinum as an anode. As a consequence of the applied potential, hydrogen gas is generated at the palladium electrode and oxygen gas at the platinum electrode.

Whereas the oxygen gas is swept out of the cell in the liquid stream, a portion of the hydrogen gas is taken up by palladium metal. An equilibrium develops between molecular hydrogen in the liquid phase and the adsorbed hydrogen in palladium. The palladium hydrogen electrode becomes a reference electrode with a half reaction of:



Where the hydrogen is supplied by the following process:



The PdH reference electrode is thus similar to the standard hydrogen electrode, which is based on the same half reaction between the hydronium ions and molecular hydrogen. However, there are also some differences. For example, palladium is not as good a catalyst for the electrode half reaction as platinum. As a consequence, under identical conditions, the reference potential of the palladium hydrogen electrode differs from that of the standard hydrogen electrode by a constant potential. In addition, because hydronium ions are involved in the reference half reaction, the potential of the PdH electrode is pH dependent.

No pH Readout with the PdH Reference Mode

If the PdH electrode were connected to another reference electrode (for example, a Ag/AgCl electrode), the PdH electrode could serve as a pH indicator electrode. However, when the PdH electrode functions as a reference electrode, it cannot indicate pH. In the same manner, a glass electrode alone cannot measure pH; it must be used with a suitable reference electrode.

The pH readout of the Dionex ICS-4000 Electrochemical Detector cell is disabled when the PdH reference mode is selected.

Dionex ICS-4000 Charge Detector (QD)

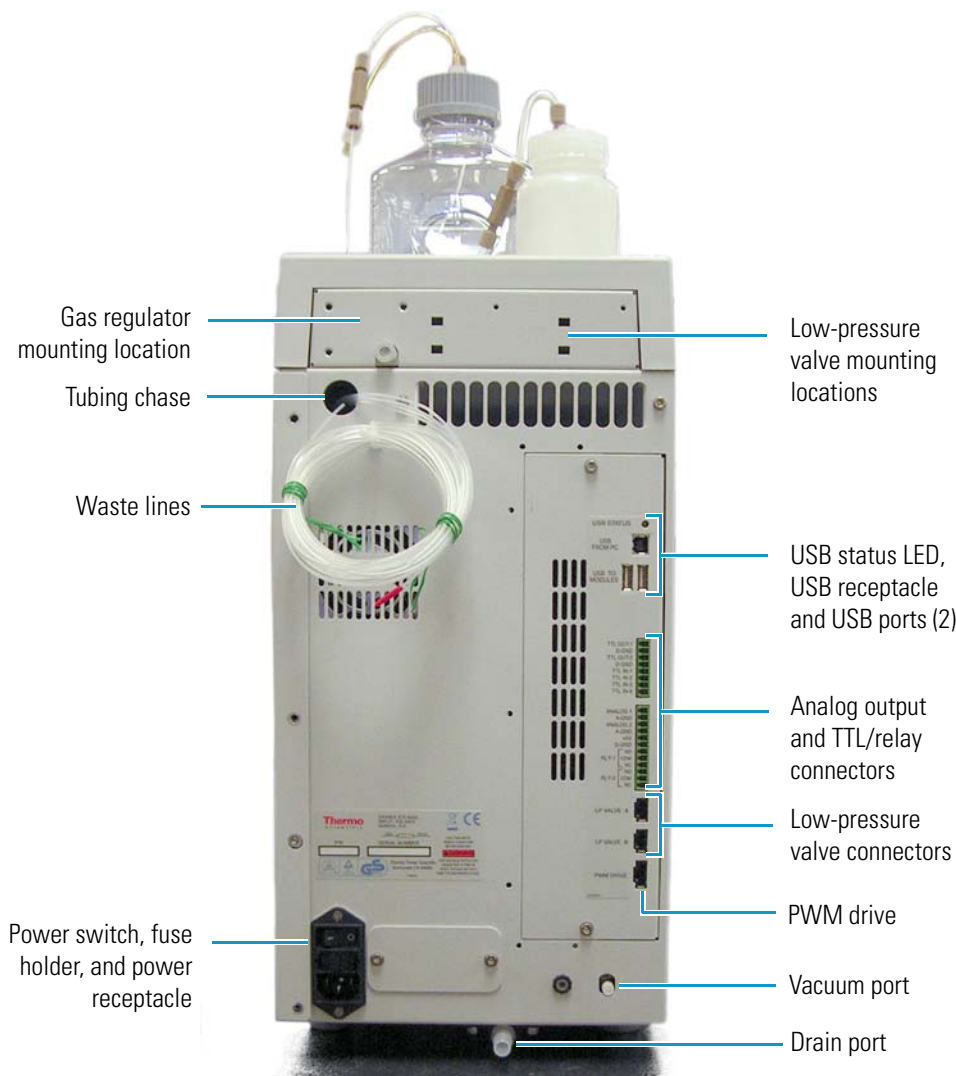
The QD is a flow-through ionic charge detector. In combination with conductivity detection, it provides improved peak identification and peak purity analysis. The QD uses a membrane-based technology and detects ions in proportion to their charge and concentration. It produces up to three times greater signal for triply charged ions (for example, phosphate) than singly charged ions, such as chloride. Ions with the same charge and concentration yield similar response factors, potentially permitting reliable quantification of known and unknown compounds with a single standard.

The QD is advantageous for the analysis of weakly dissociated ions, such as organic acid anions and amine cations. In addition, the QD provides analyte independent calibration, so potentially only one ion is needed to produce a calibration table.

Charge detectors respond to ionic species by drawing a current at a fixed potential. The current level indicates the influx of ionic content. Response is proportional to the charge; thus, weakly dissociated (suppressed) analytes will show a higher response.

Rear Panel Features

Figure 18. Dionex ICS-4000 rear panel features



Gas Regulator and Low-Pressure Valve Mounting Locations

An optional gas regulator for pressurizing the eluent reservoir can be installed. A kit (P/N 078520) that includes the regulator, mounting bracket, and other items needed must be ordered separately. For installation instructions, refer to the *Dionex ICS-4000 Capillary HPIC System Installation Instructions* (Document No. 065469) provided on the Thermo Scientific Reference Library DVD (P/N 053891).

Mounting locations for two optional low-pressure valves are provided. Low-pressure valves can be used for on/off control of liquid flow (for example, to turn flow on and off from a reagent reservoir). For valve details, see “[Low-Pressure Valves](#)” on [page 34](#).

Tubing Chase and Waste Lines

The tubing chase routes system waste lines from the front of the Dionex ICS-4000, through the interior of the system, and to the rear panel.

Power Switch, Fuse Holder, and Power Receptacle

The rear panel power switch provides on/off control of power to the Dionex ICS-4000.

The fuse cartridge contains two 5-amp IEC 60127-2 fast-blow fuses (P/N 954793). To change the fuses, see “[Changing the Main Power Fuses](#)” on page 175.

The IEC 320 C13 power cord plugs into the IEC 320 three-prong receptacle.



CAUTION The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the system and is easily accessible.



MISE EN GARDE Le cordon d'alimentation principal est utilisé comme dispositif principal de débranchement. Veillez à ce que la prise de base soit située/installée près du module et facilement accessible.



VORSICHT Das Netzkabel ist das wichtigste Mittel zur Stromunterbrechung. Stellen Sie sicher, daß sich die Steckdose nahe am Gerät befindet und leicht zugänglich ist.

USB Status LED, and USB Connectors

The USB status LED is lighted when the system is connected to Chromeleon. The LED flashes when communication is occurring between the system and Chromeleon.

The USB (Universal Serial Bus) receptacle (“B” type connector) allows connection to the PC on which Chromeleon software is installed.

The two USB ports (“A” type connectors) allow connection to other USB devices in the system (for example, an autosampler).

A 1.8 m (6 ft) USB cable (P/N 960777) is included in the Dionex ICS-4000 Ship Kit (P/N 075161). For connection instructions, refer to *Dionex ICS-4000 Capillary HPLC System Installation Instructions* (Document No. 065469), provided on the Thermo Scientific Reference Library DVD (P/N 053891).

I/O Connectors (TTL, Analog Outputs, Power, and Relay)

Two I/O connector strips are installed on the rear panel. An 8-pin connector strip provides connections for TTL output and TTL input functions. A 12-pin connector strip provides connections for analog output, power, and relay functions. For connection instructions, see “[Connecting the I/O Connectors](#)” on [page 58](#).

TTL Outputs and Inputs

TTL Outputs

The TTL outputs can be used to control functions in external devices such as an autosampler.

TTL Inputs

When connected to a controlling device, the TTL inputs can be programmed to perform the following functions:

- Switch the injection valve position (Load/Inject)
- Offset the background signal (Autozero)
- Turn the pump on and off (Pump On)
- Send a chart mark signal to the analog output (Mark). The mark is 10% of the full-scale voltage, and the duration is 0.5 seconds. A mark can be used, for example, to indicate when the injection occurred.

Analog Outputs

The two analog outputs supply a voltage signal proportional to the current measured by a detector cell. The outputs can be connected to an analog-to-digital (A/D) converter such as an integrator or other recording device.

Power, Relay, and TTL Outputs

The power, relays, and TTL outputs can be used to control functions in external devices such as an autosampler.

You can control the power, relays, and TTL outputs from the Chromeleon ePanel or the touch screen TTL/Relay screen (see “[Controlling Relay and TTL Outputs](#)” on [page 63](#)).

Low-Pressure Valve Connectors

Connectors for two optional low-pressure valves are provided. For valve details, see “[Low-Pressure Valves](#)” on [page 34](#).

PWM Drive Connector

The PWM (pulse-width modulation) Drive connector provides control of an accessory device, such as the external peristaltic pump option (P/N 082504). The device must be compatible with the power specifications for the PWM drive and connected using a correctly configured cable. The drive can be controlled from Chromeleon or the touch screen Accessory screen. For power specifications, connection instructions, and control instructions, see [“Controlling a Peristaltic Pump or Other Accessory with the PWM Drive”](#) on page 66.

Vacuum Port

The vacuum port can be connected to a vacuum degas module in a Dionex AS-AP Autosampler. The optional Dionex AS-AP degas module is used to degas the autosampler's wash liquid. A Dionex AS-AP Degas Kit (P/N 079883) is available that includes the degas module, tubing, and fittings required to install the degasser in the AS-AP and connect it to the Dionex ICS-4000. If the vacuum port is not connected, make sure the plug remains installed in the port to prevent vacuum loss from the system.

Drain Port

The drain port removes any liquid that collects in the drip tray. A clear corrugated drain line (P/N 055075) is connected to this port during installation. Place the drain line into a waste container. To maintain a positive siphon, position the waste container below the level of the Dionex ICS-4000.

IMPORTANT For correct drainage, make sure the drain line is not bent, pinched, or elevated at any point. Do not allow the end of the line to be submerged in waste liquid.

Pump and Valve Options

Peristaltic Pump

A peristaltic pump (P/N 082504) is available as an option for the Dionex ICS-4000. The pump can be used for delivering sample, external regenerant, or pump seal wash liquid.

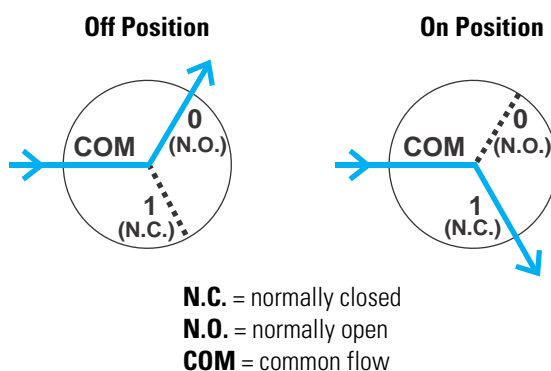
High-Pressure Valves

Two types of high-pressure valves (6-port and 10-port) are available as options for the Dionex ICS-4000. These valves must be installed by a Thermo Scientific representative.

Low-Pressure Valves

Up to two low-pressure valves can be mounted on the rear panel. The valves are either two-way (P/N 079848) or three-way valves (P/N 061971). The two-way valves provide on/off control of liquid flow in one direction, while the three-way valves provide on/off control in two directions (see [Figure 19](#)).

Figure 19. Three-way low-pressure valve flow schematics



Note: When the valve is off (not energized), port 0 is open (N.O.) and port 1 is closed (N.C.). Conversely, when the valve is on, port 1 is open and port 0 is closed.

Valve port connections to chromatography components vary, depending on the application. To control the valves, use one of the following methods:

- Select commands in the Chromeleon 7 Command window (see [Figure 9](#)) or the Chromeleon 6.8 Commands dialog box, or on the touch screen Valves screen (see “[Controlling Optional Valves](#)” on [page 85](#)).
- Add commands to a Chromeleon 7 instrument method or Chromeleon 6.8 program.

Operation

This chapter describes routine startup, operation, and shutdown procedures for the Dionex ICS-4000 Capillary HPIC System.

Note The instructions in this chapter assume that the initial setup of the Dionex ICS-4000 has already been completed (including configuring the Dionex ICS-4000 in a Chromeleon 7 instrument or Chromeleon 6.8 timebase). If this is not the case, refer to *Dionex ICS-4000 Capillary HPIC System Installation Guide* (Document No. 065469) for instructions before beginning operation.

Contents

- Starting Up the System (Overview)
- Setting Up the Reservoir
- Setting Up the Piston Seal Wash System
- Turning On the System Power
- Connecting to Chromeleon
- Priming the Pump
- Setting Operating Parameters and Equilibrating the System
- Preparing Samples
- Loading Samples Into the Sample Loop or Concentrator
- Performing Sample Analyses
- System Shutdown
- Operating the Eluent Generator (EG)
- Operating the Dionex ICS-4000 Electrochemical Detector
- Using Rear Panel Inputs and Outputs
- Controlling a Peristaltic Pump or Other Accessory with the PWM Drive

Starting Up the System (Overview)

Before starting sample analyses, perform the following tasks to start up the system and equilibrate it.

- Set up the reservoir (see [page 36](#))
- Set up the piston seal wash system (if installed) (see [page 39](#))
- Turn on the power (see [page 39](#))
- Connect to Chromeleon (see [page 39](#))
- Prime the pump (if necessary) (see [page 41](#))
- Set operating parameters (see [page 43](#))
- Equilibrate the system and verify operational readiness (see [page 43](#))

Note Because capillary IC systems use low flow rates and consume very little eluent, you can leave the Dionex ICS-4000 on at all times, allowing it to remain equilibrated and always ready to run the next sample.

Setting Up the Reservoir

IMPORTANT Always use ASTM filtered, Type I (or better) (18 megohm-cm) deionized water that meets the specifications in “[Deionized Water Requirements for IC](#)” on [page xvii](#), to rinse and fill reservoirs used for eluent generation or seal washing or to manually prepare eluent,

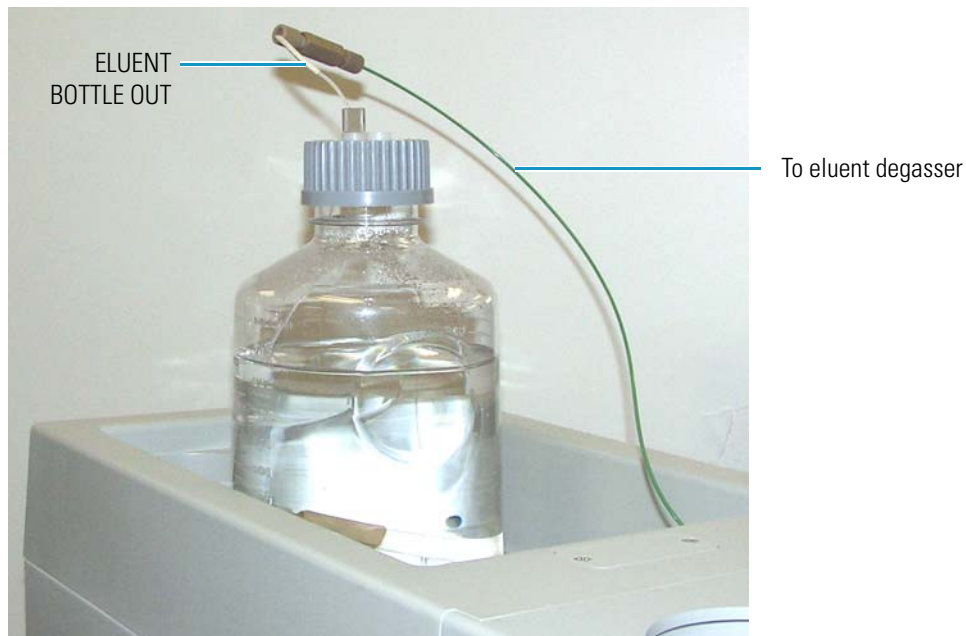
❖ To set up the reservoir

1. Rinse the eluent reservoir with deionized water.

If the reservoir still appears dirty, or has a slimy film on the inside, clean it as instructed on [page 90](#).
2. Fill the reservoir with deionized water. If you are not using the EG, fill the reservoir with the prepared eluent.
3. Place the reservoir in the reservoir organizer on top of the Dionex ICS-4000.
4. Locate the end-line filters (P/N 045987) in the Dionex ICS-4000 Ship Kit (P/N 075161).
5. Thoroughly rinse the end-line filter with deionized water and then install the filter on the end of the reservoir's eluent line.
6. Install the reservoir cap, making sure the end of the line extends to the bottom of the reservoir, and that the filter is submerged in liquid. This prevents air from being drawn through the eluent lines. Hand-tighten the cap.

7. Verify that the liquid line from the pump is connected to the **ELUENT BOTTLE OUT** line from the reservoir cap (see [Figure 20](#)).

Figure 20. Eluent reservoir connections (reservoir P/N 075313 shown)



Pressurizing Reservoirs

The eluent reservoirs available for use with the Dionex ICS-4000 can be pressurized to <math><.07\text{ MPa}</math> (<math><10\text{ PSI}</math>). For a list of reservoirs, see [page 13](#). Although not *required*, pressurizing reservoirs with helium or nitrogen is recommended under the following circumstances:

- When using eluents that are sensitive to contamination.
- When combining aqueous and nonaqueous components (for example, water and acetonitrile). Pressurizable reservoirs allow eluents to be stored under a specific atmosphere.

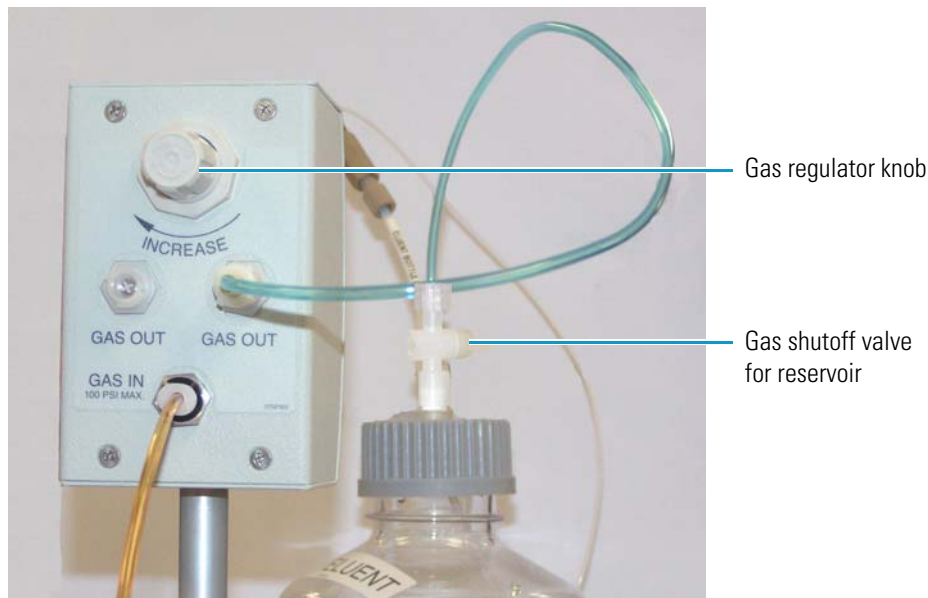
The gas regulator accessory bracket and other items needed for pressurizing the eluent reservoir must be ordered separately (P/N 078520). For installation instructions, refer to *Dionex ICS-4000 Capillary HPIC System Installation Instructions* (Document No. 065469) provided on the Thermo Scientific Reference Library DVD (P/N 053891).

❖ To pressurize the reservoir

1. Connect the liquid line from the pump to the reservoir and fill the reservoir (see “[To set up the reservoir](#)” on [page 36](#)).

2. Turn the gas regulator knob fully counterclockwise (to the left) (see [Figure 21](#)) to ensure that there is no pressure to the reservoir when the gas source is turned on.

Figure 21. Gas connections to the regulator and reservoir



3. Close the gas shutoff valve on the reservoir cap.
4. Turn on the gas source to 0.34 MPa (50 psi) \pm 0.07 MPa (10 psi).
5. Turn the gas regulator knob clockwise and adjust the pressure to between 30 and 40 kPa (5 and 6 psi).



CAUTION Never pressurize eluent reservoirs above 70 kPa (10 psi). Pressurizing reservoirs above this limit can cause the reservoir to rupture or crack.



MISE EN GARDE Ne mettez jamais les réservoirs d'éluants sous une pression supérieure à 0,07 MPa (10 lb/po²).



VORSICHT Setzen Sie den Eluentbehälter auf keinen Fall einem Druck über 0,07 MPa aus.

6. If the liquid line from the pump is connected, you may open the gas shutoff valve on the reservoir cap.

Setting Up the Piston Seal Wash System

If the optional seal wash system is installed, follow the instructions below to set up the system.

❖ To set up the seal wash system

1. Rinse the seal wash reservoir (P/N 075160) with deionized water.
2. Fill the reservoir with deionized water and place it in the reservoir organizer on top of the Dionex ICS-4000.
3. Install the reservoir cap, making sure the end of the line extends to the bottom of the reservoir.
4. Connect the seal wash inlet tubing to the fitting on the reservoir cap.

Turning On the System Power

❖ To turn on the system power

1. Flip the power switch on the rear panel (see [Figure 18](#)).
2. Turn on the power to the computer and the autosampler (if used).

When the Dionex ICS-4000 starts up, the touch screen displays a startup screen and begins an initialization process. When initialization is complete, the Home screen is displayed.

Connecting to Chromeleon

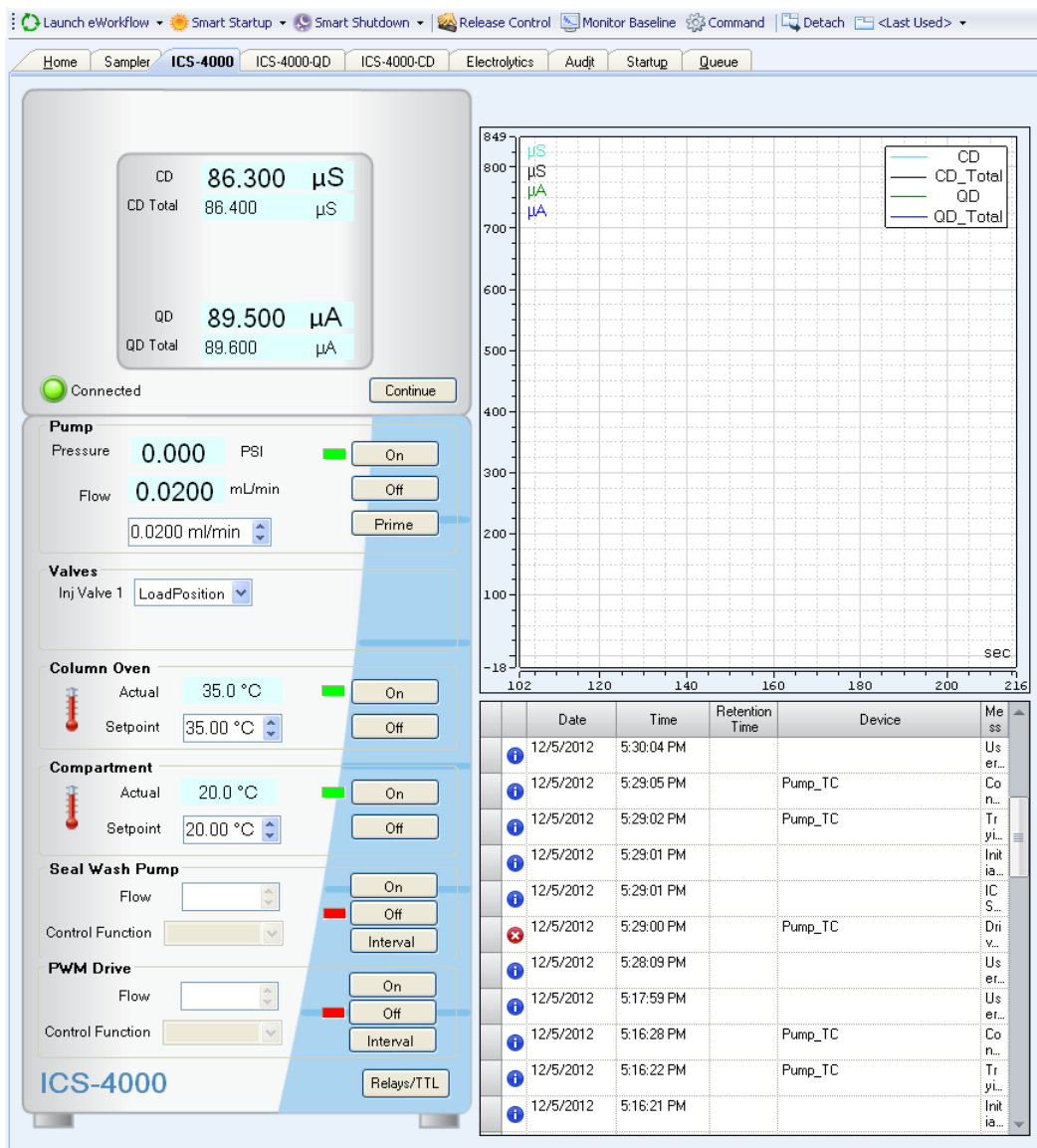
❖ To start the Chromeleon 7 or Chromeleon 6.8 client

- For Chromeleon 7, click **Start > All Programs > Chromeleon 7 > Chromeleon 7**.
- For Chromeleon 6.8, click **Start > All Programs > Chromeleon > Chromeleon**.

❖ **To display the ePanel Set (Chromeleon 7) or panel tabset (Chromeleon 6.8)**

- For Chromeleon 7, click the **Instruments** Category Bar. Select the name of the instrument in which the Dionex ICS-4000 is configured. Chromeleon 7 connects to the instrument and displays the ePanel Set. See the example in [Figure 22](#).

Figure 22. Example Chromeleon 7 ePanel Set




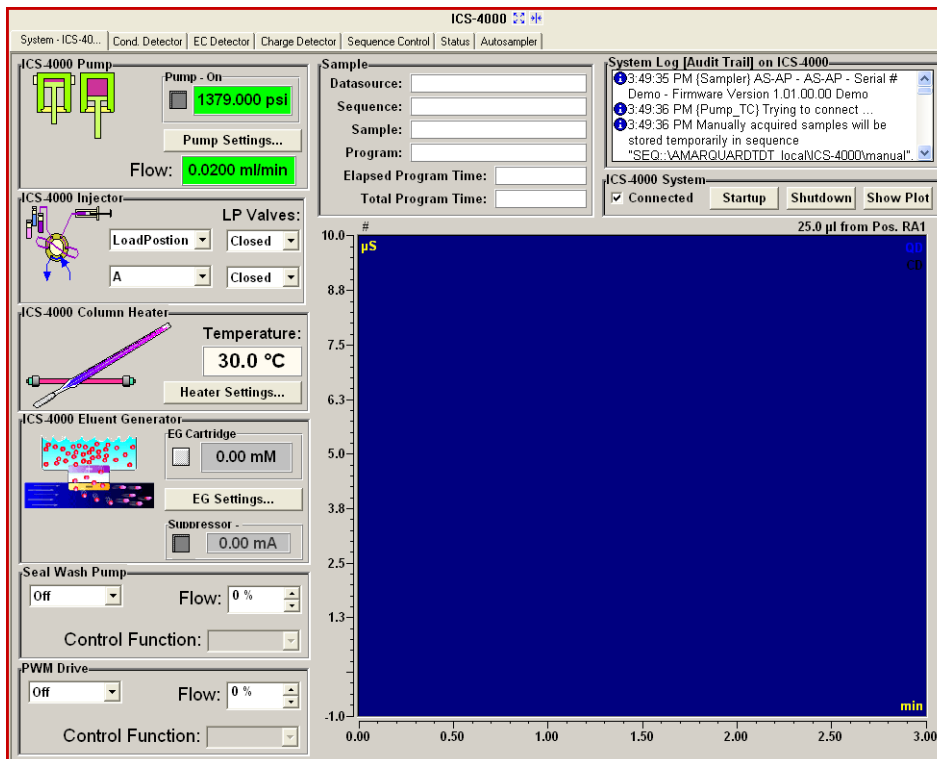
- For Chromeleon 6.8, click **View > Default Panel Tabset** or click the  toolbar button. The panel tabset opens. See the example in [Figure 23](#).

Figure 23. Example Chromeleon 6.8 panel tabset



Priming the Pump

Before beginning operation, follow the instructions in this section to prime the pump if any of the following conditions applies:

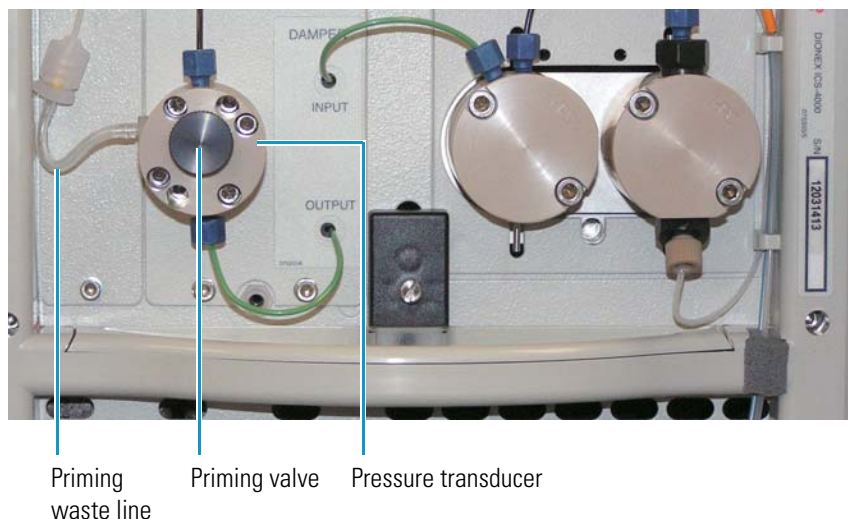
- The eluent has been changed.
- The eluent line is new (empty).
- The eluent line contains air.

Note Although a 10 mL syringe (P/N 079803) can be used to prime the pump, using a syringe is recommended only if the eluent line is 100% empty or if the pump is dry.

❖ **To prime the pump**

1. Open the priming valve on the pressure transducer (see [Figure 24](#)) by turning the knob one-quarter to one-half turn counterclockwise.

Figure 24. Pump priming valve



2. Disconnect the system from Chromeleon, if it is currently connected.
3. On the touch screen, select **Pump** on the Menu screen to open the Pump screen.
4. Under Prime/Flush, touch the flow rate list and select **2.00 mL/min**.
5. Touch **Prime**.

Prime the pump until all air is purged and no air bubbles can be seen exiting the waste line that extends from the left side of the pressure transducer (see [Figure 24](#)).

6. To stop priming, touch **Off**. Close the priming valve by turning the knob clockwise. Tighten no more than fingertight.

IMPORTANT Do not use any tools to tighten the priming valve! Overtightening may destroy the cap seal. Open or close the priming valve only when the system pressure is less than 0.7 MPa (100 psi).

Note You can also prime the pump from Chromeleon. On the Chromeleon 7 ePanel Set or the Chromeleon 6.8 panel tabset, click the **ICS-4000** tab. Click **Prime**. A message asks you to verify that the purge (priming) valve is open. Click **Execute despite warnings** (Chromeleon 7) or **OK** (Chromeleon 6.8).

Setting Operating Parameters and Equilibrating the System

This section is an overview of the steps needed to set initial system operating parameters and to equilibrate the system before beginning operation.

Setting Operating Parameters

Before beginning operation, set the following system component parameters and start each component:

- Pump flow rate
- EGC concentration
- Suppressor current (if installed)
- Temperature of each temperature device (cell heater, column heater, chromatography compartment, damper heater)

The value to set for each operating parameter depends on the application to be run. For the required settings for your application, refer to the column manual or other application documentation.

Note A Virtual Column Separation Simulator is available in Chromeleon that can help you determine the best operating parameters (column, eluent, flow rate, temperature) for a particular analysis. Refer to the Chromeleon Help for details.

You can set operating parameters on the touch screen (see “[Touch Screen Operation](#)” on [page 69](#)) or in Chromeleon.

Equilibrating the System and Verifying Operational Readiness

❖ To equilibrate the system

1. Set operating parameters and turn on devices as described in “[Setting Operating Parameters](#).”
2. In Chromeleon 7, click **Monitor Background** on the toolbar above the ePanel Set. In Chromeleon 6.8, select **Control > Acquisition On**. Chromeleon begins plotting the detector signal and pump pressure readings.
3. View the detector signal and monitor the background on the detector panel.

—or—

Monitor the detector signal on the touch screen Plot screen. See “[Viewing a Plot of the Detector Output](#)” on [page 82](#).

4. Offset the detector background and zero the reading by clicking **Autozero** on the detector panel in Chromeleon.

5. Verify that the detector baseline is at the expected reading for your application and is stable. Refer to the column manual for the appropriate background for your application. The column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
6. If the reading is too high, see “[High Background](#)” on [page 112](#) for troubleshooting information. If the baseline is drifting or is excessively “noisy” (there are large fluctuations in readings), see “[Noisy Baseline](#)” on [page 110](#) for troubleshooting information.
7. Monitor the pump pressure and make sure it is at the expected reading for the installed column and is stable.
8. Verify that all installed temperature control devices are at their set temperatures and are stable.

The system is now ready for operation.

Note

- Equilibration time varies, and it may take some time to reach the expected values.
- Because capillary IC systems use low flow rates and consume very little eluent, you can leave a capillary IC system on at all times, allowing it to remain equilibrated and always ready to run the next sample.

Preparing Samples

This section provides basic information about collecting, storing, and preparing samples for analysis.

Note Sample preparation can be performed while the system is equilibrating.

Collecting and Storing Samples

Collect samples in high density polyethylene, polystyrene, or polycarbonate containers that have been thoroughly cleaned with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “[Deionized Water Requirements for IC](#)” on [page xvii](#). Do not clean containers with strong acids or detergents because these can leave traces of ions on the container walls. The ions may interfere with the analysis.

If samples will not be analyzed on the day they are collected, filter them through clean 0.45-micron filters immediately after collection; otherwise, bacteria in the samples may cause the ionic concentrations to change over time. Refrigerating the samples at 4° C will reduce, but not eliminate, bacterial growth.

Analyze samples containing nitrite or sulfite as soon as possible. Nitrite oxidizes to nitrate, and sulfite to sulfate, thus increasing the measured concentrations of these ions in the sample. In general, samples that do not contain nitrite or sulfite can be refrigerated for at least one week with no significant change in anion concentration.

Pretreating Samples

Analyze rainwater, drinking water, and air particulate leach solutions directly with no sample preparation (other than filtering, and if required, diluting).

Filter groundwater and wastewater samples through 0.45-micron filters before injection, unless samples were filtered after collection.

A Dionex Low Volume High Pressure Inline Filter (P/N 074505) is available for removing particulates down to 0.45 micron from samples. Connect the inline filter between the autosampler outlet and the sample inlet port on the injection valve. For details, see the instructions provided with the inline filter.

Before injection, pretreat samples that may contain high concentrations of interfering substances by putting them through Dionex OnGuard™ cartridges. Refer to the installation and troubleshooting guide for the OnGuard cartridge for instructions. The guide is provided on the Thermo Scientific Reference Library DVD (P/N 053891).

Diluting Samples

Because the concentrations of ionic species in different samples can vary widely from sample to sample, no single dilution factor can be recommended for all samples of one type. In some cases (for example, many water samples), concentrations are so low that dilution is not necessary.

To dilute the sample, use eluent or ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#). When using carbonate eluents, diluting with eluent minimizes the effect of the water dip at the beginning of the chromatogram. If you dilute the sample with eluent, also use eluent from the same lot to prepare the calibration standards. This is most important for fluoride and chloride, which elute near the water dip.

To improve the accuracy of early eluting peak determinations, such as fluoride, at concentrations below 50 ppb, dilute samples in eluent or spike the samples with concentrated eluent to minimize the water dip. For example, spike a 100 mL sample with 1.0 mL of a 100 X eluent concentrate.

Loading Samples Into the Sample Loop or Concentrator

This section describes three methods for loading sample into the sample loop or concentrator column:

- Loading samples with an autosampler
- Loading samples with a syringe, using the push method
- Loading samples with a syringe, using the pull (vacuum) method

❖ To load samples with an autosampler

1. Verify that the autosampler output line is connected to the **SAMPLE IN** port on the injection valve. Direct the waste line as required for your autosampler model.
2. Prepare and fill the sample vials or well plates and place them in the autosampler tray or carousel. For detailed instructions, refer to the autosampler manuals, provided on the Thermo Scientific Reference Library DVD (P/N 053891).
3. Create a sequence in Chromeleon that specifies the vials or wells from which to take sample injections and the order in which the injections should be run.

For an overview of creating sequences, see “[Performing Sample Analyses](#).” For details about creating sequences, refer to the Chromeleon Help.

❖ To load samples with a syringe, using the push method

1. Follow the steps below to connect a sample inlet line and luer adapter to the injection valve:
 - a. Locate the manual injection tubing assembly (P/N 078493) in the Dionex ICS-4000 Ship Kit (P/N 075161). Connect the free end of the tubing to the **SAMPLE IN** port on the injection valve.
 - b. Route the end with luer adapter out of the chromatography compartment through the side slot.
2. Fill a syringe with a calibration standard or sample and insert the syringe into the luer adapter.
3. Verify that the injection valve is in the Load position. If it is not, switch the valve, using the Chromeleon 7 ePanel Set, Chromeleon 6.8 panel tabset, or the touch screen Valves screen.
4. Push sample through the tubing into the valve. Overfill the sample loop with several sample loop volumes. Excess sample will exit through the injection valve waste line.
5. Leave the syringe in the luer adapter. This prevents the sample from exiting the valve before injection.
6. Switch the injection valve to the Inject position.

