

ICS-3000 Ion Chromatography System Operator's Manual

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DP/SP Reordering Information

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EG Reordering Information

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TC Reordering Information

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1.1 **ICS-3000 System Overview**

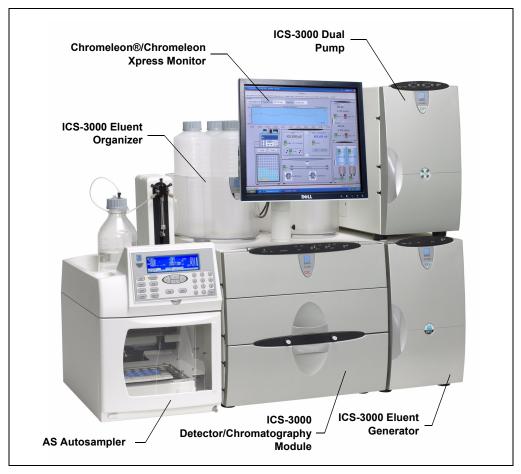


Figure 1-1. ICS-3000 System for Dual-Analysis RFIC

The Dionex ICS-3000 Ion Chromatography System offers a full range of Reagent-Free™ IC (RFIC™) components. RFIC combines automated eluent generation and self-regenerating suppression to make IC easier and more powerful than ever

before. It is no longer necessary to spend time preparing eluents and regenerants. All you need is deionized water—the IC system automatically generates eluent in the exact amount and concentration needed for your application, ensuring superior analytical results.

The dual-analysis capabilities (both simultaneous and sequential) of the ICS-3000 system let you maximize efficiency and throughput and minimize downtime. The modular system design lets you quickly configure and customize hardware.

1.1.1 ICS-3000 System Components

The table below identifies modules in the ICS-3000 product line, as well as additional products that can be added to an ICS-3000 system. Refer to the page number indicated here for a brief product overview.

Product Type	Product Name	
Pump	ICS-3000 Dual Pump (see page 3)	
	ICS-3000 Single Pump (see page 3)	
Detector/Chromatography Compartment	ICS-3000 Detector/Chromatography Module (see page 3)	
Detector (inside DC)	ICS-3000 Conductivity Detector (see page 4)	
	ICS-3000 Electrochemical Detector (see page 4)	
Accessory (inside DC)	ICS-3000 Automation Manager (see page 4)	
Detector (outside DC)	ICS-Series Photodiode Array Detector (see page 7)	
	ICS-Series Variable Wavelength Detector (see page 7)	
Thermal Compartment	ICS-3000 Thermal Compartment (see page 4)	
Eluent Generator	ICS-3000 Eluent Generator (see page 5)	
Eluent Organizer	ICS-3000 Eluent Organizer (see page 5)	

Autosampler	AS Autosampler (see page 6)	
	AS40 Automated Sampler (see page 7)	
Mass Spectrometer	MSQ Plus TM (see page 8)	

ICS-3000 Dual Pump (DP) and ICS-3000 Single Pump (SP)

With flow rates ranging from 0.001 to 10.0 mL/min and operating pressures up to 35 MPa (5000 psi), the DP/SP is designed for both standard bore and microbore applications. The pump's patented isokinetic pre-compression phase allows a precise, almost pulse-free flow.

The SP contains one isocratic pump *or* one gradient pump. The DP contains two gradient pumps, two isocratic pumps, *or* one isocratic pump and one gradient pump.

The isocratic pump delivers one eluent, while the gradient pump can deliver gradient mixtures of up to four eluent components. The selected eluent composition can be delivered as isocratic, isocratic proportioned, linear ramp, step, curved, or any combination of these.

With a DP, the second pump can be operated as a second-channel chromatography pump, an auxiliary dependent pump, or an auxiliary independent pump.

ICS-3000 Detector/Chromatography Module (DC)

The DC provides a temperature-controlled environment for ICS-3000 chromatography components. The DC can accommodate components for two channels, plumbed either serially or in parallel.

The following components may be installed in the DC:

- Conductivity detector
- Electrochemical detector
- Injection valves
- Switching valves
- Guard and separator columns
- Suppressors
- ICS-3000 Automation Manager

ICS-3000 Conductivity Detector (CD)

The CD is a modular detector with integrated cell providing dual detection capabilities (series or parallel). The CD has a signal range up to 15,000 μ S and supports high background, nonsuppressed applications. The CD is installed in the upper compartment of the DC.

ICS-3000 Electrochemical Detector (ED)

The ED is a modular detector and cell with dual-detection capabilities (series or parallel). The ED supports multiple waveforms, multiple integration times, and post-analysis data manipulation. The ED is also capable of providing 3D amperometry data. The ED cell can be configured with gold, silver, platinum, or glassy carbon working electrodes. The ED is installed in the upper compartment of the DC.

ICS-3000 Automation Manager (AM)

The AM consists of a component mounting panel on a base tray. The AM provides mounting sites for sample preparation and post-column application components: high-pressure (switching) valves, low-pressure (solenoid) valves, reaction coils, etc. The AM is installed in the upper compartment of the DC, above the detector.

ICS-3000 Thermal Compartment (TC)

The TC provides a temperature-controlled environment for ICS-3000 chromatography components. The TC is intended for applications that do not require conductivity or electrochemical detection.

The TC is available in four configurations:

- With one 2-position, 6-port high-pressure injection valve
- With two 2-position, 6-port high-pressure injection valves
- With one 2-position, 6-port high-pressure injection valve and one 2-position, 10-port high-pressure injection valve
- With no injection valves

An optional temperature stabilizer (standard bore, P/N 064548; microbore, P/N 064650) can be installed inside the TC, if necessary. The temperature stabilizer brings the eluent to the column temperature before it enters the column.

ICS-3000 Eluent Generator (EG)

The EG generates high purity acid or base eluents online from deionized water. The EG can be configured for single- or dual-channel operation. Each channel includes:

- A high precision programmable current source (power supply)
- A high pressure gas removal device that removes electrolysis gases created during eluent generation

The following options must be ordered separately for installation inside the EG:

- A disposable EluGen® cartridge to generate eluent. Each cartridge contains 900 mL of the appropriate electrolyte concentrate solution.
- A Continuously Regenerated Trap Column (CR-TC) to remove any extraneous contaminants from the deionized water source. The CR-TC is electrolytically-regenerated, which allows it to operate for extended periods without chemical regeneration.

ICS-3000 Eluent Organizer (EO)

The EO holds eluent reservoirs in a liner that contains spills and leaks. Up to two EOs can be installed on top of the DC. Each EO accommodates up to four 1-liter or 2-liter reservoirs *or* up to two 4-liter reservoirs. The EO is typically ordered configured with four 2-liter reservoirs (P/N 062629).

All eluent reservoirs available for use with the DP/SP can be pressurized. If you plan to pressurize the eluent reservoirs, the optional EO Regulator Accessory and Stand (P/N 063493) is required.

The Regulator Accessory includes a pressure regulator and gauge assembly with four outputs (for connections to four eluent reservoirs), as well as the tubing and connection fitting required. If more reservoirs are required, order a second regulator (P/N 064387).

AS Autosampler (AS)

The AS is a powerful, full-featured autosampler that precisely delivers from 1.0 to 99.9 μL (in 0.1 μL increments) or 100 to 1000 μL (in 1 μL increments) of sample to an injection valve. The autosampler can operate in several modes:

- *Concentrate*—Sample is delivered to a single analytical system for trace analysis and matrix elimination; a concentrator column is installed on the injection valve in place of a loop.
- Simultaneous—Sample is delivered to two analytical systems simultaneously; a sample loop is installed on each injection valve. Sample is delivered equally to the two systems (50% of the volume to each), allowing two complete, separate analyses to be performed with just one sample.
- Sequential—Sample is delivered to two analytical systems in sequence. This allows on-demand injection of two independent samples to two applications, using one autosampler. While the first system is being analyzed, the second system is being prepped and loaded for sample analysis.
- Sequential Concentrate—Sample is delivered to two analytical systems in sequence; a concentrator column is installed on the injection valve in place of a loop.
- Reagent Prime (available in Concentrate mode only)—Primes the lines with reagent. For example, implementing the Reagent Prime mode during the matrix elimination step ensures that the appropriate reagent is in line for matrix elimination and prevents crosscontamination between reagents.
- Reagent Flush (available in Concentrate mode only)—Flushes the concentrator column with reagent (for example, a known source of clean deionized water) to remove unwanted sample matrix.

AS40 Automated Sampler (AS40)

The AS40 is a low-cost, metal-free, sample loading device designed for ion chromatography applications. The AS40 is capable of delivering between 0.2 and 5.0 mL of sample in set increments. The AS40 holds between 66 and 88 vials, depending upon the vial size in use: 0.5 mL, 5.0 mL, or a combination of both sizes. Up to three injections can be taken from each vial.

The AS40 holds up to 11 cassettes of six 5-mL vials or eight 0.5-mL vials. Each sample is filtered during loading through a 20-µm filter in the vial cap, so there is no need to prefilter samples. Samples can be loaded against backpressures up to 690 kPa (100 psi) without an external sampling pump, thus facilitating preconcentration work.

ICS-Series Photodiode Array Detector (PDA)

The PDA optical detector is capable of measuring the absorbance spectrum from 190 to 800 nm. A deuterium lamp optimizes the UV range (190 to 380 nm) and a tungsten lamp optimizes the visible range (380 to 800 nm).

The PDA enables you to collect up to five single wavelengths (2D chromatograms) without being required to collect 3D data. Collecting individual wavelengths instead of the spectra offers two advantages: it eliminates the need to perform extractions for analyses that do not require spectral data and it conserves disk space.

ICS-Series Variable Wavelength Detector (VWD)

The VWD is a dual-beam, variable wavelength photometer with one measurement and one internal reference beam. Spectral capability from 190 to 900 nm is provided by two light sources: a deuterium lamp for ultraviolet detection and a tungsten lamp for visible wavelength operation. The four-channel detector measures at up to four wavelengths simultaneously. The VWD contains a built-in holmium oxide filter for wavelength verification. To suppress higher-order radiation, two optical filters can be inserted (automatically) into the light path.

MSQ Plus Mass Spectrometer

The MSQ Plus is an advanced analytical instrument that includes an MS detector, vacuum pumps, and data system. When integrated with an IC system, the MSQ Plus provides the separation capability of an IC and the detection capability of a single-quadrupole MS detector. This provides a strong starting point for sample analysis by offering a quick and clear mass identification for chromatographic peaks.

The MS detector contains an atmospheric pressure ionization (API) source, advanced high efficiency transmission ion optics of a square quadrupole RF lens and dual RF generators, a mass analyzer, and an ion detection system. An optional cone wash pump is available for improved performance when dealing with dirty matrices.

The MS detector is equipped with FastLockTM probes for two complementary ionization techniques: atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI). Both ionization techniques can be run in either the positive or negative ion polarity mode. The design of the interchangeable probes enables rapid switching of ionization modes.

During a scan, ions of selected mass-to-charge ratios are sequentially transmitted through a quadrupole mass filter analyzer. The MS detector has fully automatic mass scale calibration (15 to 2000 m/z) and tuning that makes optimization simple and direct. The MS detector can perform both full-range scans and selected ion monitoring (SIM) scans. When operating in conjunction with Chromeleon, the MSQ Plus can be set up to vary the scan type, the ionization technique, and the ion polarity mode.

1.1.2 ICS-3000 System Control

The ICS-3000 system is controlled by a PC configured with Chromeleon Chromatography Management System (version 6.7 or later) or Chromeleon Xpress. The Chromeleon Chromatography Management System provides complete instrument control, data acquisition, and data management. Chromeleon Xpress provides real-time control and monitoring of Dionex chromatography instruments, but does not include data management capabilities.

Chromeleon and Chromeleon Xpress provide a *panel tabset* that replaces and combines the module front panels into one centralized system control panel. A convenient **Home** panel (see <u>Figure 1-2</u>) shows the overall system status. Individual tabs provide quick access to module functions, as well as detailed status and diagnostics.

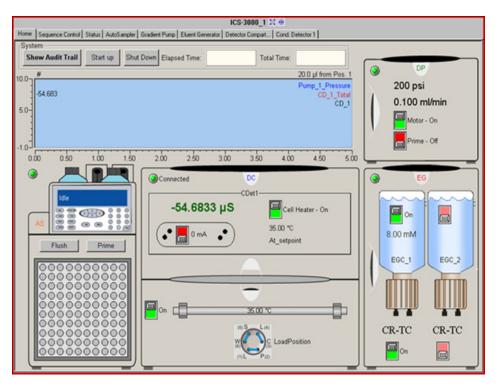


Figure 1-2. ICS-3000 Panel Tabset (Home Panel Shown)

1.2 ICS-3000 System Documentation

Every effort has been made to provide complete and accurate user documentation for the ICS-3000 system. The table below lists the primary sources of product information and the formats in which information is available.