❖ **To load samples with a syringe, using the pull (vacuum) method**

1. Follow the steps below to connect a sample inlet line and luer adapter to the injection valve:
 - a. Locate the manual injection tubing assembly (P/N 078493) in the Dionex ICS-4000 Ship Kit (P/N 075161). Connect the free end of the tubing to the **SAMPLE IN** port on the injection valve.
 - b. Route the end with luer adapter out of the chromatography compartment through the side slot.
2. Disconnect the waste line from the injection valve and replace it with a 25 to 30 cm (10 to 12 in) piece of PEEK or PTFE tubing.
3. Place the free end of this line into the sample.
4. Verify that the injection valve is in the Load position. If it is not, switch the valve using the Chromeleon 7 ePanel Set, Chromeleon 6.8 panel tabset, or the touch screen Valves screen.
5. Attach a syringe to the syringe adapter and pull out the plunger to draw the sample into the injection valve.
6. Switch the injection valve to the Inject position.

Performing Sample Analyses

This section is an overview of the steps required for performing sample analyses using Chromeleon. For details about how to perform these steps, refer to the Chromeleon Help.

To perform sample analyses with a Dionex ICS-4000, first add sample injections to a Chromeleon sequence. The sequence determines how a group of injections will be analyzed and the order in which they will be run. For each injection, the sequence typically includes the following elements:

- A predefined list of commands and parameters for controlling the system and acquiring sample data (called an *instrument method* in Chromeleon 7 and a *program* in Chromeleon 6.8).
- The chromatographic data acquired.
- A predefined set of instructions for evaluating the acquired data (called a *processing method* in Chromeleon 7 and a *quantification method* in Chromeleon 6.8).
- Templates for displaying chromatographic data on the screen and for printing reports.

Creating Sequences

Two methods are available for creating a new sequence in Chromeleon: eWorkflows (available for Chromeleon 7 only) and the Sequence Wizard.

- eWorkflows provide predefined templates and rules for creating new sequences. If they have been defined for your laboratory, eWorkflows are the preferred method for creating a new sequence.
- The Sequence Wizard provides a series of dialog boxes that walk you through the sequence creation process.

Running Sequences

After creating the sequence, load it into a Chromeleon 7 queue or Chromeleon 6.8 batch and start the run. Chromeleon performs a Ready Check to verify that the instrument is ready for operation and the instrument methods specified in the sequence are error free. If the Ready Check passes and another sequence is not currently running, the sequence is started.

System Shutdown

Because capillary IC systems use low flow rates and consume very little eluent, you can generally leave the Dionex ICS-4000 on at all times, allowing it to remain equilibrated and always ready to run the next sample.

If you need to shut down the system for any reason (for example, to move it), follow the instructions below.

❖ To shut down the system

- Follow any storage requirements for the consumables installed in the system.
- If a Dionex QDC 300 (Capillary) is installed, remove the cell from the system and dry the ports and tubing by flowing helium or nitrogen gas through them.

Consumables Storage

The columns, suppressors, and other consumable items used with a Dionex ICS-4000 system have various short- and long-term storage requirements. Refer to the manuals for the individual products for instructions. These manuals are provided on the Thermo Scientific Reference Library DVD (P/N 053891).

ED Cell Storage

Short-term Storage of the ED Cell

If the cell will not be used for a short period of time (less than 2 days), disconnect the tubing from the inlet and outlet fittings and install fitting plugs.

If the pH-Ag/AgCl reference electrode remains in the cell but eluent is not being pumped through the cell, the pH-Ag/AgCl reference electrode frit may partially dry out. If this occurs, regenerate the electrode by soaking it in a solution containing 1 M KCl and 1 M HCl.

Long-term Storage of the pH-Ag/AgCl Reference Electrode

If the cell will not be used for 2 days or more, remove the pH-Ag/AgCl reference electrode and store it in a solution of saturated KCl, as instructed in the procedure below.

1. Prepare a saturated solution of KCl in ASTM filtered, Type I (18 megohm-cm) deionized water that meets the specifications listed in “Deionized Water Requirements for IC” on page xvii.
2. Locate the cap in which the electrode was shipped and fill it two-thirds full with the prepared KCl solution.
3. Remove the pH-Ag/AgCl reference electrode from the cell.
4. Insert the electrode into the cap and screw on the cap (see Figure 25).
5. Make sure there is no air bubble in the cap. Add more KCl solution if needed.

Figure 25. pH-Ag/AgCl reference electrode in storage cap



Operating the Eluent Generator (EG)

This section describes how to select and enter the eluent concentration.

Selecting an Eluent Concentration

The allowable eluent concentration depends on the flow rate and the EGC type. Table 4 lists the concentration ranges for each EGC type.

Table 4. Eluent concentration ranges

EGC	Eluent Concentration Range
KOH (Capillary)	0.1 to 200 mM at 0.001 to 0.010 mL/min flow 0.1 to X mM at 0.010 to 0.030 mL/min where X = 2/flow
MSA (Capillary)	0.1 to 200 mM at 0.001 to 0.010 mL/min flow 0.1 to X mM at 0.010 to 0.030 mL/min where X = 2/flow

For the eluent concentration required for your application, refer to the column manual, included on the Thermo Scientific Reference Library DVD (P/N 053891). When the Dionex ICS-4000 is controlled from the front panel, only isocratic eluent delivery is possible. With isocratic delivery, the eluent composition and concentration remain constant throughout the run. When the Dionex ICS-4000 is under Chromeleon control, gradient delivery, in which the eluent concentration changes over time, is also possible.

Entering the Eluent Concentration

This section describes how to enter the eluent concentration in Chromeleon or on the touch screen.

❖ To enter the eluent concentration on the Chromeleon panel

1. On the Chromeleon 7 ePanel Set or the Chromeleon 6.8 panel tabset, click the **Electrolytics** tab.
2. In the **Setpoint** box under **Eluent Generator / EGC**, enter the concentration of eluent to be generated.
3. If the pump flow is off, turn on the flow, and then click **On** to turn on the power to the EGC.
4. If the CR-TC is off, click **On** to turn on the power to the CR-TC.

❖ To enter the eluent concentration on the touch screen

1. On the Home screen, touch the EGC concentration box.
—or—
On the Menu screen, select **Electrolytics**.
2. On the Electrolytics screen under EGC, touch the **mM** box and enter the concentration on the number keypad.
3. If the pump flow is off, go to the Pump screen and start the flow.
4. On the Electrolytics screen, touch the **On** button to turn on the power to the EGC.
5. If the CR-TC is off, touch the **On** button to turn on the power to the CR-TC.

Operating the Dionex ICS-4000 Electrochemical Detector

This section describes basic operating information for the Dionex ICS-4000 Electrochemical Detector, including cell operating precautions, detection modes, waveforms, and data collection, display, and reprocessing.

ED Cell Operating Precautions

To maintain good reproducibility of detection results:

- Prepare all eluents with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “[Deionized Water Requirements for IC](#)” on [page xvii](#).
- Avoid contamination of the cell with incompatible eluents.
- Never apply potential to the electrode unless a stream of eluent or water is flowing through the cell.
- Do not allow a pH-Ag/AgCl reference electrode to dry out. Make sure that eluent is pumped continuously through the cell. If the cell will not be used for a period of time, follow the requirements listed in “[ED Cell Storage](#)” on [page 48](#) for short term (less than 2 days) and long term (2 days or more) storage.
- Be careful to keep the polished surface of the ED cell body clean and dry when not in use. The gold, spring-loaded (pogo) contact must also remain clean and dry. If a salt bridge forms, it can cause an electrical short between the working electrode contact and the cell body.
- If the conventional (nondisposable) working electrode becomes discolored or if you notice a degradation in performance (baseline noise, tailing peaks, etc.), polish the electrode as instructed in “[Polishing a Conventional Working Electrode](#)” on [page 164](#).
- Over the lifetime of the conventional working electrode, the surface may gradually become pitted or receded. Receded electrodes can be repaired by sanding with 600 grit sandpaper. Continue sanding until the metal surface is again flush with the Kel-F electrode block surface. Then, polish the electrode (see “[Polishing a Conventional Working Electrode](#)” on [page 164](#)).
- To help determine when the pH-Ag/AgCl reference electrode needs regenerating or replacing, monitor the pH value displayed on the detector panel in Chromeleon (see [page 51](#)).
- To have an alarm displayed in the audit trail if the pH exceeds certain values, set pH limits in Chromeleon (see [page 52](#)).

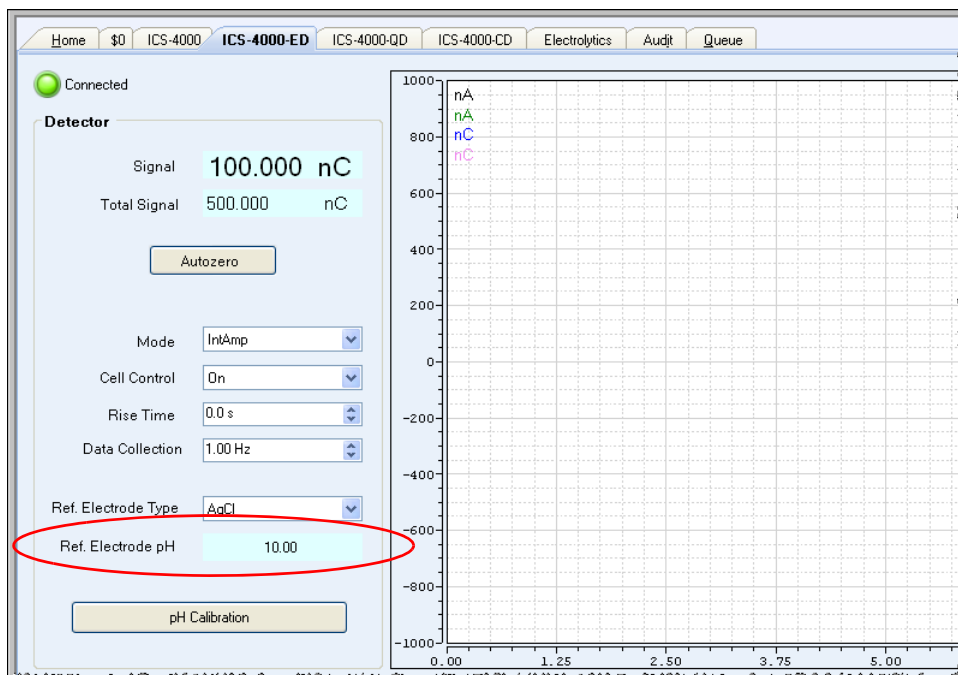
❖ To monitor the pH-Ag/AgCl reference electrode pH

1. At installation, calibrate the pH electrode (see [page 168](#)).
2. When you run your first chromatographic instrument method or program, note the pH value displayed on the Chromeleon panel (see [Figure 26](#)).

3 Operation

Operating the Dionex ICS-4000 Electrochemical Detector

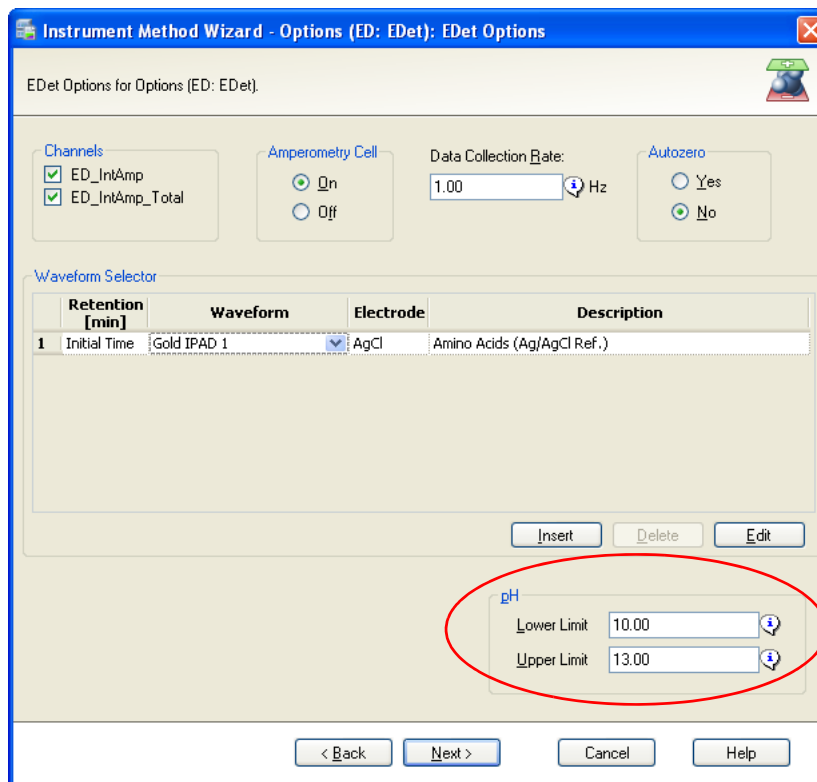
Figure 26. Monitoring the pH-Ag/AgCl Reference Electrode pH



3. Thereafter, monitor the pH value to determine whether there is a shift in the pH. A shift in the pH reading for the same eluent composition indicates a change in the Ag/AgCl reference potential.
4. If the pH value shifts by 0.5 pH unit from the value first observed, check the pH-Ag/AgCl reference electrode (see [“Using a Digital Voltmeter to Determine Reference Potential Shift”](#) on page 119).

❖ To set ED cell pH limits

You can set upper and lower pH limits in the Chromeleon 7 Instrument Method Wizard (see [Figure 27](#)) or Chromeleon 6.8 Program Wizard. The audit trail displays an alarm if the limits are exceeded.

Figure 27. Setting pH limits (Chromeleon 7 instrument method version shown)

Note To disable the alarm, set the upper limit to 14 and the lower limit to 0.

Electrochemical Detection Modes

The Dionex ICS-4000 Electrochemical Detector can perform the following electrochemical detection modes:

- DC amperometry (see [page 53](#))
- Integrated amperometry—includes pulsed amperometric detection (PAD) and integrated pulsed amperometric detection (IPAD) (see [page 56](#))

DC Amperometric Detection

In DC amperometry, a constant potential is applied to the working electrode. For automated control, enter the potential in a Chromeleon 7 instrument method (see [Figure 28](#)) or Chromeleon 6.8 program. For direct control, enter the potential on the ICS-4000 ED panel in Chromeleon or on the touch screen Electrochemical Detector screen (see “[Selecting ED Operating Parameters](#)” on [page 80](#)).

3 Operation

Operating the Dionex ICS-4000 Electrochemical Detector

Figure 28. Instrument Method Wizard page for DC amperometry in Chromeleon 7

ED Options for Options (ED: ED).

Channels: ED_1, ED_1_Total

Amperometry Cell: On, Off

Data Collection Rate: 10 Hz

Autozero: Yes, No

	Retention [min]	Voltage [V]
1	Initial Time	0.0000
2	0.000	0.0010

Buttons: Insert, Delete, < Back, Next >, Cancel, Help

The applied voltage can be changed up to 10 times during the run. The actual number of potential changes allowed depends on the available data storage capacity, which is determined by the length of the run and the data sampling rate.

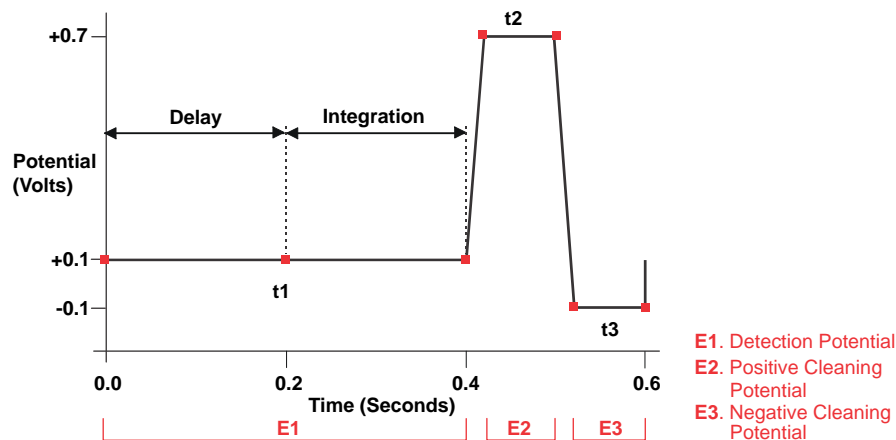
Integrated and Pulsed Amperometric Detection

Integrated and pulsed amperometric detection are similar to DC amperometry in that molecules are oxidized or reduced at the surface of an electrode. However, with these detection modes, a series of potential changes is repeated over time. By repeatedly pulsing between optimized high positive and negative potentials, the electrode surface is continually regenerated. Current is measured by integration during a portion of the repeating potential vs. time waveform. For more information about waveforms, see [page 56](#).

Pulsed Amperometric Detection

In pulsed amperometric detection (PAD), current is integrated at a single constant potential (see [Figure 29](#)).

Figure 29. Example pulsed amperometry waveform

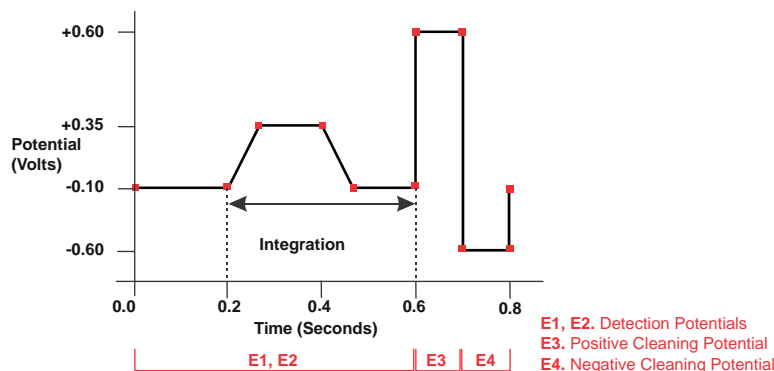


The potentials, labeled E1, E2, and E3, are applied for durations t_1 , t_2 , and t_3 , respectively. At t_1 , the E1 potential is applied. After a delay, the signal is measured by integrating the current for a fixed time. Current integrated for a fixed time is charge and the units are coulombs. At t_2 and t_3 , positive and negative cleaning pulses are added to the waveform. This waveform period repeats until the end of data acquisition or until another waveform is specified.

Integrated Pulsed Amperometric Detection

With integrated pulsed amperometric detection (IPAD), current is integrated at two or more potentials (see [Figure 30](#)).

Figure 30. Example integrated pulsed amperometry waveform



3 Operation

Operating the Dionex ICS-4000 Electrochemical Detector

With the example waveform shown in [Figure 30](#), the current is integrated both while the potential is swept across the metal oxide formation wave and also during the reverse sweep across the oxide reduction wave. This technique minimizes baseline shift and the peak dips that can occur when an eluting analyte's effect on oxide suppression is greater than the detector response from the analyte.

As with pulsed amperometric detection, the waveform period repeats until the end of data acquisition or until another waveform is specified.

Waveforms

A waveform is a series of steps, defined as points on a plot of potential vs. time. Waveforms must be defined for the integrated amperometry modes.

Integrated Amperometry Waveforms

Integrated amperometry waveforms have the following characteristics:

- The duration of one waveform period can be between 0.05 and 2.0 s, with a step resolution of 10 ms. A waveform can have no more than 100 steps.
- The maximum waveform period is 2.0 s. However, because only one data point is generated per waveform period, the effective maximum length of a waveform period depends on the data collection rate (the rate at which Chromeleon collects digital data points from the detector). The relationship is as follows:

$$\text{Data Collection Rate} = 1/\text{Waveform Period}$$

- Multiple waveforms can be defined for a single run, provided they all have the same cycle duration. Up to 15 waveform changes per run are allowed.
- Each waveform can have only one integration interval.
- The integration interval generates one integrated data point per waveform.

Pre-programmed integrated amperometry waveforms designed for quantitative analysis of specific compounds (alcohols, amino acids, carbohydrates, etc.) can be included in a Chromeleon 7 instrument method or a Chromeleon 6.8 program. The waveforms can also be selected on the touch screen Electrochemical Detector screen (see [“Selecting ED Operating Parameters”](#) on [page 80](#)). In the Chromeleon Waveform Editor, you can modify a pre-programmed waveform or define a new waveform.

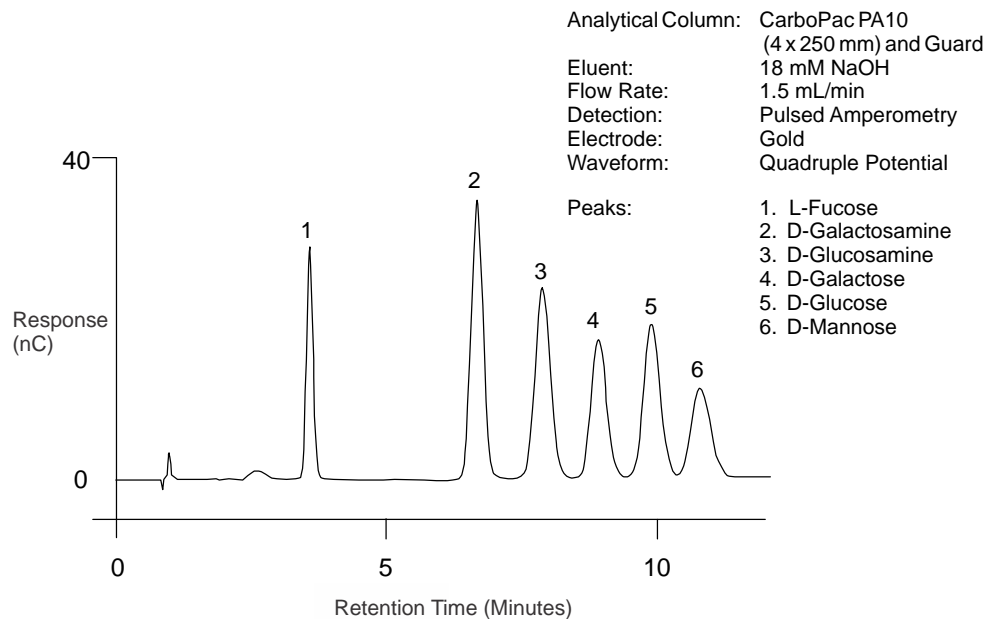
Waveforms for PdH Reference Electrodes in Chromeleon

When you create or edit an instrument method in Chromeleon 7 or program in Chromeleon 6.8, you can select a waveform created for use with the PdH reference electrode. To use a waveform that was created for a pH-Ag/AgCl electrode, select the waveform and then manually correct the potential values in the Waveform Editor. The formula for correction is provided in the Chromeleon Waveform Editor.

Collecting and Storing Amperometry Data

Chromeleon provides storage of pulsed amperometry and integrated amperometry data. Chromeleon stores the detector's response at each waveform period's integration interval. One integrated data point per waveform period is stored. This allows you to produce a chromatogram similar to the example shown in [Figure 31](#). The retention time (in minutes) is on the x-axis and the detector response (in nanoCoulombs) is on the y-axis.

Figure 31. Example chromatogram for a pulsed amperometry application



Using Rear Panel Inputs and Outputs

This section includes the following instructions:

- [Connecting the I/O Connectors](#)
- [Assigning TTL Input Functions](#)
- [Controlling Relay and TTL Outputs](#)

3 Operation

Using Rear Panel Inputs and Outputs

Connecting the I/O Connectors

Figure 32. Rear panel I/O connector strips

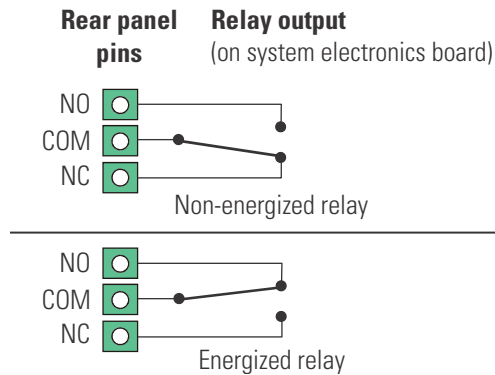
Pin Function	Connector Position	Description
TTL OUT-1	1	TTL output #1 (332 Ω pull up to +5 V, 100 mA sink)
D-GND	2	Digital ground
TTL OUT-2	3	TTL output #2 (332 Ω pull up to +5 V, 100 mA sink)
D-GND	4	Digital ground
TTL IN-1	5	TTL input #1
TTL IN-2	6	TTL input #2
TTL IN-3	7	TTL input #3
TTL IN-4	8	TTL input #4
ANALOG 1	1	16-bit analog output from detector (CD or ED)
A-GND	2	Analog ground
ANALOG 2	3	16-bit analog output from detector (QD)
A-GND	4	Analog ground
+5V	5	+5 V, 200 mA
D-GND	6	Digital ground
RLY-1	NO	Mechanical relay contacts output Connect for either normally open (NO) or normally closed (NC)
	COM	
	NC	
RLY-2	NO	Mechanical relay contacts output Connect for either normally open (NO) or normally closed (NC)
	COM	
	NC	

Note: Relays are capable of switching 2 A at 24 VDC.

Relay Output Details

Depending on which pins are connected, the relay connection can be either normally open (NO) or normally closed (NC) (see [Figure 33](#)).

Figure 33. Relay output configuration



Notes

The relays are capable of switching any low-voltage device. Switched current must be no more than 2 A at 24 VDC.

For a normally open connection, connect a COM pin and an NO pin.

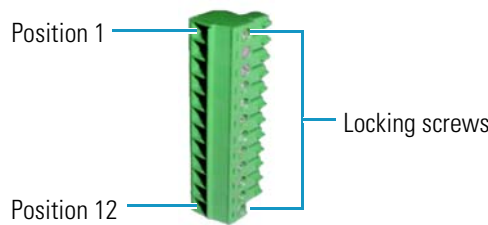
For a normally closed connection, connect a COM pin and an NC pin.

Choose NO or NC based on what you want the state of the connected device to be when the Dionex ICS-4000 power is turned off. A normally open relay is open when the relay is switched off and closed when the relay is turned on. A normally closed relay is closed when the relay is off and is open when the relay is on.

❖ **To connect to the I/O strips**

1. Locate the twisted pair of wires (P/N 043598) and a connector plug (8-position, P/N 924133; 12-position, P/N 923686) (see [Figure 34](#)) provided in the Dionex ICS-4000 Ship Kit (P/N 075161).

Figure 34. 12-position connector plug



2. For each I/O function to be used, connect an active wire (red) and a ground wire (black) to the connector plug at the pin locations for the selected I/O function. Refer to [Figure 32](#) or the label on the rear panel for the connector pin assignments for each I/O function.
3. To attach a wire to the plug, strip the end of the wire, insert it into the plug, and use a screwdriver to tighten the locking screw. If necessary, multiple ground wires can be attached to a single ground pin.



CAUTION When attaching wires to the connector plug, be careful not to allow stray strands of wire to short to an adjoining position on the connector.

4. Plug the connector plug into the corresponding connector strip on the rear panel.

5. Connect the wires from the connector plug to the appropriate connector pins on the other modules. Additional connector plugs are provided with other Dionex modules.

Note Check the polarity of each connection. Connect signal wires to signal (+) pins and ground wires to ground (-) pins.

6. If you connected a TTL input, verify that the correct function is assigned to the input and that the correct input control type is selected. Select different settings if necessary. For instructions, see [“Assigning TTL Input Functions.”](#)

Assigning TTL Input Functions

TTL input functions are assigned on the touch screen TTL/Relay screen or in the Chromeleon configuration.

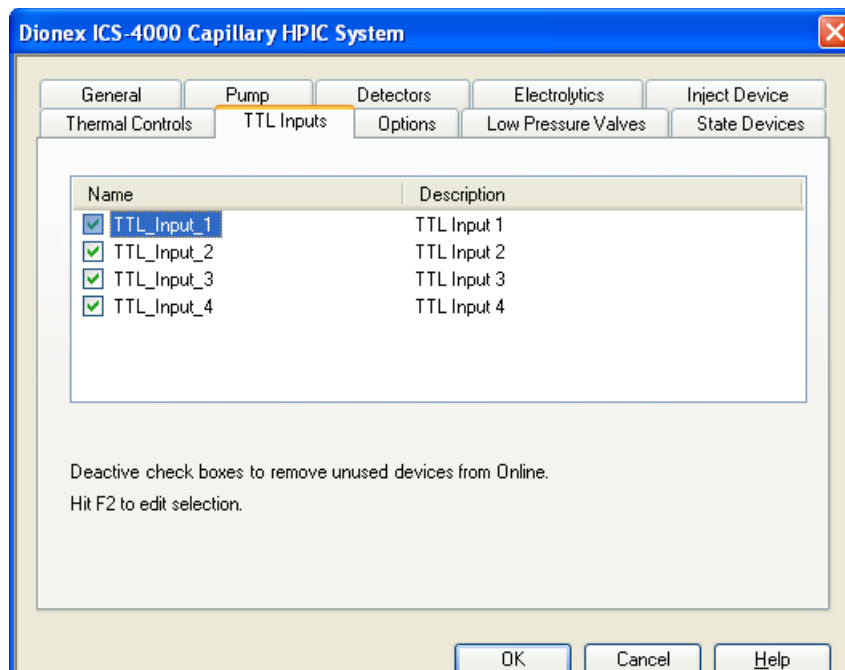
❖ To select TTL input functions from the touch screen

1. Select **TTL/Relay** on the Menu screen.
2. Under **TTL Input**, touch the down arrow next to the TTL input and select a function to be controlled by this input. When connected to a controlling device, the device can send a signal to the input to trigger the selected functions.
3. If the device connected to the TTL input does not send a normal edge signal, select the control type compatible with the device. To determine the correct type, refer to the documentation provided with the controlling device and to [“TTL Input Control Modes.”](#)

❖ To select TTL input functions from Chromeleon

1. Open the Chromeleon 7 Instrument Configuration Manager or Chromeleon 6.8 Server Configuration program.
2. Double-click the Dionex ICS-4000 icon under the instrument or timebase.
3. Select the **TTL Inputs** tab (see [Figure 35](#)).

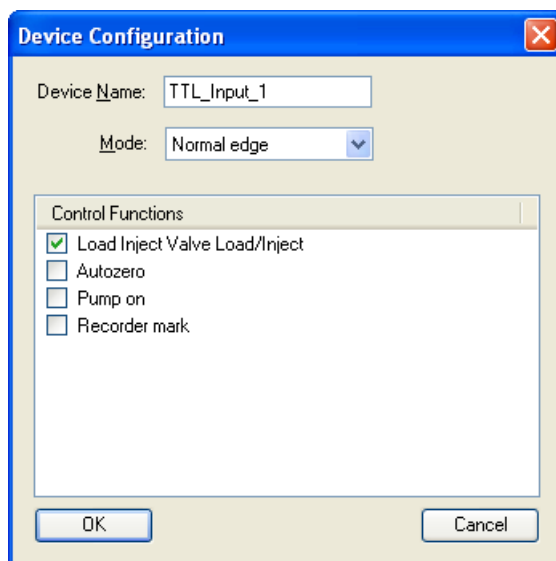
Figure 35. Instrument configuration properties: TTL Inputs



4. Select the name of the input and press the **F2** key (or double-click the name).

The Device Configuration dialog box for the selected input appears (see [Figure 36](#)).

Figure 36. Device Configuration



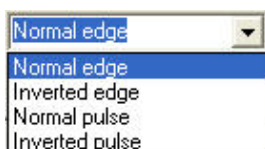
5. In the Control Functions list, select the function to be controlled by this input. When connected to a controlling device, the device can send a signal to the input to trigger the selected functions.

3 Operation

Using Rear Panel Inputs and Outputs

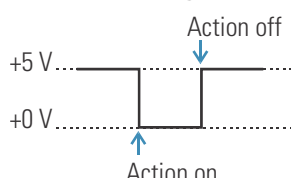
- If the device connected to the TTL input does not send a normal edge signal, select the control type compatible with the device. To determine the correct type, refer to the documentation provided with the controlling device and to “[TTL Input Control Modes.](#)”

TTL Input Control Modes



The TTL inputs respond to four types of signals to accommodate different controlling devices. The default control type, Normal Edge, is compatible with the output signals provided by Dionex modules.

TTL Normal Edge

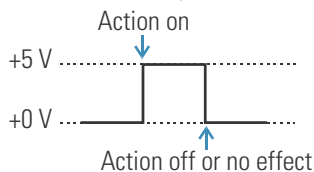


In normal edge operation, the negative (falling) edge of a signal turns on the function.

The action of the positive (rising) edge depends on the function: For on/off or other functions that have two options, the rising edge turns off the function. For functions with only one option, the rising edge has no effect.

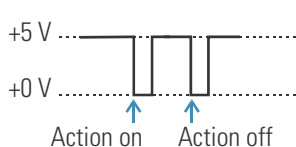
For example, for the injection valve position, the falling edge switches the valve to Load and the rising edge switches the valve to Inject. Similarly, for the pump on/off, the falling edge starts the pump and the rising edge stops it. For the offset and chart mark functions, the falling edge turns on the function and the rising edge has no effect.

TTL Inverted Edge



The inverted edge mode works identically to the normal edge mode except that the positive and negative edges are reversed in function.

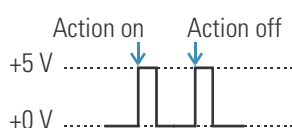
TTL Normal Pulse



In normal pulse operation, the negative (falling) edge of the TTL signal is the active edge and the positive (rising) edge is ignored.

A pulse width of 50 ms or more is guaranteed to be detected. A pulse width of 4 ms or less is guaranteed to be ignored. The action for pulse widths that are greater than 4 ms and less than 50 ms is undefined.

TTL Inverted Pulse



The inverted pulse mode operates identically to the normal pulse mode except that the positive and negative edges are reversed in function.

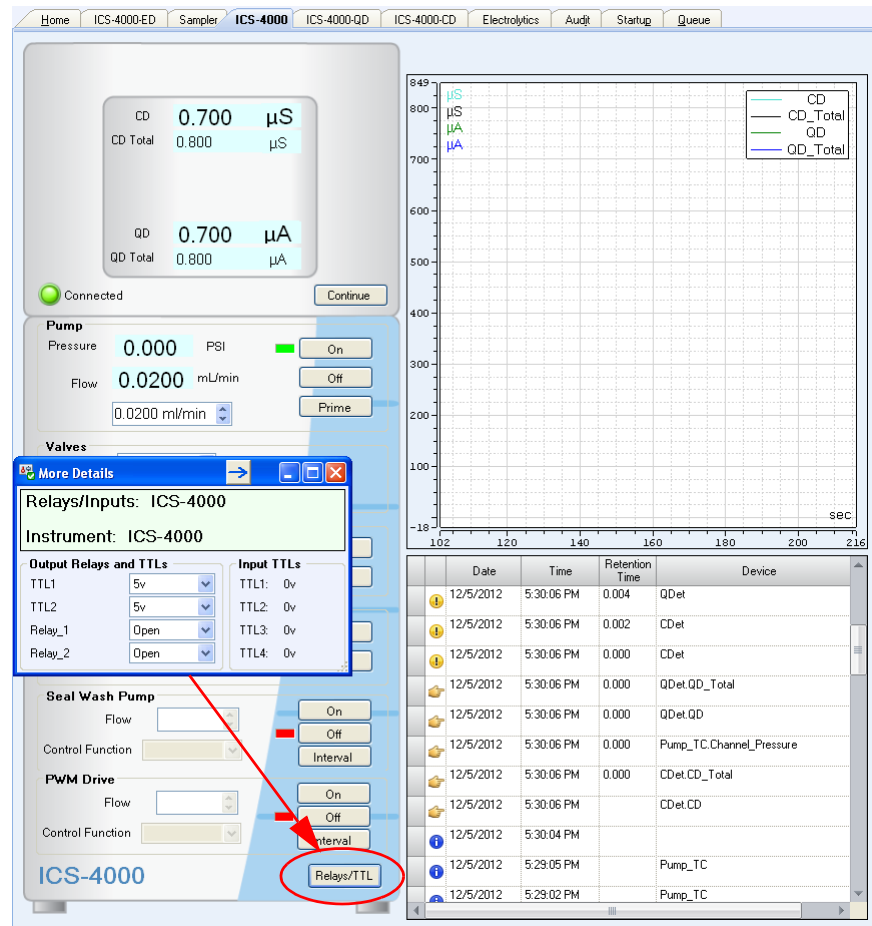
Controlling Relay and TTL Outputs

You can control the relay and TTL outputs from the Chromeleon panel or the touch screen TTL/Relay screen.

❖ To control a relay or TTL output from Chromeleon

1. On the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset, click the **ICS-4000** tab.
2. Click the **Relays/TTL** button (see Figure 37) and select the output relay and TTL settings.

Figure 37. Chromeleon 7 ePanel output relays and TTL control



Note It is possible to change the settings for the relay and TTL outputs while a Chromeleon 7 instrument method or Chromeleon 6.8 program is running.

❖ To control a relay or TTL output from the touch screen

1. Select **TTL/Relay** on the Menu screen.
2. Select the output relay and TTL settings.

Selecting Analog Output Settings

Several settings are available that allow you to configure the analog output signal for your detector and connected device (see [Table 5](#)). You can select the preferred settings on the Chromeleon panel (see [Figure 38](#)) or the touch screen Analog Output screen (see [Figure 39](#)).

Table 5. Analog output configuration settings

Analog Output Setting	Values	Description
Range	Conductivity: 0.01 to 15,000 μS DC Amperometry: 50 pA to 300 μA Integrated Amperometry: 50 pC to 200 μC Charge Detection 2 μA to 1000 μA	Sets the full-scale detector response value. The range to use depends on the detector readings expected for the application. For example, selecting a range of 20 micro Siemens (μS) will limit you to viewing conductivity readings of 20 μS or less.
Recorder calibration (Output)	Zero, Full Scale, Normal	Use this setting to calibrate a recording device. Select Zero to set the output signal to zero volts. Select Full Scale to set the output signal to the selected full-scale voltage (0.01, 0.10, or 1.00 V). For normal operation, select Normal (the default) to output a signal corresponding to the detector output.
Offset level	0 to 100%	Use this setting to adjust the zero position of the analog output when it is plotted. The value entered is a percentage of the full-scale analog output. An offset allows a recording device to plot the signal if it becomes negative. The offset level does not affect the magnitude of the output signal.