Source	Part Number	PDF File	Printed Manual
ICS-3000 Ion Chromatography System Operator's Manual	065031	Yes	No
Installing the ICS-3000 Ion Chromatography System	065032	Yes	Yes
AS Autosampler Operator's Manual	065051	Yes	No
ICS-Series Photodiode Array Detector Operator's Manual	065147	Yes	No
ICS-Series Variable Wavelength Detector Operator's Manual	065141	Yes	No
Chromeleon Xpress Quick Reference Guide	065070	Yes	No
Chromeleon Xpress online Help	N/A	N/A	N/A
Chromeleon Software User's Guide	4829.5060	Yes	No
Installing the Chromeleon Chromatography Management System with a Dionex Ion Chromatograph (IC)	031883	Yes	Yes
Chromeleon online Help	N/A	N/A	N/A

All Adobe® PDF files listed above are shipped on the Dionex Reference Library CD-ROM (P/N 053891), which is included in the ship kit of each ICS-3000 system module. In addition, the software manuals are provided as PDF files on the Chromeleon or Chromeleon Xpress CD-ROM.

A printed copy of the software installation instructions is provided in the software ship kit.

Consumables documentation: For complete information about Dionex columns, suppressors, EluGen cartridges, etc., refer to the appropriate product manual. These manuals are provided on the Reference Library CD-ROM.

1.3 The ICS-3000 System Operator's Manual

1.3.1 Overview

The electronic version (i.e., PDF file) of the ICS-3000 system operator's manual contains numerous hypertext links that can take you to other locations within the file. These links include:

- Table of contents entries
- Index entries
- Cross-references (underlined in blue) to sections, figures, tables, etc.

If you are not familiar with how to navigate PDF files, refer to the Help system for Adobe® Acrobat® or Adobe Reader® for assistance.

Chapter 1 Introduction	An overview of the ICS-3000 system; includes a brief description of the ICS-3000 modules, the software required for ICS-3000 operation, and the ICS-3000 user manuals.
Chapter 2 Description	Detailed descriptions of ICS-3000 system components and important operating features; includes an introduction to Chromeleon and Chromeleon Xpress software.
Chapter 3 System Configurations	Detailed illustrations of component plumbing for several different ICS-3000 system configurations.
Chapter 4 Getting Started	Tasks to be performed before beginning operation of the ICS-3000 system.
Chapter 5 Operation	Instructions for routine operation of the ICS-3000 system with Chromeleon or Chromeleon Xpress software.
Chapter 6 Shutdown	Short-term and long-term shutdown procedures for the ICS-3000 system.
Chapter 7 Maintenance	Routine preventive maintenance procedures for the ICS-3000 system.

Chapter 8
Troubleshooting

Minor problems that may occur during operation of the ICS-3000 system, with step-by-step procedures for how to isolate and eliminate the cause of each problem. Includes a list of Chromeleon and Chromeleon Xpress Audit Trail error messages, with an explanation of the possible cause of each message and the corrective

action to take.

Chapter 9 Service

Step-by-step instructions for routine service and parts replacement procedures the user can

perform for the ICS-3000 system.

Appendix A Specifications

Specifications and installation site requirements

for the ICS-3000 modules.

Appendix B Reordering Information Spare parts for the ICS-3000 modules.

1.3.2 Safety Messages and Notes

This manual contains warnings and precautionary statements that can prevent personal injury and/or damage to the ICS-3000 system when properly followed. Safety messages appear in bold type and are accompanied by icons, as shown below.



Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.



Indicates that the function or process of the instrument may be impaired. Operation does not constitute a hazard.

Messages d'avertissement en français



Signale une situation de danger immédiat qui, si elle n'est pas évitée, entraînera des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures mineures à modérées. Également utilisé pour signaler une situation ou une pratique qui pourrait gravement endommager l'instrument mais qui n'entraînera pas de blessures.

Warnhinweise in Deutsch



Bedeutet unmittelbare Gefahr. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.



Bedeutet eine mögliche Gefährdung. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.



Bedeutet eine mögliche Gefährdung. Mißachtung kann zu kleineren oder mittelschweren Verletzungen führen. Wird auch verwendet, wenn eine Situation zu schweren Schäden am Gerät führen kann, jedoch keine Verletzungsgefahr besteht.

Notes

Informational messages also appear throughout this manual. These are labeled NOTE and are in bold type:

NOTE NOTES call attention to certain information. They alert you to an unexpected result of an action, suggest how to optimize instrument performance, etc.

1.4 Safety and Regulatory Information

The ICS-3000 system is designed for IC (ion chromatography) and HPLC (high-performance liquid chromatography) applications and should not be used for any other purpose. Operation of an ICS-3000 module in a manner not specified by Dionex may result in personal injury.

1.4.1 Safety Labels

The TUV GS, C, US Mark safety label and the CE Mark label on the ICS-3000 modules indicate that they are in compliance with the following standards:

EMC Susceptibility and Immunity

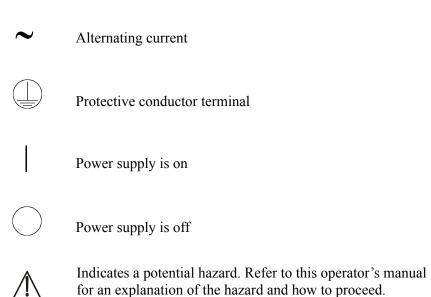
- (DC, EG, DP, SP) EN 61326 1997 including A1:1998 and A2:2001
- (TC) EN 61326 1997 including A1:1998, A2:2001, and A3:2003

Safety

- (DC, EG, DP, SP) EN 61010-1:2001, UL 61010-1:2004, CAN/CSA-C22.2 61010-1:2004
- (TC) EN 61010-1:2001, UL 61010A:2002, CAN/CSA-C22.2 61010-1:2004

NOTE The TC does not carry the TUV GS Mark safety label.

These symbols appear on the ICS-3000 modules or on labels affixed to the modules:





DP/SP Description

2.1 DP/SP Front Features

A status bar on the front of the ICS-3000 Dual Pump (DP) and ICS-3000 Single Pump (SP) includes buttons for controlling certain pump functions, as well as LEDs (light emitting diodes) that indicate the status of several pump functions (see Figure 2-1 and Figure 2-2).

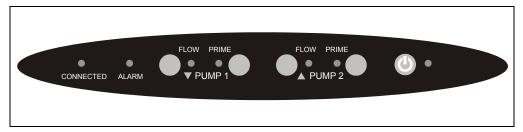


Figure 2-1. DP Status Bar

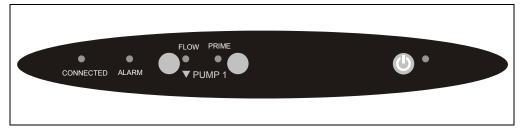


Figure 2-2. SP Status Bar

Button/LED Label	If the LED Is On	If the LED is Flashing
CONNECTED	The DP/SP is connected to a Chromeleon or Chromeleon Xpress timebase.	Does not flash.
ALARM	A DP/SP-related problem has occurred (for example, a pressure limit was activated). Check the Chromeleon or Chromeleon Xpress Audit Trail for the cause.	Does not flash.
PUMP 1 FLOW* PUMP 2 FLOW**	The DP/SP is on and is delivering flow.	Does not flash.
PUMP 1 PRIME* PUMP 2 PRIME**	Use the PRIME button to prime the pump. The LED indicates whether the pump is being primed.	The pump is being primed.
O POWER	Use this POWER button for routine on/off control of the DP/SP. When the power is on, this LED is lighted. To turn off the DP/SP, press and hold this POWER button for 2 seconds. Note: The main power switch is on the rear panel.	Does not flash.

^{*} Pump 1 is always installed in the lower half of the DP/SP enclosure.

^{**} SP only: This button is not present.

2.2 DP/SP Interior Components

The pump's mechanical components are located directly behind the front door of the module. <u>Figure 2-3</u> shows the mechanical components of a DP that contains both a gradient pump (pump 1) and an isocratic pump (pump 2).

For servicing components, the component mounting panel slides out about 5 cm (2 in) for easy access to components. To pull the panel out, use the handles on the top and bottom of the panel.



Figure 2-3. DP/SP Interior Components (DP Shown)

2.2.1 Pump Heads

The DP/SP is a zero-pulsation, serial dual-piston pump with electronic compressibility compensation. Two pump heads—a *primary* head and a *secondary* head—are connected in series. Eluent passes through both pump heads in succession.

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

The characteristic feature of the patented isokinetic pre-compression phase is the programmed overlapping of the delivery strokes of the two pump heads. When delivering compressible liquids without controlled pre-compression, the pulsation increases as the operating pressure increases, since part of the delivery stroke is required to compress eluent in the pump head.

During the pre-compression phase, pulsation is minimized by velocity modulation of the drive. A patented secondary control system (automatic compressibility compensation) ensures highly constant eluent delivery. The flow rate remains constant in relation to the pressure.

2.2.2 Pressure Transducer

The secondary pump head contains a built-in pressure transducer to measure the system pressure. The DP/SP Moduleware (the instrument control firmware installed in the pump) precisely controls the pump motor speed to ensure flow rate accuracy and to maintain constant flow and constant pressure.

Flow output from the secondary pump head passes through a static mixer (described in <u>Section 2.2.6</u>) before being directed to the remainder of the chromatography system (the injection valve, columns, and detector).

2.2.3 Proportioning Valves (Gradient pump only)

In the gradient pump, eluent flows from the eluent reservoirs, through the vacuum degas chambers, and into a four-way proportioning valve assembly (see <u>Figure 2-3</u>). Programmed percentages of each eluent are proportioned by the four valves.

2.2.4 Vacuum Degassing Module

The DP/SP vacuum degassing module provides continuous, online eluent degassing. Eluent quality significantly affects DP/SP 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 eluent proportioning valves (gradient pump only), pump heads, and detector cell. Degassing eluents is especially important when combining aqueous and nonaqueous components (for example, water and acetonitrile).

The vacuum degassing module is either single-channel (in an isocratic pump) or quad-channel (in a gradient pump). The module consists of:

- A degas chamber (with degassing membranes) with internal capacity of 670 μL per channel
- A dual-stage diaphragm vacuum pump
- An on-board vacuum sensor
- The electronics required to operate the vacuum pump
- Tubing, fittings, and other accessories

The vacuum degassing module is automatically activated when the DP/SP power is turned on. Allow about 10 minutes for the module to equilibrate.

NOTE Make sure the run time is long enough to ensure that the vacuum degassing module delivers the optimal degassing performance.

Manual Control of the Vacuum Degassing Module

The vacuum degassing module normally remains on continuously. To turn it off (for example, to investigate a leak), follow these steps:

- 1. In Chromeleon or Chromeleon Xpress, select **Command** on the **Control** menu or press the **F8** key.
- 2. In the Commands dialog box, select the pump name.
- 3. Select the **Degasser** command.
- 4. Select **Off** and click the **Execute** button.
- 5. To turn on the degasser again, select **On** and click **Execute**.

2.2.5 Piston Seal Wash System

The piston seal wash system consists of a peristaltic 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.

When seal washing is activated, the back of the main piston seal is rinsed with wash solution; this prolongs seal lifetime by preventing eluent crystallization on the seal surfaces.

Notes About the DP Piston Seal Wash System

The piston seal wash system is designed for use with only one of the two pumps in a DP module. When the DP is shipped from Dionex, the seal wash system is connected to pump 1 (the bottom pump). If necessary, connect the seal wash system to pump 2 (the top pump), instead. For instructions on how to replumb the system, refer to Section 4.5.

NOTE Dionex offers an External Seal Wash Kit (P/N 063518) for users who need to operate a piston seal wash system for both pumps in the DP. Installation instructions are provided in the kit.

2.2.6 Static Mixer

A GM-3 or GM-4 static mixer is installed after the secondary pump head (see <u>Figure 2-3</u>). In the gradient pump, the mixer helps to ensure that proportioned eluents are mixed thoroughly. In the isocratic pump, the mixer is optional, but can function as a pulse damper.

The DP/SP gradient delay volume is $600~\mu L$ when a GM-3 is installed and $380~\mu L$ when a GM-4 is installed. The gradient delay volume (or *dwell volume*) is the volume of liquid in the system between the point where the gradient is formed and the point where it enters the column. This includes the mixer, transfer tubing, and swept volume in the injector or autosampler.

2.3 DP/SP Flow Schematics

2.3.1 Isocratic Pump Flow Schematic

Figure 2-4 illustrates the liquid flow path through an isocratic pump.