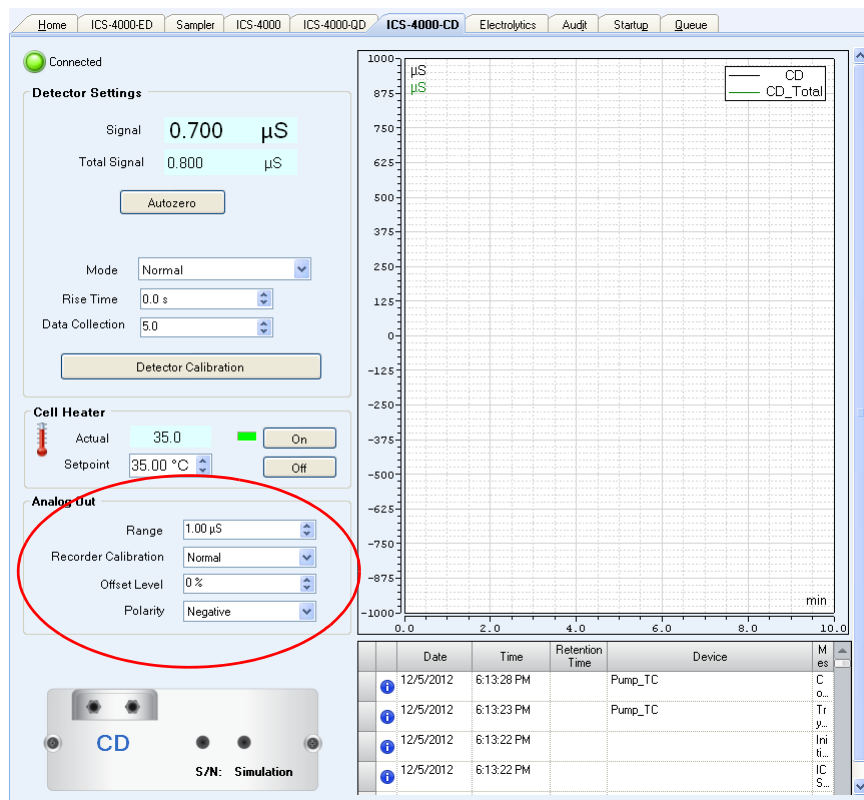
Table 5. Analog output configuration settings, continued

Analog Output Setting	Values	Description
Polarity	Positive, Negative	Use this setting to set the polarity of the analog output signal to either positive (the default) or negative. In applications in which the analyte output is lower than the background signal, the polarity must be negative to display peaks instead of dips on the chromatogram.
Mark	10% of the full-scale analog output	Use this setting to send a positive pulse to the analog output as an event marker. A mark is typically used to indicate a sample injection.

❖ **To select analog output settings from Chromeleon**

1. On the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset, click the detector tab (**ICS-4000 CD, ICS-4000 ED, ICS-4000 QD**).
2. Under Analog Out, select the parameter settings.

Figure 38. Chromeleon 7 ICS-4000 Detector ePanel (CD version shown)



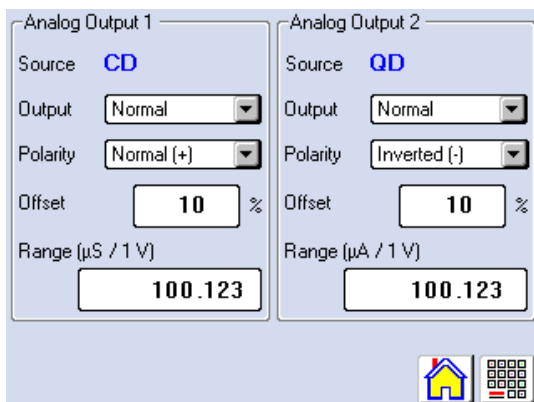
3 Operation

Controlling a Peristaltic Pump or Other Accessory with the PWM Drive

❖ To select analog output settings from the touch screen

1. Disconnect the system from Chromeleon.
2. On the touch screen Menu screen, select **Analog**. The Analog Output screen for the installed detector appears.

Figure 39. Analog Output screen (CD and QD version shown)



Controlling a Peristaltic Pump or Other Accessory with the PWM Drive

The PWM (pulse-width modulation) drive provides control of an accessory device (for example, a peristaltic pump) connected to the PWM Drive connector on the rear panel (see [Figure 18](#)). The accessory must be compatible with the power specifications of the PWM drive (see [Table 6](#)) and must be connected with a correctly configured cable. The optional external peristaltic pump (P/N 082504) is controlled by the PWM drive. A correctly configured cable is included with the pump. The PWM drive can be controlled from Chromeleon or the touch screen Accessory screen.

Table 6. PWM drive power specifications

Specification	Description
Maximum Voltage	+5 VDC <i>or</i> +24 VDC, depending on the connector pin connections (see Table 7)
PWM Drive Voltage Control %	User-controlled setting from 0 to 100%
PWM Drive Delivered Voltage	The voltage delivered to the connected device: PWM Drive Voltage Control % * Maximum Voltage

Table 7. PWM drive connector pin functions

Pin Position	Pin Function	Notes
1	+24 VDC	<ul style="list-style-type: none"> For a +24 VDC maximum voltage device, connect pins 1 and 2.
2	PWM out	
3	+5 VDC	<ul style="list-style-type: none"> For a +5 VDC maximum voltage device, connect pins 2 and 3.
4	Sense	<ul style="list-style-type: none"> Pins 4 and 5 must be connected together to allow detection of the device's presence. Never connect the pump motor or any other device to these pins.
5	Sense	

❖ **To connect an accessory to the PWM drive**

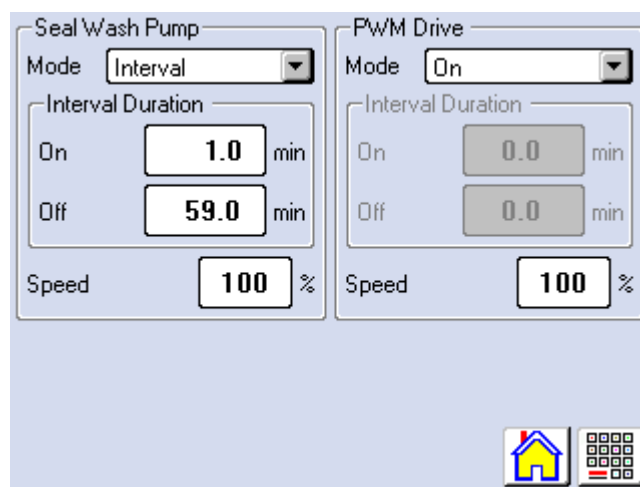
1. Verify that the cable to be used is configured correctly for the maximum voltage the device can sustain (see [Table 7](#)). If you are installing the external peristaltic pump (P/N 082504), the required cable is provided with the pump.
2. Connect the cable to the accessory device and plug the other end into the **PWM DRIVE** connector on the system rear panel.

❖ **To control the connected accessory**

1. Open the Chromeleon 7 ePanel Set or the Chromeleon 6.8 panel tabset and click the **ICS-4000** tab.

—or—

Open the touch screen Accessory screen (see [Figure 40](#)).

Figure 40. Accessory screen

1. Under PWM Drive, select a Mode:
 - **On** turns on the pump; it remain on continuously until turned off.

3 Operation

Controlling a Peristaltic Pump or Other Accessory with the PWM Drive

- **Interval** turns the pump on and off at a fixed interval (for example, 1 minute on, 59 minutes off). To specify the interval, enter values in the On and Off boxes under Interval Duration.
 - **Off** turns off the accessory.
2. To set the speed of the pump, touch the Speed box and enter a percentage. 100% is full speed and 0% is off.

Touch Screen Operation

This chapter describes operating tasks you can perform from the front panel touch screen.

Contents

- Overview of Touch Screen Operation
- Controlling the Pump
- Controlling the Eluent Generator
- Controlling the Suppressor
- Controlling the Injection Valve
- Controlling Temperature Zones
- Selecting Detector Operating Parameters
- Viewing a Plot of the Detector Output
- Setting Analog Output Options
- Controlling TTLs and Relays
- Controlling Optional Valves
- Controlling Accessories
- Selecting Setup Options
- Viewing System Information
- Opening Calibration Screens

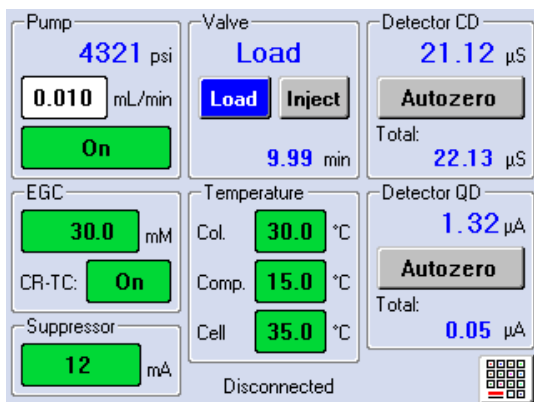
Overview of Touch Screen Operation

The touch screen provides direct control of most Dionex ICS-4000 operating functions. When you select an operating command or parameter from the touch screen, the command or parameter is executed immediately.

Using the Touch Screen

The Home screen (see [Figure 41](#)) provides controls for the most commonly used operating functions.

Figure 41. Home screen example



- To select a control (button, edit box, menu, etc.), touch and release the control with your fingertip. When you lift your finger, the action is taken.

Tip To cancel and not initiate the action, slide your finger away from the control before lifting.


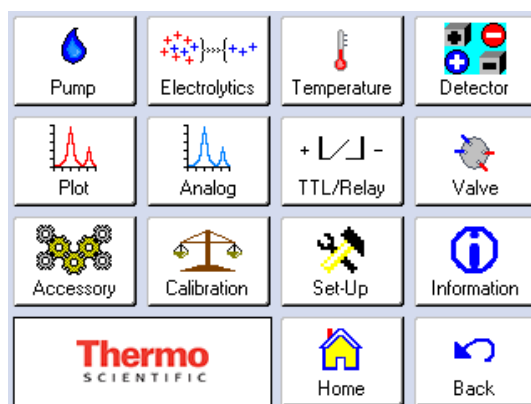


- To access operating functions not available from the Home screen, touch the menu  button. The Menu screen opens (see [Figure 42](#)). Touch the button for the screen you want to view.

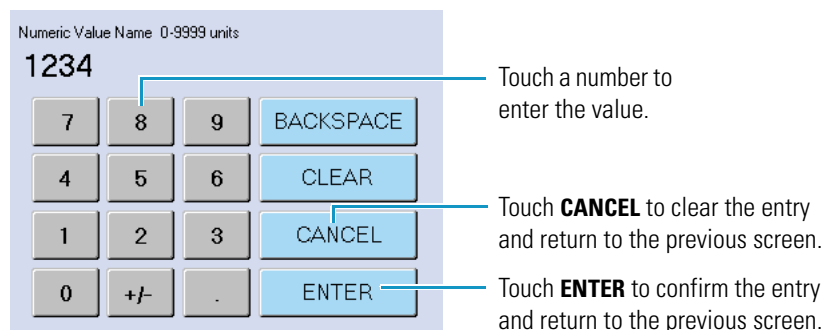
Figure 42. Menu screen



- To return to the Home screen, touch the home  button.
- To return to the previous screen, touch the back  button.

- To change a numerical value (for example, the flow rate), touch the box showing the current value (for example, **0.010** mL/min). A number keypad opens (see Figure 43). Touch the numbers on the keypad and then touch **ENTER**.

Figure 43. Number keypad



Tip The background color on some controls indicates the status of the corresponding device.

- Red means the device is off.
- Amber means the device is on, but not ready for operation.
- Green means the device is on and is ready for operation.

Using the Touch Screen with Chromeleon

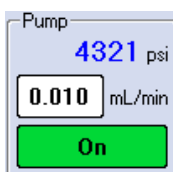
When the Dionex ICS-4000 is connected to Chromeleon, Connected is displayed on the Home screen. In this mode, most of the controls on the touch screens are disabled. This prevents touch screen commands from conflicting with Chromeleon commands.

In general, when the Dionex ICS-4000 is connected to Chromeleon, you can:

- View all operational status information (pressure, flow rate, conductivity readings, etc.)
- Go to any of the touch screens
- View a plot of the detector data from the Plot screen and adjust the plot display using the controls on the screen

To restore touch screen (local) control, disconnect the Dionex ICS-4000 from Chromeleon. To disconnect from Chromeleon, click the **Connected** button on the Chromeleon 7 ePanel or the **Connected** check box on the Chromeleon 6.8 panel tabset.

Controlling the Pump



The Pump controls on the Home screen display the current pump pressure, the flow rate setting, and the status of the pump (**On**, **Off**, **Fast Ramp**, or **Prime**). To change the flow rate, touch the flow rate set point box.

To control other pump functions, go to the Pump screen (see [Figure 44](#)).

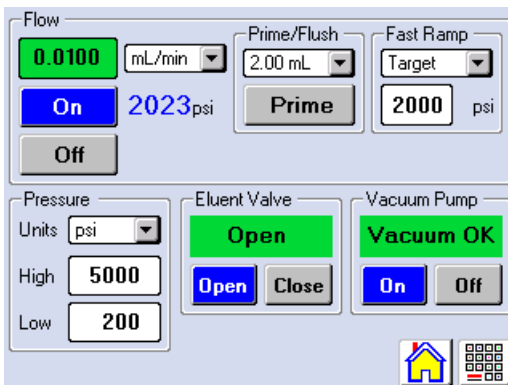
❖ To open the Pump screen

Touch the pump status box on the Home screen.

–or–

Select **Pump** on the Menu screen.

Figure 44. Pump screen example



❖ To set the pump flow rate

On the Home screen or Pump screen, touch the flow rate set point box and enter the flow rate on the number pad.

Capillary IC applications typically use flow rates from 0.01 to 0.02 mL/min. Refer to the column manual for the flow rate to use with your column and application. Column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

❖ To select the flow rate units

On the Pump screen under Flow, touch the units list and select either **mL/min** or **µL/min**.

❖ To start and stop the pump

On the Pump screen under Flow, touch **On** to start the pump at the currently selected flow rate or **Off** to stop the flow.

❖ To prime the pump

1. Open the priming valve on the pressure transducer by turning the knob one-quarter to one-half turn counterclockwise.
2. On the Pump screen under Prime/Flush, touch the flow rate list and select a prime flow rate (**2.00 mL/min**, **1.00 mL/min**, or **0.1 mL/min**).

3. Touch **Prime**.
4. To stop priming, touch **Off**. Close the priming valve.
For priming instructions using Chromeleon, see “[Priming the Pump](#)” on page 41.

Note If you select a flow rate below 0.1 mL/min in the Prime/Flush flow rate list, the button is labeled **Flush**. Use this function when flushing and hydrating consumable devices (for example, a suppressor). This function allows the pump to run below the low pressure limit without triggering a low pressure error.

❖ **To select fast ramp settings**

1. On the Pump screen under Fast Ramp, touch the mode list and select either **Target**, **Step**, or **Off**.
2. If you selected **Target**, touch the pressure box and enter the target pressure on the number pad.
For details about fast ramping, see “[Fast Ramping](#)” on page 15.

❖ **To select the pressure unit**

On the Pump screen under Pressure, touch the Units list and select the preferred unit (**psi**, **bar**, or **MPa**). This sets the pressure unit for all touch screens that display pressure values.

❖ **To set the pump pressure limits**

1. On the Pump screen under Pressure, touch the High box and enter the maximum pressure limit on the number pad. The high limit must be at least 0.7 MPa (100 psi) above the low limit.
2. Touch the Low box and enter the minimum pressure limit on the number pad.
When you configure the Dionex ICS-4000 in Chromeleon, the software sets the maximum and minimum limits for the pump automatically. If you are not using Chromeleon to control the Dionex ICS-4000, set the limits on the Pump screen.
The first time the power to the Dionex ICS-4000 is turned on, the maximum system pressure limit is 41 MPa (6000 psi) and the minimum pressure limit is 0. The eluent generator requires a maximum high limit of 35 MPa (5000 psi) and a minimum low limit of 1.4 MPa (200 psi).

❖ **To control the eluent valve**

On the Pump screen under Eluent Valve, touch **Open** or **Close**. For more information about the valve, see “[Eluent Valve](#)” on page 14.

❖ **To enable and disable vacuum degassing**

On the Pump screen under Vacuum Pump, touch **On** to enable vacuum degassing. Touch **Off** to disable vacuum degassing. For more information about vacuum degassing, see “[Eluent Degasser](#)” on page 14.

Controlling the Eluent Generator



The EGC controls on the Home screen display the eluent concentration currently being generated. If the EGC is not operating, **Off** is displayed and the background of the box is red. The status of the CR-TC (**On** or **Off**) is also displayed. To control the EGC and CR-TC, go to the Electrolytics screen (see Figure v).

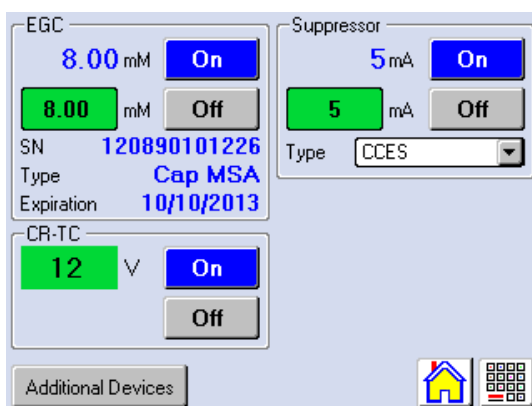
❖ To open the Electrolytics screen

Touch the suppressor status box on the Home screen

–or–

Select **Electrolytics** on the Menu screen.

Figure 45. Electrolytics screen



❖ To set the eluent concentration

On the Electrolytics screen under EGC, touch the **mM** set point box and enter the concentration on the number keypad.

To determine the eluent concentration required for your application, refer to the column manual included on the Thermo Scientific Reference Library DVD (P/N 053891).

❖ To turn the EGC power on or off

On the Electrolytics screen under EGC, touch the **On** or **Off** button. When you turn on the power, the EGC begins generating eluent at the currently selected concentration setting.

Note The EGC power is always turned off when the pump flow is off.

❖ To view EGC information

The Electrolytics screen displays the following information about the EGC:

- Actual concentration of the eluent currently being generated
- Serial number of the installed EGC

- Type of EGC installed
 - Expiration date of the installed EGC. The date is 18 months from the date of installation. An EGC is best if used within two years from the date of manufacture. Although you can continue operation with the cartridge after this date, performance may be impaired until a new cartridge is installed.
- ❖ **To turn the CR-TC power on or off**
On the Electrolytics screen under CR-TC, touch the **On** or **Off** button.

Note The CR-TC power is always turned off when the pump flow is off.

Controlling the Suppressor



The Suppressor control on the Home screen displays the current status of the suppressor. When the suppressor is on, the current (mA) setting is displayed. The background color of the box indicates the suppressor status:

- When the suppressor is off, the background is red.
- When the suppressor is on but the current setting has not yet been reached, the background is amber.
- When the suppressor is on and the current setting has been reached, the background is green.

To control the suppressor, go to the Electrolytics screen (see [Figure v](#)).

❖ **To open the Electrolytics screen**

Touch the suppressor status box on the Home screen

–or–

Select **Electrolytics** on the Menu screen.

❖ **To turn the suppressor current on or off**

On the Electrolytics screen under Suppressor, touch the **On** or **Off** button.

Note The suppressor power is always turned off when the pump flow is off.

❖ **To select the suppressor type**

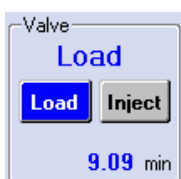
On the Electrolytics screen under Suppressor, touch the suppressor type list and select the type (**None/MMS**, **Dummy**, **ACES**, or **CCES**).

❖ **To set the suppressor current**

On the Electrolytics screen under Suppressor, touch the current set point box and enter a value on the number pad.

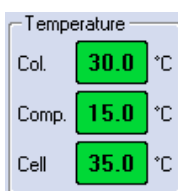
The appropriate suppressor current setting depends on the suppressor type, the column, and other variables. Refer to the suppressor manual for the recommended current setting for your application. The suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

Controlling the Injection Valve



The Valve controls on the Home screen display the current position of the high-pressure injection valve installed in the system and the elapsed time since the last injection. You can also switch the position of this valve from the Home screen (touch **Load** or **Inject**). For details about the injection valve, see [page 20](#).

Controlling Temperature Zones



The Temperature controls on the Home screen display the temperature setting of each controlled temperature zone (column heater, chromatography compartment, conductivity cell). The background color of the temperature box indicates the status of the zone.

- When the temperature zone is off, the background is red.
- When the temperature zone is on and the set temperature has not yet been reached, the background is amber.
- When the temperature zone is on and the set temperature has been reached, the background is green.

For details about the temperature control ranges for the column heater, chromatography compartment, and conductivity cell, see [“Temperature Control Zones”](#) on [page 19](#).

Note The temperature of the column heater must be set to at least 2 °C above the compartment temperature. The temperature of the CD cell heater must be set to at least 7 °C above the compartment temperature.

To set the temperature of a control zone or turn a zone on or off, go to the Temperature screen (see [Figure 46](#)).

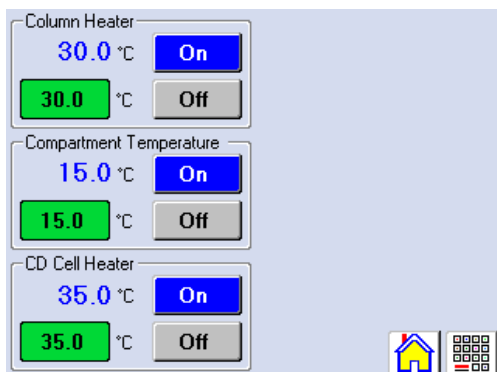
❖ **To open the Temperature screen**

Touch a temperature status box on the Home screen

—or—

Select **Temperature** on the Menu screen.

Figure 46. Temperature screen



❖ **To set the temperature of a temperature zone**

On the Temperature screen, touch the temperature box of the zone you want to set and enter the temperature on the number pad.

❖ **To turn a temperature zone on or off**

To turn on a zone, touch the **On** button of the zone you want to control. The zone begins heating or cooling to the current setting. The background color of the temperature box turns amber while the zone is reaching the temperature setting and green when the setting is reached.

To turn off a zone, touch the **Off** button of the zone. The background color of the temperature box turns red.

Selecting Detector Operating Parameters

The Home screen provides status information about each detector installed in the Dionex ICS-4000. Additional screens for selecting detector operating parameters are available. For a CD, see [page 77](#); for a QD, see [page 79](#); for an ED, see [page 80](#).

Selecting CD Operating Parameters



When a Dionex ICS-4000 Conductivity Detector is installed, the Home screen displays the conductivity of the fluid flowing through the detector (in μS). The reading on top is the offset reading (the conductivity with the background conductivity subtracted). The Total reading is the conductivity with no offset.

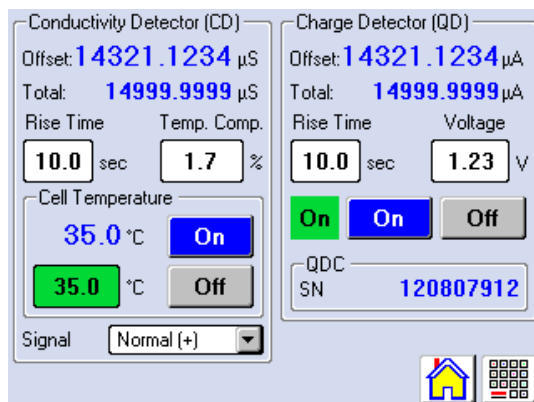
4 Touch Screen Operation

Selecting Detector Operating Parameters

To determine the offset, allow the system to equilibrate after start up. At equilibration, the conductivity reading is the background conductivity (the conductivity of the eluent before sample injection). To offset the background conductivity and zero the top reading, touch **Autozero**.

To set operating parameters for the CD, go to the Detector screen (see [Figure 47](#)). To open the Detector screen, select **Detector** on the Menu screen.

Figure 47. Detector screen (CD and QD detectors installed)



❖ To select the data rise time

On the Detector screen, under Conductivity (CD), touch the Rise Time list and select the preferred rise time.

The data rise time determines the amount of filtering performed on the CD. The rise time is a measure of how quickly the detector responds to a change in signal, and is defined as the time it takes the output signal to rise from 10% of its final value to 90% of its final value. The selected data rise time is used to filter both the digital data output, which is sent to the computer and the Plot screen, and the analog data output. The default rise time is 2 seconds. Choosing an appropriate rise time value can optimize performance by keeping the signal-to-noise ratio at a minimum level. A longer rise time allows averaging of the noise frequencies, and subsequently, the baseline will contain much less short-term noise. However, longer rise times may have the following effects on peaks:

- Peak shape will become asymmetric.
- The peak maximum will be shifted.
- The peak height will be reduced.

The rise time should be approximately 25% of the peak width at one-half the height of the narrowest peak of interest.

For example, for a peak width of 5 seconds, calculate the rise time as: (5 sec) 25% = 1.25 sec. Because 1.25 sec is not one of the available settings for rise time, select the next fastest rise time, 1 sec.

❖ **To change the temperature compensation for the CD**

On the Detector screen, under Cell Temperature, touch the **Temp. Comp.** box and enter the temperature compensation on the number pad.

The CD has built-in temperature compensation that helps minimize changes in the baseline or in peak heights if the operating temperature is different from the temperature at which the cell was calibrated. The default temperature compensation is 1.7% per °C. This can be reset to between 0% and 3.0% per °C, depending on the eluent. If you notice that the baseline shifts up when the temperature increases, the temperature compensation is too low and should be reset to a higher value.

❖ **To control the cell temperature**

- To set the cell temperature, touch the temperature box on the Detector screen under Cell Temperature, and enter the temperature on the number pad.
- To turn the cell temperature on or off, touch the **On** or **Off** button, respectively.

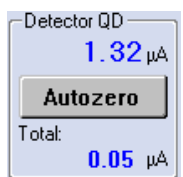
You can also set the temperature and control the cell on the Temperature screen (see “Controlling Temperature Zones” on page 76).

❖ **To change the polarity of the conductivity data**

On the Detector screen under Signal, touch **Normal (+)** or **Inverted (-)**.

Note In applications in which the analyte output is lower than the background conductance, the polarity must be inverted to have peaks instead of dips on the chromatogram.

Selecting QD Operating Parameters



When a Dionex ICS-4000 Charge Detector is installed, the Home screen displays the current of the fluid flowing through the detector (in μA). The reading on top is the offset reading (the current with the background current subtracted). The Total reading is the current without subtracting the background.

To determine the offset, allow the system to equilibrate after start up. At equilibration, the current reading is the background current (the current of the eluent before sample injection). To offset the background and zero the top reading, touch **Autozero**.

To set operating parameters for the QD, go to the Detector screen (see Figure 47). To open the Detector screen, select **Detector** on the Menu screen.

❖ **To select the data rise time**

On the Detector screen, under Charge Detector (QD), touch the **Rise Time** list and select the preferred rise time. For details about rise time, see page 78.

4 Touch Screen Operation

Selecting Detector Operating Parameters

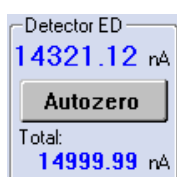
❖ To turn the Dionex ODC 300 (Capillary) on or off

On the Detector screen, under Charge Detector (QD), touch **On** or **Off**.

❖ To set the voltage

On the Detector screen, under Charge Detector (QD), touch the Voltage box and enter the voltage on the number keypad. The default value is 6.0 V.

Selecting ED Operating Parameters



When a Dionex ICS-4000 Electrochemical Detector is installed, the Home screen displays the current of the fluid flowing through the detector (in nC for integrated amperometry mode and nA for DC amperometry mode). The reading on top is the offset reading (the current with the background current subtracted). The Total reading is the current without subtracting the background.

To determine the offset, allow the system to equilibrate after startup. At equilibration, the current reading is the background current (the current of the eluent before sample injection). To offset this background and zero the top reading, touch **Autozero**.

To set other ED operating parameters, go to the Detector screen (see [Figure 48](#) and [Figure 49](#)). To open the Detector screen, select **Detector** on the Menu screen.

Figure 48. ED screen (integrated amperometry mode)

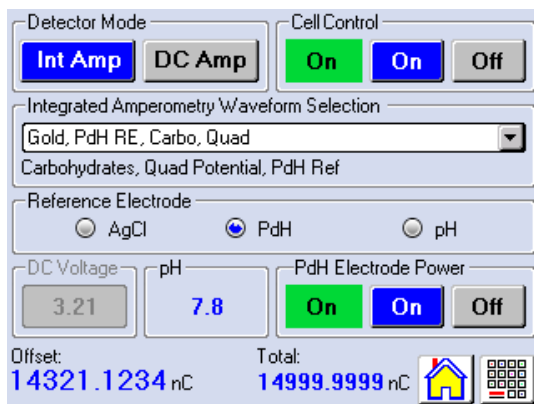
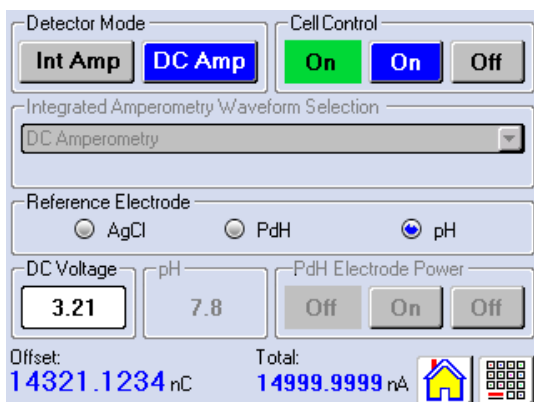


Figure 49. ED screen (DC amperometry mode)



❖ **To select the amperometry mode**

On the ED screen, under Detector Mode, touch either **Int Amp** or **DC Amp**.

❖ **To turn the cell on or off**

On the ED screen, under Cell Control, touch either **On** or **Off**.

For integrated amperometry: When you turn on the cell, the selected waveform starts running.

For DC amperometry: When you turn on the cell, the selected voltage is applied constantly to the working electrode.

❖ **To select the waveform for integrated amperometry**

On the ED screen, touch the **Waveform Selection** list, and select the pre-defined waveform that you want.

–or–

Select **Set from Chromeleon** to run the waveform that was last downloaded from Chromeleon.

❖ **To select the reference electrode**

On the ED screen, under Reference Electrode, touch either **AgCl**, **PdH**, or **pH**.

Two reference electrode types are available for the ED:

- Combination pH-Ag/AgCl reference electrode
- Palladium hydrogen (PdH) reference electrode

The combination pH-Ag/AgCl reference electrode has two modes: AgCl or pH.

❖ **To set the DC voltage**

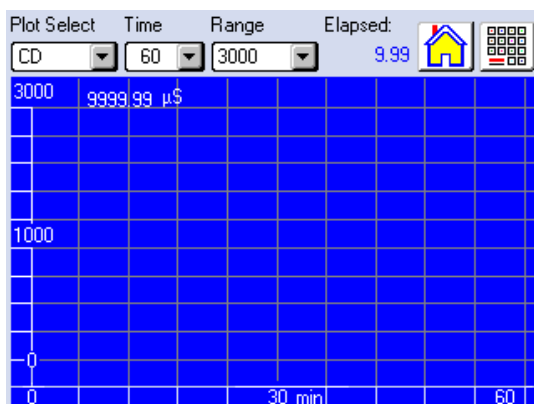
On the ED screen, touch the **DC Voltage** box and enter the voltage on the number keypad. If the cell voltage is on, the selected DC voltage is applied.

Viewing a Plot of the Detector Output

The Plot screen (see [Figure 50](#)) provides a real-time plot of the detector output. The screen can display up to 60 minutes of data. Data older than 60 minutes is discarded. You can view the plot and adjust the display when the Dionex ICS-4000 is under either local or computer control. The plot screen also displays the elapsed time (in minutes) since injection. If more than one detector is installed, you can switch between the different plots.

To open the screen, select **Plot** on the Menu screen.

Figure 50. Plot screen



❖ **To select the detector plot to view**

If a CD and a QD are installed, the plot shows the CD total data by default. To view the QD plot, touch the Plot Select list and select QD.

❖ **To set the length of time plotted (horizontal scale)**

On the Plot screen, touch the Time list and select the number of minutes to display on the plot.

❖ **To set the signal range**

On the Plot screen, touch the Range list and select the range to display on the plot.

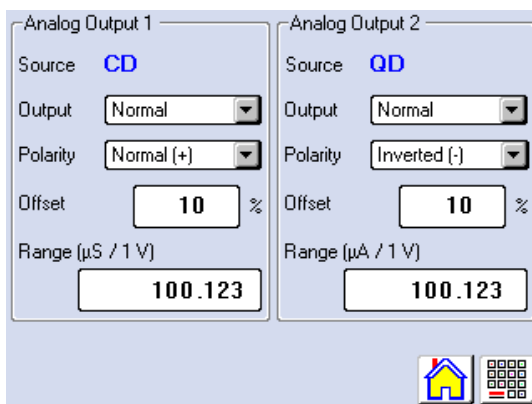
The Range sets the highest detector reading displayed on the plot (the vertical scale). If the tops of peaks are cut off, select a higher range. If peaks are short, select a lower range. To have the scaling of the signal plot automatically adjusted to the height of the signal, select **Auto**.

Setting Analog Output Options

Two analog output connectors are provided on the Dionex ICS-4000 rear panel. Each analog output supplies a voltage signal proportional to the current measured by a detector cell. The outputs can be connected to an analog-to-digital (A/D) converter, such as an integrator or other recording device.

The Analog Output screen (see Figure 51) provides options for configuring the analog outputs for your connected recording device and application. To open the screen, select **Analog** on the Menu screen.

Figure 51. Analog Output screen (CD and QD version shown)



❖ **To select the analog output signal type**

On the Analog screen, touch the Output list and select **Normal**, **Zero**, or **Full**.

Select **Normal** to output a signal corresponding to the offset reading from the detector and the other selected Analog parameters. Use the other two settings to calibrate an analog-to-digital converter device. Select **Zero** to set the output signal to zero volts. Select **Full** to set the output signal to a full-scale response.

❖ **To select the analog output polarity**

On the Analog screen, touch the Polarity list and select **Normal(+)** or **Inverted (-)**.

The polarity of the analog output signal can be set to either positive (the default) or negative. In applications in which the analyte output is lower than the background signal, the polarity must be negative to display peaks instead of dips on the chromatogram.

❖ **To enter the analog output offset**

On the Analog screen, touch the **Offset %** box and enter the percentage offset on the number keypad.

The offset adjusts the zero position of the analog output when it is plotted. The value entered is a percentage of the full-scale analog output. An offset allows a recording device to plot the signal if it becomes negative, but does not affect the magnitude of the output signal.

❖ **To enter the analog output range**

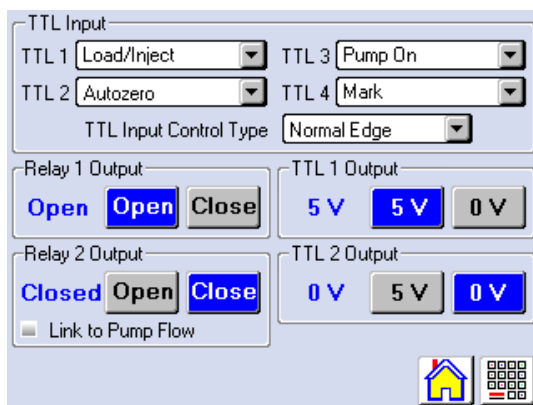
On the Analog screen, touch the Range box and enter the range of a full-scale detector response. The range to use depends on the detector readings expected for the application. For example, if you expect detector readings for your conductivity detection application to be less than 50 μS , enter 50.

Controlling TTLs and Relays

The TTL/Relay screen (see [Figure 52](#)) provides control of the TTL inputs and relay and TTL outputs.

To open the screen, select **TTL/Relay** on the Menu screen.

Figure 52. TTL/Relay screen



For TTL and relay connection instructions, see “[Connecting the I/O Connectors](#)” on [page 58](#).

❖ **To assign a function to a TTL input**

On the TTL/Relay screen under TTL Input, touch the TTL function list and select a function:

- **Load/Inject** switches the injection valve position.
- **Autozero** offsets the background signal.
- **Pump On** turns the pump on and off.
- **Mark** sends a chart mark signal to the analog output. The mark is 10% of the full-scale voltage, and the duration is 0.5 seconds. A mark can be used, for example, to indicate when the injection occurred.

❖ **To select a TTL input control type**

On the TTL/Relay screen under TTL Input, touch the TTL Input Control Type list and select a type (**Normal Edge**, **Inverted Edge**, **Normal Pulse**, or **Inverted Pulse**).

The TTL inputs respond to four types of signals to accommodate different controlling devices. The default control type, Normal Edge, is compatible with the output signals provided by Thermo Scientific Dionex modules.

If the controlling device connected to the input does not send a normal edge signal, select the appropriate control type. To select the correct type, refer to the documentation provided with the controlling device and to the information in “[TTL Input Control Modes](#)” on [page 62](#).

❖ **To control a relay output**

On the TTL/Relay screen, under the relay output you want to control, touch **Open** or **Close**.

The relay outputs can be connected for either normally open or normally closed. For connection instructions, see “[Connecting the I/O Connectors](#)” on [page 58](#).

To link Relay 2 to the pump flow, select the **Link to Pump Flow** check box. When this check box is selected, Relay 2 closes automatically when the pump motor is on and the flow is greater than 0.

❖ **To control a TTL output**

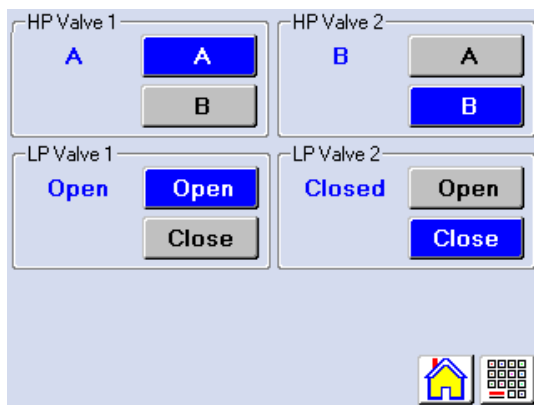
On the TTL/Relay screen, under the TTL output you want to control, touch **5 V** or **0 V**.

The TTL outputs are normally at 5 volts. Setting a TTL output to 0 volts turns on the function in the connected device.

Controlling Optional Valves

To control optional high-pressure and low-pressure valves installed in the system, touch **Valve** on the Menu screen to go to the Valve screen (see [Figure 53](#)).

Figure 53. Valve screen



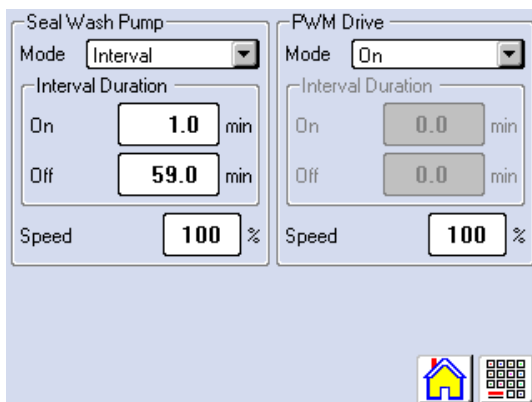
For details about each valve type, see:

- “[High-Pressure Valves](#)” on [page 33](#)
- “[Low-Pressure Valves](#)” on [page 34](#)

Controlling Accessories

Use the Accessory screen to control the optional seal wash pump or a device connected to the PWM drive (for example, an external peristaltic pump). To open the screen, select **Accessory** on the Menu screen.