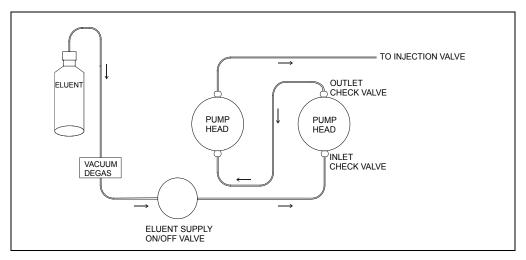


Figure 2-4. Isocratic Pump Flow Schematic

- Eluent flows from the reservoir, through the vacuum degas chamber (if the vacuum degas module is installed), through the eluent supply on/off valve, and into the inlet check valve on the primary pump head.
- The inlet check valve opens, drawing eluent into the primary pump head. At the same time, the secondary piston pushes forward, pushing eluent into the system. After completing the intake, the primary piston pushes eluent through the outlet check valve and into the secondary pump head.
- Flow exits the secondary pump head and is directed to the remainder of the chromatography system (the injection valve, columns, and detector).

2.3.2 Gradient Pump Flow Schematic

<u>Figure 2-5</u> illustrates the liquid flow path through a gradient pump.

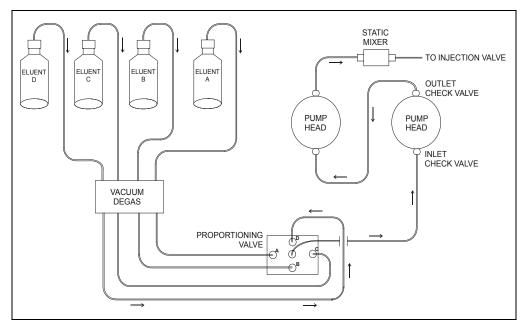


Figure 2-5. Gradient Pump Flow Schematic

- Eluent flows from the reservoirs and through the vacuum degas chambers. The selected proportions of eluent flow out of the proportioning valve assembly and into the inlet check valve on the primary pump head.
- The inlet check valve opens, drawing eluent into the primary pump head. At the same time, the secondary piston pushes forward, pushing eluent into the system. After completing the intake, the primary piston pushes eluent through the outlet check valve and into the secondary pump head.
- Flow exits the secondary pump head, continues through the static mixer, and is then directed to the remainder of the chromatography system (the injection valve, columns, and detector).

2.4 DP/SP Rear Panel

Figure 2-6 illustrates the rear panel of the DP/SP.

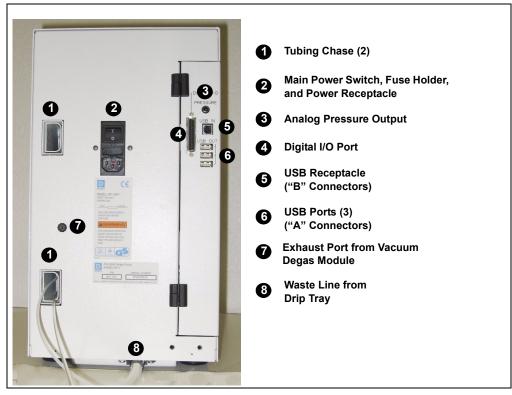


Figure 2-6. DP/SP Rear Panel

Tubing Chases

The tubing chases route tubing from the front of the DP/SP, through the interior of the module, and to the rear panel.

Main Power Switch, Fuse Holder, and Power Receptacle

The rear panel power switch is the main power switch for the DP/SP. Turn on the main power switch before initial operation and leave it on unless instructed to turn it off (for example, before performing a service procedure).

NOTE For routine on/off control, use the POWER button on the front of the DP/SP (see Figure 2-1 and Figure 2-2). To

turn off the pump, press and hold the POWER button for 2 seconds.

The fuse cartridge contains two 2-amp IEC 60127-2 slow-blow fuses (P/N 954773). For instructions on how to change the fuses, see Section 9.9.

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



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



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.



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

Digital I/O Port

The digital I/O port provides a connection to an AS40 Automated Sampler or other auxiliary device. The connector includes three TTL inputs and four relay outputs. <u>Table 2-1</u> indicates the functions assigned to the connector pins.

For instructions on connecting the pump to an AS40, refer to the *ICS-3000 Ion Chromatography System Installation Instructions* (Document No. 065032). The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).



The maximum switching voltage of the relays is 24 V. The switching current must not exceed 100 mA.

Pin Number	Signal Name	Signal Level	Description
1			Not used
2			Not used
3	Relay 3 Out	Potential-free	Normally open
4	Relay 1 Out	Potential-free	Normally closed
5	Relay 2 Out	Potential-free	Normally closed
6	Relay 3 Out	Potential-free	Normally closed

Table 2-1. 25-Pin D-Sub I/O Port (Female)

Pin Number	Signal Name	Signal Level	Description
7	Relay 1 Out	Potential-free	Common
8	Relay 2 Out	Potential-free	Common
9	Gnd	Ground	Ground
10	Gnd	Ground	Ground
11	Gnd	Ground	Ground
12	Gnd	Ground	Ground
13			Not used
14	Relay 4 Out	Potential-free	Normally open
15	Relay 4 Out	Potential-free	Common
16	Relay 4 Out	Potential-free	Normally closed
17			Not used
18	Relay 3 Out	Potential-free	Common
19	Relay 1 Out	Potential-free	Normally open
20	Relay 2 Out	Potential-free	Normally open
21	Vcc Out	+5V	+5V, 500 mA
22	TTL Input 1	TTL	Hold/Run
23	TTL Input 2	TTL	Stop
24	TTL Input 3	TTL	Start
25			Not used

Table 2-1. 25-Pin D-Sub I/O Port (Female) (Continued)

Analog Pressure Output

The analog pressure output indicates the operating pressure of the pump. The pressure output is set to 50 mV/MPa (5 mV/14.51 psi). To monitor the pressure, connect the pressure output to a recorder or an A/D converter. These are the pin assignments for the 2-pin Cinch connector (P/N 8005.9001A):

Signal Level	Function
Inner ring:	Signal (pressure)
Outer ring:	Ground

The analog pressure output defaults to the pump 1 (bottom pump) output signal. To redirect the output to pump 2 (the top pump), follow these steps:

- 1. In Chromeleon or Chromeleon Xpress, select **Command** on the **Control** menu or press the **F8** key.
- 2. In the Commands dialog box, select the pump name.
- 3. Select the **AnalogOut** command.
- 4. Select **PumpTop** in the **AnalogOut** drop-down box and click **Execute**.

USB Connections

- One USB receptacle ("B" type connector) allows a connection from the PC on which Chromeleon or Chromeleon Xpress software is installed.
- Three USB (Universal Serial Bus) ports ("A" type connectors) are provided for connections to other ICS-3000 modules.

One 1.8-m (6-ft) USB cable (P/N 960777) is provided in the ship kit (DP Ship Kit, P/N 062463; SP Ship Kit, P/N 063342).

Waste Lines

- One waste line is connected to the secondary pump head.
- One waste line exits the drip tray and is routed to the rear panel.

Place the free ends of the waste lines into one waste container. To maintain a positive siphon, position the waste container below the level of the pump.

NOTE To ensure proper drainage, the waste lines must remain above the liquid level in the waste container.

2.5 Eluent Reservoirs

The following reservoirs are available for use with the DP/SP:

- 1-liter plastic reservoir (P/N 063291)
- 2-liter plastic reservoir (P/N 062510)
- 4-liter plastic reservoir (P/N 063292)



Do not use the 2-liter plastic reservoir (P/N 062510) for offline vacuum degassing of eluents. The reservoir was not designed for this purpose.



N'utilisez pas le réservoir en plastique de 2 litres (N/P 062510) pour le dégazage à vide hors ligne d'éluants. Le réservoir n'a pas été conçu à cette fin.



Verwenden Sie den 2-Liter Plastikbehälter (Bestell-Nr. 062510) nicht zum Offline Vakkum-Entgasen von Eluenten. Der Behälter ist dafür nicht ausgelegt.

All eluent reservoirs listed above are pressurizable. Although the DP/SP does not *require* pressurized reservoirs, Dionex recommends pressurizing reservoirs with helium or nitrogen 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.



Never pressurize eluent reservoirs above 0.07 MPa (10 psi). Pressurizing reservoirs above this limit can cause the reservoir to explode.



Ne mettez jamais les réservoirs d'éluants sous une pression supérieure à 0,07 MPa (10 psi).



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

2.6 EO (Optional)

The ICS-3000 Eluent Organizer (EO) holds eluent reservoirs in a liner that contains spills and leaks. Up to two EOs can be installed on top of the DC. Each EO accommodates up to four 1-liter or 2-liter reservoirs *or* up to two 4-liter reservoirs. The EO is typically ordered configured with four 2-liter reservoirs (P/N 062629).

All eluent reservoirs available for use with the DP/SP can be pressurized. If you plan to pressurize the eluent reservoirs, an optional regulator kit is required. The kit is available in two versions:

- When the DC is installed as the topmost module in the system, the EO Regulator Kit (P/N 063493) is required. The kit includes a pressure regulator and gauge assembly with four outputs (for connections to four eluent reservoirs), as well as the tubing and connection fitting required.
- When the TC, ICS-Series Variable Wavelength Detector, or ICS-Series Photodiode Array Detector is installed as the topmost module in the system, the TC/VWD/PDA Regulator Bracket Kit (P/N 064792) is required. The kit includes the EO Regulator Kit described above, as well as a right-angle regulator bracket and mounting hardware. After attaching the bracket to the TC or detector, you will mount the gas regulator assembly on the bracket.

If more reservoirs are required, order a second regulator (P/N 062345).



EG Description

2.7 EG Front Features

The status bar on the front of the ICS-3000 Eluent Generator (EG) includes buttons that provide control of certain EG functions, as well as LEDs that indicate the status of several EG functions (see Figure 2-7).

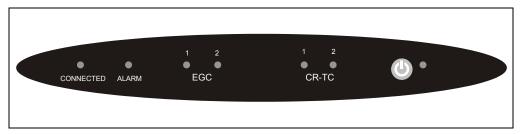


Figure 2-7. EG Status Bar

Button/LED Label	If the LED is On	Comment
CONNECTED	The EG is connected to a Chromeleon or Chromeleon Xpress timebase.	
ALARM	An EG-related problem has occurred (for example, the EluGen cartridge was disconnected).	Check the Chromeleon or Chromeleon Xpress Audit Trail for the cause.
EGC 1 EGC 2	The EluGen cartridge is on and is generating eluent.	EGC 1 is always installed in the left section of the component compartment. EGC 2, if present, is installed in the right section of the compartment.
CR-TC 1 CR-TC 2	The CR-TC is on.	CR-TC 1 is always installed in the left section of the component compartment. CR-TC 2, if present, is installed in the right section of the compartment.

ICS-3000 Ion Chromatography System

Button/LED Label	If the LED is On	Comment
(b) POWER	Use this POWER button for routine on/off control of the EG. When the power is on, this LED is lighted. To turn off the EG, press and hold this POWER button for 2 seconds.	The main power switch is on the EG rear panel.
NOTE: The EG status bar LEDs do not flash.		

2.8 EG Interior Components

<u>Figure 2-8</u> shows the EG component compartment, which is located directly behind the front door. The component mounting panel divides the compartment into two sections. In a single-channel EG, components are installed in the left section only.

To access components for maintenance or service procedures, push down on the slide release latch and pull the tray forward until it reaches the stop.

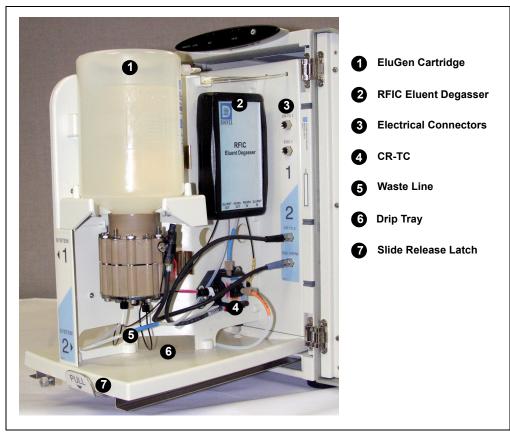


Figure 2-8. EG Interior Components (Second (Right Side) Channel of a Dual-System EG Shown)

EluGen Cartridge

Several types of EluGen cartridges are available for use with the EG (see the table below). Each cartridge contains 900 mL of the appropriate electrolyte concentrate solution for eluent generation.

EluGen Cartridge	Part Number	Function
EGC II K ₂ CO ₃ EluGen Cartridge	058904	Generates potassium carbonate eluent for anion exchange separations. Note: Produces a carbonate/bicarbonate mixture when installed with the EPM Electrolytic pH Modifier (P/N 063175) and EGC-CO ₃ Mixer (P/N 061686).
EGC II KOH EluGen Cartridge	058900	Generates potassium hydroxide eluent for anion exchange separations.
EGC II LiOH EluGen Cartridge	058906	Generates lithium hydroxide eluent for anion exchange separations.
EGC II MSA EluGen Cartridge	058902	Generates methanesulfonic acid eluent for cation exchange separations.
EGC II NaOH EluGen Cartridge	058908	Generates sodium hydroxide eluent for anion exchange separations.

For more information, refer to the EluGen cartridge manual. The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).

RFIC Eluent Degasser

The RFIC Eluent Degasser (P/N 062137) contains a tubing assembly that purges the electrolysis gas from the freshly-generated eluent before it is directed to the separator column.

Backpressure Coil (Optional)

The EluGen cartridge requires at least 14 MPa (2000 psi) of system backpressure for removal of electrolysis gas from the eluent produced by the cartridge. A system backpressure of 16 MPa (2300 psi) is ideal.

If necessary, increase the system backpressure by installing a backpressure coil between the injection valve and the EluGen cartridge **OUTLET** port. For details, refer to Section 9.15.

Continuously Regenerated Trap Column (CR-TC)

The CR-TC is a high pressure, electrolytically-regenerated trap column. The CR-TC is designed to remove anionic or cationic contaminants in the eluent or deionized water and to reduce drift during gradient separations. Two versions of the CR-TC can be used with the EG:

- CR-ATC (Continuously Regenerated Anion Trap Column, P/N 060477)
- CR-CTC (Continuously Regenerated Cation Trap Column, P/N 060478)

For more information, refer to the CR-TC manual. The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).

- NOTE Do not install a CR-TC in the same channel as an EGC K₂CO₃ EluGen Cartridge or an EPM Electrolytic pH Modifier.
- NOTE The IonPac® ATC-HC Trap Column (P/N 059604) or IonPac CTC-1 Trap Column (P/N 040192) may be used with the EG. However, both IonPac trap columns require off-line chemical regeneration. Contact Dionex for more information.

EPM Electrolytic pH Modifier and EGC-CO₃ Mixer

The EGC II K₂CO₃ EluGen Cartridge can be used with an EPM Electrolytic pH Modifier (P/N 063175) and an EGC-CO₃ Mixer (P/N 061686) to produce a carbonate/bicarbonate mixture for use with anion exchange separations on carbonate-based IonPac columns.

After the cartridge generates potassium carbonate eluent, the EPM adjusts the eluent concentration to produce the carbonate/bicarbonate mixture. The EGC- $\rm CO_3$ Mixer provides mixing necessary to produce a homogenous solution of electrolytically-generated $\rm K_2\rm CO_3$ and KHCO₃ eluent.

For more information about these products, refer to the EluGen cartridge manual. Cartridge manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

Leak Sensor

If liquid collects in the drip tray in the bottom of the EG, a leak sensor reports the leak to Chromeleon or Chromeleon Xpress, and an error message is displayed in the Audit Trail. In addition, the **Alarm** LED on the EG front panel lights.

Electrical Connections

Electrical connectors provide a connection from the components installed in the EG to the EG power supplies. There are two sets of electrical connectors (one per channel). Connections for the following components are provided:

 Two EluGen cartridges or one cartridge and one EPM Electrolytic pH Modifier

and

• Two Continuously Regenerated Trap Columns (CR-TC)

When operating with an EGC II K_2CO_3 EluGen Cartridge and the EPM, the cartridge is connected to the EGC power supply for one channel and the EPM is connected to the EGC power supply for the second channel. Thus, the EG can accommodate only one such configuration.

Waste Line

The module waste line (white tubing) discharges liquid and gas waste from the RFIC Eluent Degasser. This line is marked **WASTE-GAS SEPARATOR** inside the EG.

NOTE A gas separator waste tube (P/N 045460) should be connected to the EG waste line during installation. For details, refer to the ICS-3000 Ion Chromatography System Installation Instructions (Document No. 065032).

Place the free end of the waste line into a waste container. To maintain an active drain, position the waste container below the level of the EG.

NOTE To ensure proper drainage, each waste line must remain above the liquid level in the waste container.

2.9 EG Rear Panel

<u>Figure 2-9</u> illustrates the rear panel of the ICS-3000 Eluent Generator (EG).

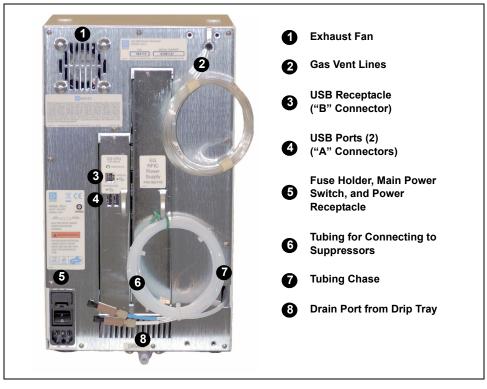


Figure 2-9. EG Rear Panel

Exhaust Fan

The exhaust fan cools the interior of the EG and exhausts any hydrogen and oxygen gases that escape during operation.

USB Connections

- One USB receptacle ("B" type connector) allows a connection from the PC on which Chromeleon or Chromeleon Xpress software is installed.
- Two USB (Universal Serial Bus) ports ("A" type connectors) are provided for connections to other ICS-3000 modules.

One 1.8-m (6-ft) USB cable (P/N 960777) is provided in the EG Ship Kit (P/N 062453).

Fuse Holder, Main Power Switch, and Power Receptacle

The fuse cartridge contains two 2-amp slow-blow fuses (P/N 954773). For instructions on how to change the fuses, see Section 9.16.

The rear panel power switch is the main power switch for the EG. Turn on the main power switch before initial operation and leave it on unless instructed to turn it off (for example, before performing a service procedure).

NOTE For routine on/off control, use the POWER button on the front of the EG (see <u>Figure 2-7</u>). To turn off the EG, press and hold the POWER button for 2 seconds.

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



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



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.



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

Tubing Chase

The tubing chase routes tubing from the front of the EG, through the interior of the module, and to the rear panel.

Vent, Suppressor, and Drain Lines

- The gas vent line (clear tubing) discharges the electrolysis gas (H₂ or O₂) that is vented from the electrolyte reservoir of the EluGen cartridge.
- The lines exiting the tubing chase (white tubing) are for connecting the REGEN IN port on the CR-TC or EPM Electrolytic pH Modifier to the REGEN OUT port on the suppressor.
- The drip tray drain port removes any liquid that collects in the drip tray in the bottom of the EG. A clear corrugated tubing line is connected to this port during installation.

Place the free ends of the vent and drain lines into a waste container. To maintain a positive siphon, position the waste container below the level of the EG.

NOTE To ensure proper drainage, each waste line must remain above the liquid level in the waste container.

2.10 EG Flow Schematic

Figure 2-10 illustrates the liquid flow path through the EG.

Deionized water is delivered by the DP/SP to the EluGen cartridge. DC current is applied to the cartridge to produce eluent.

The electrolytically-generated eluent flows through the Continuously Regenerated Trap Column (CR-TC) into the RFIC Eluent Degasser, through the backpressure coil (if installed), through the injection valve to the separator column, and finally to the detector cell.

The detector cell effluent is directed through the Atlas or SRS suppressor regenerant chamber to the CR-TC regenerant chamber, through the RFIC Eluent Degasser inside the EG, and finally to waste.

NOTE Refer to the EGC-CO₃ Mixer manual for a flow schematic showing the components required to generate a carbonate/bicarbonate mixture. The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).

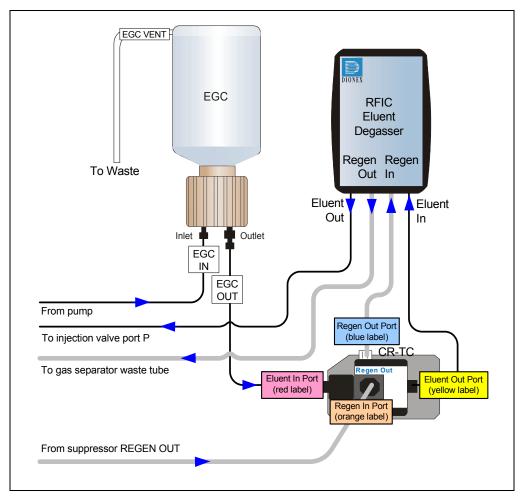


Figure 2-10. EG Flow Schematic Example



2.11 DC Front Features

A status bar on the front of the ICS-3000 Detector/Chromatography Module (DC) (see <u>Figure 2-12</u>) includes buttons for controlling certain DC functions, as well as LEDs that indicate the status of several DC components and functions.

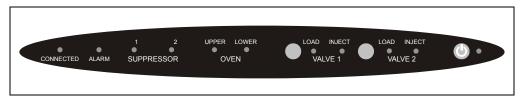


Figure 2-11. DC Status Bar

Button/LED Label	If the LED Is On	If the LED Is Flashing
CONNECTED	The DC is connected to a Chromeleon or Chromeleon Xpress timebase.	Does not flash.
ALARM	The leak sensor is wet or there was a valve or suppressor error. Check the Chromeleon or Chromeleon Xpress Audit Trail for the cause.	Does not flash.
SUPPRESSOR 1 SUPPRESSOR 2	The suppressor is on and current is being applied.	Does not flash.
OVEN UPPER	The upper compartment is at its set temperature.	The upper compartment is transitioning to the set temperature. The compartment is not ready for operation.
OVEN LOWER	The lower compartment is at its set temperature.	The lower compartment is transitioning to the set temperature. The compartment is not ready for operation.

Button/LED Label	If the LED is On	If the LED is Flashing
VALVE 1 LOAD VALVE 2 LOAD VALVE 1 INJECT VALVE 2 INJECT	Use the VALVE 1 and VALVE 2 buttons to manually switch the position of the DC injection valves. The LEDs indicate whether the valve is in the Load or Inject position.	Valve error. See Section 8.25 for troubleshooting.
O POWER	Use this POWER button for routine on/off control of the DC. When the power is on, this LED is lighted. To turn off the DC, press and hold this POWER button for 2 seconds. Note: The main power switch is on the DC rear panel.	Does not flash.

NOTE The VALVE 1 and VALVE 2 buttons can be enabled and disabled in Chromeleon or Chromeleon Xpress. When disabled, the valve can be controlled only from the software. To enable or disable a button, open the Commands dialog box (press F8) and select the Valve1Button (or Valve2Button) command in the list of commands for the DC.

Sample Loading Ports

The front of the DC has two sample loading ports (see <u>Figure 2-12</u>) that can be connected to injection valves installed inside the DC. A syringe is used to manually load sample through the ports. For automated sample injection, the DC can be connected to an autosampler. For more information about sample injection, see <u>Section 5.1</u>.



Figure 2-12. DC Sample Loading Ports

2.12 DC Interior Components

Figure 2-13 illustrates the interior compartments of the DC.

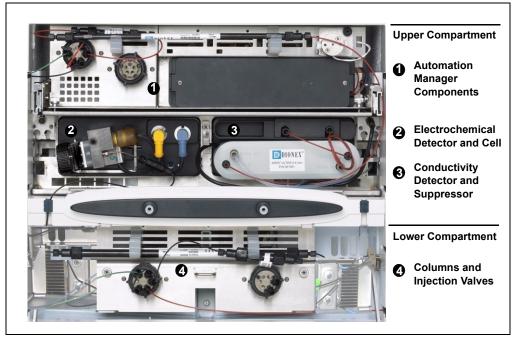


Figure 2-13. DC Interior View Example

Upper Compartment

The upper compartment consists of two sections:

- The top section houses an optional ICS-3000 Automation Manager (AM). The AM contains various components required for performing matrix elimination, large volume pre-concentration, post-column reagent addition, and other functions. See Section 2.17 for details about the AM.
- The section below the AM section houses one or two detectors. The detectors can be either two ICS-3000 Conductivity Detectors (CDs), two ICS-3000 Electrochemical Detectors (EDs), or one detector of each type. See
 Section 2.15 for details about the CD and Section 2.16 for details about the ED.

Suppressors for conductivity detection are installed in this compartment also. The following types of suppressors can be used:

- SRS Self-Regenerating Suppressor (SRS 300 or ULTRA II) (2- and 4-mm)
- AES Atlas Electrolytic Suppressor
- MMSTM MicroMembraneTM Suppressor (MMS 300 or MMS II)

Lower Compartment

The lower compartment can hold up to two column sets (four columns) with IDs of 1 mm to 9 mm and lengths of 100 mm to 250 mm.

The lower compartment also holds one or two injection valves. Two valve versions (6-port and 10-port) are available. Both versions are electrically-activated, two position valves. See <u>Section 2.14</u> for details about the injection valves

The compartment tray slides forward about 10 cm (4 in) for easy access to columns and valves. To pull the tray forward, use the handle installed in the center of the compartment (see <u>Figure 2-14</u>).