Figure 54. Accessory screen



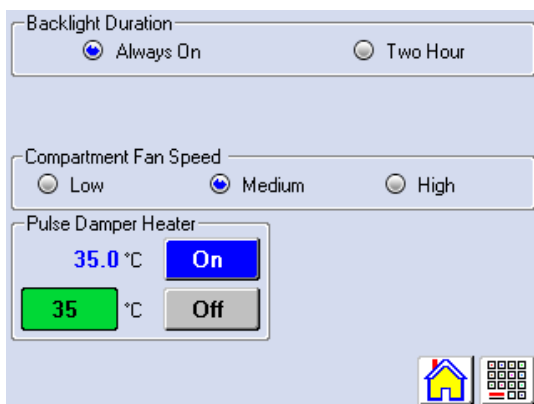
❖ To control the seal wash pump or PWM drive

1. Under Seal Wash Pump or PWM Drive, select the Mode:
 - **On** turns on the pump or drive; it remains on continuously until turned off.
 - **Interval** turns the pump or drive on and off at a fixed interval (for example, 1 minute on, 60 minutes off). To specify the interval, enter values in the On and Off boxes under Interval Duration.
 - **Off** turns off the accessory.
2. To set the speed, touch the **Speed** box and enter a percentage. 100% is full speed and 0% is off.

Selecting Setup Options

Use the Setup screen to select general options for the system. To open the screen, select **Setup** on the Menu screen.

Figure 55. Setup screen



❖ To select the backlight duration

On the Set-Up screen under Backlight Duration, select an option:

- **Always On** sets the backlight to remain on continuously. Selecting this option will shorten the lifetime of the backlight.
- **Two Hours** sets the backlight to turn off after two hours if the touch screen is not used during that time. This is the default option.

❖ To select the chromatography compartment fan speed

On the Set-Up screen, under Compartment Fan Speed, select **Low**, **Medium**, or **High**.

❖ To control the pulse damper heater

To set the temperature, touch the temperature box under Pulse Damper Heater and enter a temperature on the number keypad.

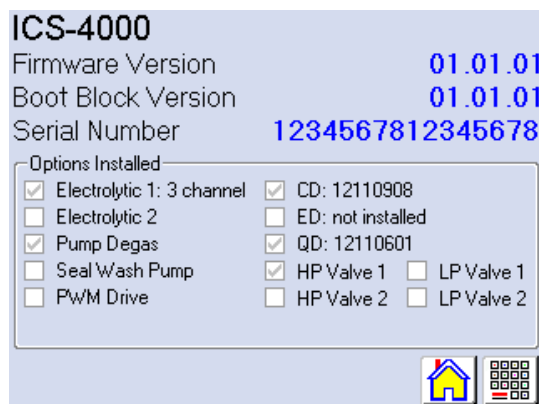
To turn the heater on or off, select the **On** or **Off** button.

Under typical operating conditions, the pulse damper should be on and set to the default temperature (37 °C). If ambient temperature in the laboratory rises above 30 °C, increase the pulse damper heater temperature to ensure that the temperature of the pulse damper remains above ambient and does not fluctuate.

Viewing System Information

The Information screen (see [Figure 56](#)) displays version and configuration information about the Dionex ICS-4000. To open this screen, touch **Information** on the Menu screen.

Figure 56. Information screen



The Information screen displays the following information:

- **Firmware Version:** The firmware program controls instrument functions, sends status and data to Chromeleon, and receives control parameters from Chromeleon.
- **Boot Block Version:** The boot block program executes when the Dionex ICS-4000 power is turned on. The boot block program starts the firmware.
- **Serial Number:** The serial number of the Dionex ICS-4000.
- **Options Installed:** Checked boxes indicate the options installed in the Dionex ICS-4000.

Opening Calibration Screens

To open a calibration screen, select **Calibration** on the Menu screen and select either **Conductivity Cell** or **pH Reference Electrode**, depending on the type of detector installed.

Calibration can be performed using the touch screen or the Chromeleon Wellness Panel. For instructions, see [“To calibrate the offset”](#) on [page 155](#) or [“To calibrate the pH-Ag/AgCl reference electrode”](#) on [page 168](#).

Note The Service button on the Calibration screen provides access to calibration functions that are performed only by Thermo Fisher Scientific personnel. An access code is required to go to the service screens.

Maintenance

This chapter describes routine maintenance procedures for the Dionex ICS-4000 Capillary HPIC System that users may perform. All other maintenance procedures must be performed by Thermo Fisher Scientific personnel.

Daily Maintenance

- Check for leaks and tighten or replace any leaking fittings.
- Wipe up spills.
- Rinse dried eluent off of components with deionized water. Dry the leak sensor thoroughly; if the sensor is not dry, it will remain activated and continue to report a leak to the Chromeleon audit trail.

Weekly Maintenance

- Check fluid lines for crimping or discoloration. Replace any pinched or damaged lines.
- Check the liquid level in the eluent reservoir and refill as required.
- Check the waste container and empty when needed.
- Check the end-line filter (P/N 045987) and change if needed. When end-line filters are new, they are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent reservoir does not contain solvent.

Note It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Periodic Maintenance

Clean the eluent reservoir every 3 months, as instructed below.

❖ To clean a reservoir

1. Dispose of any remaining chemicals according to municipal regulations.
2. Rinse the reservoir (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications in that meets the specifications in “Deionized Water Requirements for IC” on page xvii.
3. Rinse the inside of the reservoir with isopropyl alcohol or methanol.
4. If algae or bacteria have left a slimy film on the reservoir, use an algicide or disinfectant (dilute hydrogen peroxide, etc.).
5. Rinse cleaning chemicals out of the reservoir with ASTM Type I (18 megohm-cm) filtered and deionized water.
6. Dry the reservoir with clean, particulate-free air.

Annual Maintenance

Thermo Fisher Scientific recommends performing preventive maintenance annually, as well as before scheduled Performance Qualification tests. A Dionex ICS-4000 Preventive Maintenance Kit (P/N 075156) is available for this purpose.

Troubleshooting

This chapter is a guide to troubleshooting issues that may arise during operation of the Dionex ICS-4000 Capillary HPIC System.

If you are unable to resolve a problem by following the instructions here, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

Contents




- [Error Messages](#)
- [Troubleshooting Error Messages](#)
- [No Communication with Chromeleon](#)
- [Liquid Leaks/Leak Alarm](#)
- [Low System Backpressure](#)
- [High System Backpressure](#)
- [Pump Does Not Start](#)
- [Pump Stops](#)
- [Degasser Low Vacuum](#)
- [Degas Vacuum Pump Does Not Run](#)
- [EG Stops Operation](#)
- [Noisy Baseline](#)
- [High Background](#)
- [Low Detector Output](#)
- [No Peaks](#)
- [Tailing Peaks](#)
- [Poor Retention Time Reproducibility](#)
- [Peak Retention Times Are Too Early](#)
- [Peak Retention Times Are Too Late](#)
- [ED Cell Troubleshooting](#)

Error Messages

The instrument control firmware installed in each Dionex ICS-4000 periodically checks the status of certain parameters. If a problem is detected, it is displayed on the touch screen.

Error messages are also reported to Chromeleon and logged in the audit trail. Each error message displayed in the Chromeleon audit trail is preceded by an icon that identifies the severity of the underlying problem (see [Table 8](#)).

Table 8. Chromeleon audit trail severity levels

Icon	Default Severity Level	Description
	Warning	A message is displayed in the audit trail, but the current run is not interrupted.
	Error	A message is displayed in the audit trail, and the system attempts to correct the problem (sometimes by using an alternative parameter), but the current run is not interrupted. If an error occurs during the Ready Check, the queue or batch will not be started until the error is resolved.
	Abort	A message is displayed in the audit trail, and the running queue or batch is aborted.

The table below lists the most frequently observed Dionex ICS-4000 audit trail error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

Table 9. Audit trail error messages

Message	Severity	See
CD cell option disconnected	Abort	page 93
Cell heater exceeds safe temperature	Abort	page 94
Column heater exceeds safe temperature	Abort	page 94
Compartment Peltier exceeds safe temperature	Abort	page 94
CR-TC over current	Abort	page 94
CR-TC short circuit	Abort	page 95
CR-TC stopped for EG off	Warning	page 95
CR-TC stopped for zero flow	Warning	page 96
ED cell current exceeds limits	Warning	page 96
ED cell turned of due to zero flow	Abort	page 96
ED cell reference electrode disconnected	Abort	page 97
ED cell working electrode disconnected	Abort	page 97
ED pH offset calibration failed	Warning	page 97
ED pH slope calibration failed	Warning	page 98

Table 9. Audit trail error messages, continued

Message	Severity	See
EGC cartridge disconnected	Abort	page 98
EGC activation date not valid	Abort	page 98
EGC invalid concentration	Abort	page 99
EGC invalid flow rate	Abort	page 99
EGC invalid serial number	Abort	page 99
EGC ion count not valid	Abort	page 100
EGC over-current	Abort	page 100
EGC over-power	Abort	page 100
EGC over-voltage	Abort	page 101
EGC wrong cartridge error	Abort	page 101
High pressure valve 1 error	Abort	page 101
Leak detected	Warning	page 101
Load/Inject valve error	Abort	page 102
Pump pressure over limit	Abort	page 102
Pump pressure under limit	Abort	page 103
QDC off for zero flow	Abort	page 103
QD cell option disconnected	Abort	page 103
Suppressor open circuit	Abort	page 104
Suppressor over-current	Abort	page 104
Suppressor over-power	Abort	page 104
Suppressor over-voltage	Abort	page 104
Suppressor stopped due to flow rate	Warning	page 105
Suppressor type wrong	Warning	page 105

Troubleshooting Error Messages

 **CD cell option disconnected.**

❖ **To troubleshoot**

1. Check the detector connection. An electronics connector on the back of the detector plugs into a receptacle on the chromatography compartment. Push on the detector to make certain the connector is securely connected and then tighten the thumbscrews.
2. If the error appears again, contact Technical Support for Dionex products for assistance. The detector electronics may have malfunctioned.

 **Cell heater exceeds safe temperature.**

This error occurs when the temperature of the conductivity detector is higher than the maximum allowed. This error may occur if the system is operating in an environment in which the temperature is greater than 40 °C.

❖ **To troubleshoot**

1. For environmental specifications, refer to [Appendix A, “Specifications.”](#)
2. Verify that the chromatography compartment temperature setting is at least 5 °C less than the CD cell temperature setting.
3. Check the suppressor current setting. Running the suppressor at a higher current than is recommended for the application can cause the CD cell to heat up.

 **Column heater exceeds safe temperature.**

This error occurs when the temperature of the column heater exceeds the maximum allowed. This error may occur if the system is operating in an environment in which the temperature is greater than 40 °C.

❖ **To troubleshoot**

For environmental specifications, refer to [Appendix A, “Specifications.”](#)

 **Compartment exceeds safe temperature.**

This error occurs when the temperature of the chromatography compartment is higher than the maximum allowed. This error may occur if the system is operating in an environment in which the temperature is greater than 40 °C.

❖ **To troubleshoot**

For environmental specifications, refer to [Appendix A, “Specifications.”](#)

 **Compartment temperature calibration error.**

❖ **To troubleshoot**

Repeat the calibration procedure. Follow the instructions provided in the DC Temperature Calibration Kit (P/N 063782). If the error appears again, contact Technical Support for Dionex products for assistance.

 **CR-TC over current.**

This error occurs when the current applied to the CR-TC exceeds the maximum current allowed. (The CR-TC current is automatically turned off to prevent damage to the CR-TC.) This error may also occur if liquid flow to the CR-TC is interrupted.

❖ **To troubleshoot**

1. Make sure the CR-TC is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.
2. To determine why liquid flow stopped, refer to “[Pump Stops](#)” on [page 108](#).
3. If the error appears again, contact Technical Support for Dionex products for assistance. The CR-TC control electronics may have malfunctioned.

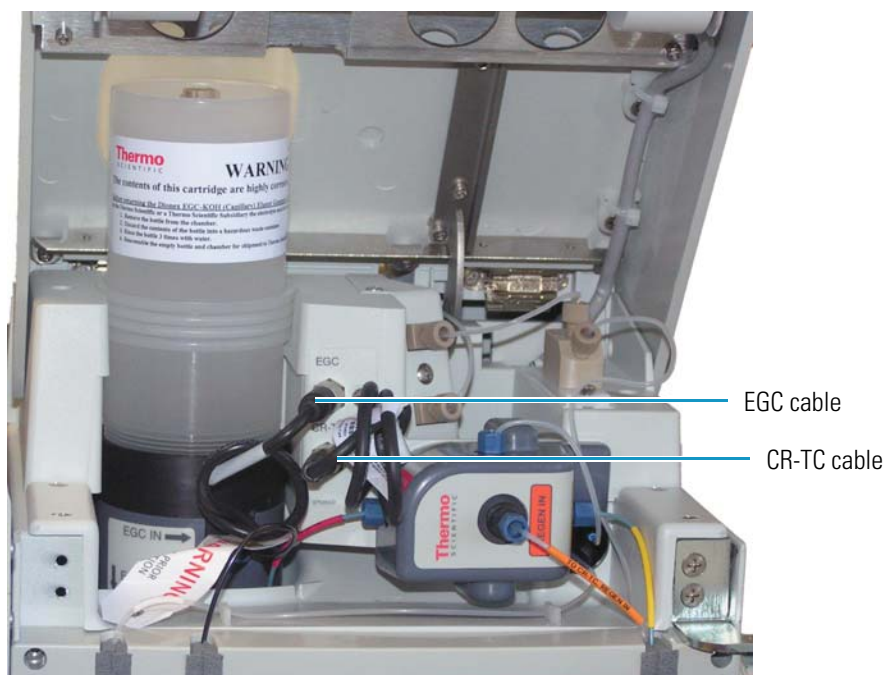
Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

 **CR-TC short circuit.**

❖ **To troubleshoot**

1. Make sure the CR-TC is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.

Figure 57. EGC and CR-TC installed on top tray of Dionex ICS-4000



2. If the error appears again, the CR-TC may be faulty. Replace the CR-TC (see “[Replacing a CR-TC](#)” on [page 144](#)).

 **CR-TC stopped because EG is off.**

This error occurs if you attempt to turn on the CR-TC when the EG is turned off. The CR-TC current is automatically turned off to prevent damage to the CR-TC.

❖ **To troubleshoot**

Verify that the pump flow and the EGC are on before turning on the CR-TC.

 **CR-TC stopped due to zero flow.**

This error occurs if you turn off the pump flow while the EGC current (and CR-TC) are on. The CR-TC current is automatically turned off to prevent damage to the CR-TC.

❖ **To troubleshoot**

If the pump stopped unexpectedly, follow the troubleshooting steps in “[Pump Stops](#)” on [page 108](#).

 **ED cell current exceeds limits.**

This error can have multiple causes (for example, a cell potential that is too high for a given salt concentration, a cell potential that is too high because the reference mode is incorrect, injection of excessive amounts of electroactive analytes, a damaged or incorrectly installed cell gasket, or an electrical short between two of the three electrodes).

❖ **To troubleshoot:**

1. Turn off the cell voltage. Excessive currents can change or even damage the working electrode.
2. Turn off the pump flow.
3. Disassemble the cell (for instructions, see [page 162](#)) and check for evidence of liquid and salt bridges that can cause shorts. Rinse the cell surface and dry it with a clean, lint-free towel. Replace the cell gasket.
4. Verify that the correct electrode material, waveform potentials, and reference mode are selected for the application being run.
5. Restart the flow and select the DC amperometry mode. Apply cell potential in steps increasing toward the detection potential (the potential of the integration period in integrated amperometric detection). If the current becomes excessive again, try a new working electrode or another cell, if available.

 **ED cell turned off due to zero flow.**

This error occurs if the pump flow is turned off while the ED cell is on. The cell is automatically turned off to prevent damage to the cell.

❖ **To troubleshoot**

If the pump stopped unexpectedly, follow the troubleshooting steps in “[Pump Stops](#)” on [page 108](#).

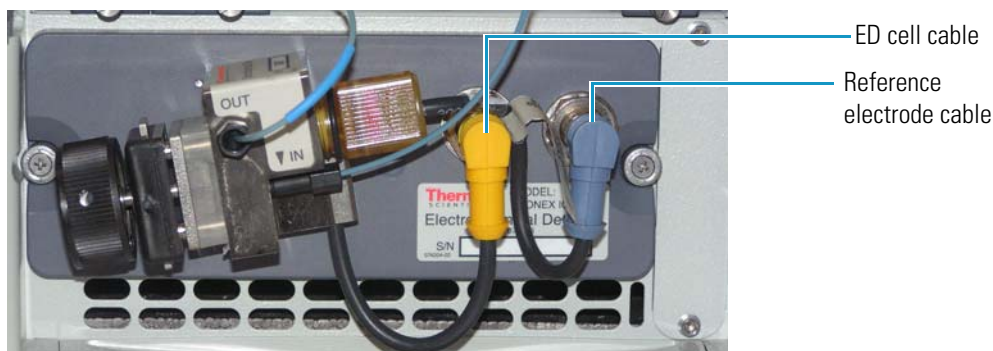
Stop **ED cell option disconnected.**

This error indicates that the electrochemical detector cell is unplugged.

❖ **To troubleshoot**

1. Check the detector connections: Verify that the signal cable from the ED cell is securely connected to the electrical connector on the detector (see [Figure 58](#)). Also, push firmly on the detector to ensure the electrical connector on the rear of the detector is securely connected to the electrical receptacle inside the chromatography compartment.

Figure 58. ED cell connections



2. If the error appears again, contact Technical Support for Dionex products for assistance. The detector electronics may have malfunctioned.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

Stop **ED cell reference electrode disconnected.**

❖ **To troubleshoot**

Check the cable connection: Verify that the cable from the reference electrode is securely connected to the electrical connector on the detector (see [Figure 58](#)).

Stop **ED cell working electrode disconnected.**

This error occurs when the ED cell cable is disconnected from the detector electronics.

❖ **To troubleshoot**

Check the cable connection: Verify that the signal cable from the ED cell is securely connected to the electrical connector on the detector (see [Figure 58](#)).

! **ED pH offset calibration failed.**

This error occurs when the pH reading differs by more than 1.0 pH unit from 7.0, which is the value specified for offset calibration. This can be caused by too large a change in the Ag/AgCl reference potential or by a damaged glass membrane in the pH sensing part of the reference electrode.

❖ **To troubleshoot**

1. Check the buffer selection.
2. Verify that the electrode is properly immersed in the calibration buffer.
3. Repeat the calibration procedure (see [page 168](#)). If the error recurs, repeat the calibration at least one more time.
4. If the error recurs after repeating the calibration at least two times, replace the Ag/AgCl reference electrode (see [page 166](#)).

 **ED pH slope calibration failed.**

This error occurs when the pH-sensing glass membrane of the Ag/AgCl reference electrode is broken or otherwise affected, causing the calibration slope to deviate by more than $\pm 10\%$ from the theoretical slope of 59 mV/pH unit at 25 °C.

❖ **To troubleshoot:**

1. Make sure the calibration temperature is as close as possible to 25 °C.
2. Verify that the correct buffer is being used and that the electrode is properly immersed in the calibration buffer.
3. Repeat the calibration procedure (see [page 168](#)). If the error recurs, repeat the calibration at least one more time.
4. If the error recurs after repeating the calibration at least two times, replace the Ag/AgCl reference electrode (see [page 166](#)).

 **EGC activation date not valid.**

This error occurs if the activation date for the EGC is not a valid date. This may indicate a problem with the memory chip in the EGC.

❖ **To troubleshoot**

1. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.
2. If the error message appears again, contact Technical Support for Dionex products for assistance.

 **EGC cartridge disconnected.**

This error occurs if the EGC is disconnected from the eluent generator electronics.

❖ **To troubleshoot**

1. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.

2. If the error message appears again, contact Technical Support for Dionex products for assistance. The EGC control electronics may have malfunctioned.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

 **EGC invalid concentration.**

This error occurs if the eluent concentration is outside the range allowed by the EG. This may indicate corrupted memory or a problem in the instrument control firmware.

❖ **To troubleshoot**

Contact Technical Support for Dionex products for assistance.

Note The Dionex ICS-4000 electronics components and instrument control firmware cannot be serviced by the user.

 **EGC invalid flow rate or stopped due to zero flow**

This error occurs if the flow rate is set to a value the EG does not support. This error also occurs if the pump flow is turned off while the EGC is on. The EGC is automatically turned off to prevent damage to the cell.

❖ **To troubleshoot**

1. Set the flow rate to a value within the allowed range. For the allowed flow rate range, see [Appendix A, “Specifications.”](#)
2. If the pump stopped unexpectedly, follow the troubleshooting steps in [“Pump Stops”](#) on [page 108](#).

 **EGC invalid flow rate-concentration.**

This error occurs if the selected concentration is too high for the current flow rate.

❖ **To troubleshoot**

Set the concentration to a value within the allowed range. The allowable eluent concentration for a particular application depends on several factors: the flow rate, suppressor type, EGC type, and cartridge configuration. For details, see [“Selecting an Eluent Concentration”](#) on [page 49](#).

 **EGC invalid serial number.**

This error occurs if the serial number reported by the EGC is invalid. This may indicate a problem with the memory chip in the EGC.

❖ **To troubleshoot**

1. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.
2. If the error message appears again, contact Technical Support for Dionex products for assistance.

 **EGC ion count not valid.**

This error occurs if the ion count reported by the EGC is invalid. This may indicate a problem with the memory chip in the EGC.

❖ **To troubleshoot**

1. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.
2. If the error message appears again, contact Technical Support for Dionex products for assistance.

 **EGC over current.**

This error occurs when the current applied to the EGC exceeds the maximum current allowed. (The EGC current is automatically turned off to prevent damage to the cartridge.) This error may also occur if the liquid flow to the cartridge is interrupted.

❖ **To troubleshoot**

1. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.
2. To determine why liquid flow stopped, refer to “[Pump Stops](#)” on [page 108](#).
3. If the error appears again, contact Technical Support for Dionex products for assistance. The EG control electronics may have malfunctioned.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

 **EGC over power.**

This error occurs when, in order to maintain the selected current, the power supply is required to supply a higher voltage than the EGC can support.

❖ **To troubleshoot**

Replace the EGC (see [page 136](#)).

 **EGC over voltage.**

This error occurs when the current applied to the EGC exceeds the maximum current allowed. (The EGC current is automatically turned off to prevent damage to the cartridge.) This error may also occur if the liquid flow to the cartridge is interrupted.

❖ **To troubleshoot**

1. To determine why liquid flow stopped, refer to “[Pump Stops](#)” on [page 108](#).
2. If the error message appears again, contact Technical Support for Dionex products for assistance. The EGC control electronics may have malfunctioned.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

 **EGC wrong cartridge installed.**

This error occurs when an EGC that is not supported by the Dionex ICS-4000 is plugged into the EG.

❖ **To troubleshoot**

Refer to [Table 1](#) for a list of the supported EGC types for capillary IC systems.

 **High-pressure valve 1 error.**

This error occurs if an optional high-pressure valve fails to switch position within 1 second of being toggled.

❖ **To troubleshoot**

1. If a sequence is being executed, terminate the sequence by selecting Stop on the Chromeleon 7 ePanel or Chromeleon 6.8 Control panel.
2. Flip the power switch on the rear panel to turn off the system power and then flip the switch again to turn on the power.
3. Try to toggle the valve from position on the Valves touch screen.
4. If the problem persists, contact Technical Support for Dionex products for assistance.

 **Leak detected.**

The leak sensor is installed above the drip tray in the chromatography compartment (see [Figure 3](#)). If liquid accumulates in the drip tray, the sensor signals the problem and this error message appears.

❖ **To troubleshoot**

1. Locate the source of the leak by visually inspecting the tubing, fittings, and components.

2. Tighten fittings or replace tubing and fittings as required. For detailed troubleshooting of various types of leaks, see “[Liquid Leaks/Leak Alarm](#)” on [page 106](#).
3. After fixing the leak, dry the drip tray thoroughly to prevent the leak sensor from triggering additional error messages.

 **Load/Inject valve error.**

This error occurs if the injection valve installed in the Dionex IC Cube fails to switch position within 1 second of being toggled.

❖ **To troubleshoot**

1. If a sequence is being executed, terminate the sequence by selecting Stop on the Chromeleon 7 ePanel or Chromeleon 6.8 Control panel.
2. Flip the power switch on the rear panel to turn off the system power and then flip the switch again to turn on the power.
3. Try to toggle the valve from Load to Inject by pressing the **Load** and **Inject** buttons on the touch screen Home screen.
4. If the problem persists, contact Technical Support for Dionex products for assistance.

 **Pump pressure over limit.**

This error may be caused by a blockage in the system or by a problem with the column.

❖ **To troubleshoot**

1. Check all tubing connections for signs of blockage; replace as needed (see “[Replacing Tubing and Fittings](#)” on [page 122](#)). Be careful not to overtighten fittings.
2. To determine the source of the high backpressure, isolate segments of the flow path:
 - a. Remove the pump outlet tubing at the injection valve.
 - b. Set the flow rate to 0.01 mL/min, start the pump flow, and record the backpressure.
 - c. Disconnect all components after the injection valve.
 - d. One at a time, reconnect each component of the flow path. If reconnecting a component causes an abnormal increase in backpressure, replace the component. Replace as many components as necessary to resume operation at the standard operating backpressure.
3. As columns age, their backpressure increases. It may be necessary to compensate for this by increasing the high pressure limit. If the column is the source of the high backpressure, clean the column. Refer to the column manual provided on the Thermo Scientific Reference Library DVD (P/N 053891) for instructions. If this does not eliminate the problem, replace the column.

4. Observe a run to see whether the high pressure limit is triggered when injection occurs; if it is, the injection valve may be the source of the blockage (i.e., the rotor seal or stator may need to be replaced). Contact Technical Support for Dionex products for assistance.

 **Pump pressure under limit.**

❖ **To troubleshoot**

1. The eluent supply may be depleted. Check the eluent reservoir and refill it if needed. Prime the pump before resuming operation (see [page 41](#)).
2. Check the eluent line for air bubbles. If air is trapped in the line, replace the end-line filter (P/N 045987). Verify that the end of the line extends to the bottom of the reservoir and the filter is submerged in eluent. Prime the pump (see [page 41](#)).
3. Eluents may be insufficiently degassed. Check the vacuum degassing module.
4. Check all tubing connections for leaks; tighten loose fitting connections. Be careful not to overtighten fittings. For tightening requirements, refer to “[Fitting and Tube Connection Guidelines](#)” on [page 123](#).
5. Make sure the priming valve is closed (see [Figure 24](#)). To close the valve, turn the knob clockwise until closed. Tighten no more than fingertight.
6. A check valve may be defective. Replace the check valve cartridges (see [page 124](#)) and then prime the pump (see [page 41](#)).

 **QDC off for zero flow.**

This error occurs if you turn off the pump flow while the QD cell is on. The cell is automatically turned off to prevent damage to the cell.

 **QD cell option disconnected.**

❖ **To troubleshoot**

1. Check the cell connection. An electronics connector on the back of the cell plugs into a receptacle on the QD detector. Push on the cell to make certain the connection is secure.
2. If the problem persists, there may be a problem in the detector electronics. Contact Technical Support for Dionex products for assistance.

❖ **To troubleshoot**

If the pump stopped unexpectedly, follow the troubleshooting steps in “[Pump Stops](#)” on [page 108](#).

 **Suppressor open circuit.**

❖ **To troubleshoot**

1. When the cartridge is installed in the Dionex IC Cube, pins on the rear of the cartridge connect the suppressor to its power source. Make sure the suppressor cartridge is fully inserted into the Dionex IC Cube and the thumbscrews are tightened.
2. If the error persists, replace the suppressor (see “[Replacing a Dionex IC Cube Cartridge](#)” on [page 148](#)).

 **Suppressor over current.**

This error may be caused by a depleted or dirty suppressor or by a malfunction in the suppressor controller electronics.

❖ **To troubleshoot**

1. Follow the instructions in the suppressor manual to regenerate the suppressor. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
2. Follow the instructions in the suppressor manual to clean the suppressor.
3. If you suspect a malfunction in the suppressor controller, contact Technical Support for Dionex products for assistance.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

 **Suppressor over power.**

This error appears when, in order to maintain the selected current, the Dionex ICS-4000 is required to apply a higher voltage than the suppressor can support.

❖ **To troubleshoot**

1. Reduce the flow rate.
2. Rehydrate the suppressor. Refer to the suppressor manual for instructions. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
3. If the error persists, replace the suppressor (see “[Replacing a Dionex IC Cube Cartridge](#)” on [page 148](#)).

 **Suppressor over voltage.**

This error appears if you turn on the suppressor and the system cannot establish a connection with the suppressor.

❖ **To troubleshoot**

1. When the cartridge is installed in the Dionex IC Cube, pins on the rear of the cartridge connect the suppressor to its power source. Make sure the suppressor cartridge is fully inserted into the Dionex IC Cube and the thumbscrews are tightened.
2. If the error persists, replace the suppressor (see “[Replacing a Dionex IC Cube Cartridge](#)” on [page 148](#)).

ⓘ **Suppressor stopped due to flow rate.**

This message occurs if the pump flow stops while the suppressor is on. The suppressor is automatically turned off to prevent damage to the suppressor.

❖ **To troubleshoot**

If the pump stopped unexpectedly, see the troubleshooting steps in “[Pump Stops](#)” on [page 108](#).

ⓘ **Suppressor type wrong.**

This message occurs if the selected suppressor type does not match the type of eluent being generated.

❖ **To troubleshoot**

Select the suppressor type required for your EGC. For EGC KOH, select the ACES suppressor. For EGC MSA, select the CCES suppressor.

No Communication with Chromeleon

1. Check the USB connections. The Dionex ICS-4000 should be connected by a USB cable to the PC on which Chromeleon is installed.
2. Check that the Dionex ICS-4000 is configured in Chromeleon and is assigned to an instrument.

For Chromeleon connection and configuration instructions, refer to *Dionex ICS-4000 Capillary HPIC System Installation Instructions* (Document No. 065469).

Liquid Leaks/Leak Alarm

Note After eliminating the source of a leak, always dry the leak sensor thoroughly. If the leak sensor is not dry, it will remain activated and will continue to report a leak to the audit trail.

Leaking fitting or broken liquid line

Locate the source of the leak. Tighten or replace the fitting or replace the broken line (see [page 122](#)).

Blocked or improperly installed liquid line

Make sure the lines are not crimped or otherwise blocked. Also, if the line is a waste line, make sure it is not elevated at any point after it exits the Dionex ICS-4000. If a line is blocked, replace it (see [page 122](#)).

Defective piston seal

If the optional seal wash tubing is not installed, a defective piston seal will cause liquid to drip out of bottom seal wash tube. Replace the piston seal (see [page 127](#)).

Leaking check valve

If the leaking check valve is securely tightened but allows leaks despite this, the valve is defective. Replace both check valves (see [page 124](#)).

❖ To tighten a loose check valve

1. Turn off the pump.
2. Loosen the check valve enough to allow it to turn freely.
3. Tighten the check valve fingertight, and then tighten it one-quarter turn with a 1/2-inch wrench.
4. If the check valve continues to leak, tighten it an additional one-quarter turn with the 1/2-inch wrench.
5. If the check valve continues to leak, it is defective. Replace both check valves.

Leaking priming valve knob

- If leaks occur when the priming valve knob is open, close the knob *completely* and then open it one-half to three-quarters turn. If this does not stop the leakage, replace the seal (see [page 134](#)).
- If leaks occurs when the priming valve knob is closed, replace the seal (see [page 134](#)). If this does not stop the leakage, either the transducer housing or the knob is damaged and should be replaced. Contact Technical Support for Dionex products for assistance.

Leaking EGC

Replace the EGC (see [page 136](#)).

Leaking injection valve

1. Make sure the liquid line connections to the valve are tight. Replace any damaged fittings (see [page 122](#)).
2. If the leak is from behind the valve stator, the rotor seal may be scratched. Rebuild the valve (see [page 146](#)).

Leaking cell

1. Check the waste lines for blockage; trapped particles can plug the lines and cause a restriction and/or leak. If necessary, clear the waste lines by reversing the direction of flow.
2. Make sure the plumbing downstream from the cell is clear; a blockage may overpressurize the cell and cause it to leak. If the problem continues, contact Thermo Fisher Scientific for assistance.

Leaking suppressor

Refer to the suppressor manual for troubleshooting procedures. Suppressor manuals are included on the Dionex Reference Library CDROM (P/N 053891).

Low System Backpressure

Low pressure limit was tripped

Refer to the troubleshooting steps for “[Pump pressure under limit.](#)” on [page 103](#).

Loose fitting

Make sure there are no liquid leaks in the flow system. Check tubing connections throughout the system (including the check valves, injection valves, and columns) and tighten or replace fittings as needed (see [page 122](#)).

Internal EGC leak (from membrane barrier)

Leakage from the membrane barrier may trip the pump low pressure limit and shut down the pump. If all other causes of the low system pressure have been eliminated, replace the EGC (see [page 136](#)).

Note The leak sensor cannot immediately detect leakage from the membrane barrier because these leaks exit through the EGC vent line.

High System Backpressure

Restriction in the system plumbing

1. Begin pumping eluent through the system (including the columns) at the flow rate normally used.
2. Follow the flow schematic in [Figure 4](#) (for conductivity detection) or [Figure 5](#) (for electrochemical detection) and work backward through the system, beginning at the flow cell exit. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops abnormally indicates the point of restriction.

Note If the restriction has caused such high pressure that the system cannot be operated, you must work forward through the system, adding parts one at a time until an abnormal pressure increase (and hence, the restriction) is found.

3. If the restriction is in the tubing or fitting, remove the restriction by back flushing or by replacing the tubing or fitting (see [page 122](#)).

Pump Does Not Start

Flow rate is set to zero

1. Select a flow rate on the Chromeleon panel or the Pump touch screen.
2. Verify that a flow rate is set in the Chromeleon instrument method or program.

While being primed, pump starts briefly and an alarm sounds

1. If the high pressure limit was tripped, refer to the troubleshooting steps for “[High System Backpressure](#)” on [page 108](#).
2. If the low pressure limit was tripped, refer to the troubleshooting steps for “[Low System Backpressure](#)” on [page 107](#) and to the steps for “[Liquid Leaks/Leak Alarm](#)” on [page 106](#).
3. If another audit trail error message is displayed, refer to the troubleshooting steps listed for the particular message.

Pump Stops

Instrument method, program, or TTL input instructed the pump to stop

If no error message is displayed in the audit trail, the pump was probably instructed to stop by a command in the Chromeleon instrument method or program or by a TTL signal from a remote device. If you do not want the pump to stop, edit the instrument method or program or change the settings of the remote TTL signal source.

Low pressure limit was tripped

Refer to the troubleshooting steps for “[Pump pressure under limit.](#)” on [page 103](#).

High pressure limit was tripped

Refer to the troubleshooting steps for “[Suppressor type wrong.](#)” on [page 105](#).

Degasser Low Vacuum

Leak in the vacuum degas module

Check all tubing connections for leakage; tighten loose fitting connections (see “[Fitting and Tube Connection Guidelines](#)” on [page 123](#)).

Degas Vacuum Pump Does Not Run

Electrical connections incorrectly installed

There may be a problem with the connections from the degas vacuum pump to the CPU board. Contact Technical Support for Dionex products for assistance.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

EG Stops Operation

Pump flow is off

Turning off the pump automatically turns off the EG and the suppressor. Current to the CR-TC is automatically turned on and off when the EG power is turned on and off. If the pump stopped unexpectedly, refer to the troubleshooting steps in “[Pump Stops](#)” on [page 108](#).

Pump flow rate is too low or too high

Select a flow rate between 0.001 and 0.030 mL/min.

Electrical error detected (ALARM LED is lighted)

To prevent damage to the EGCs, the pump automatically turns off electrical power to the cartridge when excessive current or voltage is detected. Contact Technical Support for Dionex products for assistance.

Note The Dionex ICS-4000 electronics components cannot be serviced by the user.

EGC is expended

Replace the cartridge (see [page 136](#)).

Noisy Baseline

Eluent is contaminated

1. Clean all eluent reservoirs thoroughly (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed on [page xvii](#). Dry with clean, particulate-free air. If a reservoir still appears dirty, or if there is a slimy film on the interior, follow the cleaning instructions on [page 90](#).
2. Flush the system with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “[Deionized Water Requirements for IC](#)” on [page xvii](#).
3. Replace all end-line filters (P/N 045987).
4. If you are not using the EG, prepare new stock solution.
5. If you are not using the EG, prepare fresh eluent. To ensure eluent purity, prepare all eluents with spectro-grade eluents, reagent-grade chemicals, and ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “[Deionized Water Requirements for IC](#)” on [page xvii](#).

Pump not properly primed

Prime the pump (see [page 41](#)).

Piston seal is damaged

If the piston seal is damaged, it allows liquid leaks. Check for moisture around the pump head. If necessary, replace the piston seal (see [page 127](#)).

Flow system leak ahead of cell

Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace liquid line connections (see [page 122](#)).

Rapid changes in ambient temperature

Make sure the door of the temperature-controlled chromatography compartment is kept closed.

Insufficient system equilibration following changes to operating parameters; especially apparent when operating at high sensitivities

Allow a longer system equilibration time (up to 2 hours) before starting operation.

Noisy Baseline: CD Only

- **Incorrect suppressor operating conditions**

Refer to the suppressor manual for troubleshooting information. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

- **Cell above or below temperature**

Contact Technical Support for Dionex products for assistance.

- **Detector electronics not functioning correctly**

Contact Technical Support for Dionex products for assistance.

- **Temperature compensation setting not optimized**

Optimize the selected setting (see [“To change the temperature compensation for the CD”](#) on [page 79](#)).

Noisy Baseline: ED Only

- (DC Amperometry and Integrated Amperometry modes) **Air bubbles trapped inside cell**

- ❖ **To clear trapped bubbles**

1. While wearing gloves and eye protection and with the pump running and all plumbing connected, generate a slight temporary backpressure by putting your finger over the end of the cell outlet tubing for 2 to 3 seconds.
2. Repeat two or three times.
3. If the baseline does not improve, check the other causes of baseline instability described in this section.

IMPORTANT Do not block the end of the cell outlet tubing for longer than the recommended 2 to 3 seconds. Doing so creates high backpressure, which can break the pH-Ag/AgCl reference electrode glass membrane.

Note To prevent air from becoming trapped in the cell in the future, increase the backpressure on the cell by connecting backpressure tubing to the cell outlet. The backpressure limit for the Dionex ICS-4000 Electrochemical Detector cell is 690 kPa (100 psi). Do not exceed this limit.

- (DC Amperometry and Integrated Amperometry modes) **Frequent, random spikes in the baseline**

The pH-Ag/AgCl reference electrode diaphragm is plugged. First, try regenerating the pH-Ag/AgCl reference electrode frit by soaking the electrode in a solution of 1 M KCl plus 1 M HCl. If this does not eliminate the spiking, replace the electrode (P/N 061879).

- (DC Amperometry and Integrated Amperometry modes) Regular baseline oscillation on high-sensitivity ranges
- (DC Amperometry and Integrated Amperometry modes) Dirty or pitted conventional (nondisposable) working electrode

Polish the working electrode (see “Polishing a Conventional Working Electrode” on page 164). If you are using a disposable electrode, replace it.

- (Integrated Amperometry mode) Regular baseline oscillations
 1. Check the pump pressure plot to verify that the pump is working properly.
 2. Air bubbles may be trapped inside the cell (see “To clear trapped bubbles” on page 111).
 3. The water used to prepare the eluent may contain trace contaminants. Remake the eluent, using ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “Deionized Water Requirements for IC” on page xvii.

High Background

CR-TC is contaminated

Clean the device as instructed in the CR-TC manual. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

Wrong eluent

Check that you are using the correct eluent for your application. For an Dionex ICS-4000 Electrochemical Detector, verify that the pH readout is correct for your eluent.

Background signal not offset from detector signal

1. Before injecting sample, allow the background signal to equilibrate, and then select **Autozero** on the Chromeleon 7 ePanel, Chromeleon 6.8 control panel, or touch screen Home screen.
2. Verify that an Autozero command is included in the Chromeleon instrument method or program.

CD Only

- Background not suppressed by suppressor

Verify that the suppressor is turned on and the current is set to the correct value. Refer to the suppressor manual for additional troubleshooting guidance. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

- Regenerant fails to suppress background

Use a higher regenerant flow rate. Refer to the suppressor manual for the suggested regenerant flow rate for your application.

ED Only

- (Integrated Amperometry mode) Excessive number or length of integration intervals and/or incorrect potential for the integration

Verify that the length and potential of the integration interval is correct (refer to the column manual for the settings required for your application). Column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

- (DC Amperometry and Integrated Amperometry modes) Amperometric detection cell working electrode shorted to counter electrode
 - Clean the working electrode with water and dry with pressurized air or nitrogen.
 - Remove any precipitate on the counter electrode by cleaning the spot directly opposite the working electrode with a lint-free tissue.
- (DC Amperometry and Integrated Amperometry modes)—Leak between gasket and electrode, or between gasket and cell body
 - Remove any precipitate with water and a lint-free tissue and then install a new gasket (see “Replacing a Disposable Working Electrode Gasket” on page 161 or “Replacing a Conventional Working Electrode Gasket” on page 162).
 - Turn the yoke knob (see Figure 95) a full 360 degrees to completely engage the knob.

Low Detector Output

Insufficient sample injected

Increase the injection size or concentration.

Working electrode fouled (ED Only)

1. If a disposable working electrode is being used, replace the electrode.
2. For conventional (nondisposable) electrodes, clean the working electrode with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in “Deionized Water Requirements for IC” on page xvii. Dry with pressurized air or nitrogen. If the electrode is pitted, polish the electrode (see “Polishing a Conventional Working Electrode” on page 164).
3. Check the value of the reference potential shift (see “To monitor the pH-Ag/AgCl reference electrode pH” on page 51). If the value fluctuates by more than 30 mV, electrode passivation may occur because potentials that are too high are being applied.

Recalibrate the pH-Ag/AgCl reference electrode (see [page 168](#)). If the problem persists, replace the pH-Ag/AgCl reference electrode (see [page 166](#)).

Analog output range set too high

Select a more sensitive analog output range.

No Peaks

EGC is not on

1. Verify that the EGC concentration is specified in the Chromeleon instrument method program, or on the Electrolytics touch screen. If you are operating the Dionex ICS-4000 from the touch screen, enter the eluent concentration on the Electrolytics screen (see [“Controlling the Eluent Generator”](#) on [page 74](#)).
2. Make sure the EGC cable is securely plugged in (see [Figure 57](#)) and the cable locking ring is tightened.

Injection valve is not actuating

Test the valve by manually switching the position from the Chromeleon 7 ePanel, Chromeleon 6.8 control panel, or the Valves touch screen.

Cell voltage is off (ED Only)

Turn on the cell voltage from the Chromeleon 7 ePanel, Chromeleon 6.8 control panel, or the Detector touch screen.

Tailing Peaks

Excess tubing void volumes

Check tubing connections for void volumes.

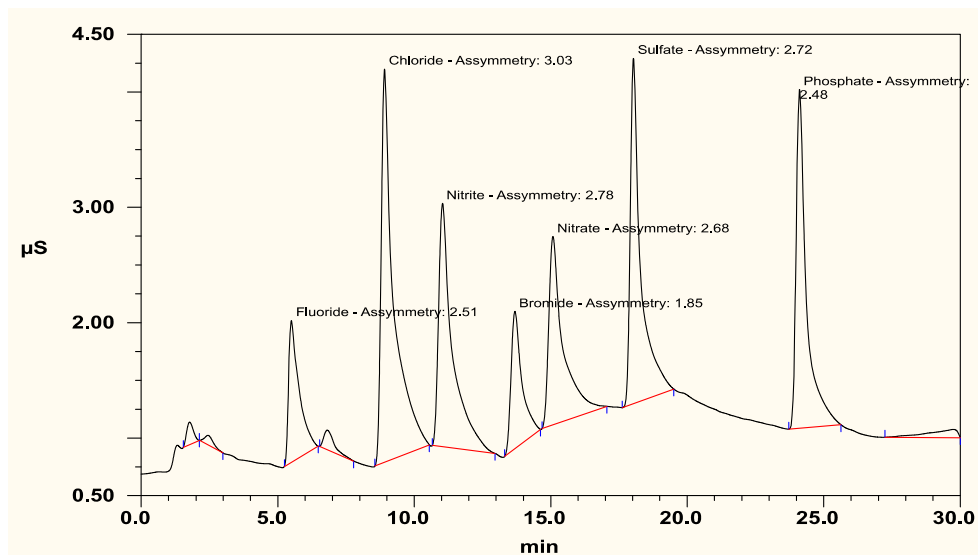
(DC Amperometry and Integrated Amperometry modes) Dirty or pitted conventional (nondisposable) working electrode

1. Clean the working electrode with water and dry with pressurized air or nitrogen.
2. If you are using a conventional working electrode, polish the electrode (see [“Polishing a Conventional Working Electrode”](#) on [page 164](#)) or if you are using a disposable electrode, replace it. See [Appendix B, “Reordering Information.”](#)

Capillary tubing fittings incorrectly installed

Incorrectly installed fittings on capillary tubing can increase void volumes, causing chromatograms with tailing peaks (see [Figure 59](#)). For installation instructions, see [“Replacing Tubing and Fittings”](#) on page 122.

Figure 59. Tailing peaks caused by incorrectly installed capillary tube fittings



Poor Retention Time Reproducibility

Liquid leaks

Check for leaks throughout the system (see [“Liquid Leaks/Leak Alarm”](#) on page 106). Tighten or replace tubing and fittings as needed (see [“Replacing Tubing and Fittings”](#) on page 122).

Pump not primed

Prime the pump (see [“Priming the Pump”](#) on page 41).

Liquid lines incompletely flushed after an eluent change

Attach a 10 mL syringe (P/N 079803) to the priming waste line exiting the pressure transducer (see [Figure 24](#)). Open the priming valve (turn the knob one-quarter to one-half turn counterclockwise). Draw at least 10 mL of the new eluent through the liquid lines before operation.

Inoperative check valves

Replace the check valves (see [“Replacing Pump Check Valves”](#) on page 124).

Eluent is contaminated

1. Clean all eluent reservoirs thoroughly (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#). Dry with clean, particulate-free air. If a reservoir still appears dirty, or if there is a slimy film on the interior, follow the cleaning instructions on [page 90](#).
2. Flush the system with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#).
3. Replace all end-line filters (P/N 045987) (see [“Setting Up the Reservoir”](#) on [page 36](#)).
4. If you are not using the EG, prepare new stock solution.
5. If you are not using the EG, prepare fresh eluent. To ensure eluent purity, prepare all eluents with spectro-grade eluents, reagent-grade chemicals, and ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#).

Incorrect eluent concentration

1. Check the EGC concentration specified in the Chromeleon instrument method. The correct eluent concentration setting depends on the flow rate and the EGC type. See [“Selecting an Eluent Concentration”](#) on [page 49](#) for a list of valid concentration ranges for each cartridge type, and then edit the instrument method as required.

If you are operating the Dionex ICS-4000 from the touch screen, enter the eluent concentration on the EGC screen (see [“Controlling the Eluent Generator”](#) on [page 74](#)).

2. If you are preparing eluent manually, prepare the eluent with spectro-grade eluents, reagent-grade chemicals, and ASTM Type I (or better) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#).

Peak Retention Times Are Too Early

Eluent concentration setting is too high

The correct eluent concentration setting depends on the flow rate and EGC type. See [“Selecting an Eluent Concentration”](#) on [page 49](#) for a list of valid concentration ranges for the cartridges, and then edit the Chromeleon instrument method or program as required.

Flow rate is too low

Increase the flow rate.

Peak Retention Times Are Too Late

Eluent concentration setting is too low

The correct eluent concentration setting depends on the flow rate and EGC type. See [“Selecting an Eluent Concentration”](#) on [page 49](#) for a list of valid concentration ranges for the cartridges, and then edit the Chromeleon instrument method or program as required.

If you are operating the Dionex ICS-4000 from the touch screen, enter the eluent concentration on the EGC screen (see [“Controlling the Eluent Generator”](#) on [page 74](#)).

Flow rate is too high

Decrease the flow rate.

ED Cell Troubleshooting

ED Cell pH Readout Always 7.0

Disconnected pH-Ag/AgCl reference electrode

Verify that the reference electrode cable is securely connected.

Reference electrode short circuit

Replace the reference electrode (see [page 166](#)).

pH-Ag/AgCl reference electrode glass membrane broken or cracked

Replace the pH-Ag/AgCl reference electrode (see [page 166](#)).

Cannot Set ED Cell pH Readout to 7.0

Inaccurate calibration buffer

Use a pH meter to check the pH of the buffer.

Dry pH-Ag/AgCl reference electrode

1. Soak the pH-Ag/AgCl reference electrode in a solution containing 1 M KCl and 1 M HCl long enough to restore the electrode potential to within 30 mV when compared to an unexposed Ag/AgCl reference electrode. To test the pH-Ag/AgCl reference electrode potential, see [“Using a Digital Voltmeter to Determine Reference Potential Shift”](#) on [page 119](#).
2. Replace the pH-Ag/AgCl reference electrode (see [page 166](#)).

To prevent a pH-Ag/AgCl reference electrode from drying out, make sure that eluent is being pumped continuously through the cell. If the cell will not be used for a short time (less than 2 days), disconnect the tubing from the inlet and outlet fittings and all fitting plugs. For longer shutdowns, see “ED Cell Storage” on page 48.

Shift in ED Cell pH Readout

The pH readout is considered to have shifted if it is 0.5 pH units or more different from the value observed when the pH-Ag/AgCl reference electrode was new.

Faulty pH-Ag/AgCl reference electrode

1. Check the electrode by following the instructions in “Shift in Ag/AgCl Reference Potential” on page 119.
2. Regenerate the electrode by soaking in a solution containing 1 M KCl and 1 M HCl long enough to restore the electrode potential to within 30 mV when compared to an unexposed Ag/AgCl reference electrode. To test the pH-Ag/AgCl reference electrode potential, see “Using a Digital Voltmeter to Determine Reference Potential Shift” on page 119.
3. If soaking does not fix the problem, replace the electrode (see page 166).

No ED Cell pH Readout or Intermittent Readout

The pH reading is displayed on the ED ePanel in Chromeleon 7 or the EC Detector Control panel in Chromeleon 6.8.

PdH reference electrode selected

Select the AgCl reference electrode mode on the ED ePanel in Chromeleon 7 or the EC Detector Control panel in Chromeleon 6.8, or in the Chromeleon 7 instrument method or the Chromeleon 6.8 program.

Disconnected reference electrode

Verify that the reference electrode cable is securely connected.

Uncalibrated pH-Ag/AgCl reference electrode

Calibrate the pH-Ag/AgCl reference electrode (see page 168).

Dry pH-Ag/AgCl reference electrode

1. Soak the pH-Ag/AgCl reference electrode in a solution containing 1 M KCl and 1 M HCl long enough to restore the electrode potential to <30 mV when compared to an unexposed Ag/AgCl reference electrode. To test the electrode potential, see “Using a Digital Voltmeter to Determine Reference Potential Shift” on page 119.

2. If soaking the pH-Ag/AgCl reference electrode does not fix the problem, replace the electrode (see [page 166](#)).

To prevent a pH-Ag/AgCl reference electrode from drying out, make sure that eluent is being pumped continuously through the cell. If the cell will not be used for a short time (less than 2 days), disconnect the tubing from the inlet and outlet fittings and all fitting plugs. For longer shutdowns, see “[ED Cell Storage](#)” on [page 48](#).

Contaminated pH-Ag/AgCl reference electrode

Replace the pH-Ag/AgCl reference electrode (see “[Replacing a pH-Ag/AgCl Reference Electrode](#)” on [page 166](#)).

Shift in Ag/AgCl Reference Potential

Faulty pH-Ag/AgCl reference electrode

A shift in reference potential causes a shift in the effective potential applied to the working electrode. For example, when using an electrode with a shift of 50 mV, an applied potential of 0.1 V, is equivalent to an applied potential of 0.15 V for a new pH-Ag/AgCl reference electrode with no shift.

Following the steps below, measure the pH-Ag/AgCl reference electrode potential shift by comparing it to the potential shift of an unexposed electrode. A spare pH-Ag/AgCl reference electrode (P/N 061879) stored in 3 M KCl can be kept on hand for this purpose.

Using a Digital Voltmeter to Determine Reference Potential Shift

1. For each pH-Ag/AgCl reference electrode (the unexposed electrode and the electrode being tested), use a straightened paper clip or short piece of wire of a suitable diameter to connect the voltmeter’s voltage inputs to pin 1 on the reference electrode’s cable connector.

To identify the cable connector pins, refer to Product Information Update for the Electrochemical Detector (PIU_ED_1). The document is included on the Thermo Scientific Reference Library DVD (P/N 053891).

2. Immerse both electrodes in a solution of 0.1 M KCl.
3. Read the potential difference (in mV) between the unexposed electrode and the electrode being tested. If it is greater than 30 mV, try regenerating the electrode by soaking it in a solution containing 1 M KCl and 1 M HCl. If this does not reduce the potential shift, replace the electrode (see [page 166](#)).

Service

This chapter describes Dionex ICS-4000 Capillary HPIC System service and repair procedures that users may perform. All procedures not included here, including electronics-related repair procedures, must be performed by Thermo Fisher Scientific personnel. For assistance, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

Before replacing any part, verify the cause of the problem by referring to the information in [Chapter 6, “Troubleshooting.”](#)

IMPORTANT Substituting non-Dionex/Thermo Fisher Scientific parts may impair system performance, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

Contents

- [Replacing Tubing and Fittings](#)
- [Fitting and Tube Connection Guidelines](#)
- [Servicing Pump Components](#)
- [Servicing Eluent Generator Components](#)
- [Servicing High-Pressure \(Injection\) Valves](#)
- [Replacing a Dionex IC Cube Cartridge](#)
- [Replacing Capillary Columns](#)
- [Servicing Dionex ICS-4000 Conductivity and Charge Detectors](#)
- [Servicing a Dionex ICS-4000 Electrochemical Detector](#)
- [Changing the Main Power Fuses](#)

Replacing Tubing and Fittings

To replace tubing and fittings, refer to the tables below and the information in “Fitting and Tube Connection Guidelines” on page 123.

Table 10. Tubing and fittings for the Dionex ICS-4000

Part	Length	Part Number	Used to connect...
Dionex IC Cube Tubing Kit	N/A	072186	See Table 11
0.25 mm (0.010 in) ID PEEK tubing, black	19 cm (7.5 in)	078431	Pressure transducer to EGC inlet
0.062 mm (0.0025 in) ID PEEK tubing, blue	18 cm (7 in)	072204	EGC outlet to CR-TC eluent inlet
0.062 mm (0.0025 in) ID PEEK tubing, blue	33.65 cm (13.25 in)	078432	CR-TC eluent outlet to EG degas eluent inlet
1.58 mm (0.062 in) ID ETFE (ethylene tetrafluoroethylene) tubing, clear	30.5 cm (12 in)	078433	EG degas regen outlet to CR-TC regen inlet
1.58 mm (0.062 in) ID ETFE tubing, clear	2.1 m (7 ft)	072225	CR-TC regen outlet to waste
ED Cell Inlet Tubing Kit	N/A	074221	ED cell inlet to column outlet

[Table 11](#) lists the tubing and fittings in the Dionex IC Cube Tubing Kit (P/N 072186).

Table 11. Contents of the Dionex IC Cube Tubing Kit (P/N 072186)

Part	Length/Quantity	Part Number	Used to connect...
Precision cut 0.062 mm (0.0025 in) ID PEEK tubing, blue	65 mm (2.56 in)	072188	50 mm guard column outlet to 250 mm analytical column inlet
Precision cut 0.062 mm (0.0025 in) ID PEEK tubing, blue	115 mm (4.53 in)	072189	Guard column inlet to injection valve
Precision cut 0.062 mm (0.0025 in) ID PEEK tubing, blue	75 mm (2.93 in)	074603	35 mm guard column outlet to 150 mm analytical column inlet
Precision cut 0.062 mm (0.0025 in) ID PEEK tubing, blue	210 mm (8.27 in)	072187	Analytical column inlet to injection valve (if a guard column is not present)
0.25 mm (0.010 in) ID PEEK tubing, black	610 mm (24 in)	042690	EG degas cartridge regen outlet to waste (if an EG is not present)

Table 11. Contents of the Dionex IC Cube Tubing Kit (P/N 072186), continued

Part	Length/ Quantity	Part Number	Used to connect...
0.125 mm (0.005 in) ID PEEK tubing, red	610 mm (24 in)	044221	Inject valve waste to AS-AP waste
High-pressure 10-32 fitting bolt, blue	7	074449	All connections
High-pressure 10-32 ferrule, double-cone, blue	7	074373	All connections

Fitting and Tube Connection Guidelines

The following sections provide guidelines and instructions for tube fitting connections used in the Dionex ICS-4000.

Ferrule Fitting Guidelines

Use blue, high-pressure 10-32 fitting bolts (P/N 074449) and blue, high-pressure 10-32 double-cone ferrules (P/N 074373) for all connections. Fitting bolts and ferrules are included in the Dionex ICS-4000 Ship Kit (P/N 075161).

Follow the steps below to tighten a fitting for a high-pressure connection.

❖ To tighten a high-pressure fitting

1. Use your fingers to tighten the fitting bolt as tight as you can.
2. After finger-tightening, use a wrench to tighten the fitting an additional three-quarter turn (270 degrees).
3. If leaks occur, replace the fitting bolt (P/N 074449), ferrule (P/N 074373), and tubing.

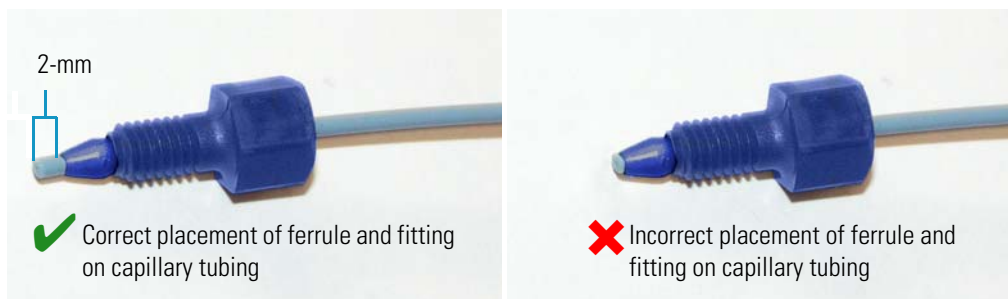
Capillary Tube Fitting Guidelines

The 0.062 mm (0.0025 in) ID blue PEEK tubing (capillary tubing) used for Dionex IC Cube connections is cut at the factory using a specialized precision cutter. This tubing is provided in the Dionex IC Cube Tubing Kit (P/N 072186). Do not shorten or lengthen this tubing or substitute different tubing.

❖ **To install capillary tube fittings**

1. Install the fitting bolt and ferrule onto the tubing. Position the ferrule at least 2 mm (0.1 in) from the end of the tubing (see [Figure 60](#)).

Figure 60. Ferrule and fitting bolt placement for capillary tubing connections



2. Insert the tubing into the port until it stops.

Figure 61. Fitting installation



3. While maintaining pressure on the tubing to keep it in place in the port, tighten the fitting bolt fingertight and then tighten the fitting further by following the instructions in [“Ferrule Fitting Guidelines.”](#)

Cutting Non-Capillary Tubing

If a connection does not require precision-cut capillary tubing, you can use a tubing cutter to cut tubing to the required length. Make sure the cut is at a right angle to the length of the tubing and there are no nicks or burrs on the end. A tubing cutter (P/N 049584) is included in the Dionex ICS-4000 Ship Kit (P/N 075161). Refer to the instructions provided with the cutter.

Servicing Pump Components

Replacing Pump Check Valves

A dirty check valve causes erratic flow rates and pressures; in addition, it may cause the pump to lose prime and/or be difficult to reprime. If a check valve leaks or is dirty, it should be replaced.

Items needed

- Inlet check valve assembly (P/N 044541)
- Outlet check valve assembly (P/N 044540)
- 1/2-inch wrench (P/N 062336)
- Cleanroom gloves (lint-free, particle-free, and oil-free)
- 0.2 micron filtered, Class 10, isopropyl alcohol (IPA)
- 0.2 micron filtered, canned air duster (ITW Chemtronics Ultrajet® or equivalent)

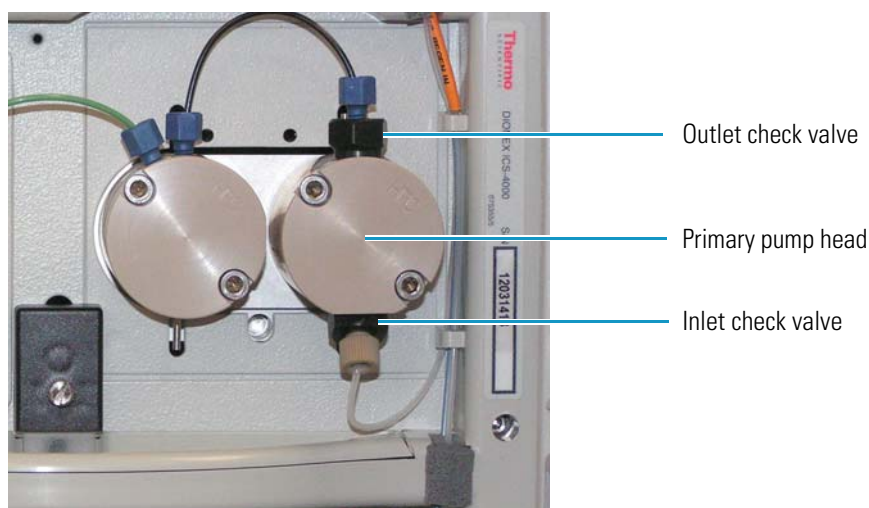
❖ To remove the check valves

1. Turn off the pump flow.
2. Begin monitoring the Pressure reading on the ICS-4000 panel on the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. When the system pressure reaches zero:
 - Disconnect the pump from the software by clicking **Connected** on the ICS-4000 panel.
 - Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door to access pump components.
4. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

Note Never disassemble the pump head with bare hands. Even minute particles of dust, dirt, etc., on the check valves or piston can contaminate the inside of the pump head and result in poor pump performance.

5. The check valves are installed in the primary pump head (see [Figure 62](#)).

Figure 62. Pump components

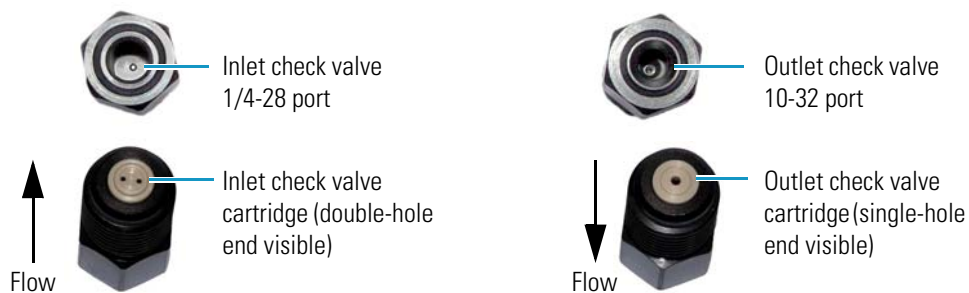


6. Disconnect the tubing connections from the inlet and outlet check valves.
7. Loosen both check valve assemblies using a 1/2-inch wrench.
8. Remove both check valve assemblies from the pump head.

❖ **To install the new check valves**

1. Identify the *inlet* check valve assembly, which has a 1/4-28 port, and verify that the double-hole end of the cartridge is visible (see Figure 63). If the double-hole end is not visible, remove the cartridge from the housing and install it correctly.
2. Identify the *outlet* check valve assembly, which has a smaller (10-32) port, and verify that the single-hole end of the cartridge is visible (see Figure 63). If the single-hole end is not visible, remove the cartridge from the housing and install it correctly.

Figure 63. Inlet and outlet check valve assemblies



Note For correct pump operation, the check valve cartridges must be installed in their respective housings in the correct orientation. Liquid enters through the check valve in the large single hole and exits through the small double holes.

3. Before installing the inlet and outlet check valves, rinse the bottom of the check valve ports in the pump head with isopropyl alcohol and blow them clean using a 0.2 micron filtered, canned air duster. Inspect the bottom of each port for any particulate matter. If necessary, rinse and dry again until they are clean.
4. Install the inlet check valve assembly on the *bottom* of the primary pump head. Install the outlet check valve assembly on the *top* of the pump head.
5. Tighten the check valves fingertight, and then use the 1/2-inch wrench to tighten an additional one-quarter to one-half turn.

IMPORTANT Overtightening may damage the pump head and check valve housing and crush the check valve seats.

6. Reconnect the inlet and outlet tubing to the check valves.
7. Close the compartment door and flip the power switch to turn on the system power.
8. Reconnect the pump to the software by clicking **Connected** on the ICS-4000 panel.

Replacing Pump Piston Seals

A defective piston seal allows leakage past the piston. This may cause unstable flow rates and baseline noise; in addition, it may make it difficult to prime the pump.

The piston seal replacement procedure consists of:

- Removing the pump head and piston
- Cleaning the piston
- Removing the main piston seal
- Removing the piston seal wash seal
- Installing new seals
- Reinstalling the piston and pump head

Although they look similar, the seals for capillary IC pumps and analytical IC pumps are different. Before continuing, verify that you have the correct seals for a capillary pump.

- Main piston seal for capillary pumps (P/N 075493)
- Seal wash seal for capillary pumps (P/N 068628)
- O-ring (P/N 040695) (used for capillary or analytical pumps)

Items needed from the ICS-4000 Ship Kit (P/N 075161)

- 10-32 fitting plugs (P/N 042772)
- 10 mL syringe (P/N 079803)
- Seal insertion tool (P/N 063675)

Additional items needed

- Cleanroom gloves (lint-free, particle-free, and oil-free)
- Small beaker
- ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on page xvii.
- Methanol (optional)
- 0.2 micron filtered, Class 10, isopropyl alcohol (IPA)
- Lint-free paper towels (KIMWIPES® or equivalent)
- Large flat-blade screwdriver
- 3.0 mm hex key (P/N 062338)
- 0.2 micron filtered, canned air duster (ITW Chemtronics Ultrajet or equivalent)

❖ **To remove the pump head and piston**

1. Turn off the pump flow.
2. Begin monitoring the Pressure reading on the ICS-4000 panel on the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. When the system pressure reaches zero:
 - Disconnect the pump from the software by clicking **Connected** on the ICS-4000 panel.
 - Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door to access pump components.
4. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

Note Never disassemble the pump head with bare hands. Even minute particles of dust, dirt, etc., on the check valves or piston can contaminate the inside of the pump head and result in poor pump performance.

5. Disconnect all tubing connections to the pump head with the defective piston seal.
6. The primary and secondary pump heads have different components. When disassembling a pump head, refer to [Figure 64](#) for a primary pump head or [Figure 65](#) for a secondary pump head.

Figure 64. Primary pump head assembly

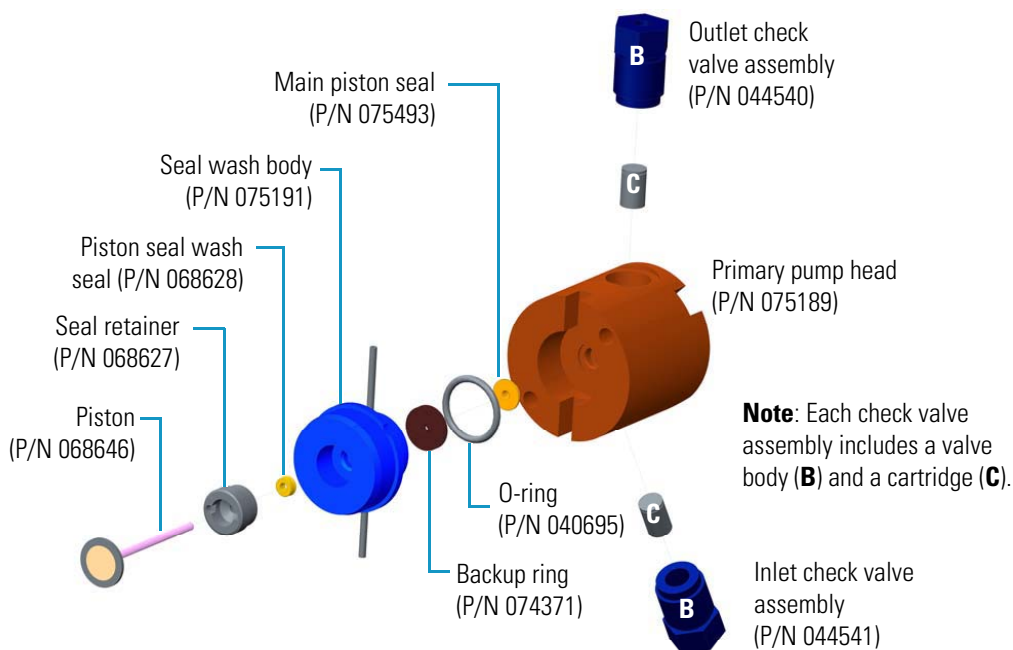
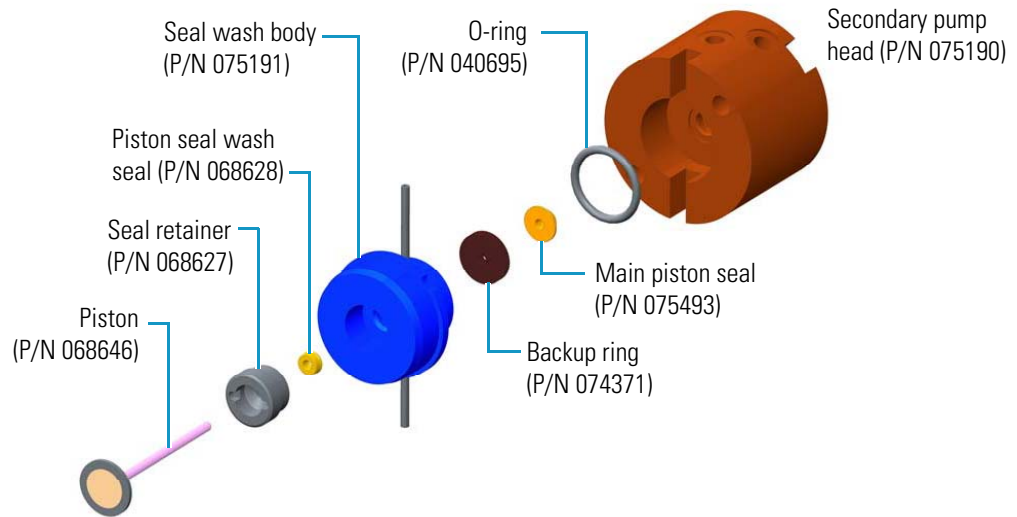


Figure 65. Secondary pump head assembly



7. Using a 3.0 mm hex key (P/N 062338), loosen the two screws on the pump head with the defective seal. Remove the screws, and then carefully remove the head and place it on a clean surface.
8. Remove the seal wash body from the pump head. Or, if the seal wash body was not removed with the pump head in [Step 7](#), pull it straight out of the pump mechanism now.
9. Pull the piston out of the pump mechanism.

Note A magnet secures the piston in place. If the magnetic force makes the piston difficult to remove, tilt the piston to one side and then pull it out of the pump mechanism.

❖ **To clean the piston**

1. Place the piston in a beaker containing either ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications in [“Deionized Water Requirements for IC”](#) on [page xvii](#) or methanol. Sonicate for several minutes.
2. After cleaning, rinse the piston thoroughly with ASTM Type I (18 megohm-cm) filtered and deionized water. Dry it with a lint-free paper towel.
3. Inspect the piston for signs of damage. If the piston is scratched or scored, replace it (see [page 132](#)).

IMPORTANT Even minute scratches or particles of dust, dirt, etc. on the check valves or piston can contaminate the inside of the pump head and result in poor pump performance.

❖ **To remove the main piston seal**

1. If this is the *primary* pump head, insert a 10-32 fitting plug (P/N 042772) into the 10-32 outlet hole of the check valve nut.

If this is the *secondary* pump head, insert a 10-32 fitting plug (P/N 042772) into both the 10-32 inlet and outlet holes.

2. Using a 10 mL syringe (P/N 079803), inject a few drops of ASTM Type I (18 megohm-cm) filtered and deionized water through the main piston seal and into the piston cavity in the pump head.
3. Reinsert the piston approximately 3 mm (0.125 in) into the piston seal and press gently. The seal should pop out of the head and onto the piston.

IMPORTANT Do not use a sharp tool (such as tweezers) to remove the main piston seal. This will scratch the inside of the pump housing; these scratches will prevent a proper seal and cause leakage.

4. If the piston seal was not removed in [Step 3](#), follow these steps:
 - a. Verify that the 10-32 fitting plugs in the inlet and outlet holes are tightened enough to prevent any leaks from the pump head.
 - b. Fill the piston cavity with water and check for bubbles.
 - c. If there are no bubbles, repeat [Step 3](#).

❖ **To remove the piston seal wash seal**

1. Remove the O-ring (P/N 040695) and the backup ring (P/N 074371) from the seal wash body.
2. Follow these steps to remove the piston seal wash seal from the seal wash body:
 - a. Using a large flat-blade screwdriver, remove the seal retainer (P/N 068627) from the seal wash body.
 - b. Insert the piston into the seal wash body from the O-ring side and gently push the seal out of the retainer.

IMPORTANT Do not use a sharp tool (such as tweezers) to remove the piston seal wash seal. This may scratch the seal and the inside of the pump housing; scratches will prevent a proper seal and cause leakage.

❖ **To install the piston seals and O-ring**

1. Follow these steps to reassemble the seal wash body:
 - a. Place the seal wash body on a clean work surface.

- b. Slide the new seal wash seal, with the open side of the seal facing upward, onto the seal insertion tool (P/N 063675) (see [Figure 66](#)).

Figure 66. Piston seal wash seal and seal insertion tool



- c. Insert this end of the tool partway into the seal wash body. Make sure the tool is centered and does not rock back and forth. Then, press firmly on the tool and the seal wash body until they snap together.
- d. Remove the seal insertion tool from the seal wash body.
- e. The seal wash seal is now partially installed. To complete the seal installation, place the retainer in the seal wash body and use the large flat-blade screwdriver to tighten the retainer.
- f. Place the new O-ring (P/N 040695) on the seal wash body.

Note When replacing a piston seal, always replace the O-ring, also. This will prevent leaks.

- g. Place the new backup ring (P/N 074371) on the seal wash body.
2. Rinse the new main piston seal (P/N 075493) with isopropyl alcohol (IPA) or dip it into a container of IPA. (The seal is easier to install when it is moist.)
3. Insert the piston through the seal wash assembly, and then through the new main piston seal.
4. Make sure the piston seal is centered.

IMPORTANT If the piston seal is not centered, applying pressure to it in [Step 7](#) will damage the seal and make it unusable.

5. Place the front of the pump head, flat side down, on a clean work surface.
Make sure the open side of the piston seal faces away from the retainer for the seal wash seal.
6. Using IPA, rinse inside the pump head cavity where the seal will be installed. Blow inside the cavity to dry it using the 0.2 micron filtered, canned air duster. Inspect the cavity for any particulate matter. If necessary, rinse and dry again, until the cavity is clean. Fill the cavity with ASTM Type I (18 megohm-cm) filtered and deionized water.
7. Place the components on the pump head and *gently* press the housing until the piston seal snaps into place.

When pressing the seal in place, ensure that the piston is free to move out. This will relieve the pressure in the pump head during seal installation.

IMPORTANT Do not use a sharp tool (such as tweezers) to install the piston seal. This will scratch the seal and the inside of the pump housing; these scratches will prevent a proper seal and cause leakage.

8. Remove the 10-32 fitting plugs from the pump head.

❖ **To reinstall the piston and pump head**

1. Slide the piston partway into the pump head; approximately 6 mm (1/4 in) of the sapphire part of the piston should extend from the head.
2. Place the pump head back on the pump.
3. Reinstall the screws in the pump head. Using a 3.0 mm hex key, tighten the screws just until they come into contact with the pump head. Then, tighten the screws another one-quarter to one-half turn, one-eighth of a turn at a time.
4. Reconnect all tubing connections to the pump head. Tighten connections fingertight, and then tighten an additional one-quarter turn only.
5. Close the compartment door.
6. Flip the power switch on the rear panel to turn on the system power.
7. Reconnect the system to the software by clicking **Connected** on the ICS-4000 panel.
8. Turn on the pump flow.

Replacing a Pump Piston

If a new piston seal leaks (assuming that the pump head is tight), it indicates that the piston is dirty, scratched, or broken, and should be replaced.

The piston replacement procedure consists of:

- Removing the pump head and the old piston
- Reinstalling a new piston
- Reinstalling the pump head

Before continuing, verify that you have the correct piston for a capillary pump (P/N 068646).

Items needed

- Cleanroom gloves (lint-free, particle-free, and oil-free)
- ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on page xvii.

❖ **To remove the pump head and piston**

1. Turn off the pump flow.
2. Begin monitoring the Pressure reading on the ICS-4000 panel on the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. When the system pressure reaches zero:
 - Disconnect the pump from the software by clicking **Connected** on the ICS-4000 panel.
 - Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door to access pump components.
4. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

Note Never disassemble the pump head with bare hands. Even minute particles of dust, dirt, etc., on the check valves or piston can contaminate the inside of the pump head and result in poor pump performance.

5. Disconnect all tubing connections to the pump head with the defective piston seal.
6. The primary and secondary pump heads have different components. When disassembling a pump head, refer to [Figure 64](#) for a primary pump head or [Figure 65](#) for a secondary pump head.
7. Locate the 3.0 mm hex key (P/N 062338) in the ICS-4000 Ship Kit (P/N 075161) and then loosen the two screws on the pump head with the defective seal. Remove the screws, and carefully remove the head and place it on a clean surface.
8. If the seal wash body was not removed with the pump head in [Step 7](#), pull it straight out of the pump mechanism now.
9. If the piston was not removed with the pump head or seal wash body, remove the piston now by pulling it straight out of the pump mechanism.