Figure 2-14. DC Side View with Lower Compartment Tray Pulled Out

Temperature Control

The following temperature control zones are possible with the DC, depending on the options installed:

- In the single-zone (shared) configuration, the upper and lower compartments are in the same temperature zone. The temperature can be set to between 15 and 40 °C. Air flow passages between the compartments allow the temperature to stabilize equally in both compartments.
- In the dual-zone configuration, the lower compartment is isolated from the upper compartment in a separate temperature zone. There is no common air flow between the upper and lower compartments. The temperature of the lower compartment can be set to between 10 and 70 °C. The temperature of the upper compartment can be set to between 15 and 40 °C.
- When a CD is installed, the heated conductivity cell can be set to 5 °C above the upper compartment temperature, up to 60 °C.
- If an RCH-1 Reaction Coil Heater is installed in the AM, the temperature of the RCH-1 heater can be set to 5 °C above the upper compartment temperature, up to 80 °C.

The temperatures achieved for a particular zone can vary from the control range, depending on the ambient temperature (see the table below).

Temperature Zone	Control Range	Achieved Temperatures (Based on Ambient)
Upper compartment (or shared zone)	15 to 40 °C	If the RCH-1 is off: Minimum temperature ≥ (ambient – 10 °C) If the RCH-1 is at 80 °C or the CD cell is at 60 °C Minimum temperature ≥ (ambient – 5 °C)
Lower compartment	10 to 70 °C	Minimum temperature ≥ (ambient – 5 °C) Maximum temperature ≥ (ambient + 50 °C)
CD cell	15 to 60 °C	Minimum temperature ≥ (upper compartment temperature + 5 °C)
RCH-1	15 to 80 °C	Minimum temperature ≥ (upper compartment temperature + 5 °C)

For example, if the ambient air temperature in your lab is 30 °C and you set the upper compartment temperature to 15 °C, the actual temperature achieved in the upper compartment will be about 20 °C.

NOTE A DC Temperature Calibration Kit (P/N 063782) is available. The kit includes the parts and instructions required to verify the temperature calibration of the DC upper and lower compartments and to recalibrate, if required.

2.13 DC Rear Panel

Figure 2-15 illustrates the rear panel of the DC.

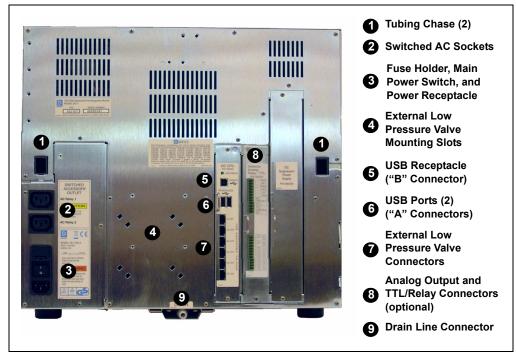


Figure 2-15. DC Rear Panel

Tubing Chases

The tubing chases route tubing from the front compartment, through the DC interior, and to the rear panel. Foam inserts insulate the chases.

Switched AC Sockets

The two AC receptacles can be used to control the power to external devices. Use Chromeleon or Chromeleon Xpress to switch the power on and off. TTL inputs can also be used to control the AC sockets (see Section 2.18.4).

Fuse Holder, Main Power Switch, and Power Receptacle

The fuse holder contains two 10-amp slow-blow fuses (P/N 954772). For instructions on how to change the fuses, see Section 9.19.

The rear panel power switch is the main power switch for the DC. Turn on the main power switch before initial operation and leave the switch on unless instructed to turn it off (for example, before performing a service procedure).

NOTE For routine on/off control, use the POWER button on the front of the DC (see Figure 2-12). To turn off the DC, press and hold the POWER button for 2 seconds.

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



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



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.



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

USB Connections

The USB (Universal Serial Bus) receptacle ("B" type connector) allows connection to the PC on which Chromeleon or Chromeleon Xpress software is installed

The two USB ports ("A" type connectors) allow connection to other USB devices in the system (for example, the pump and eluent generator).

A 1.8-m (6-ft) USB cable (P/N 960777) is included in the DC Ship Kit (P/N 062614, dual; P/N 063408, single).

External Low Pressure Valve Outputs

Six outputs allow connection to externally-installed low-pressure (solenoid) valves. 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). You control the outputs with Chromeleon or Chromeleon Xpress.

Two low-pressure valves can be installed on a regulator stand and four valves can be installed on the rear of the DC.

Analog Output, TTL, and Relay Connectors (Optional)

If the I/O option (P/N 062201) is installed, the following connections are provided:

- Two analog outputs (one for each detector)
- One +5V power output
- Two relay outputs
- Two TTL outputs
- Eight programmable TTL inputs

See Section 2.18 for details about the I/O option.

2.14 Injection Valves

One or two injection valves can be installed in the lower compartment of the DC. The following models are available: 6-port (P/N 061961) and 10-port (P/N 061962).

2.14.1 Injection Valve Operation

Each ICS-3000 Ion Chromatography System injection valve has two operating positions: Load and Inject. Liquid flows through either the Load or Inject path, depending on the valve position.

Figure 2-16 shows flow schematics for the 6-port valve.

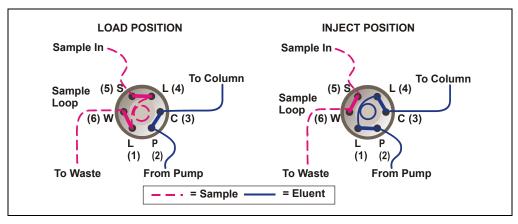


Figure 2-16. Injection Valve Flow Schematics (6-Port Valve)

- In the Load position, sample is loaded into the sample loop, where it is held until injection. Eluent flows from the pump, through the valve, and to the column, bypassing the sample loop. Sample flows from the syringe or autosampler line (if installed), through the valve, and into the sample loop. Excess sample flows out to waste.
- In the Inject position, sample is swept to the column for analysis. Eluent flows from the pump, through the sample loop, and on to the column, carrying the contents of the sample loop with it. Section 5.3 describes how to inject samples manually and Section 5.2 describes how to inject samples with an autosampler.

<u>Figure 2-17</u> shows example flow schematics for the 10-port valve when it is connected for an application that uses a concentrator column.

NOTE Other plumbing configurations for the 10-port valve are possible, depending on the components to be connected to the valve and the application to be run. Refer to the appropriate Dionex Application Note for more information.

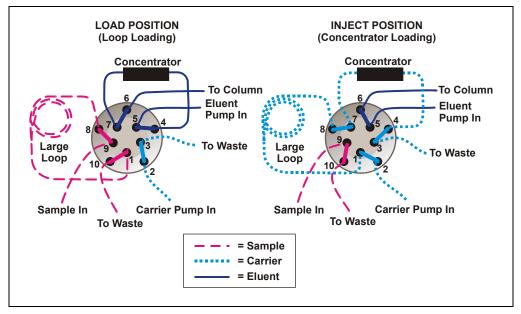


Figure 2-17. Injection Valve Flow Schematics (10-Port Valve) Example Connections: Large Loop to Concentrator

Liquid flows through either the Load or Inject path, depending on the valve position. For the example shown in <u>Figure 2-17</u>, the flow occurs as follows:

• In the Load position, sample flows from the syringe or autosampler line, through the valve, and into the sample loop. Excess sample flows out to waste. Eluent flows from the eluent pump, through the valve, through the concentrator column, and to the separator column. If sample was previously loaded onto the concentrator column, it is swept to the separator column for analysis.

• In the Inject position, carrier liquid flows through the sample loop and on to the concentrator column, carrying the contents of the sample loop with it. Excess flows out to waste. Eluent flows from the pump, through the valve, and to the separator column, bypassing the concentrator column.

2.14.2 Injection Valve Plumbing

Figure 2-18 shows the injection valve connections for the 6-port valve. The injection valve is plumbed at the factory with all tubing and fittings for connection to the pump, sample loading port, column, and waste. A 10 μ L PEEKTM sample loop (P/N 042949) is installed between ports L (1) and L (4).

Dionex offers sample loops in various sizes. If needed, the pre-installed $10~\mu L$ loop can be replaced with a loop that has a different sample injection volume.

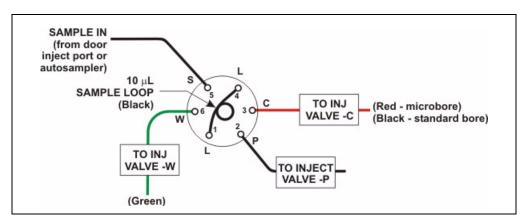


Figure 2-18. Injection Valve Plumbing (6-Port Valve)

2.15 CD Conductivity Detector

One or two ICS-3000 Conductivity Detectors (CDs) can be installed in the DC. Each 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 (if installed). A suppressor (optional) can be installed on the clips on the lower part of the CD. The cell and suppressor are installed in the upper compartment of the DC (see <u>Figure 2-13</u>). The detector electronics are not accessible to the user.



Figure 2-19. Conductivity Detector with Suppressor

2.15.1 Heated Conductivity Cell

The DC 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.

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 DC provides temperature control of both the DC 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 middle and upper compartment to a high of 60 °C.

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.

Follow these steps to change the temperature compensation factor:

- 1. In Chromeleon or Chromeleon Xpress, select **Command** on the **Control** menu or press the **F8** key.
- 2. In the Commands dialog box, select the conductivity detector.
- 3. Select the **Temperature_Compensation** command and enter the desired value.
- 4. Click Execute.

2.15.2 Suppressor

The suppressor reduces the eluent conductivity and enhances the conductivity of the sample ions, thereby increasing detection sensitivity. The CD can operate with an AES, SRS 300, SRS ULTRA II, MMS 300, or MMS III suppressor.

The suppressor is installed below the cell on the CD (see <u>Figure 2-19</u>). The suppressor cable plugs into the connector next to the detector (on either the left or the right side, depending on which side the CD is installed) (see <u>Figure 2-20</u>).



Figure 2-20. Suppressor Connection (Right-Side Installation)

NOTE It is also possible to control a suppressor with an ICS-3000 Electrochemical Detector (ED). Tabs for installing the suppressor are provided on the front of the DC (next to the ED). The cable connection is the same as for the CD.

For details about any of the suppressors, including guidelines for selecting a suppressor for your application, refer to the suppressor manuals. The manuals are on the Dionex Reference Library CD-ROM (P/N 053891).

NOTE A gas separator waste tube (P/N 045460) should be connected to the suppressor waste line during installation to prevent accumulation of oxygen or hydrogen gasses. For details, refer to the ICS-3000 Ion Chromatography System Installation Instructions (Document No. 065032).

2.15.3 System Flow Schematic for Conductivity Detection

<u>Figure 2-21</u> illustrates the flow path through a DC for a conductivity detection application using suppression in recycle mode. For information about other suppression modes, refer to the suppressor manuals. The manuals are on the Dionex Reference Library CD-ROM (P/N 053891).

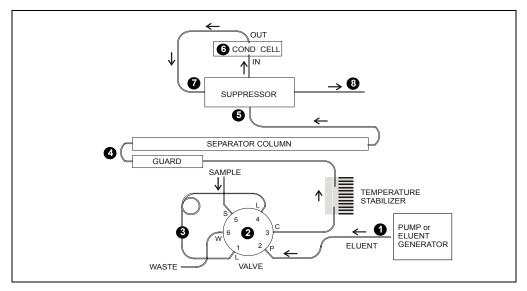


Figure 2-21. DC Flow Schematic for Conductivity Detection (Suppression in Recycle Mode)

- Eluent from the pump or EG flows into the injection valve 2.
- After sample is loaded into the sample loop 3 and the injection valve is toggled to the Inject position, eluent passes through the loop.
- The eluent/sample mixture is pumped first through a temperature stabilizer, then on to the guard and separator columns 4, and then through the suppressor 5.
- From the suppressor, the mixture flows through the cell, where the analytes are detected. A digital signal is sent to Chromeleon or Chromeleon Xpress software.
- Finally, 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. Flow is then routed to waste 3. If an EG is installed, flow is routed to the CR-TC (see Section 2.10 for details).

2.16 ED Electrochemical Detector

One or two ICS-3000 Electrochemical Detectors (EDs) can be installed in the DC. Each complete ED assembly consists of an amperometry cell and the detector electronics required to collect data and send it to the computer and the analog output (if installed). The detector is installed in the upper compartment of the DC (see <u>Figure 2-13</u>). The cell mounts on the front of the detector. The detector electronics are not accessible to the user.

The ED can perform the following electrochemical detection modes:

- DC amperometry (see <u>Section 2.16.3</u>)
- Integrated amperometry (includes pulsed amperometry) (see <u>Section 2.16.4</u>)
- Cyclic voltammetry (see <u>Section 2.16.5</u>)

2.16.1 Amperometry Cell

The ED amperometry cell is a three-electrode voltammetric cell. It is a miniature flow-through cell with a titanium cell body (the counter electrode), a working electrode, and a combination pH-Ag/AgCl reference electrode (see Figure 2-22). The type of working electrode used depends on the application. Four conventional (non-disposable) working electrode types are available: gold, platinum, silver, and glassy carbon. Three disposable working electrodes types are available: gold, silver, and platinum.