Note A magnet secures the piston in place. If the magnetic force makes the piston difficult to remove, tilt the piston to one side and then pull it out of the pump mechanism.

10. If the piston is broken, be sure to remove all broken pieces. If necessary, flush the pump head with ASTM Type I (18 megohm-cm) filtered and deionized water.

❖ **To install the new piston and reinstall the pump head**

1. Slide the new piston (P/N 068646) partway into the pump head; approximately 6 mm (1/4 in) of the sapphire part of the piston should extend from the head.
2. Place the pump head back on the pump.

3. Reinstall the screws in the pump head. Using the 3.0 mm hex key (P/N 062338), tighten the screws just until they come into contact with the pump head. Then, tighten the screws another one-quarter to one-half turn, one-eighth of a turn at a time.
4. Reconnect all tubing connections to the pump head. Tighten connections fingertight, and then tighten an additional one-quarter turn only.
5. Close the compartment door.
6. Flip the power switch on the rear panel to turn on the system power.
7. Reconnect the pump to the software by clicking **Connected** on the ICS-4000 panel.
8. Turn on the pump flow.

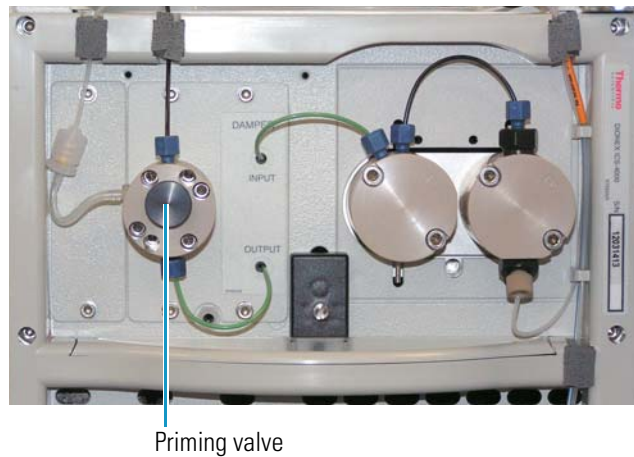
Replacing the Seal on the Pump Priming Valve Knob

Replace the seal in the priming valve knob if there is leakage around the valve threads when the valve is open or if the waste port leaks when the valve is closed.

❖ To replace the seal

1. Turn off the pump flow.
2. Begin monitoring the Pressure reading on the ICS-4000 panel on the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. When the system pressure reaches zero:
 - Disconnect the pump from the software by clicking **Connected** on the ICS-4000 panel.
 - Flip the power switch on the rear panel to turn off the system.
3. Open the chromatography compartment door to access pump components.
4. The priming valve knob is located
5. on the pressure transducer (see [Figure 67](#)). To remove the knob, turn it counterclockwise all the way and then pull it straight off.

Figure 67. Pump components



6. Pull the old seal off the end of the priming valve knob.
7. Hold the new seal (P/N 078455) with the groove in the priming valve seal facing away from the knob (see [Figure 68](#)). Carefully slide the seal onto the knob; avoid scratching or nicking the sides.

Figure 68. Priming valve and seal



IMPORTANT Do not use a sharp tool (such as tweezers) to install the seal. This may scratch the seal and the surface of the priming valve knob. These scratches will prevent a proper seal and cause leakage.

8. Insert the priming valve knob into the transducer housing, turn the knob clockwise, and tighten fingertight.
9. Turn on the system power, reconnect the pump to the software, and turn on the pump flow.

Servicing Eluent Generator Components

Replacing an EGC

Replace the EGC when the cartridge is expended or when it leaks. For a list of the EGC models that can be used with the Dionex ICS-4000, see “[Eluent Generator Cartridge \(EGC\)](#)” on [page 18](#).



CAUTION The EGC contains one of the following: a corrosive base (KOH, LiOH, or NaOH), a corrosive acid (MSA), or a concentrated K_2CO_3 solution. Wear protective eyewear and gloves when handling the cartridge.



MISE EN GARDE La cartouche d'EGC contient un de ce qui suit: une base corrosive (KOH, LiOH, ou NaOH), un acide corrosif (MSA), ou une solution concentrée de K_2CO_3 . Porter des lunettes et des gants protectives en manipulant la cartouche.

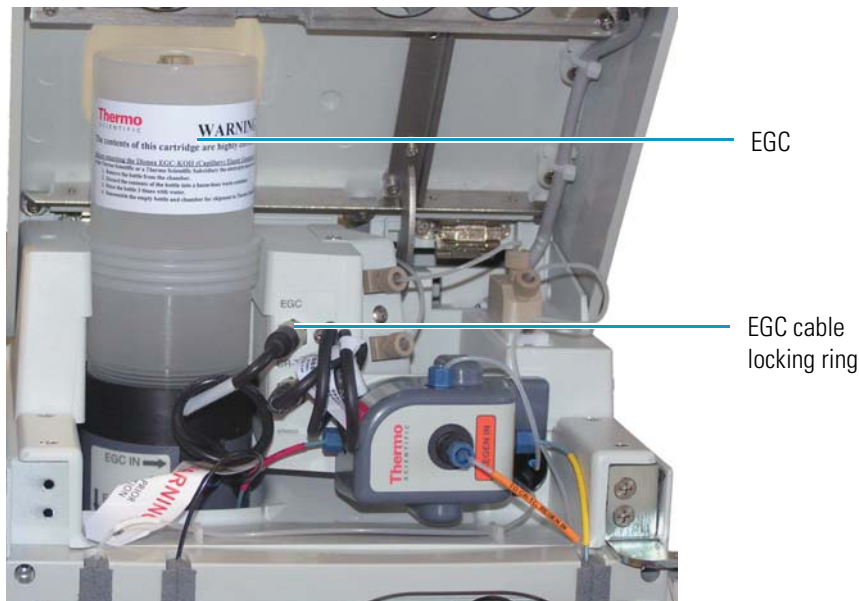


VORSICHT Die EGC-Kartusche enthält eine korrodierende Base (KOH, LiOH oder NaOH), eine korrodierende Säure (MSA) oder eine konzentrierte K_2CO_3 -Lösung. Tragen Sie daher beim Umgang mit Kartusche eine Schutzbrille und Handschuhe.

For reference, [Figure 69](#) shows the final eluent generator plumbing after all setup steps are complete.

❖ To remove the old EGC

1. Turn off the pump flow. This turns off the power to the EGC and the suppressor, also.
2. Lift up the touch screen panel to access the EG components (see [Figure 70](#)).

Figure 70. EGC installed on top tray of Dionex ICS-4000

3. Twist the metal locking ring on the EGC electrical cable counterclockwise to loosen it, and then pull the cable straight out of the electrical connector.
4. Remove the luer fitting from the top of the EGC and install the vent fitting supplied with the EGC (see [Figure 71](#)). If the vent fitting is not available, install a fitting plug. This prevents leakage if the cartridge is turned over during removal.

Figure 71. EGC vent fitting

5. Lift the EGC straight up and then slide it forward to remove it from its holder.
6. Disconnect the inlet and outlet lines from the ports on the side and bottom of the EGC.

❖ **To dispose of the old EGC**

If the EGC is expended, follow these steps to dispose of it:

1. Make sure the vent fitting is tightened and then turn over the cartridge so that the eluent generation chamber is on top. Unscrew the eluent generation chamber from the electrolyte reservoir and pour the remaining electrolyte solution into an appropriate hazardous waste container.

Note Refer to the Material Safety Data Sheet (MSDS) shipped with the EGC for the chemical description.

2. Rinse the electrolyte reservoir and membranes *three times* with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications in “[Deionized Water Requirements for IC](#)” on [page xvii](#). Rinsing should render the reservoir and membranes nonhazardous; however, check your local, state, and federal regulatory agency regulations for proper disposal.

❖ **To store an old EGC**

If the EGC is not expended, follow these steps to prepare the cartridge for storage:

1. Plug all fittings. Tighten the vent fitting. This will prevent the evaporation of water in the electrolyte reservoir.
2. Store the cartridge in an upright position (with the electrolyte reservoir on top) at 4 to 40 °C (39 to 104 °F) until its next use. The original shipping container is ideal for storage. Before resuming operation, condition the cartridge. For instructions, see [page 143](#).

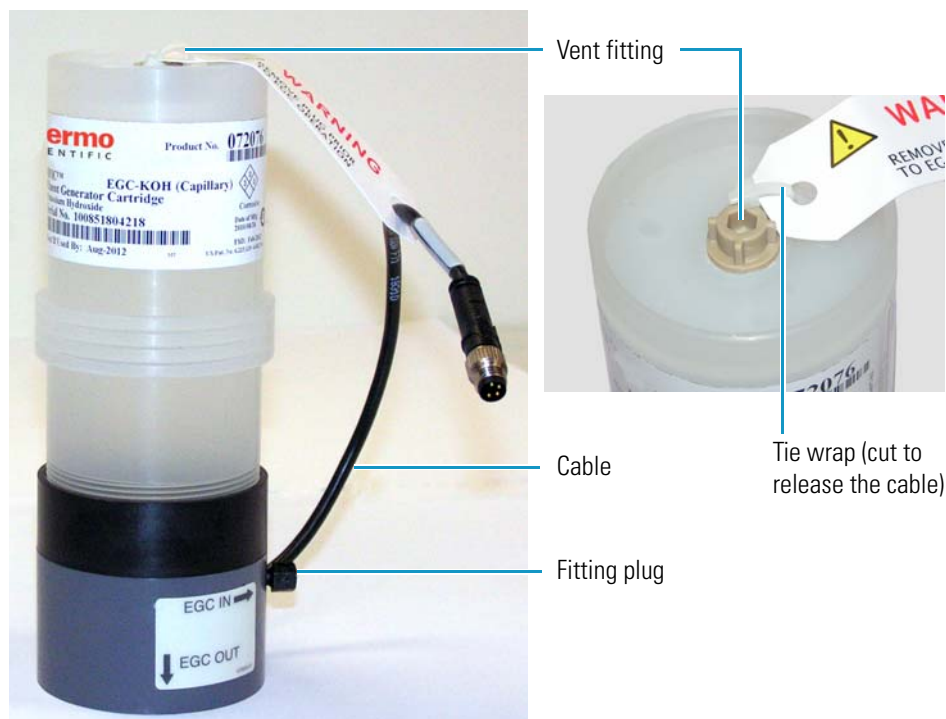
❖ To flush the new EGC

1. Remove the new EGC from the shipping container.

Note Save the shipping container; it may be required for storage or disposal of the EGC.

2. The EGC cable is attached to the vent fitting on the top of the cartridge with a cable tie. Cut the cable tie to release the cable (see [Figure 72](#)).

Figure 72. EGC with cable attached to vent fitting

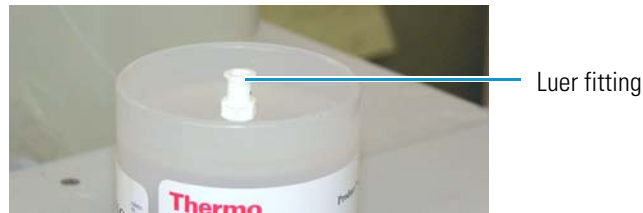


3. Verify that the vent fitting is tightened. This prevents cartridge leaks during installation.
4. Remove the fitting plugs from the **EGC IN** and **EGC OUT** ports on the EGC (see [Figure 72](#)).
5. While holding the EGC with the ports on the bottom, shake the EGC vigorously and tap it with the palm of your hand 10 to 15 times to dislodge air bubbles that may be trapped in the chamber.

- Unscrew the vent fitting from the top of the EGC and install the luer adapter shipped with the EGC (see [Figure 73](#)). Save the vent fitting.

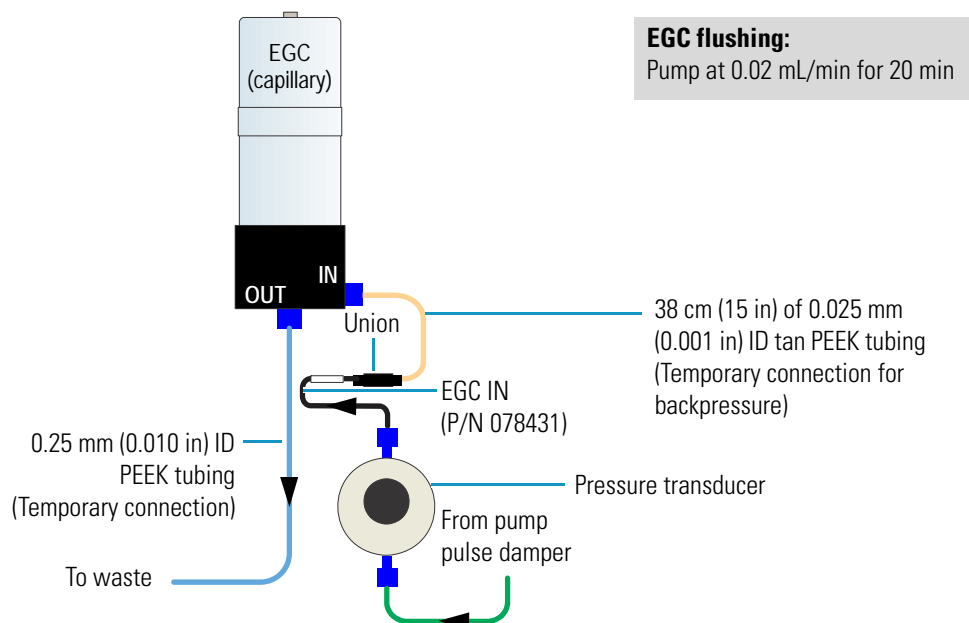
IMPORTANT To ensure proper ventilation, always install the luer fitting on the top of the EGC before operation. If you need to remove the EGC from the system, install the vent fitting to prevent leaks.

Figure 73. Top of EGC with luer fitting installed



- Obtain the following items from the Dionex ICS-4000 Ship Kit (P/N 075161):
 - 0.25 mm (0.010 in) ID black PEEK tubing (P/N 042690)
 - 38 cm (15 in) piece of 0.025 mm (0.001 in) ID tan PEEK tubing (P/N 074582)
 - Two 10-32 fitting bolts and two double-cone ferrules
- Cut a piece of black tubing long enough to connect from the EGC to a waste container and connect this tubing to the **EGC OUT** port (see [Figure 74](#)). Direct the free end to a waste container. This is a temporary connection.
- Connect the tan PEEK tubing (P/N 074582) between the **EGC IN** line (P/N 078431) (pre-plumbed to the pump pulse damper) and the **EGC IN** port (see [Figure 74](#)). This is a temporary connection for backpressure.

Figure 74. Flow schematic for flushing the EGC



10. Slide the EGC into its holder (see [Figure 70](#)).
11. Align the four pins inside the cable connector with the holes in the EGC connector on the front tray. Push the cable firmly onto the front tray connector and then twist the locking ring fingertight to secure it.
12. Unscrew the fitting on the top of the EGC a few turns to vent the EGC.



4-pin EGC cable connector

IMPORTANT To ensure proper ventilation, always loosen the fitting on the top of the EGC before operation.

13. Verify that the suppressor and the CR-TC are off.

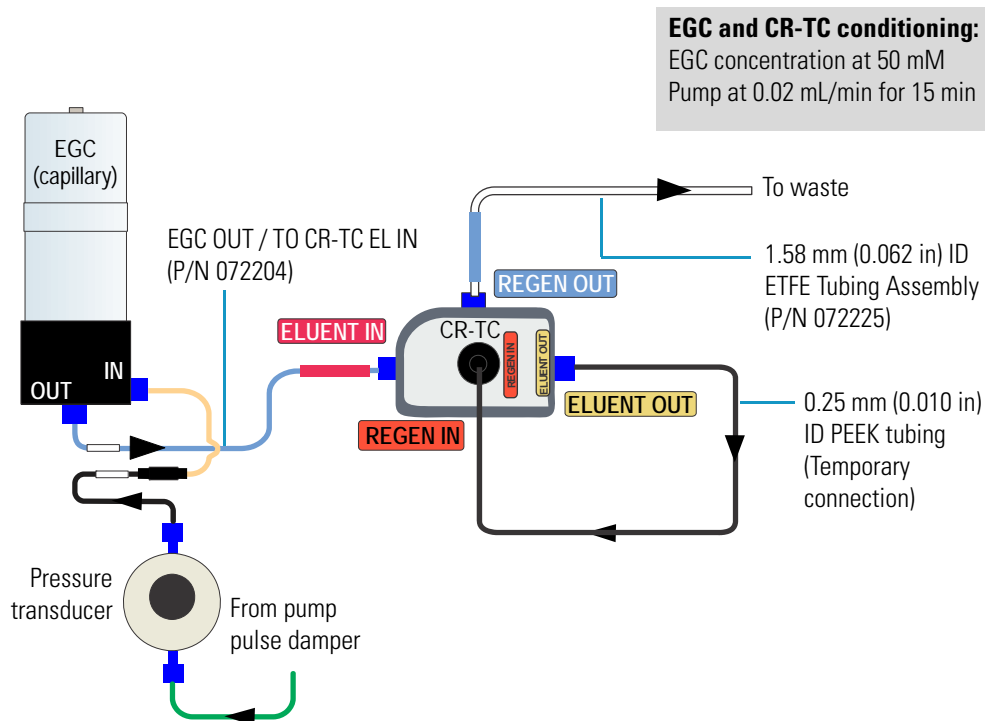
IMPORTANT To avoid damaging the suppressor and CR-TC, always turn them off before flushing the EGC. The pump flow is on during flushing; however, no flow reaches the suppressor or CR-TC.

14. Set the pump flow to **0.02 mL/min** and turn on the pump.
15. Flush the EGC for 20 minutes and then turn off the pump flow.
16. Remove the temporary black PEEK tubing from the **EGC OUT** port.

❖ **To condition the EGC**

1. Disconnect the lines from the **ELUENT OUT** and **REGEN IN** ports of the CR-TC and connect a piece of black PEEK tubing between these ports (see [Figure 75](#)). This is a temporary connection.
2. Connect the **EGC OUT / TO CR-TC EL IN** line (P/N 072204) between the **EGC OUT** and CR-TC **ELUENT IN** ports (see [Figure 75](#)).

Figure 75. Flow schematic for conditioning the EGC



3. Verify that the suppressor is off.
4. Verify that the pump flow rate is **0.02 mL/min**.

IMPORTANT To avoid damaging the suppressor, always turn off the suppressor before conditioning the EGC. The pump flow is on during conditioning; however, no flow reaches the suppressor.

5. Set the EGC concentration to **50 mM** and turn on the EGC and CR-TC power.
6. Condition the EGC for 15 minutes and then turn off the EGC and CR-TC power, and the pump flow.
7. Remove the temporary tan backpressure tubing and reconnect the line from the pressure transducer to the **EGC IN** port.
8. Disconnect the temporary tubing from the **ELUENT OUT** and **REGEN IN** ports of the CR-TC and reconnect the **ELUENT IN** and **REGEN OUT** lines from the EG Degas to their respective ports.

Replacing a CR-TC

Refer to [Figure 69](#) for the Dionex ICS-4000 EG flow schematic.

Removing the Old CR-TC

1. Turn off the pump flow. This turns off the power to the EGC and the suppressor, also.
2. Lift up the touch screen panel to access the EG components (see [Figure 70](#)).
3. Twist the metal locking ring on the CR-TC electrical cable counterclockwise to loosen it (see [Figure 76](#)), and then pull the cable straight out of the connector.

Figure 76. CR-TC electrical cable



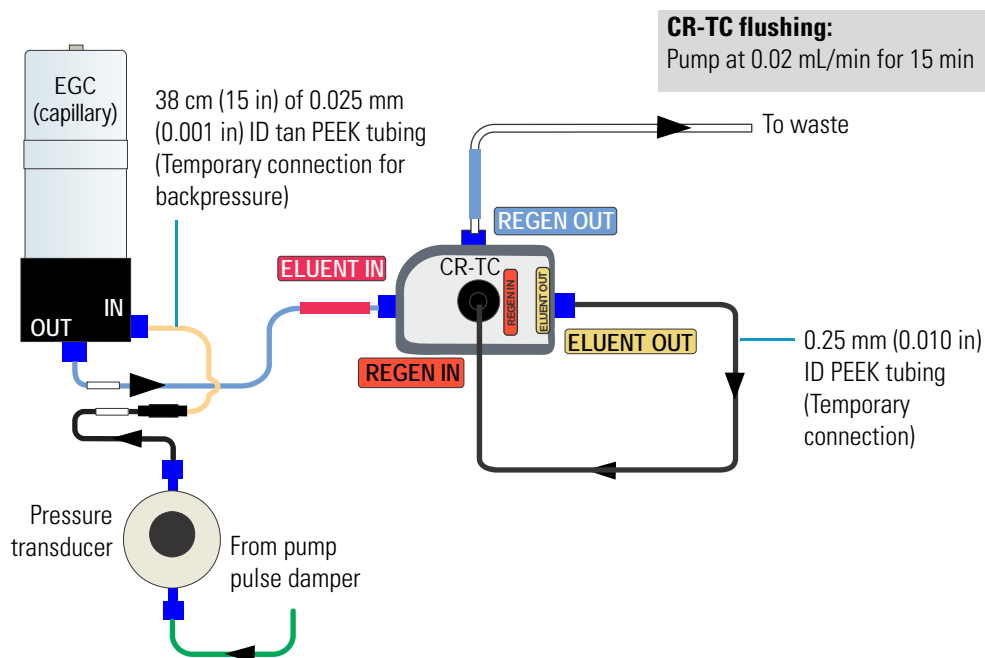
4. Disconnect the liquid lines from the four ports on the CR-TC.
5. Pull the CR-TC straight off of its mounting location and remove the CR-TC from the Dionex ICS-4000.

Installing and Flushing the New CR-TC

1. Remove the plugs from the ports on the new CR-TC.
2. Obtain the following items from the Dionex ICS-4000 Ship Kit (P/N 075161):
 - 0.25 mm (0.010 in) ID black PEEK tubing (P/N 042690)
 - 38 cm (15 in) piece of 0.025 mm (0.001 in) ID tan PEEK tubing (P/N 074582)
 - Two 10-32 fitting bolts and two double-cone ferrules

3. Connect the tan PEEK tubing (P/N 074582) between the **EGC IN** line (P/N 078431) (pre-plumbed to the pump pulse damper) and the **EGC IN** port (see Figure 77). This is a temporary connection for backpressure.
4. Connect the **EGC OUT / TO CR-TC EL IN** line (P/N 072204) between the **EGC OUT** and CR-TC **ELUENT IN** ports (see Figure 77).
5. Connect a piece of 0.25 mm (0.010 in) ID black PEEK tubing between the **ELUENT OUT** and **REGEN IN** ports of the CR-TC (see Figure 77). This is a temporary connection.
6. Connect the waste line to the **REGEN OUT** port of the CR-TC.

Figure 77. Flow schematic for flushing the CR-TC



7. Verify that the suppressor is off.

IMPORTANT To avoid damaging the suppressor, always turn off the suppressor before flushing the CR-TC. The pump flow is on during conditioning; however, no flow reaches the suppressor.

8. Set the flow rate to **0.02 mL/min** and turn on the pump flow. Allow the pump to run for 15 minutes.
9. Turn off the pump flow.
10. Remove the tan backpressure tubing from the eluent inlet line from the pump.
11. Connect the eluent inlet line from the pump to the **EGC IN** port.
12. Disconnect the temporary tubing from the **ELUENT OUT** and **REGEN IN** ports of the CR-TC.

Completing the New CR-TC Plumbing

1. Connect the **ELUENT IN** and **REGEN OUT** lines from the EG degas cartridge to their respective ports on the CR-TC.
2. Align the two mounting holes on the CR-TC back plate with the ball studs on the mounting plate, check that no liquid lines are caught under the CR-TC, and then push the CR-TC firmly onto the mounting ball studs. The CR-TC clicks into place when properly installed.
3. Align the three pins inside the cable connector with the holes in the CR-TC connector on the front tray. Push the cable firmly onto the front tray connector and then twist the locking ring fingertight to secure it.



3-pin CR-TC
cable connector

Servicing High-Pressure (Injection) Valves

Rebuilding a High-Pressure Valve

Thermo Fisher Scientific recommends rebuilding the high-pressure (injection) valve annually. The Injection Valve Rebuild Kit (P/N 074698; 4-port valve; P/N 075974, 6-port valve) contains all required replacement parts.

Note Substitution of non-Dionex/Thermo Fisher Scientific parts may impair valve performance and void the product warranty.

Note If you prefer, you can replace the high-pressure valve “pod,” instead of rebuilding the valve. Replacing the pod is easier and faster than rebuilding the valve.

❖ To rebuild a high-pressure valve

1. Turn off the pump flow.
2. Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door.
4. Disconnect each liquid line connected to the valve.
5. Follow the instructions provided in the Rebuild Kit to replace the rotor seal, isolation seal, and stator face.
6. Reconnect all liquid lines to the injection valve.
7. Turn on the system power.
8. Turn on the pump flow. Check for leaks from the valve. For tightening guidelines for high-pressure fittings, see [page 123](#).
9. Close the door.

Replacing a High-Pressure Valve Pod

This procedure describes how to replace the mechanical parts (the “pod”) of a high-pressure valve (P/N 074699, 4-port; P/N 075971, 6-port).

Note If the valve electronics require service, contact Thermo Fisher Scientific. Electronics-related repair procedures must be performed by Thermo Fisher Scientific personnel.

Note Substitution of non-Dionex/Thermo Fisher Scientific parts may impair valve performance and void the product warranty.

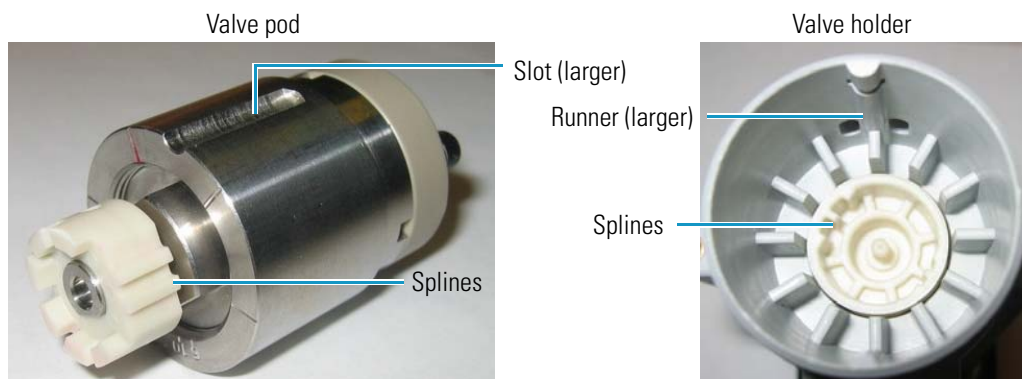
❖ To replace a high-pressure valve pod

1. Turn off the pump flow.
2. Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door.
4. Disconnect each liquid line connected to the valve.
5. Unscrew the locking ring on the outside of the valve (see [Figure 78](#)) and remove the ring.

Figure 78. High-pressure valve locking ring



6. Grasp the front of the valve pod and pull out firmly to remove it from the Dionex IC Cube.
7. Align the slots in the new pod with the runner in the valve holder on the Dionex IC Cube (see [Figure 79](#)). Valve pods are keyed to fit only one way (one slot is narrower than the other). Verify that the slots are aligned with their matching runners.
8. Also verify that the two splines on the pod align with the matching splines inside the valve holder (see [Figure 79](#)). If necessary, twist the end of the pod to adjust the position of the splines.

Figure 79. High-pressure valve pod and pod holder

9. Push the pod into the holder until it clicks into place. Replace the black locking ring.
10. Reconnect all liquid lines to the valve.
11. Turn on the system power and start the pump flow. Check for leaks from the valve. Tighten fittings as required. For tightening guidelines for high-pressure fittings, see [page 123](#).
12. Close the door.

Replacing a Dionex IC Cube Cartridge

❖ To replace a Dionex IC Cube cartridge

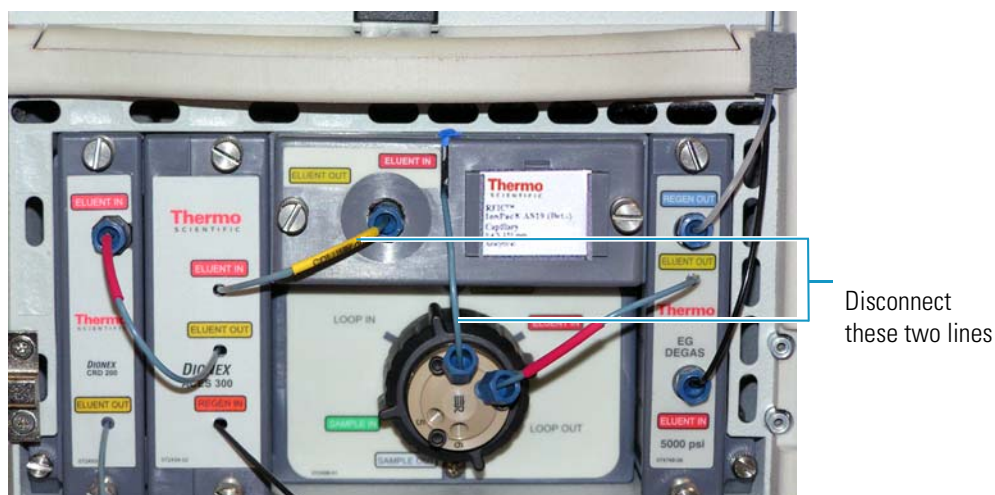
1. Turn off the pump flow.
2. Open the chromatography compartment door.
3. Disconnect each liquid line connected to the Dionex IC Cube cartridge to be replaced.
4. Loosen the two thumbscrews on the cartridge and pull the cartridge out of the Dionex IC Cube.
5. Remove the caps from the regenerant ports on the rear of the cartridge and then slide the new cartridge into the Dionex IC Cube. Push the cartridge firmly into the Dionex IC Cube and then tighten the thumbscrews.
6. If you are installing a suppressor or CRD cartridge, hydrate them before use. Refer to the instructions in the suppressor or CRD manual. The manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891)
7. Reconnect the liquid lines that were disconnected in [Step 3](#).

Replacing Capillary Columns

❖ To remove capillary columns from the column cartridge

1. Turn off the pump flow.
2. Open the Dionex ICS-4000 door.
3. Disconnect the line from the **ELUENT OUT** port of the injection valve and the line from the **ELUENT OUT** port of the column cartridge (see [Figure 80](#)).

Figure 80. Column cartridge eluent lines



4. Loosen the two thumbscrews on the column cartridge and pull the cartridge out of the Dionex IC Cube.
5. Lift the lid of the column cartridge to open it.
6. Remove the guard column (if present) and analytical column from the column clips. [Figure 81](#) shows a column cartridge with both a guard column and an analytical column installed. [Figure 82](#) shows a column cartridge with only an analytical column installed.

Figure 81. Analytical and guard columns installed in column cartridge

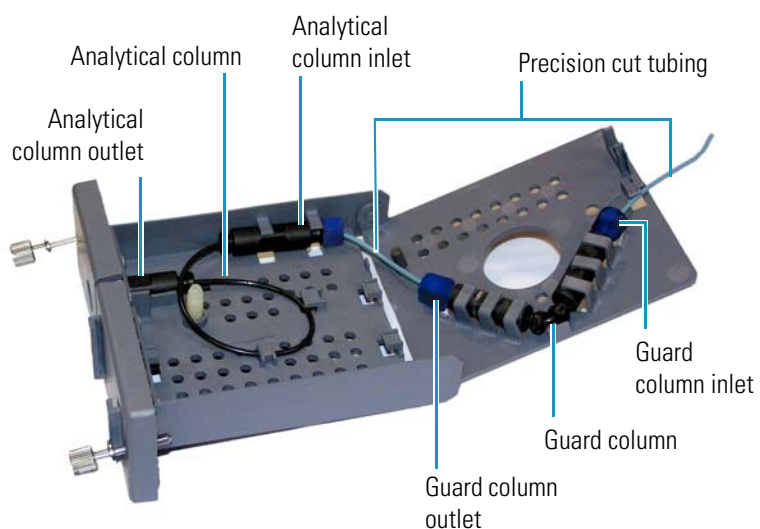
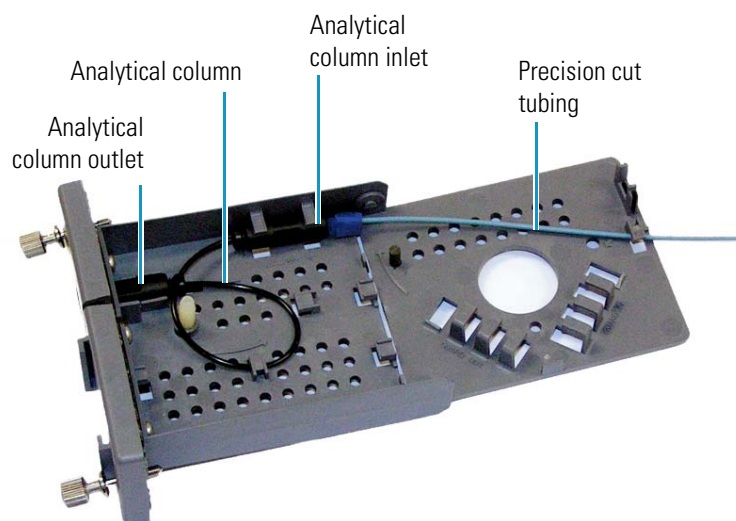
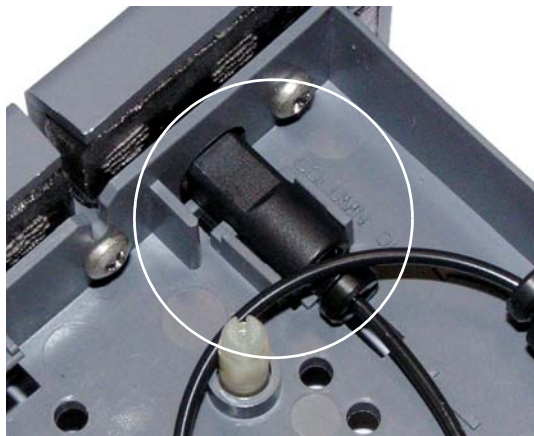


Figure 82. Analytical column only installed in column cartridge



7. Lift up the analytical column outlet fitting (see [Figure 83](#)) slightly to free it from the clips and then slide the fitting out of the cartridge.

Figure 83. Analytical column outlet fitting



8. Remove the columns from the cartridge.
9. Disconnect the tubing from the columns. Save the tubing.

IMPORTANT Do not cut the tubing that you removed from the columns and do not substitute different tubing. To ensure good chromatographic results, capillary tubing is cut at the factory using a specialized precision cutter. If you need to replace the tubing or to install a different length tubing (to accommodate a new column configuration), order a Dionex IC CubeTubing Kit (P/N 072186).

10. If the new column configuration is the same as the previous, connect the precision cut tubing that you removed in [Step 9](#) to the new columns.
11. If you are changing the column configuration, connect the precision cut tubing required for your configuration. See the following figures.

Figure 84. Tubing connections for 250 mm analytical column and 50 mm guard column

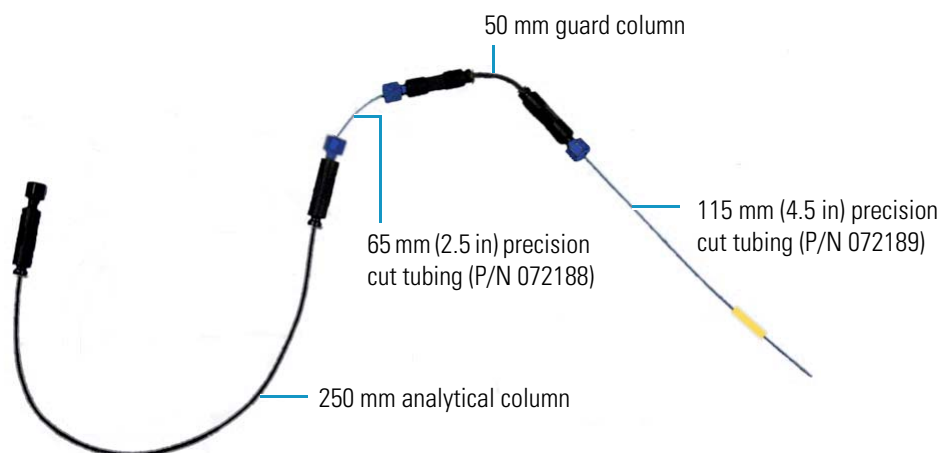


Figure 85. Tubing connections for 150 mm analytical column and 35 mm guard column

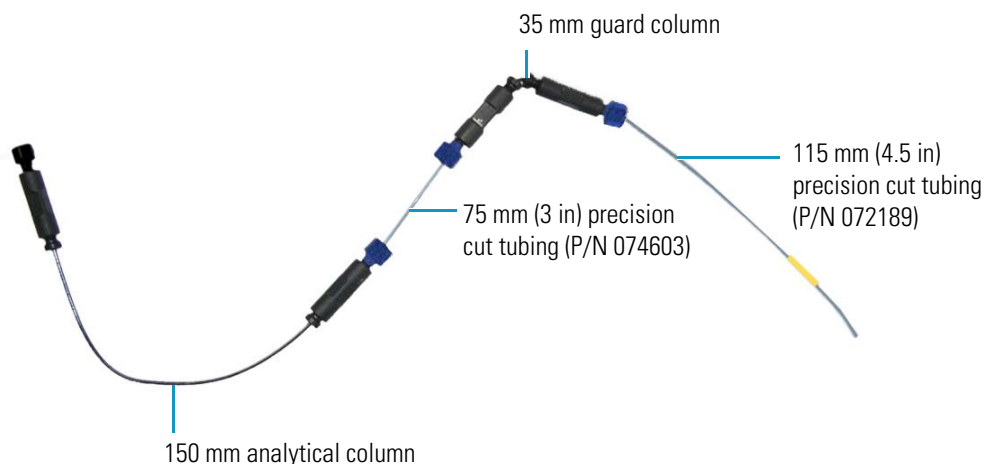
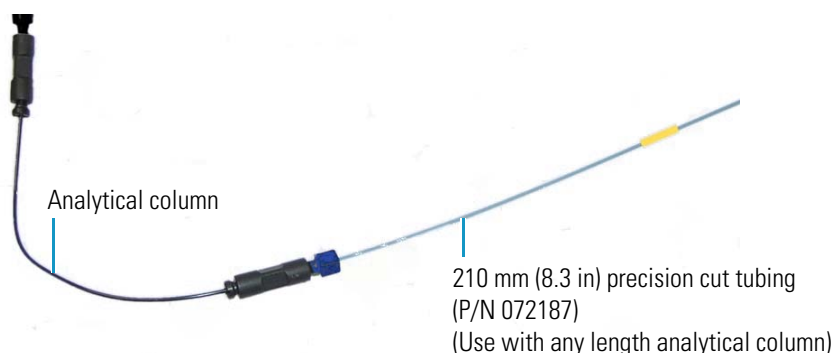


Figure 86. Tubing connections for analytical column only



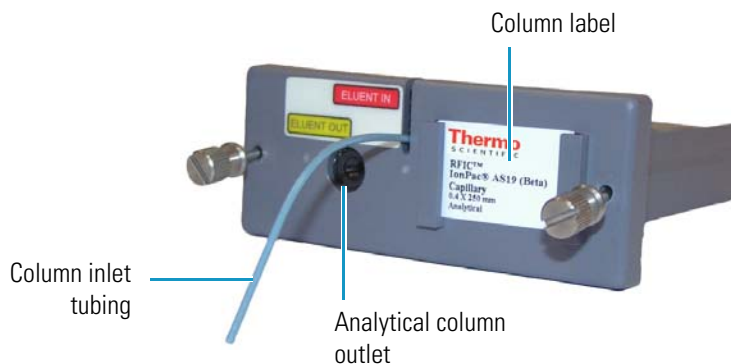
❖ **To install the new columns in the column cartridge**

IMPORTANT Before continuing, refer to the manual for the column you are installing. Make sure to complete all startup steps described in the manual. Column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

1. Before installing the new analytical column, tear off the column label and slide it into the holder on the front of the cartridge (see [Figure 87](#)).
2. Remove the fitting plug from the outlet fitting on the analytical column. Orient the fitting with a flat side up and push the fitting into the opening at the front of the column cartridge until it stops (see [Figure 83](#)).
3. Coil the analytical column tubing inside the cartridge as shown in [Figure 81](#) or [Figure 82](#). Secure the column and the inlet fitting in the clips on the column cartridge.
4. If a guard column is used, secure the inlet and outlet fittings on the guard column in the column clips on the lid of the column cartridge.

5. Route the column inlet tubing from the guard column (if used) or the analytical column through the clip on the top edge of the cartridge lid.
6. Close the lid (you should hear a click) and route the tubing into the slot on the front of the column cartridge (see [Figure 87](#)).

Figure 87. Column cartridge closed



Note If the columns are installed correctly, the cartridge lid snaps closed easily. If the lid does not close easily, do not force it. Open the lid and verify that the columns and tubing are installed correctly and secured in the clips.

❖ **To reinstall the column cartridge and connect the plumbing**

1. Slide the column cartridge into the Dionex IC Cube column oven and tighten the two thumbscrews.
2. Turn on the pump flow.
3. Wait until liquid is flowing out of the **ELUENT OUT** port of the injection valve.
4. Connect the tubing from the **ELUENT IN** port of the column cartridge to the **ELUENT OUT** port of the injection valve.
5. Wait until liquid is flowing out of the **ELUENT OUT** port of the column cartridge.
6. Reconnect the line to the **ELUENT OUT** port of the column cartridge.
7. Close the compartment door.

Servicing Dionex ICS-4000 Conductivity and Charge Detectors

This section provides instructions for the following procedures:

- “Calibrating a CD Cell” on [page 154](#)
- “Replacing a CD” on [page 156](#)
- “Replacing a Dionex QDC 300 (Capillary) (QDC)” on [page 157](#)
- “Replacing a QD” on [page 158](#)

Calibrating a CD Cell

To calibrate the CD cell, first calibrate the offset and then calibrate the slope.

Note Do not use this procedure to calibrate a new cell. New cells are calibrated at the factory.

❖ To prepare the system

1. Prepare a 1.0 mM KCl solution by dissolving 0.07456 g of reagent-grade KCl in 1 liter of 18 megohm-cm deionized water.
2. Open either the touch screen Conductivity Cell Calibration screen or the Chromeleon Calibration panel.

To open the touch screen, disconnect the Dionex ICS-4000 from Chromeleon. On the touch screen, select **Calibration** on the Menu screen. Under Detector, touch **Conductivity Cell**. The Conductivity Cell Calibration screen opens (see [Figure 88](#)).

To open the Chromeleon panel, open the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. Click the **ICS-4000 CD** tab and click **Detector Calibration**. The Calibration panel opens (see [Figure 89](#)).

Figure 88. Conductivity Cell Calibration touch screen

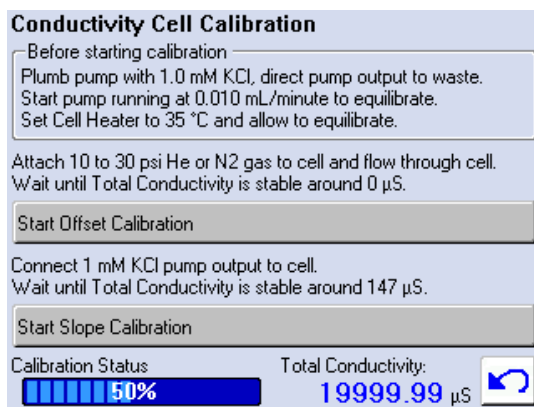
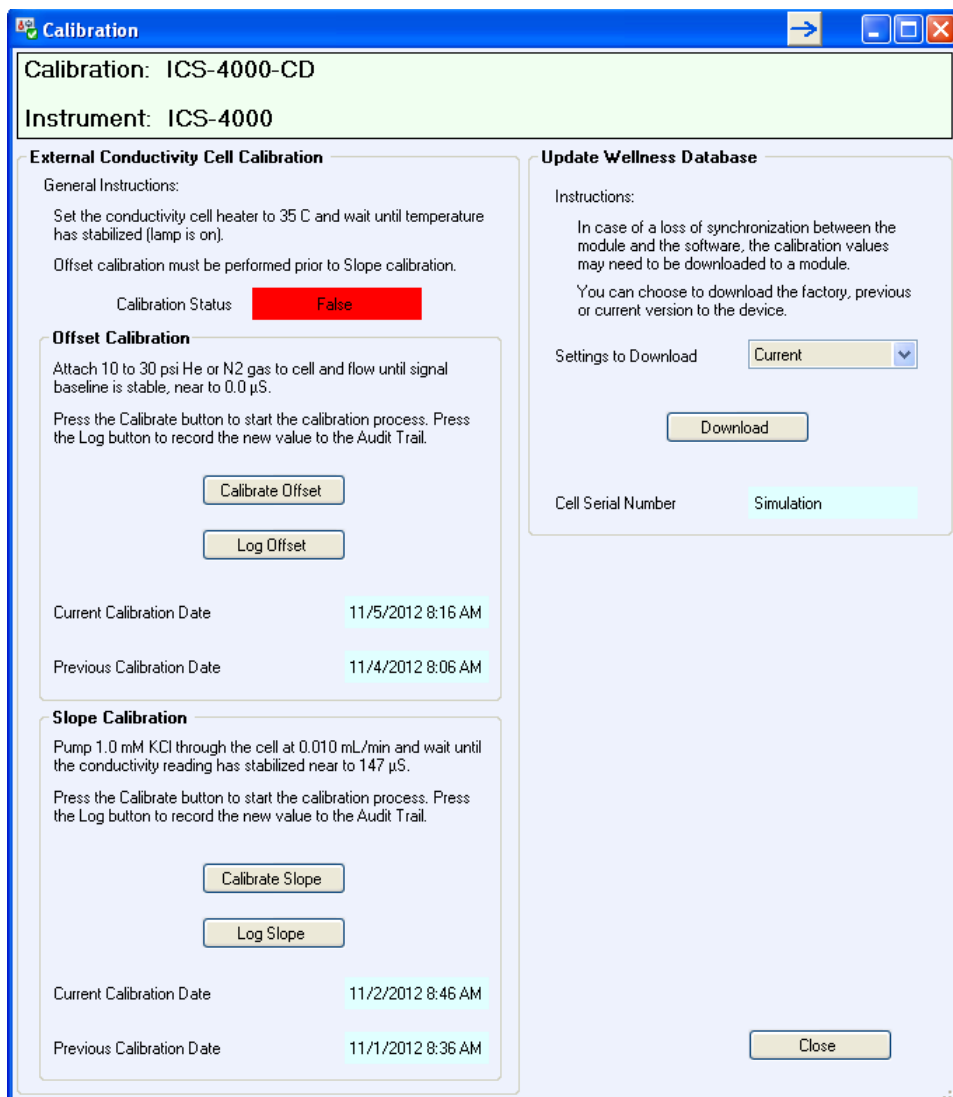


Figure 89. ICS-4000 CD Calibration ePanel (Chromeleon 7 version shown)

3. Connect the pump inlet line to a reservoir filled with 1.0 mM KCL solution.
4. Disconnect the pump output line from the **Eluent In** port of the injection valve. Direct the pump output to waste.
5. Start the pump running at 0.10 mL/min and allow the pump to equilibrate.
6. Set the cell heater to 35 °C and allow the temperature to equilibrate before continuing.

❖ **To calibrate the offset**

1. Connect a source of helium or nitrogen gas to the cell inlet.
2. Turn on the gas pressure to between 0.07 and 0.2 MPa (10 and 30 psi) and allow the signal baseline to stabilize at or near 0.0 μ S.

3. Click **Start Offset Calibration** (on the touch screen) or **Calibrate Offset** (in Chromeleon).
4. If you are using Chromeleon, click **Log Offset** to record the new calibration value in the audit trail.

❖ To calibrate the slope

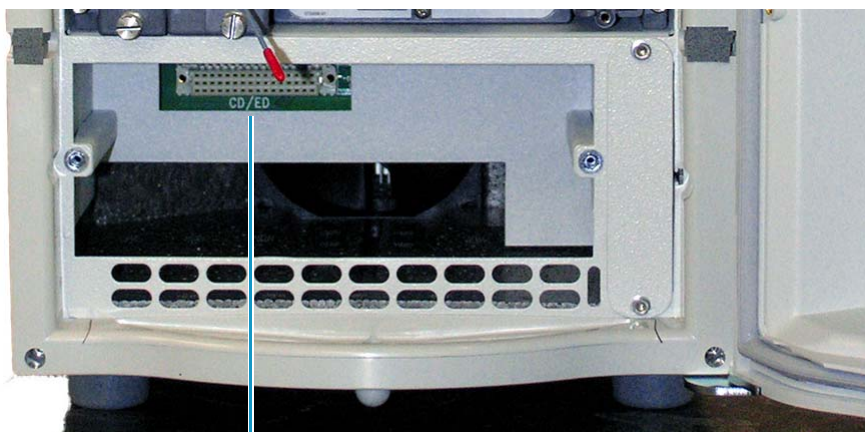
1. Connect the pump output line directly to the cell inlet.
2. Pump 1.0 mM KCl through the cell at 0.01 mL/min.
3. Wait until the total conductivity reading stabilizes at $147.00 \pm 2 \mu\text{S}$.
4. Click **Start Slope Calibration** (on the touch screen) or **Calibrate Slope** (in Chromeleon).
5. After calibration, the conductivity reading should be $147.00 \pm 2 \mu\text{S}$. If this is not the case, repeat the calibration.
6. If you are using Chromeleon, click **Log Slope** to record the new calibration value in the audit trail.
7. Flush the KCl solution from the system by pumping ASTM Type I (18 megohm-cm) filtered and deionized water through the cell. When the conductivity drops to less than $1 \mu\text{S/cm}$, stop the pump.
8. Reconnect the pump to the injection valve and reconnect the line from the suppressor to the cell inlet.

Replacing a CD

❖ To replace a CD

1. Stop the pump flow.
2. Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door and disconnect the tubing from the inlet and outlet ports on the CD cell.
4. If a Dionex ICS-4000 Charge Detector is installed, disconnect the tubing from the **REGEN OUT** port on the Dionex QDC 300 (Capillary) (QDC). Pull the cell straight off the CD.
5. Loosen the two thumbscrews on the CD and pull the detector straight out to remove it from the compartment.
6. Note the electrical plug on the back of the new detector and the receptacle inside the Dionex ICS-4000 (see [Figure 90](#)).

Figure 90. Lower section of the chromatography compartment without a detector



Electrical receptacle for a CD or ED

7. Push the new CD (P/N 075120) into the opening and press firmly to ensure the electrical connection is secure. Tighten the thumbscrews.
8. If included, reinstall the QDC and reconnect the tubing to the **REGEN OUT** port.
9. Reconnect the cell inlet and outlet lines. Close the compartment door.
10. Turn on the system power and restart the pump flow.

Replacing a Dionex QDC 300 (Capillary) (QDC)

❖ To replace a QDC

1. Stop the pump flow.
2. Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door and disconnect the tubing from the **OUT** port on the CD cell and from the **REGEN OUT** port on the QDC.
4. Pull the cell straight off the CD and QD.
5. Hold the new Dionex QDC 300 (Capillary) (P/N 078814) with the label on the left side and press the ball studs on the back of the cell onto the mounting location on the front of the CD and QD (see [Figure 91](#)).
6. Press firmly to ensure the cell's electrical connection to the QD is secure.

7 Service

Servicing Dionex ICS-4000 Conductivity and Charge Detectors

Figure 91. Dionex QDC 300 (Capillary) installed on Dionex ICS-4000 Conductivity Detector



7. Reconnect the tubing. Close the compartment door.
8. Turn on the system power and restart the pump flow.

Replacing a QD

❖ To replace the Dionex ICS-4000 Charge Detector

1. Stop the pump flow.
2. Flip the power switch on the rear panel to turn off the system power.
3. Open the chromatography compartment door and disconnect the tubing from the **OUT** port on the CD cell and from the **REGEN OUT** port on the Dionex QDC 300 (Capillary).
4. Pull the Dionex QDC 300 (Capillary) straight off of the CD and QD.
5. Loosen the two thumbscrews on the QD and then pull the detector straight out of the compartment.
6. Push the new QD (P/N 078449) into the slot and tighten the thumbscrews (see [Figure 92](#)).

Figure 92. QD installed in chromatography compartment



7. Reinstall the Dionex QDC 300 (Capillary) and reconnect all tubing. Close the compartment door.
8. Turn on the system power and restart the pump flow.

Servicing a Dionex ICS-4000 Electrochemical Detector

This section provides instructions for the following procedures:

- “Replacing an ED” on page 159
- “Disconnecting an ED Cell” on page 161
- “Replacing a Disposable Working Electrode Gasket” on page 161
- “Replacing a Conventional Working Electrode Gasket” on page 162
- “Polishing a Conventional Working Electrode” on page 164
- “Replacing a pH-Ag/AgCl Reference Electrode” on page 166
- “Replacing a PdH Reference Electrode” on page 171

Note To prevent contamination, wear cleanroom gloves (lint-free, particle-free, and oil-free) when handling the ED cell. Never touch the electrode surface.

Replacing an ED

❖ To replace an ED

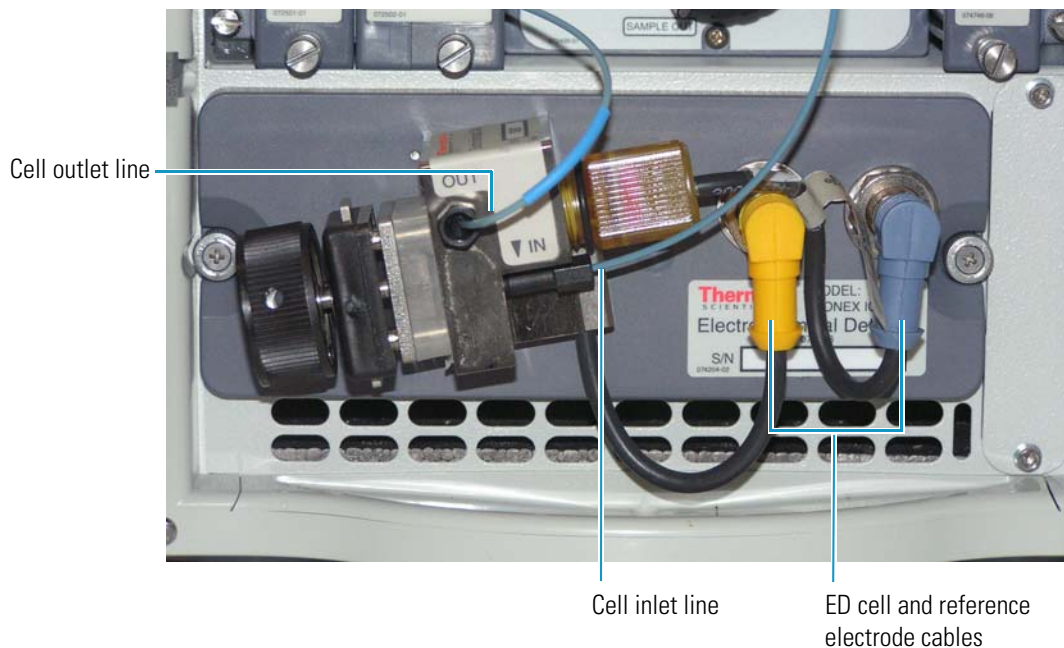
1. Turn off the cell voltage from the Chromeleon panel or the touch screen Electrochemical Detector screen.
2. Stop the pump flow.
3. Flip the power switch on the rear panel to turn off the system power and then open the chromatography compartment door.
4. To prevent contamination, wear cleanroom gloves when handling the ED cell.

7 Service

Servicing a Dionex ICS-4000 Electrochemical Detector

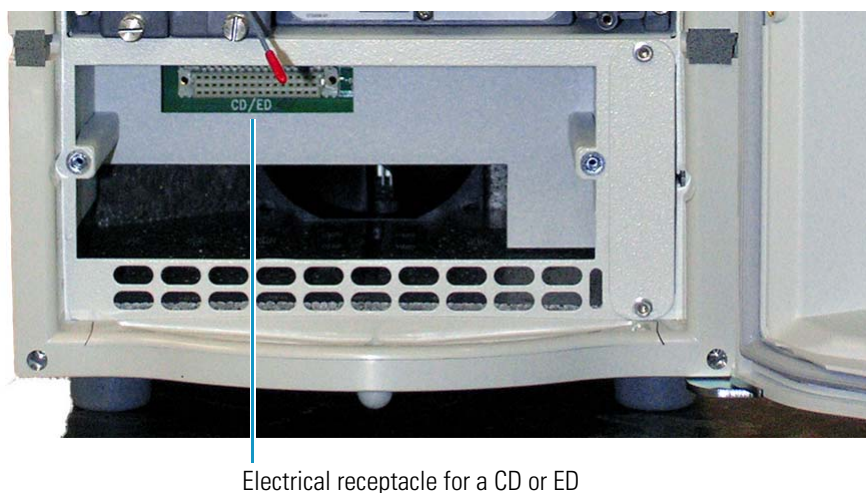
5. Disconnect the cell outlet and inlet lines from the ED cell and disconnect the two electrical cables (see [Figure 93](#)).

Figure 93. ED cell connections (cell with pH-Ag/AgCl reference electrode shown)



6. Grasp the cell body and pull straight out to remove the cell from the detector.
7. Loosen the two thumbscrews on the ED and pull the detector straight out to remove it from the chromatography compartment.
8. Note the electrical plug on the back of the new detector and the receptacle inside the Dionex ICS-4000 (see [Figure 94](#)).

Figure 94. Lower section of the chromatography compartment without a detector



9. Push the new ED (P/N 075121) into the opening and press firmly to ensure the electrical connection is secure. Tighten the thumbscrews.
10. If you are installing an ED in a system that previously was set up for conductivity detection, remove the suppressor cartridge from the IC Cube and replace it with a suppressor bypass cartridge (P/N 072055). For IC Cube cartridge installation instructions, see [page 148](#).
11. Reinstall the cell and reconnect the cell inlet and outlet lines and the cables.
12. Close the compartment door. Turn on the system power, and restart the pump flow.

Disconnecting an ED Cell

Before performing an ED cell service procedure, follow these instructions to disconnect the cell.

Note To prevent contamination, wear cleanroom gloves (lint-free, particle-free, and oil-free) when handling the ED cell. Never touch the electrode surface.

❖ To disconnect an ED cell

1. Turn off the cell voltage from the Chromeleon panel or the touch screen Electrochemical Detector screen.
2. Stop the pump flow and open the chromatography compartment door.
3. Disconnect the cell outlet and inlet lines from the ED cell and disconnect the two electrical cables (see [Figure 93](#)).
4. Grasp the cell by the cell body and pull straight out to remove it from the detector.

Replacing a Disposable Working Electrode Gasket

For installation instructions for disposable working electrodes, refer to the installation guide shipped with the electrodes (listed below) or to the *Product Manual for Disposable Electrodes* (Document No. 065040), which is included on the Thermo Scientific Reference Library DVD (P/N 053891).

- *Disposable Silver Electrode Installation Guide for Dionex ICS-4000 Electrochemical Detector* (Document No. 065137)
- *Disposable Platinum Electrode Installation Guide for Dionex ICS-4000 Electrochemical Detector* (Document No. 065139)
- *Disposable Gold Electrode Installation Guide* (Document No. 065191)

IMPORTANT When you install a disposable electrode, make sure that you install the correct gasket for your system. For the Dionex ICS-4000 Capillary HPIC System, use a 0.001 in PTFE gasket (P/N 072117, Pkg. of 2).

Replacing a Conventional Working Electrode Gasket

Replace the ED cell gasket for conventional working electrodes (P/N 045972) if there is a leak between the gasket and electrode, or between the gasket and cell body.

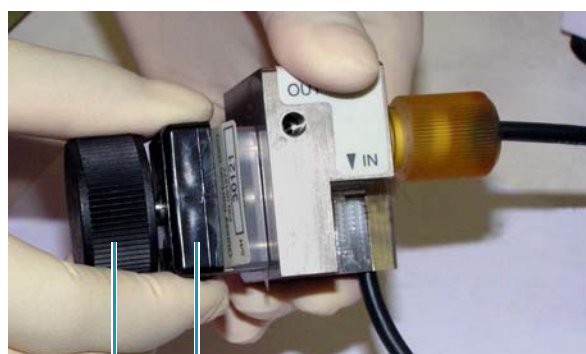
Items needed

- Cleanroom gloves (lint-free, particle-free, and oil-free)
- Blunt-end tweezers

❖ To replace the gasket

1. Follow the instructions in “[Disconnecting an ED Cell](#)” on [page 161](#) to turn off the cell voltage, stop the pump flow, and disconnect the cell from the detector.
2. Unscrew the yoke knob on the yoke block two to three turns to loosen it (see [Figure 95](#)).

Figure 95. ED cell (shown with a pH-Ag/AgCl reference electrode)



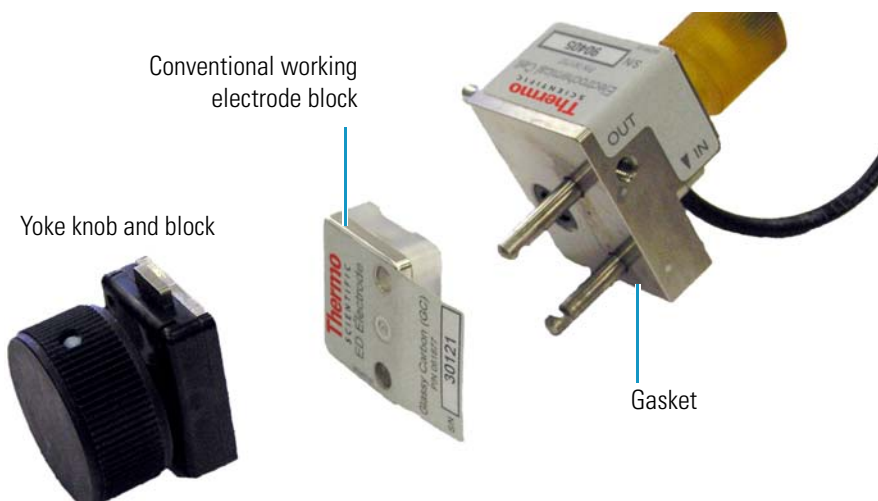
Yoke knob Yoke block

Note Handle the cell gasket and the inside surfaces of the cell carefully to prevent scratches which may subsequently cause leaks.

3. Squeeze the tabs on the sides of the yoke block and pull the block and knob off the working electrode (see [Figure 95](#)).

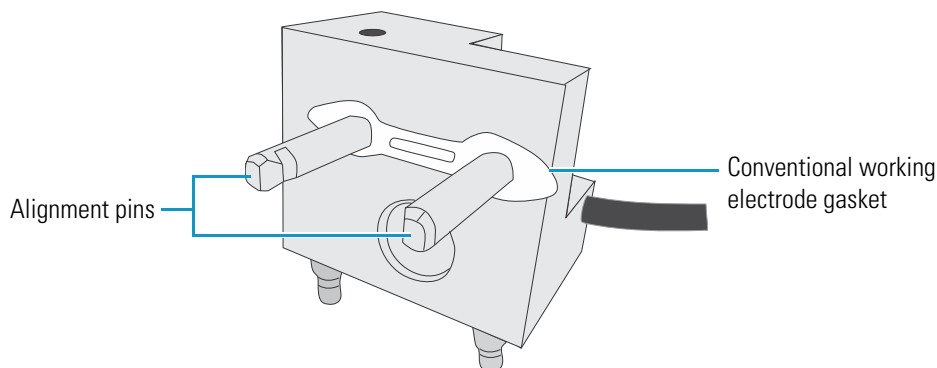
- Carefully separate the parts (see [Figure 96](#)).

Figure 96. ED cell components



- Use tweezers to remove the old cell gasket from the cell body (see [Figure 97](#)).

Figure 97. ED cell gasket for conventional working electrodes



- Rinse the surface of the cell with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in [“Deionized Water Requirements for IC”](#) on [page xvii](#).
- Clean the polished surface of the cell with a clean, damp, lint-free tissue.
- Install the new gasket over the alignment pins on the cell body. When correctly installed, one end of the gasket extends beyond the cell body. This facilitates gasket installation and removal.
- Verify that the gasket is flat against the cell body and is not wrinkled.
- Reinstall the working electrode block and the yoke knob and block. Fingertighten the yoke knob by turning it a full 360 degrees.

Note It is not possible to overtighten the yoke knob. Once the knob clicks into place, it does not tighten any further. The yoke knob ensures constant pressure is applied to the cell.

❖ To reinstall the cell and reconnect tubing

1. Start the pump flow.
2. Wait until liquid is flowing out of the cell inlet line and then reconnect the tubing to the ED cell inlet port.
3. Wait about 60 seconds before tightening the connection.
4. Orient the cell assembly with the yoke knob on the left and then push the cell onto its mounting location on the ED.
5. Reconnect the two electrical cables.
6. Reconnect the tubing to the ED cell outlet port.
7. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.

Polishing a Conventional Working Electrode

These instructions are for conventional (nondisposable) working electrodes only. **Do not polish disposable electrodes.**

Guidelines for Polishing a Conventional Working Electrode

- **Do not polish new conventional working electrodes before installation.**
- After an electrode has been used for a period of time, a layer of contamination may build up. When this occurs, the electrode must be polished.
- After the working electrode is polished and installed, background signal and analyte sensitivity require several hours to stabilize. Once stabilized, do not polish the electrode unless you observe a loss of signal or severe electrode recession.

Items needed

- Polishing kit (P/N 036313) shipped with the electrode. The kit contains polishing pads (P/N 036321), a bottle of fine polishing compound (P/N 036318), and a bottle of coarse polishing compound (P/N 036319).
- Cleanroom gloves (lint-free, particle-free, and oil-free)
- Tweezers

Note To prevent contamination, wear cleanroom gloves (lint-free, particle-free, and oil-free) when handling the working electrode. Never touch the electrode surface.

❖ **To polish a conventional working electrode**

1. Remove the working electrode from the cell. See the instructions in [“Replacing a Conventional Working Electrode Gasket,” Step 1 through Step 4.](#)
2. Prepare the polishing pads:
 - a. Designate a pad for use with the coarse polishing compound.
 - b. Designate another pad for fine polishing compound; also designate the working electrode type with which it will be used.

Note Do not use the same fine polishing pad to polish more than one type of working electrode; this can contaminate the electrode surface with microparticles from the other working electrodes.

- c. Designate a pad that will not be used with polishing compound. This pad is for removal of particles after polishing (see [Step 5](#)).
 - d. Moisten the suede side of the polishing pad slightly with water and place the pad on a smooth, flat surface, with the suede side facing up.
3. Polish the electrode:

Note

- If you are polishing the electrode before initial installation, use only the fine polishing compound.
- If you are polishing the electrode because of degradation of performance, such as increased baseline noise or tailing peaks, first use the coarse polishing compound. Then, repeat with the fine compound.

- a. Sprinkle about one-half gram of polishing compound in the center of the suede side of the polishing pad. Add enough ASTM Type I (18 megohm-cm) filtered and deionized water to make a thick paste.
 - b. Using the working electrode block, spread the paste evenly over the pad. Then, applying firm pressure in a figure eight motion, polish the surface of the electrode block for about 1 minute. If the pad dries out while polishing, add water sparingly. However, never allow the polishing compound to dry on the electrode.
 - c. Use ASTM Type I (18 megohm-cm) filtered and deionized water to rinse off all traces of polishing compound from the electrode block. An ultrasonic cleaner is effective for thoroughly cleaning the electrode block. Carefully rinse the surface of the block with ASTM Type I (18 megohm-cm) filtered and deionized water.
4. If you used the coarse polishing compound in [Step 3](#), repeat the step with the fine compound.

5. Using a moist piece of polishing cloth (with no polishing compound added), rub the polished surface free of residual polishing compound particles.
6. Inspect the surface of the working electrode to make sure that it is clean. Repeat [Step 5](#), if necessary.

Note The polishing pads are reusable. Do not rinse the polishing compound from the pads. After initial use, add only enough polishing compound to maintain the coating on the pad.

❖ To reinstall the cell and reconnect tubing

1. Replace the working electrode block and yoke knob. Fingertighten the knob by turning it a full 360 degrees.

Note It is not possible to overtighten the yoke knob. Once the knob clicks into place, it does not tighten any further. The yoke knob ensures constant pressure is applied to the cell.

2. Start the pump flow.
3. Wait until liquid is flowing out of the cell inlet line and then reconnect the tubing to the ED cell inlet port.
4. Wait about 60 seconds before tightening the connection.
5. Orient the cell assembly with the yoke knob on the left and then push the cell onto its mounting location on the ED.
6. Reconnect the tubing to the ED cell outlet port.
7. Reconnect the two electrical cables.
8. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.
9. Reapply the electrode potential. The baseline will drift for more than 1 hour as the cell re-equilibrates. Peak area values may require up to 12 hours to stabilize.

Replacing a pH-Ag/AgCl Reference Electrode

When to Replace a pH-Ag/AgCl Reference Electrode

Replace the pH-Ag/AgCl reference electrode (P/N 061879) if performance problems occur that are not corrected by regenerating the electrode. Performance problems can include no pH readouts, a shift in Ag/AgCl reference potential or incorrect readouts, baseline spikes, or a decreased response even with a freshly polished working electrode. The pH-Ag/AgCl reference electrode typically lasts from 3 months to 1 year, depending on use.

Note To regenerate a pH-Ag/AgCl reference electrode, soak it in a solution of 1 M KCl and 1 M HCl.

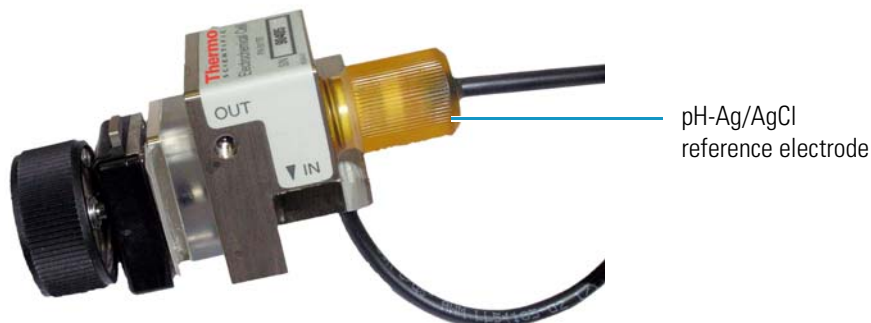
Items Needed

- Cleanroom gloves (lint-free, particle-free, and oil-free)

❖ To replace the pH-Ag/AgCl reference electrode

1. Follow the instructions in “[Disconnecting an ED Cell](#)” on [page 161](#) to turn off the cell voltage, stop the pump flow, and disconnect the cell from the detector.
2. To prevent contamination, wear cleanroom gloves (lint-free, particle-free, and oil-free) when handling the ED cell.
3. Unscrew the pH-Ag/AgCl reference electrode and remove it from the cell body (see [Figure 98](#)).

Figure 98. ED cell with pH-Ag/AgCl reference electrode



4. Unscrew the storage cap from the new pH-Ag/AgCl reference electrode (see [Figure 99](#)). Save the cap.

Figure 99. pH-Ag/AgCl reference electrode with storage cap



IMPORTANT Always store the electrode in the storage cap filled with saturated KCl solution when the cell is not in use. This prevents the pH-Ag/AgCl reference electrode membrane from drying out and damaging the electrode. For storage instructions, see [page 48](#).

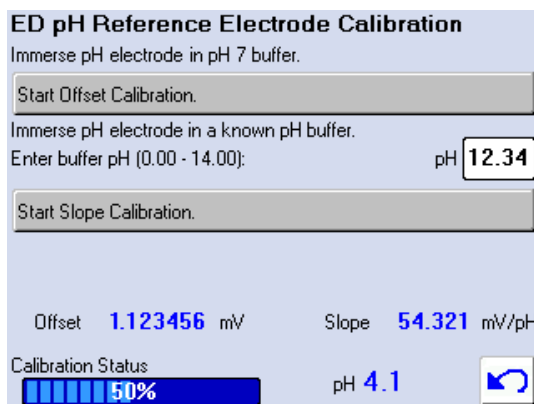
5. Rinse the new electrode thoroughly in ASTM Type I (18 megohm-cm) filtered and deionized water to remove any precipitated salt.
6. Calibrate the pH-Ag/AgCl reference electrode.

❖ **To calibrate the pH-Ag/AgCl reference electrode**

1. Obtain the following items:
 - A buffer solution with a pH of 7.00
 - A second buffer solution with a different pH (typically one that matches the pH of the eluent used in your application; in most cases, this is either pH 10 or pH 4)
 - Cleanroom gloves (lint-free, particle-free, and oil-free)
2. Connect the cell and electrode electrical cables to the detector. Do not install the electrode in the cell yet.
3. Open either the touch screen Calibration screen or the Chromeleon Calibration panel:

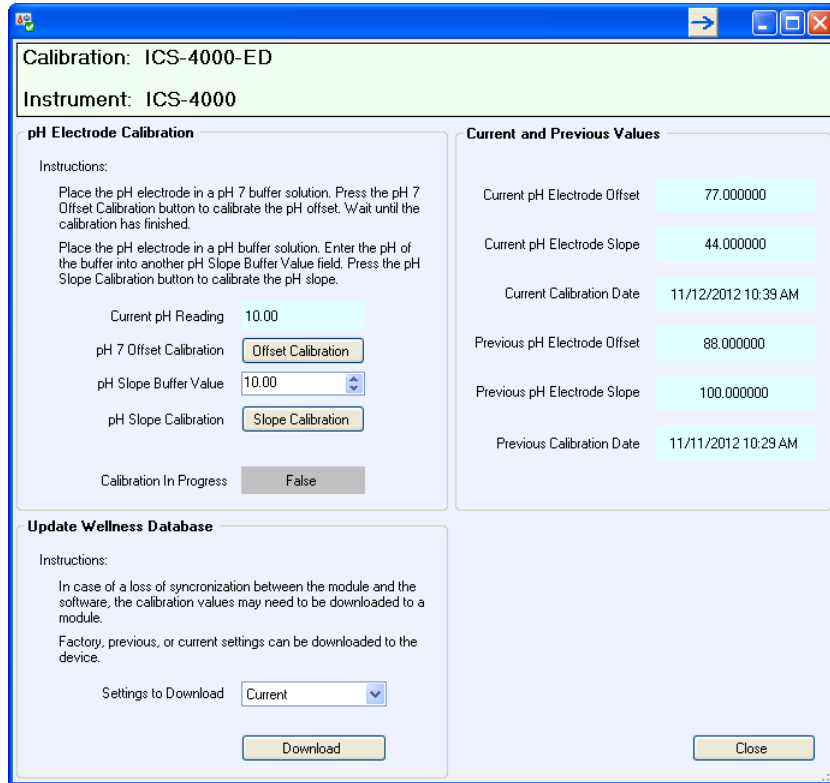
For touch screen operation: Disconnect the Dionex ICS-4000 from Chromeleon. On the touch screen, select **Calibration** on the Menu screen. Under Detector Calibration, touch **pH Reference Electrode**. The ED pH Reference Electrode Calibration screen opens (see [Figure 100](#)).

Figure 100. ED pH Reference Electrode Calibration touch screen



For Chromeleon operation: Open the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset. Click the **ICS-4000 ED** tab and click **pH Calibration**. The ICS-4000 ED Calibration panel opens (see [Figure 101](#)).

Figure 101. ICS-4000 ED Calibration panel (Chromeleon 7 version shown)

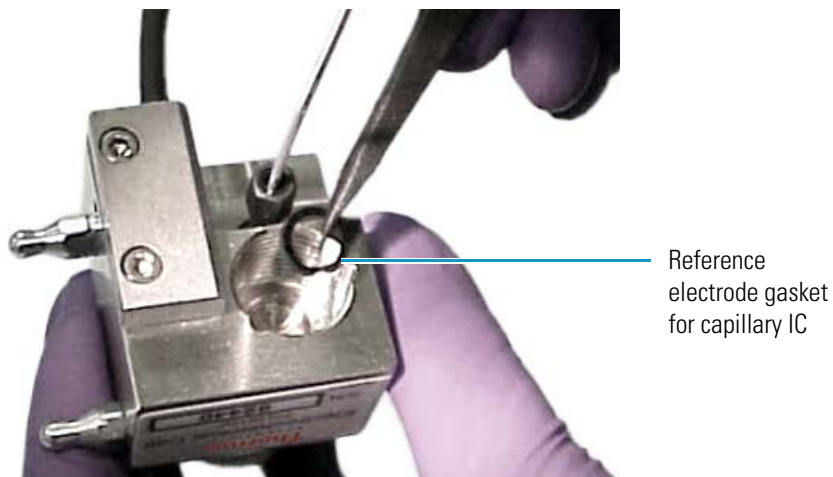


4. Follow the instructions on the screen to calibrate the offset and then the slope. After calibrating the electrode, disconnect the electrode cable and the cell cable.
5. Grasp the cell body and pull straight out to remove the cell from the detector.
6. Install the electrode in the cell (see the following instructions).