Figure 2-22. Amperometry Cell (Titanium Cell Shown)

Cell Design

The ED amperometry 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 (0.25 μ L) of the channel also allows operation with high efficiency, narrow bore 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. The inlet tubing is in electrical contact with the counter electrode and through it to ground. The working electrode current is processed using low noise analog amplifiers and filters.

Amperometry Cell Solvent Compatibility

The amperometry cell can be used with common reversed-phase solvents such as methanol and acetonitrile. If a disposable working electrode is used, the percentage of methanol should not exceed 30% and the percentage of acetonitrile should not exceed 10%. 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.

2.16.2 Combination pH-Ag/AgCl Reference Electrode

The 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.

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.

IMPORTANT

Do not allow the 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 KCI solution. See Section 6.4 for detailed storage instructions.

Monitoring the Amperometry 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 reference electrode needs regenerating or replacing, thus improving the reproducibility of your analyses. See Section 4.1.2 for how to monitor the pH readout.

NOTE Monitor the pH when the reference electrode is used in the Ag mode as well as in the pH mode.

2.16.3 DC Amperometric Detection

In DC amperometry, a constant potential is applied to the working electrode. The potential can be entered into a program file (PGM) (see <u>Figure 2-23</u>) or on the Control panel for direct control.

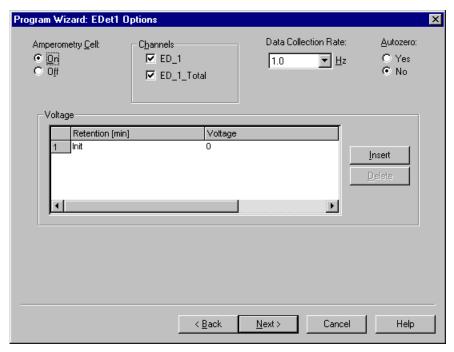


Figure 2-23. DC Amperometry Program Wizard Page

The applied voltage can be programmed 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.

2.16.4 Integrated and Pulsed Amperometric Detection

Integrated and pulsed amperometric detection are similar to DC amperometry (see Section 2.16.3) 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 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. See Section 2.16.6 for more information about waveforms.

Pulsed Amperometric Detection

In pulsed amperometric detection (also known as PAD), current is integrated at a single constant potential (see Figure 2-24).

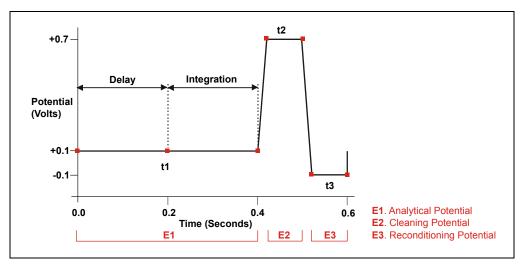


Figure 2-24. Example Pulsed Amperometry Waveform

The potentials, labeled E1, E2, and E3, are applied for durations t1, t2, and t3, respectively. At t1, 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 t2 and t3, 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 Amperometric Detection

With integrated amperometric detection (also known as IA or IPAD), current is integrated at two or more potentials (see Figure 2-25).

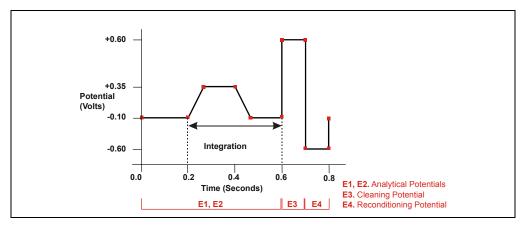


Figure 2-25. Example Integrated Amperometry Waveform

With the example waveform shown in <u>Figure 2-25</u>, 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.

2.16.5 Cyclic Voltammetry Detection

The determination of the optimum potentials to use in amperometry begins with an electrochemical technique called voltammetry, in which the current that results from oxidation or reduction reactions is measured against the voltage applied to the system. The applied voltage is changed (scanned) within preset limits.

In cyclic voltammetry, the voltage is first scanned in one direction and then reversed so that the voltage at the end of the scan is the same as at the beginning. This results in a triangular waveform (see Section 2.16.6).

In the cyclic voltammetry mode, the detector measures the current at a rate of 1 KHz (i.e., 1000 raw data points per second are collected). The data is compressed before storage to 20 Hz.

2.16.6 Waveforms

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

Cyclic Voltammetry Waveforms

Cyclic voltammetry waveforms consist of three steps, each with a time and a potential. The waveform forms a triangle, with the first step always at time zero and the first and third steps always having the same potential. Figure 2-26 shows an example of a triangular waveform used in cyclic voltammetry.

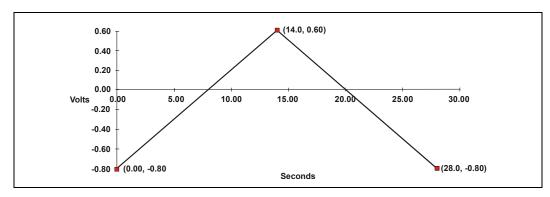


Figure 2-26. Example Cyclic Voltammetry Waveforms

In this example, the potential is scanned from -0.80 to +0.60 V and then from +0.60 to -0.80 V. The total time for one waveform period is 28 s. This provides a sweep rate of 0.1 V/s as shown in the equation below.

$$\frac{2 \times \Delta V}{CV \, Cycle \, Time} = Sweep \, Rate$$

$$\frac{2 \times 1.4V}{28s} = 0.1 \, \frac{V}{s}$$

Cyclic voltammetry waveforms are defined manually on the Chromeleon or Chromeleon Xpress electrochemical detector Control panel. Click the **CV Mode** button to display the following window.

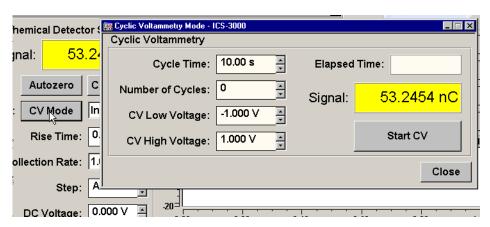


Figure 2-27. Cyclic Voltammetry Control Panel

Integrated Amperometry Waveforms

Integrated amperometry waveforms are included in a Program file in Chromeleon or Chromeleon Xpress. 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, for 2D data, 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:

Data Collection Rate = 1/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.

Chromeleon and Chromeleon Xpress provide several pre-programmed waveforms (see Figure 2-28).

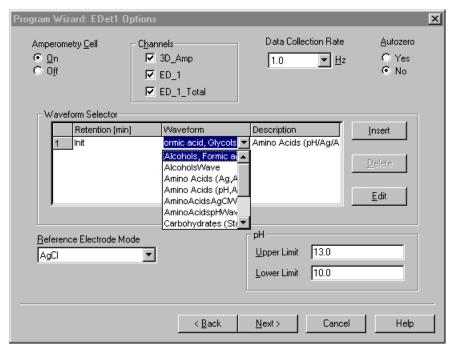


Figure 2-28. Pre-Programmed Waveforms

You can use a pre-programmed waveform, without modification, or modify it for your application. You can also define a new waveform. Waveforms are defined and modified in the Waveform Editor (see Figure 2-29). Click the Edit button on the **Program Wizard: ED** Options page to view the Waveform Editor.

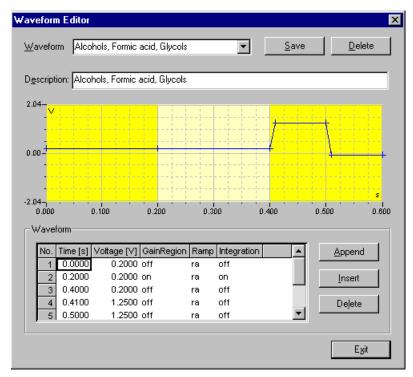


Figure 2-29. Waveform Editor

Analytical vs. Scanning Waveforms

The pre-programmed analytical waveforms supplied with Chromeleon are designed for quantitative analysis of specific compounds (alcohols, amino acids, carbohydrates, etc.). With analytical waveforms, integration occurs either while a single constant potential is being applied over time (see the example waveform in <u>Figure 2-24</u>) or while a series of stepped potentials are being applied (see the example waveform in <u>Figure 2-25</u>).

With scanning waveforms, integration occurs while the potential is being increased linearly over time (see the example scanning waveform in Figure 2-30). Although scanning waveforms are typically not as useful for quantitative analysis as analytical waveforms, when used to collect 3D amperometric data, scanning waveforms can be optimized to provide characteristic I-t plots (see Section 2.16.7). Comparison of I-t plots of a known substance with an unknown can help to identify unknown peaks or to determine whether a substance is coeluting with another.

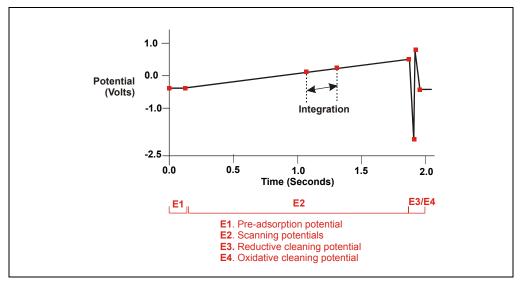


Figure 2-30. Example Scanning Waveform

2.16.7 Storing and Reprocessing Amperometry Data

NOTE Chromeleon Xpress cannot store or reprocess amperometry data.

Chromeleon provides storage of both 2D and 3D pulsed amperometry and integrated amperometry data. For 2D 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 2-31. The retention time (in minutes) is on the x-axis and the detector response (in nanoCoulombs) is on the y-axis.

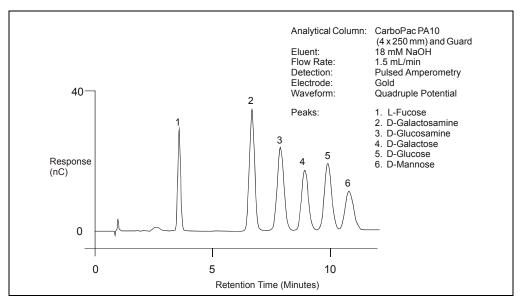


Figure 2-31. Example Chromatogram for a Pulsed Amperometry Application

For 3D integrated amperometry data, Chromeleon stores raw data points at a rate of 1 KHz throughout the entire run. Each raw 3D data point is defined by the following attributes: the detector current (1) recorded at a particular waveform time (2) and at a specific retention time (3). As a result, the two-dimensional view of an integrated amperometry chromatogram (retention time vs. integrated current) is extended by a third dimension (waveform time). Thus, data for the entire waveform period—not just the integration intervals—is collected. For details about

how to acquire 3D amperometry data, refer to the 3D Amperometry User's Manual (Document No. 065218).

Chromeleon displays and lets you reprocess the 3D data in the 3D Amperometry View window (see <u>Figure 2-32</u>). To open the window, double-click the sample in the Browser to open the chromatogram. On the **View** menu, click **3D-Amperometry**,

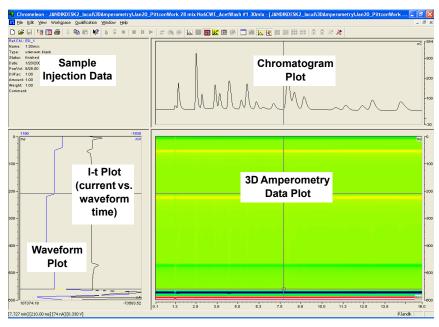


Figure 2-32. 3D Amperometry View Window in Chromeleon

The window is divided into several display areas:

Sample injection data	Displays information about the sample.
Chromatogram plot	Displays a chromatogram of data selected in the 3D amperometry data plot.
3D amperometry data plot	Displays the raw 3D amperometry data.
Waveform/I-t plot	Displays an I-t plot (current vs. waveform time) of selected data and optionally a waveform plot.

The raw 3D amperometry data plot can be viewed as either an Iso or a 3D (wireframe) plot. For both plot types, colors are used to represent the ranges of response values.

The Iso view is the default view for the 3D raw data. This is a top down view of the data (imagine you are looking down on the data from above the plot) (see <u>Figure 2-33</u>). The x-axis is the retention time (min) and the y-axis is the waveform period (ms). The z-axis, which is not visible on the plot, is the response (nA).

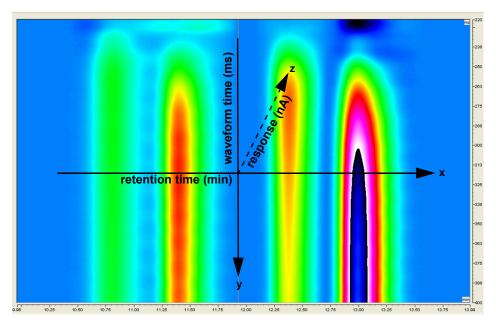


Figure 2-33. 3D Amperometry Data in Chromeleon: Iso View

NOTE The 3D data shown in Figures 2-33 through 2-36 was collected using an analytical waveform (see "Analytical vs. Scanning Waveforms" on page 67).

To switch to the 3D plot view, right-click on the Iso plot, select **Decoration** and click the **Iso/3D Plot** tab. The 3D view projects the response values in the third dimension, which allows you to see the height

of responses as well as the color mappings (see <u>Figure 2-34</u>). For this view, imagine you are standing in front and slightly to the left of the plot.

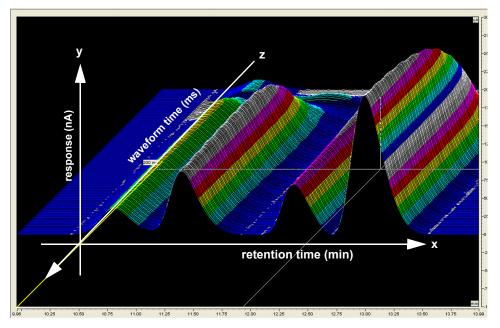


Figure 2-34. 3D Amperometry Data in Chromeleon: 3D View

Baseline Correction

Based on the peak recognition algorithm, Chromeleon can calculate a baseline I-t plot for each data point of a peak. Each I-t plot measured at a given retention time can be corrected by subtracting the calculated baseline I-t plot from the 3D amperometry data. By definition, if baseline correction is enabled, 3D amperometry data is zero unless a peak is present.

To enable baseline correction, select **Baseline correction** on the **General** tab page of the 3D Amperometry Decoration dialog box.

<u>Figure 2-35</u> is an example of 3D amperometry data before baseline correction is enabled.

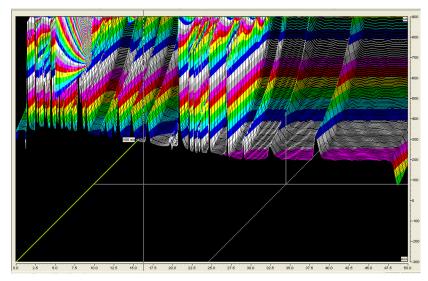


Figure 2-35. 3D Amperometry Data Before Baseline Correction

Figure 2-36 shows the same data after baseline correction is enabled.

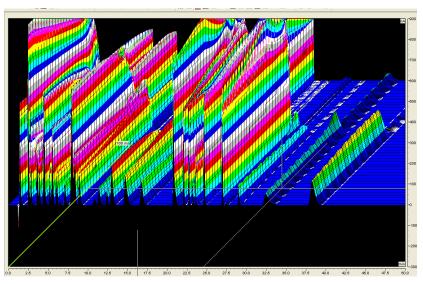


Figure 2-36. 3D Amperometry Data After Baseline Correction

2.17 Automation Manager

The ICS-3000 Automation Manager (AM) provides a mounting location for various components used for performing matrix elimination, large volume preconcentration, post-column reagent addition, and other functions.

Each AM consists of a tray on which valves and other components are installed (see <u>Figure 2-37</u>). The tray is installed in the upper compartment of the DC (see <u>Figure 2-13</u>). Connections to other components in the system (pump, injection valve, reagent reservoirs, etc.) depend on the application. Refer to <u>Chapter 3</u> for the configuration schematic for your application.

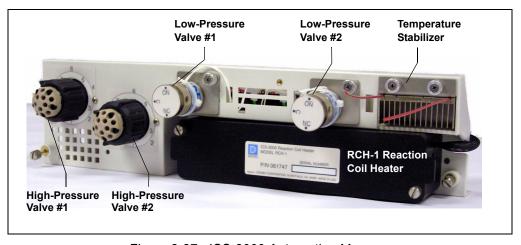


Figure 2-37. ICS-3000 Automation Manager

The AM is available in the following configurations:

Components Included	AM Part Number
Two 10-port high-pressure valves Two low-pressure 3-way valves	061738
One 10-port high-pressure valve One low-pressure 3-way valve	061736
One 6-port high-pressure valve One low-pressure 3-way valve	061740
AM tray with no valves	061734

In addition to the configurations described above, you can order the following components separately for installation on an AM:

AM Component	Part Number
High-pressure valve, 6-port	061961
High-pressure valve, 10-port	061962
Low-pressure valve, 3-way	061971
Low-pressure valve, 2-way	061745
RCH-1 Reaction Coil Heater	061746
Temperature stabilizer, standard bore, 0.25-mm (0.010-in) ID	062561
Temperature stabilizer, microbore, 0.125-mm (0.005-in) ID	062562

2.17.1 High-Pressure Switching Valves

Up to two high-pressure switching valves can be installed on an AM. Two models are available: 6-port (P/N 061961) and 10-port (P/N 061962). Both models are electrically-activated, two-position valves. Figure 2-38 and Figure 2-39 show the liquid flow path through the valve ports at each valve position.

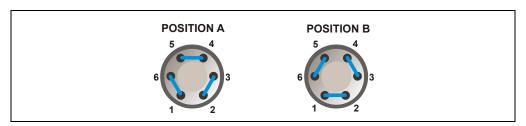


Figure 2-38. High-Pressure Switching Valve Flow Schematics: 6-Port Valve

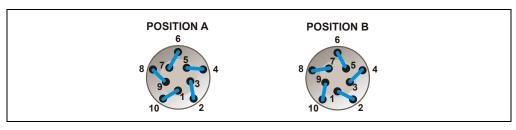


Figure 2-39. High-Pressure Switching Valve Flow Schematics: 10-Port Valve

Valve port connections to chromatography components vary, depending on the application. Refer to <u>Chapter 3</u> for the configuration schematic for your application.

Valves are controlled by Chromeleon or Chromeleon Xpress (see Section 2.17.3).

2.17.2 Low-Pressure Valves

Up to two low-pressure valves can be mounted on an AM. The valves are either two-way or three-way valves. 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 2-40).

Valve port connections to chromatography components vary, depending on the application. Refer to <u>Chapter 3</u> for the system flow schematic for your application.

Valves are controlled by Chromeleon or Chromeleon Xpress (see Section 2.17.3).

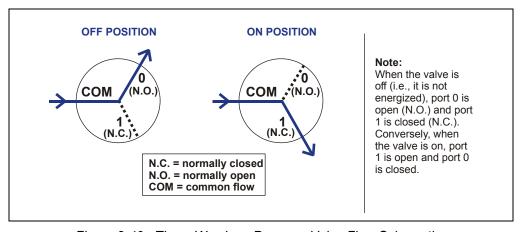


Figure 2-40. Three-Way Low-Pressure Valve Flow Schematics

2.17.3 High- and Low-Pressure Valve Control

Chromeleon or Chromeleon Xpress is used to control the high- and low-pressure valves. Commands for valve control can be included in a program (see <u>Figure 2-41</u>) for automated control, or the valves can be controlled manually from a Control panel (see <u>Figure 2-42</u>).

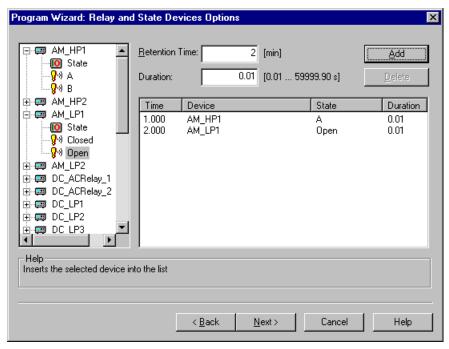


Figure 2-41. Program Wizard Relay and State Devices Options Page

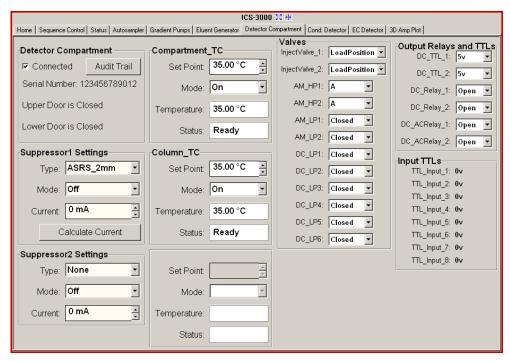


Figure 2-42. DC Control Panel

2.17.4 RCH-1 Reaction Coil Heater

The RCH-1 Reaction Coil Heater (P/N 061746) can hold up to two reaction coils. The heater has an operating temperature range of from 5 °C above the temperature of the upper compartment up to 80 °C.

Commands for heater control can be included in a program (see <u>Figure 2-43</u>) for automated control, or the heater can be controlled manually from the DC Control panel (see Figure 2-42).

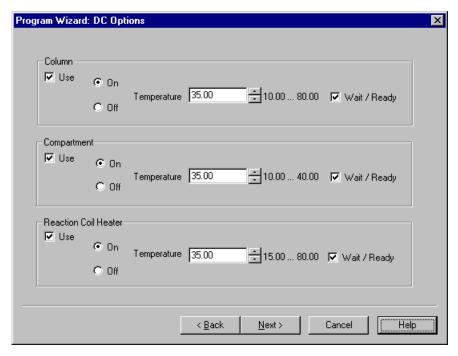


Figure 2-43. Program Wizard DC Options Page

2.18 I/O Option

When the I/O option (P/N 062201) is installed, two 12-pin connector strips are on the DC rear panel. Figure 2-44 describes the functions assigned to each connector pin.

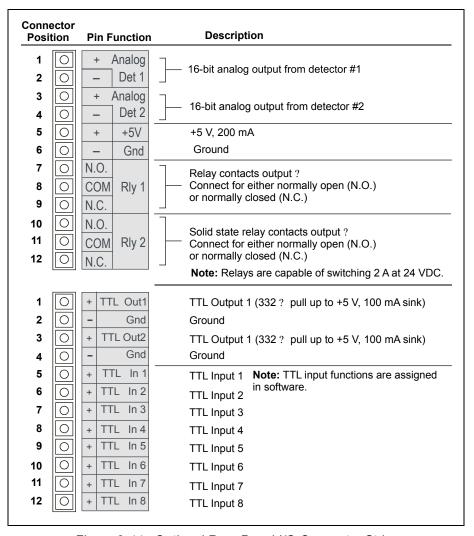
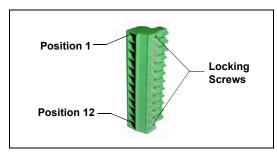


Figure 2-44. Optional Rear Panel I/O Connector Strips

2.18.1 I/O Option Connections

1. Locate the twisted pair of wires (P/N 043598) and 12-position connector plugs (P/N 923686) (see Figure 2-45) provided with the I/O option board.



2. For each I/O function to be used, connect

Figure 2-45. 12-Position Connector Plug

an active wire (red) and a ground wire (black) to the 12-position connector plug at the appropriate pin locations. Refer to <u>Figure 2-44</u> or the label on the DC rear panel for the connector pin assignments.

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.



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.

- 3. Plug the connector into the appropriate 12-pin connector on the DC rear panel.
- 4. Connect the wires from the DC 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.

5. 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. Input functions and control types are assigned from the Chromeleon Server Configuration program (see Section 2.18.4).

2.18.2 Analog Outputs

When the I/O option is installed, two analog outputs (one for each detector) are installed on the DC rear panel (see <u>Figure 2-15</u>). The analog outputs supply a voltage signal proportional to the current measured by the detector cell. The outputs can be connected to an analog-to-digital (A/D) converter such as an integrator or other recording device. Refer to <u>Section 2.18.1</u> and the documentation for the device for connection instructions.

Several settings are available that allow you to configure the analog output signal for your detector and connected device. <u>Table 2-2</u> describes the settings. You select the desired settings on the detector Control panel (see <u>Figure 2-46</u>).

Analog Output Setting	Values	Description
Full-scale voltage	0.01, 0.10, or 1.00 V	Sets the voltage output of a full-scale detector response. The voltage to use depends on the recording device to which the analog output is connected. For example, if the analog output is connected to a device that accepts input voltages up to 1 V, select a full-scale voltage output of 1 V.
Range	Conductivity: 0.01 to 15,000 μS DC Amperometry: 50 pA to 300 μA Integrated Amperometry: 50 pC to 200 μC	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 microSiemens (μ S) will limit you to viewing conductivity readings of 20 μ S or less.

Table 2-2. Analog Output Configuration Settings

Analog Output Setting	Values	Description
Recorder calibration	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.
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.

Table 2-2. Analog Output Configuration Settings (Continued)

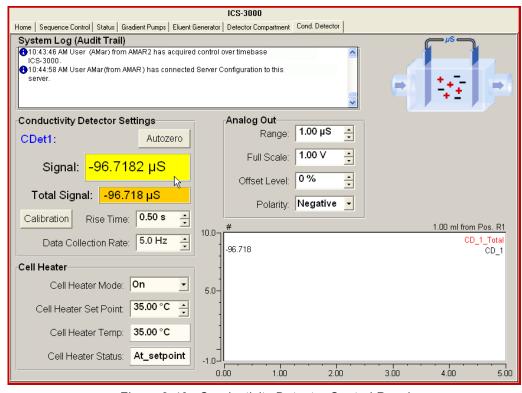


Figure 2-46. Conductivity Detector Control Panel

2.18.3 Power, Relay, and TTL Outputs

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

Depending on which pins are connected, the relay connection can be either normally open (N.O.) or normally closed (N.C.) (see <u>Figure 2-47</u>). Choose N.O. or N.C. based on what you want the state of the connected device to be when the DC 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.