❖ **To install the pH-Ag/AgCl reference electrode in the cell**

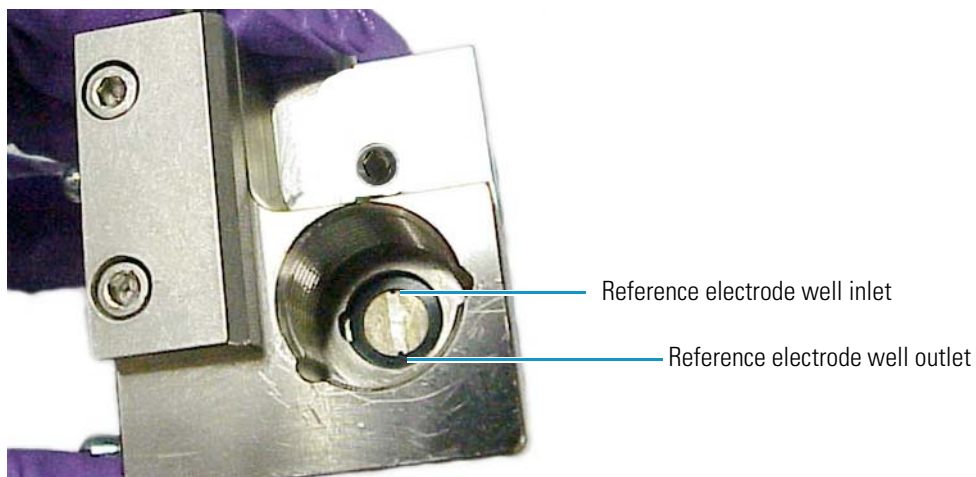
1. To avoid any hydraulic pressure buildup when inserting the reference electrode into the cell, make sure that fitting plugs are not installed on the cell inlet and outlet fittings.
2. Using tweezers, install the reference electrode gasket for capillary IC (P/N 072162) into the bottom of the reference electrode well (see [Figure 102](#)).

Figure 102. pH-Ag/AgCl reference electrode O-ring



3. Make sure the gasket is centered in the bottom of the well and that it does not block the inlet or outlet of the well. The inlet and outlet are small round openings in the bottom of the well (see [Figure 103](#)).

Figure 103. Reference electrode well with gasket for capillary IC installed



4. Verify that the reference electrode O-ring has been removed and the reference electrode gasket is correctly installed in the bottom of the well.
5. Screw the pH-Ag/AgCl reference electrode into the reference electrode well and tighten it fingertight (see [Figure 104](#)).

Figure 104. Installation of the pH-Ag/AgCl reference electrode in the reference electrode well



6. Start the pump flow. Wait until liquid is flowing out of the cell inlet line and then reconnect the tubing to the ED cell inlet port. Wait about 1 minute before tightening the connection fully.
7. Orient the cell assembly with the yoke knob on the left and then push the cell onto its mounting location on the ED.
8. Reconnect the tubing to the ED cell outlet port and reconnect the two electrical cables.
9. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.

Replacing a PdH Reference Electrode

When to Replace a PdH Reference Electrode

- Replace the PdH reference electrode if its sensing surface is damaged or if the electrode no longer seals properly. Replace the PdH electrode if performance has degraded (for example, you observe a lower response, higher background, or spikes).
- The PdH reference electrode typically lasts several years, depending on use.

Items Needed

- PdH reference electrode (P/N 072075)
- Tweezers
- Wrench
- Cleanroom gloves (lint-free, particle-free, and oil-free)

❖ To replace a PdH reference electrode

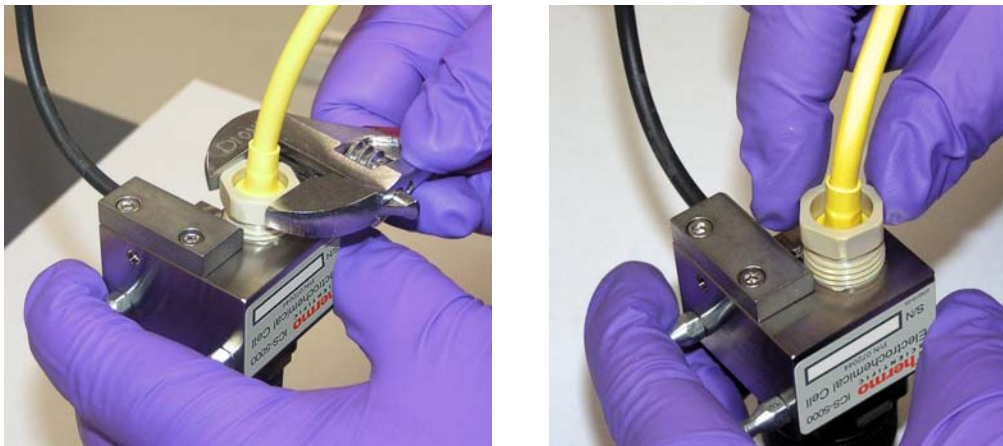
1. Follow the instructions in [“Disconnecting an ED Cell”](#) on [page 161](#) to turn off the cell voltage, stop the pump flow, and disconnect the cell from the detector.

7 Service

Servicing a Dionex ICS-4000 Electrochemical Detector

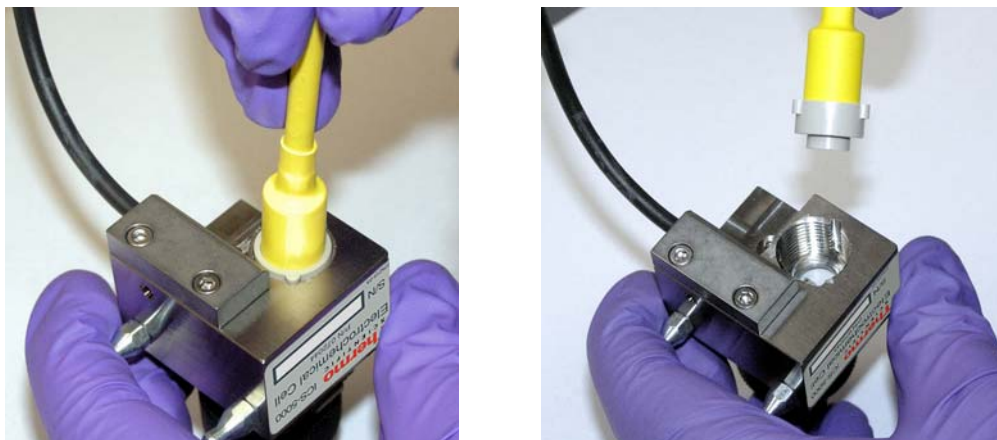
2. Use a wrench to unscrew the PdH reference electrode nut and then remove the nut from the reference electrode well (see [Figure 105](#)).

Figure 105. Removal of the PdH reference electrode nut



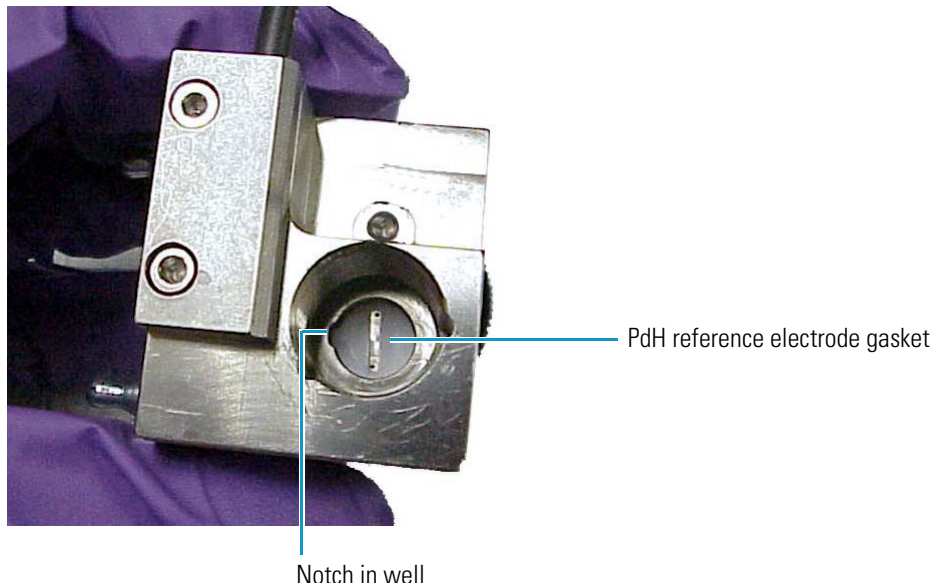
3. Pull the reference electrode out of the well (see [Figure 106](#)).

Figure 106. Removal of the PdH reference electrode



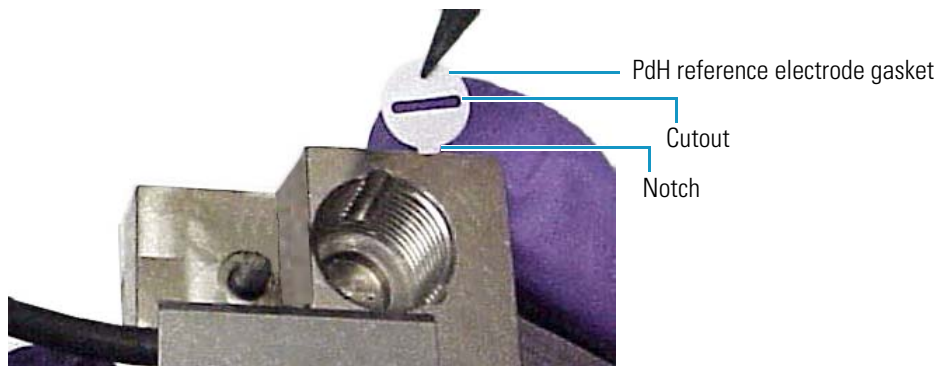
4. Using tweezers, remove the PdH reference electrode gasket from the well (see [Figure 107](#)).

Figure 107. PdH reference electrode gasket installed in the reference electrode well

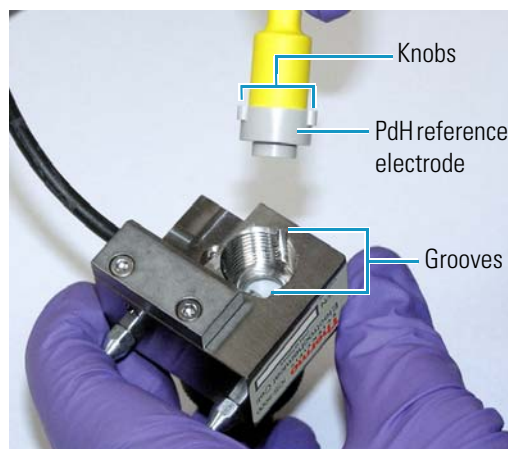


5. Using tweezers, grasp the new PdH reference electrode gasket (P/N 072214) on its edge (see [Figure 108](#)). To avoid deforming the gasket cutout, do not place the tweezer tips on the cutout.

Figure 108. Installation of the PdH reference electrode gasket



6. Align the gasket so that the notched edge of the gasket aligns with the corresponding notch in the reference electrode well and press the gasket into the well.
7. To avoid any hydraulic pressure buildup when inserting the reference electrode into the cell, make sure that fitting plugs are not installed on the cell inlet and outlet fittings.
8. Align the fitting on the end of the PdH reference electrode so that the knobs on the fitting align with the grooves in the reference electrode well (see [Figure 109](#)).
9. Insert the fitting into the well.

Figure 109. Installation of the PdH reference electrode

10. Screw the nut on the PdH reference electrode into the reference electrode well and tighten it fingertight. After fingertightening, use a wrench to tighten the nut an additional 20 to 30 degrees.
11. Start the pump flow.
12. Wait until liquid is flowing out of the cell inlet line and then reconnect the tubing to the ED cell inlet port. Wait about 60 seconds before tightening the connection fully.
13. Orient the cell assembly with the yoke knob on the left and then push the cell onto its mounting location on the ED.
14. Reconnect the tubing to the ED cell outlet port and reconnect the two electrical cables.
15. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.
16. Condition the PdH reference electrode (refer to the instructions below).

Conditioning the PdH Reference Electrode

Condition the PdH reference electrode after plumbing the cell.

❖ To condition the PdH reference electrode

1. On the Chromeleon 7 ePanel Set or Chromeleon 6.8 panel tabset, select the following settings:
 - a. Set the pump flow rate to **0.01 mL/min** and turn on the pump flow.
 - b. Set the concentration to **100 mM** and turn on the EGC.
 - c. Verify that eluent is exiting the cell.
 - d. Set the ED reference electrode type to **PdH** and turn on the PdH power.

- e. Select **DC Amperometry** mode, set the DC voltage to **0.000 V**, and turn on the cell voltage. Switch to **Integrated Amperometry** mode, click **Set Integrated Amperometry**, and select the **Gold, PdH RE, Carbo, Quad** waveform.
2. Condition the electrode for 1 hour at these settings.
3. Turn off the pump flow. This turns off the EGC power and the cell voltage.

Changing the Main Power Fuses

1. If the pump flow is currently on:
 - a. Turn off the flow.
 - b. Begin monitoring the current pressure reading. When the system pressure reaches zero, disconnect the system from the software by clicking **Connected** on the **ICS-4000** panel.
2. Flip the power switch on the rear panel to turn off the system power.
3. Disconnect the main power cord from both its source and from the rear panel.



WARNING HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the Dionex ICS-4000.



CAUTION The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the IC system and is easily accessible.



CAUTION Operation at AC input levels outside of the specified operating voltage range may damage the IC system.



AVERTISSEMENT HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du Dionex ICS-4000.



MISE EN GARDE Le cordon d'alimentation principal est utilisé comme dispositif principal de débranchement. Veillez à ce que la prise de base soit située/installée près du module et facilement accessible.



WARNUNG STROMSCHLAGGEFAHR—Zur Vermeidung von elektrischen Schlägen ist eine geerdete Steckdose zu verwenden. Das Gerät darf nicht ohne Erdung betrieben bzw. an Wechselstrom angeschlossen werden.

7 Service

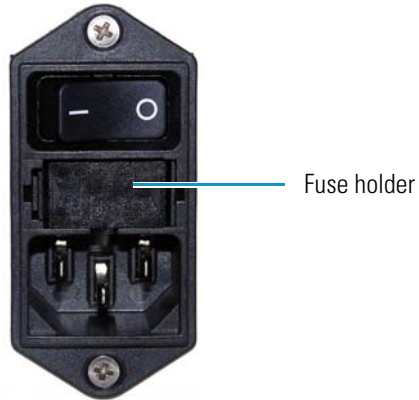
Changing the Main Power Fuses



VORSICHT HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des Dionex ICS-4000.

- The fuse holder is located below the power switch on the rear panel (see [Figure 110](#)). Use a small screwdriver to remove the fuse holder.

Figure 110. Power switch, fuse holder, and power receptacle



- Replace the two fuses with new 5-amp IEC 60127-2 fast-blow fuses (P/N 954793). Thermo Fisher Scientific recommends always replacing *both* fuses.
- Reinstall the fuse holder. Reconnect the power cord and turn on the power.

Specifications

Pump	
Type	Dual-piston (in series), dual-motor, digital cam, microprocessor controlled, variable speed
Construction	Chemically inert, metal-free PEEK pump heads and flow path; compatible with aqueous eluents from pH 0–14 and reversed-phase solvents
Pressure Range	0–41 MPa (0–6000 psi)
Flow Rate Range	0.001–0.100 mL/min with settable flow increments at 0.0001 mL/min, typical working range of 5–20 μ L/min
Flow Rate Precision	< 0.1%
Flow Rate Accuracy	< 0.1%
Pressure Ripple	< 0.2% at 10 μ L/min
Reservoir Pressure	None required; optional regulator assembly available
Vacuum Degasser	Integrated
Piston Seal Wash	Option, automatic operation
RFIC-EG Gradients	0.1–200 mM
Gradient Profiles	Any combination of an unlimited number of linear, convex, and concave positive and negative gradient profiles
Eluent On/Off Valve	Electrically actuated, standard
Leak Sensor	Built-in, optical
Eluent Generator	
Eluent Concentration Range	0.1–200 mM
Flow Rates	0.001–0.100 mL/min
Maximum Operating Pressure	35 MPa (5000 psi)

Eluent Generator

Maximum Solvent Concentration	Cations: None Anions: 25% methanol
Gradient Profiles	Standard – any combination of an unlimited number of linear, convex, and concave positive and negative gradient profiles

Thermal Compartment (Single Temperature Zone)

Temperature Range	10–40 °C (minimum temperature: ambient -15 °C) (maximum temperature: ambient +20 °C)
Temperature Accuracy	±0.5 °C
Temperature Stability	±0.2 °C
Temperature Precision	±0.2 °C

Dionex IC Cube

Components in the Dionex IC Cube	Injection valve EG degasser Analytical column and guard column Carbonate Removal Device Capillary suppressor Capillary column heater: 15–80 °C
Flow Path	Plumbing configuration 0.4 mm columns, fully inert, (PEEK)

Suppression Modes

Electrolytic Suppression, Self-Regenerating, Recycle Mode
Electrolytic Suppression, Self-Regenerating, External Water Mode
Non-Suppressed Conductivity

Conductivity Detector

Electronics Type	Microprocessor-controlled digital signal processing, autoranging
Cell Drive	8 kHz square wave
Linearity	1%
Resolution	0.00238 nS/cm
Output Range	Digital signal range: 0–15,000 µS/cm Analog signal range: 0–15,000 µS/cm

Conductivity Detector, continued	
Noise, Wet	< 0.2 nS at 23 μ S/cm background < 0.1 nS at 1 μ S/cm background
Filter	Rise times 0 to 10 s, programmable
Sampling Rate	1 to 100 Hz, user-settable or automatic
Cell Temperature	5 °C above compartment temperature to 60 °C maximum. User-settable, working range is identical to settable range
Cell Temperature Stability	< 0.001 °C
Cell Temperature Compensation	Default 1.7% per °C; programmable from 0–3% per °C
Flow Cell Maximum Pressure	10 MPa (1500 psi)
Flow Cell Volume	0.02 μ L
Cell Electrodes	Passivated 316 stainless steel, compatible with MSA
Cell Body	Chemically inert polymer
Heat Exchanger	Inert, tortuous-path for low axial dispersion
Electrochemical Detector	
Electronics Type	Microprocessor-controlled digital signal processing
Electronic Noise (Wet Noise)	IPAD (Au electrode) < 50 pC at 10 mM KOH DC Amperometry (GC) < 10 pA at catecholamine eluent
Potential Range	-2.0 to 2.0 V in 0.001 V increments
Signal Range (Digital and Analog)	Integrated Amperometry: 50 pC to 200 μ C DC Amperometry: 5 pA to 74 μ A
Filter	0–10 s response time, user settable
Control Mode	Local or remote control using relay closures or TTL, or control using Chromeleon software
Cell Body	Titanium body with PEEK inlet tubing Compatible with 0.2–0.6 mm ID columns
Working Electrodes	Conventional: gold, glassy carbon, platinum, and silver Disposable: gold, platinum, carbon, and silver
Reference Electrodes	pH-Ag/AgCl combination, one-piece design PdH, one-piece design

Electrochemical Detector, continued

Autoranging	Yes
Analog Output	User-selectable full scale of 10, 100, or 1000 mV
Cell Volume at Working Electrode	< 0.2 μ L
Maximum Cell Operating Pressure	0.7 MPa (100 psi)

Charge Detector

Electronics Type	Microprocessor controlled digital signal processing, autoranging
Linearity	1%
Output Range	Digital signal range: 0–20 μ A Analog signal range: 0–20 μ A
QD Cell Maximum Pressure	0.7 MPa (100 psi)

System Software

Chromeleon Chromatography Data System

Physical Specifications

Power Requirements	100–240 Vac, 50-60 Hz autoranging
Operating Temperature	4–40 °C (40–104 °F); cold-room compatible (4 °C) as long as system power remains on
Operating Humidity Range	5–95% relative, noncondensing
Control Modes	Full control through front panel and Chromeleon software; alternative control through TTL or relay closures; two relay outputs, two TTL outputs, four programmable inputs
USB Communication Protocol	One USB input; one built-in two output USB hub
Dimensions (h × w × d)	56.1 cm × 22.4 cm × 53.3 cm (22.1 in × 8.8 in × 24.5 in)
Weight	25 kg (55 lb)

Reordering Information

Part Number	Item
Pump Head Components	
075189	Primary pump head
075190	Secondary pump head
044541	Inlet check valve assembly (for primary pump head) (includes inlet nut and 1/8 in cartridge)
044520	Outlet check valve assembly (for primary pump head) (includes outlet nut and 1/8 in cartridge)
075493	Main piston seal
074371	Backup ring
040695	O-ring
075191	Seal wash body
068628	Piston seal wash seal
068627	Retainer for piston seal wash seal
068646	Piston
075195	Priming valve knob
078455	Priming valve knob seal
Piston Seal Wash System	
078434	Seal Wash Pump Kit (includes Seal Wash Tubing Kit and reservoir)
078435	Seal Wash Tubing Kit
075300	Seal wash reservoir
Eluent Reservoirs and Accessories	
063291	Eluent reservoir (polypropylene), 1-liter
075313	Eluent reservoir (polystyrene) 1-liter reservoir
062510	Eluent reservoir (plastic), 2-liter
078520	Regulator Kit

Part Number	Item
045987	Filter, end-line
044105	Filter, high-pressure inline
Eluent Generator Consumables	
072076	EGC KOH (Capillary)
072077	EGC MSA (Capillary)
072078	CR-ATC (Capillary)
072079	CR-CTC (Capillary)
Valves and Accessories	
074525	High-pressure injection valve, 4-port
075917	High-pressure injection or switching valve, 6-port
079848	Low-pressure valve, 3-way
061745	Low-pressure valve, 2-way
074698	High-pressure valve rebuild kit, 4-port
075974	High-pressure valve rebuild kit, 6-port
IC Cube and Consumables	
076055	Dionex IC Cube
072186	IC Cube Tubing Kit (includes precision cut tubing for column connections)
074459	EG degas cartridge (capillary); can be used with system pressures up to 35 MPa (5000 psi)
072052	ACES 300 Anion Capillary Electrolytic Suppressor
072053	CCES 300 Cation Capillary Electrolytic Suppressor
072055	Suppressor bypass cartridge
072054	CRD 200 Carbonate Removal Device (Capillary)
072056	CRD bypass cartridge
Detectors and Detector Cells	
075120	Dionex ICS-4000 Conductivity Detector
078449	Dionex ICS-4000 Charge Detector
078814	Dionex QDC 300 (Capillary)
075121	Dionex ICS-4000 Electrochemical Detector (without cell)
072044	ED cell
067541	ED PEEK cell with reference electrode (no working electrode)

Part Number	Item
ED Detector Consumables	
061879	pH-Ag/AgCl reference electrode
072162	Gasket for pH-Ag/AgCl reference electrode (for capillary)
072075	PdH reference electrode
072214	Gasket for PdH reference electrode
061749	ED gold conventional working electrode, with gasket and polishing kit
061751	ED platinum conventional working electrode, with gasket and polishing kit
061753	ED glassy carbon conventional working electrode, with gasket and polishing kit
061755	ED silver conventional working electrode, with gasket and polishing kit
045972	Gasket, PTFE, 0.001 in (for conventional working electrode)
063722	ED AAA gold working electrode, with gasket and polishing kit
060082	Gold AAA-Direct disposable working electrodes (6 electrodes and 2 gaskets)
060139	Gold carbohydrate disposable working electrodes (polyester substrate) (6 electrodes and 2 gaskets)
066480	Gold carbohydrate disposable working electrodes (PTFE substrate) (6 electrodes and 2 gaskets)
064440	Platinum disposable working electrodes (6 electrodes and 2 gaskets)
063003	Silver disposable working electrodes (6 electrodes and 2 gaskets)
069336	Carbon disposable working electrodes (6 electrodes and 2 gaskets)
072117	Gasket for disposable working electrodes, PTFE, 0.001 in (2 gaskets)
062158	ED cell polypropylene support block (for use with disposable electrodes)
036313	Polishing kit for conventional working electrodes
036319	Coarse polishing compound
036318	Fine polishing compound
036321	Polishing pads

Part Number	Item
Tubing and Fittings	
074373	Ferrule fitting, 10-32 double-cone, high pressure
074449	Fitting bolt, 10-32, high pressure (for double-cone ferrule fitting)
062511	Ferrule fitting, 1/16 in
052230	Fitting bolt, 1/16 in
071358	Fitting bolt, 1/16 in (short)
042772	Plug
024305	Luer adapter fitting, 1/4-28
Analog Output/Relay/TTL Accessories	
043598	Twisted pair of wires
924133	Connector plug, 8-position
923686	Connector plug, 12-position
Kits	
075156	Dionex ICS-4000 Preventive Maintenance Kit
082504	External Peristaltic Pump Kit
Miscellaneous Items	
960777	USB cable, 1.8 m (6 ft)
954793	Fuse, 5-amp IEC 60127-2 fast-blow
079803	Syringe, 10 mL
016388	Syringe, 1 mL

Index

Symbols

- % Offset (analog out) 64
- +5V output 32

Numerics

- 10-port high-pressure valve 33
- 4-port high-pressure valve 20
- 6-port high-pressure valve 33

A

- Accessory screen 86
- ACES 300 22, 105
- Acetonitrile 27
- Ag/AgCl half-cell 27
- Air particulate samples 45
- Air pressure requirements 36
- Alarm LED (EG) 109
- Ambient humidity 19
- Ambient temperature 19
- Amperometry cell
 - See ED cell
- Analog output 32
 - Connection instructions 58
 - Connectors 32
 - Description 32
 - Full-scale 64
 - Mark 65
 - Offset level 64
 - Polarity 65
 - Range 64
 - Recorder calibration 64
 - Selecting settings 64
 - Touch screen operation 82
- Analog-to-digital converter 32
- Analyses 47
- Analytical column 22
- Audit trail 92
 - Icons 92

- Autosampler
 - Controlling with relays/TTLs 32
 - Injecting samples 46
 - Loading samples 46
- Autozero 43

B

- Background conductivity
 - High 112
 - Offsetting 43
- Background signal 43
- Backlight on touch screen 87
- Backpressure, excessive
 - Causes of 108
- Backpressure, low
 - Causes of 107
- Baseline
 - Noisy or drifting 110
 - Spikes 111
- Baseline, monitoring 44
- Batch (Chromleon) 48
- Bottles
 - See Reservoirs

C

- Calibration
 - CD cell 154
 - pH-Ag/AgCl reference electrode 168
 - Recorder (analog) output 64
- Capillary IC applications
 - CR-TC trap column for 18
 - Replacing capillary columns 149–153
- Capillary tubing
 - Installation guidelines 123
- Carbonate removal device (CRD) 23
- CCES 22
- Charge Detector (QD) 25, 29
 - Replacement procedure 158
 - Touch screen operation 79

Index: D

Check valve replacement 124
Chromatogram 2
 Dips instead of peaks 65
 Water dip in 45
Chromleon 10
 Audit trail 92
 Command window 13
 Connecting to 39
 Control modes 12
 Creating sequences 47
 Instrument method 47
 Panels 11
 Programs 47
 Troubleshooting communication problems 105
 USB connections 31
 Virtual Column Separation Simulator 43
Column cartridge 22
Column heater 19, 22
Columns 22
Command window 13
Commands
 Creating a program 47
Compartment fan speed 87
Compartment temperature control 19
Compliance
 FCC iii
 Regulatory iii
 WEEE v
Concentration of eluent 49
Conductivity Detector (CD) 25–26
 Cell calibration 154
 Replacement procedure 156
 Temperature control 26
 Temperature control of cell 19
 Touch screen operation 77
Consumables
 Storage of 48
Contacting Technical Support 91
Continuously Regenerated Trap Column (CR-TC) 17–18
 Contamination 112
 Replacement procedure 144, 146
Counter electrode 27
CR-ATC (Capillary) 18
CR-CTC (Capillary) 18
CRD bypass cartridge 23
CRD cartridge 23

D

Daily maintenance 89
Data analysis description 2
Data rise time 78

Degasser 14
Detection description 2
Detector output
 Low 113
Detectors 25
 Charge Detector (QD) 29
 Conductivity Detector (CD) 26
 Electrochemical Detector (ED) 26
Disposable working electrodes 27
Drifting baseline 110

E

ED cell
 Cell design 27
 Description 27
 Electrodes 27
 Monitoring the pH readout 28
 Operating precautions 51
 Solvent compatibility 27
 Storage of 48
EG degas cartridge 17
EGC
 See Eluent generator cartridges
Electrochemical Detector (ED) 25
 Detection modes 26
 ED cell description 27
 ED replacement procedure 159
 Operating 50
 Operating modes 53
 Palladium hydrogen (PdH) reference electrode 28
 pH-Ag/AgCl reference electrode 27
 Touch screen operation 80
 Waveforms 56
 Working electrode gasket replacement 162
 Working electrodes 27
 See also ED cell
Electrodes 27
 Reference 27
 Working 27
Electrolysis gases 17
 Purging from eluent 20
Electrolytics 50
Electromagnetic compatibility iii
Eluent bottles
 See Reservoirs
Eluent Generator (EG) 17
 Alarm LED is lighted 109
 Component description 18
 Concentration selection 49
 Degas cartridge 20
 Electrolysis gases 20
 Operating 49

- Touch screen operation 74
- Eluent generator cartridges 18
 - Concentration ranges allowed 49
 - Disposal 139
 - Entering the concentration 50
 - Leaks 107
 - Replacement procedure 136–140, 143
 - Storage 139
- Eluent reservoirs
 - See Reservoirs
- Eluent valve 14

Eluents

- Aqueous 89
- Delivery process 2
- Flushing lines after eluent change 115
- KOH 49
- Monitoring pH 27
- MSA 49
- Vacuum degasser 14
- EMC compliance iii
- End-line filters 36
- ePanel Set 11
- Equilibration 43
- Error messages 92

F

- Fan speed 87
- Fast ramping 15
- FCC compliance iii
- Ferrule fittings
 - Tightening 123
- Filter, end-line 36
 - Bacterial contamination 89
 - Dirty or clogged 89
- Fittings 122
 - High-pressure 123
 - Tightening 123
- Flow schematics
 - Electrochemical detection 9
 - IC Cube 23–25
 - Injection valves 22
 - Low-pressure valves 34
- Full-scale analog output 64
 - Response range 64
- Fuses 31
 - Replacement procedure 175

G

- Gas regulator 30, 37
- Glassy carbon electrodes 27
- Gold electrodes 27

- Guard column 22

H

- Half-cells 27
- High pressure limit tripped 108
- High-pressure valve
 - Rebuilding 146
 - See also Valves
- Home screen 70
- Humidity 19

I

- I/O connectors
 - Installation instructions 58–59
- IC Cube 19–20
 - Cartridge replacement procedure 148
 - Column cartridge 22
 - Column heater 22
 - CRD bypass cartridge 23
 - CRD cartridge 23
 - EG degas cartridge 20
 - Flow schematic for conductivity detection 23
 - Suppressor bypass cartridge 22
 - Suppressor cartridge 22
 - Tubing and fittings 122
- Information screen 88
- Injection valve 20
 - Flow schematics 21–22
 - Rebuilding 146
 - Replacing mechanical parts (pod) 147
 - Touch screen operation 76
- Instrument method (Chromleon) 47
- Integrated and pulsed amperometric detection (IPAD) 54
- Ion chromatography overview 1
- Ion exchange 2
- IonPac ATC-HC Trap Column 18
- IonPac CTC-1 Trap Column 18
- IPAD 54

K

- KOH eluent 18, 49

L

- Leak sensor 16
- Loading samples
 - With a syringe 46–47
 - With an autosampler 46
- Low detector output 113
- Low pressure limit tripped 107

Index: M

Low-pressure valves 30, 32, 34
Flow schematic 34

M

Main power receptacle 31
Main power switch 31
Maintenance
Daily 89
Weekly 89, 110
Yearly 90
Manual injections 46–47
Mark (analog output) 65
Methanesulfonic acid eluent (MSA) 18, 49
Methanol 27
Mobile phases
See Eluents
Monitoring background 43

N

Nitrite in samples 45
Noisy baseline 110
Number keypad 71

O

Offset percentage (analog out) 64
Offsetting background conductivity 43
Operation 35
Creating sequences 47
Equilibrating the system 43
Performing analyses 47
Priming the pump 41
Reservoir setup 36
Sample preparation 44
Selecting eluent concentration 49
Setting operating parameters 43
Shutting down 48
Touch screen 69
Organic solvent 27

P

PAD 55
Palladium hydrogen (PdH) reference electrode 28
Replacement procedure 171
Waveforms for 56
Panel Tabset 12
Panel tabset 12
Panels
Chromeleon 11
Parameters

Chromeleon commands 47
Peaks
Reversed 65
Tailing 114
Peristaltic pump 33
Controlling 66
pH gradient 27
pH half-cell 27
pH reading
Adjusting potentials for 28
Effect on potentials 28
Monitoring 28
Setting pH limits 52
pH-Ag/AgCl reference electrode 27
Calibration procedure 168
Monitoring the pH 51
Preventing from drying out 28
Replacement procedure 166
See also Reference electrodes
Piston replacement 132
Piston seal replacement 127
Piston seal wash 17, 39
Platinum working electrodes 27
Plot screen 82
Polarity
Analog output 65
Potassium hydroxide eluent 18
Potential shifting 28
Power output 32
Power receptacle 31
Power switch (rear panel) 31
Powering up the system 39
Pressure limit tripped 107–108
Pressure transducer 16
Pressurizing reservoirs 37
Priming the pump 41
Priming valve
Leakage from knob 106
Priming valve seal replacement 134
Problems
See Troubleshooting
Processing samples 47
Product warranty, voiding 121
Programs (Chromeleon)
Creating programs 47
Proportioning valves
Leaks 106
Malfunction 115, 117
Pulse damper 16
Pulse damper heater 19
Pulsed amperometric detection (PAD) 55

Pump 15
 Components 16
 Fast ramping 15, 73
 High pressure limit tripped 108
 Low pressure limit tripped 107
 Piston seal wash 17
 Priming 41
 Seal wash 39
 Touch screen control 72
 Troubleshooting 108
 Pump check valve replacement 124
 Pump piston replacement 132
 Pump piston seal replacement 127
 Pump priming valve seal replacement 134
 PWM drive 33
 Connection instructions 67
 Controlling accessories with 66
 Touch screen operation 86

Q

QD 29
 QDC 300 Cell (Capillary)
 Replacement procedure 157
 Queue (Chromeleon 7) 48

R

Rear panel 30
 Analog outputs 32
 I/O connectors 57
 Power switch 31
 TTL and relay connectors 32
 USB connections 31
 Waste lines 33
 Recorder
 Analog output setting 64
 Calibrating 64
 Redox reactions 28
 Reference electrodes
 Palladium hydrogen (PdH) 28
 pH-Ag/AgCl 27–28
 Reference potential shift 28
 Regulatory compliance iii
 Relay connectors 32
 Configuring 32
 Installation instructions 58–59
 Relay outputs 32
 Connections for normally open/normally closed 59
 Controlling 32, 63
 Reproducibility
 Affected by amperometry cell pH 28
 Reproducibility is poor 112

Reservoirs 13
 Cleaning 110, 116
 End-line filters 36
 Pressurizing 37
 Setting up 36
 Retention time
 Troubleshooting 116–117
 Reversed-phase solvents 27
 Running samples 47

S

Safety standards iii
 Sample analyses 47
 Sample loop 20
 Injecting 21
 Sample processing 47
 Samples
 Collecting and storing 44
 Diluting 45
 Filtering 44
 Pretreating 45
 Seal wash pump 17
 Setting up 39
 Touch screen operation 86
 Separation process 2
 Separator column 22
 Sequences 47
 Service procedures 121
 Capillary columns replacement 149–153
 CD cell calibration 154
 CD replacement 156
 CR-TC replacement 144, 146
 ED replacement 159
 EGC replacement 136–140, 143
 Fuse replacement 175
 IC Cube replacement 148
 Injection valve pod replacement 147
 Palladium hydrogen (PdH) reference electrode
 replacement 171
 pH-Ag/AgCl reference electrode replacement 166
 Pump check valve replacement 124
 Pump piston replacement 132
 Pump piston seal replacement 127
 Pump priming valve seal replacement 134
 QD replacement 158
 QDC 300 Cell (Capillary) replacement 157
 Replacing capillary columns 152
 Setup screen 87
 Ship Kit 31
 Short-term storage
 ED cell 48
 Shutdown 48

Index: T

- Silver electrodes 27
- Solvent compatibility (ED) 27
- Start-up procedures
 - Offsetting the background 43
- Sulfite in samples 45
- Support 91
- Suppressor
 - Touch screen operation 75
- Suppressor bypass cartridge 22
- Suppressor cartridge 22
- Syringe
 - Manual injections 46–47
 - Using to prime pump 41
- System flow descriptions 7
- System power-up 39
- System shutdown 48

T

- Technical Support 91, 121
- Temperature control 19
 - Column heater 19
 - Compartment 19
 - Conductivity cell 26
 - Conductivity Detector (CD) cell 19
 - Pulse damper 19
 - Touch screen operation 76
- Three-way valves 34
- Tightening fittings 123
- Touch screen operation 10, 69
 - Analog output options 82
 - Backlight duration 87
 - Charge Detector (QD) 79
 - Conductivity Detector (CD) 77
 - Electrochemical Detector (ED) 80
 - Eluent generator 74
 - Fan speed selection 87
 - Home screen 70
 - Information screen 88
 - Injection valve 76
 - Menu screen 70
 - Number keypad 71
 - Overview 69
 - Plot screen 82
 - Pump control 72
 - PWM drive 86
 - Seal wash pump 86
 - Setup screen 87
 - Suppressor 75
 - Temperature control 76
 - TTL/Relay screen 84
 - Using with Chromeleon 71
 - Valve control 85

- Troubleshooting 91
 - Chromatographic reproducibility 112
 - EG stops operation 109
 - Error message 92
 - High background 112
 - Low detector output 113
 - Noisy baseline 110
 - Pump stops unexpectedly 108
 - Retention times too early 116
 - Retention times too late 117
 - Tailing peaks 114
 - USB connections 105
 - Vacuum degasser low vacuum 109
- TTL and relay connectors 32
 - Installation instructions 58–59
- TTL and relay control 32
 - Touch screen operation 84
- TTL inputs
 - Assigning functions to 60
 - Control types 62
 - Functions available 32
- TTL outputs 32
 - Controlling 32, 63
- TTL/Relay control 32
- Tubing and fittings
 - Capillary tubing guidelines 123
 - Replacing 122

U

- USB cable 31
- USB connections 31
 - Troubleshooting 105

V

- Vacuum degasser 14
 - Controlling 14
 - Vacuum is low 109
- Valves
 - 10-port high-pressure 33
 - 4-port high-pressure (injection) 20
 - 6-port high-pressure 33
 - Eluent valve 14
 - Injection 20
 - Low-pressure 30, 34
 - Rebuilding high-pressure valves 146
 - Replacing a high-pressure valve pod 147
 - Touch screen operation 85
 - Two-way, three-way 34
- Virtual Column Separation Simulator 43

W

Waste lines 33

Water dip 45

Water samples 45

Waveforms 56

For PdH reference electrode 56

WEEE compliance v

Weekly maintenance 89

Working electrode gasket

Replacement procedure 161–162

Working electrodes 27

Materials 27

Polishing conventional electrodes 164

Y

Yearly maintenance 90

Z

Zero position (analog out) 64