The relays can be programmed to switch any low-voltage device. Switched current must be no more than 2 A at 24 VDC. Refer to Section 2.18.1 and the documentation for the external device for connection instructions.

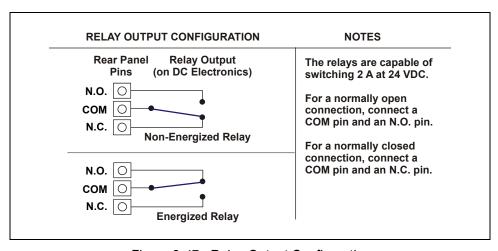


Figure 2-47. Relay Output Configuration

You can control the power, relays, and TTL outputs from the DC Control panel in Chromeleon or Chromeleon Xpress (see Figure 2-48).

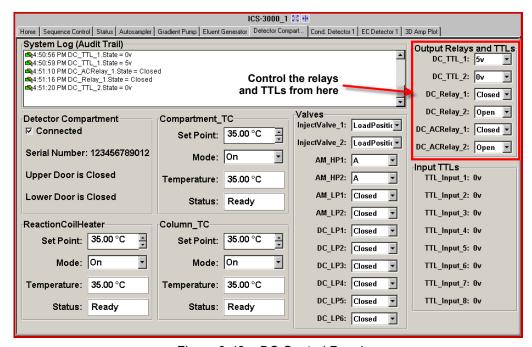


Figure 2-48. DC Control Panel

NOTE It is possible to change the settings for the power, relays, and TTL outputs while a Chromeleon or Chromeleon Xpress program is running.

2.18.4 TTL Inputs

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

- Injection valves left and right (load/inject)
- AM high-pressure valves A and B (A/B)
- AM low-pressure valves A and B (open/closed)
- DC low-pressure valves A through F on the rear panel (open/closed)
- ED detectors 1 and 2 (on/off)
- CD/ED detectors 1 and 2 auto-offset
- CD/ED detectors 1 and 2 mark
- Suppressors 1 and 2 (on/off)
- Reaction coil heater (on/off)
- A/C relays 1 and 2 (open/closed)

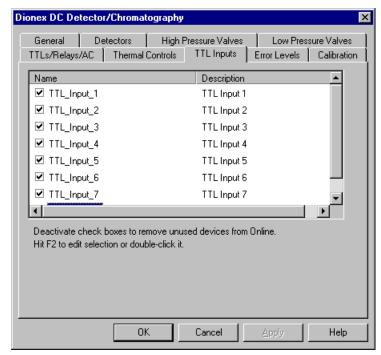
Assigning TTL Input Functions

TTL input control functions are assigned in the Chromeleon Server Configuration program. You can assign one or more functions to each input.

NOTE If two timebases are sharing the DC, the TTL input functions are shared between the timebases. For example, if TTL input one is assigned to control injection valve 1, that function is assigned to both timebases. You do not need to associate a TTL input with a particular timebase.

To select TTL input control functions:

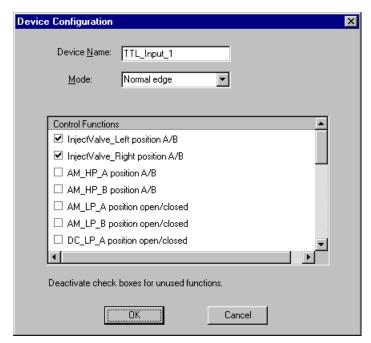
- 1. Open the Chromeleon Server Configuration program.
- 2. Double-click the DC icon under the timebase.



3. Select the **TTL Inputs** tab (see <u>Figure 2-49</u>).

Figure 2-49. DC Server 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.

Figure 2-50. Assign TTL Input Control Functions

- In the Control Functions list, select the check box of one or more functions 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.
- 6. By default, several functions are selected. Scroll down the list and disable all unwanted functions.

TTL Input Control Types

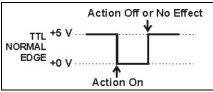


The DC TTL inputs respond to four types of signals to accommodate different controlling devices. The default control type, **Normal Edge**, is compatible with the output signals provided by Dionex modules.

If the device connected to the DC does not send a normal edge signal, select the appropriate control type. Refer to the documentation provided with the controlling device and the information below to determine the

correct type. Select the input control type in the Device Configuration dialog box for each TTL input (see Figure 2-50).

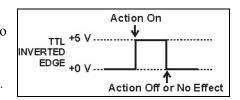
 Normal Edge: In normal edge operation, the negative (falling) edge of a signal turns on the function.



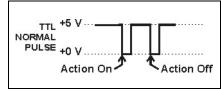
The action of the positive Action On (rising) edge depends on the function: For on/off or other functions that have two options, the rising edge turns off the function. However, for functions with only one option, the rising edge has no effect.

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 detector start/stop, the falling edge starts the detector and the rising edge stops it. For the detector mark and offset functions, the falling edge turns on the function and the rising edge has no effect.

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

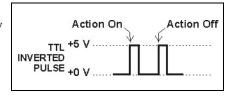


 Normal Pulse: In normal pulse operation, the negative (falling) edge of the TTL signal is the active edge and the positive (rising) edge is ignored.



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.

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





2.19 TC Front Features

A status bar on the front of the ICS-3000 Thermal Compartment (TC) includes LEDs (light emitting diodes) that indicate the status of several TC components and functions (see Figure 2-51).

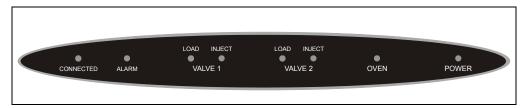


Figure 2-51. TC Status Bar

LED Label	If the LED is On	If the LED is Flashing
CONNECTED	The TC is connected to a Chromeleon or Chromeleon Xpress timebase.	Does not flash.
ALARM	A sensor detected the presence of a gas or humidity leak inside the TC, or a valve or oven error was detected. Check the Chromeleon or Chromeleon Xpress Audit Trail for the cause.	Does not flash.
VALVE 1 LOAD VALVE 2 LOAD VALVE 1 INJECT VALVE 2 INJECT	Indicates whether the injection valve is in the Load or Inject position.	Valve error. See <u>Section 8.30</u> for troubleshooting.

LED Label	If the LED Is On	If the LED is Flashing
OVEN	The TC is at its set temperature.	The TC is transitioning to the set temperature. The TC is not ready for operation. If the LED is flashing and the ALARM LED is lighted, an oven error has been detected. See Section 8.30 for troubleshooting.
POWER	When the power is on, this LED is lighted. Use the POWER button on the front of the TC for routine on/off control. To turn on the TC, press the POWER button. To turn off the TC, press and hold the POWER button for 2 seconds.	Does not flash.

NOTE The main power switch is on the TC rear panel (see Figure 2-53).

2.20 TC Interior Components

<u>Figure 2-52</u> illustrates the interior of the TC. The components installed in your TC may vary, depending on your application.

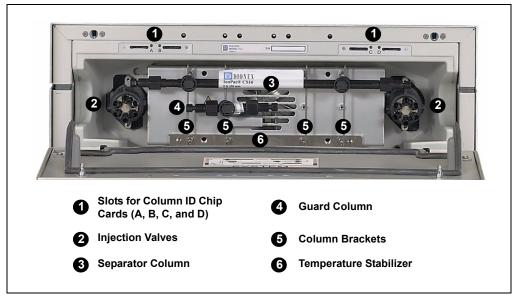


Figure 2-52. TC Interior View: Example Configuration

Injection Valves

The TC can hold one or two electrically-actuated, high-pressure injection valves. Two valve models are available:

- A 2-position, 6-port Rheodyne TitanHPTM injection valve
- A 2-position, 10-port Rheodyne TitanHP™ injection valve

See Section 2.22 for details about the injection valves.

Column Brackets

The column brackets are installed in the TC at the factory. The TC can hold up to three columns with an inner diameter of 1 to 9 mm and a maximum length of 30 cm (11.8 in). Columns are attached to the brackets with special column clips (P/N 064786; set of six clips).

Temperature Stabilizers (Optional)

Installation of a temperature stabilizer (standard bore, P/N 064548; microbore, P/N 064650) ensures that (a) the temperature of the stationary phase remains constant over the total column length and (b) the column and the eluent have the same temperature during the analysis.

When these conditions are met, the analytical separation is performed at the nominal temperature. This minimizes fronting and tailing of peaks, as well as retention time variations

Column ID Chip Card Slots

The TC column ID (identification) system lets you store column-specific information on a memory card and read the information at any time. Up to four column ID chip cards can be installed at the same time. The TC is shipped with two column ID chip cards (P/N 5710.1500). An optional Column ID Kit (P/N 066353) containing five chip cards is available.

See Section 2.23 for details about the column ID system.

2.21 TC Rear Panel

<u>Figure 2-53</u> illustrates the rear panel of the TC.

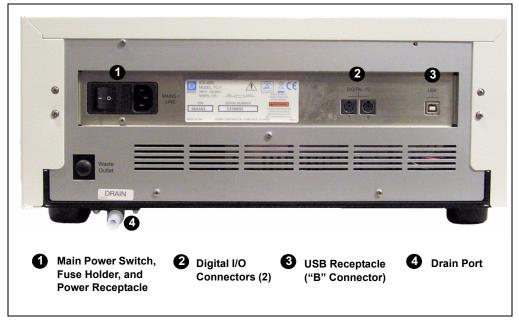


Figure 2-53. TC Rear Panel

Main Power Switch, Fuse Holder, and Power Receptacle

The rear panel power switch is the main power switch for the TC. Turn on the main power switch before initial operation, and leave the switch on unless instructed to turn it off (for example, before performing a service procedure).

NOTE For routine on/off control of the TC, use the POWER button on the front of the module. To turn off the TC, press and hold the POWER button for 2 seconds.

The fuse holder contains two 2-amp fuses (P/N 954776). For instructions on how to change the fuses, see <u>Section 9.29</u>.

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



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



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.



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

Digital I/O Connectors

The two 6-pin mini-DIN connectors can be used to exchange digital signals with any external device capable of being controlled through a relay or TTL input. The two connectors are identical in function.

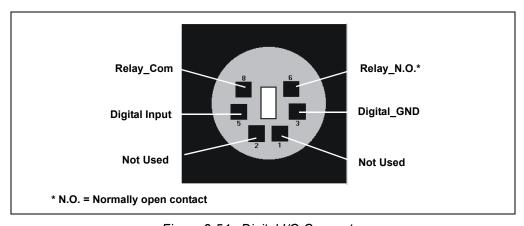


Figure 2-54. Digital I/O Connector

When the relay is activated, the connection is between Com and N.O.

Connection	Description
Digital Input <> Digital_GND	0 to +5 V
Relay_Com <> Relay_N.O.	Switching voltage: 100 VDC Switching current: 0.25 A Carry current: 1.0 A Switching capacity: 3 W Contact resistance: Max. 150 megohms

Use the 6-pin mini-DIN signal cable (P/N 6000.1004) provided in the TC Ship Kit (P/N 064789) to connect the TC to an external device. For details about the pin assignments, refer to <u>Table 2-3</u>.

Pin	Signal Name	Signal Level	Core Color	Core Label
1	Not occupied		Pink	Analog High
2	Not occupied		Gray	Analog Low
3	Digital_GND	0 V	Green	Digital GND
5	Digital Input	0 to +5 V	Yellow	Digital Input
6	Relay_N.O.	$100 \text{ V}_{DC}/0.25 \text{ A/3 W}$	Brown	Digital Output
8	Relay_Com	$100 \text{ V}_{DC}/0.25 \text{ A/3 W}$	White	Dig./Out (GND)
			Black	Shield

Table 2-3. Pin Assignments for the TC Mini-DIN Signal Cable

USB Receptacle

The USB (Universal Serial Bus) receptacle ("B" type connector) provides a connection to the PC on which Chromeleon or Chromeleon Xpress is installed. Select one of the following methods to establish the connection:

- Connect the TC directly to the USB receptacle on the PC.
- Connect the TC to the PC through another USB device in the system (for example, the pump).

The TC Ship Kit (P/N 064789) includes a 1-m (3-ft) USB cable (P/N 063246).

Drain Port

The drain port provides a connection for the waste line (P/N 014157) that removes condensing water and liquid leaks from the interior of the TC. The tubing should be directed to an appropriate waste container.



The waste line must remain below the drain port. If the waste line is above the drain port, liquid may flow back into the TC and cause damage to the instrument. Make sure the waste line is not bent or kinked at any point.

2.22 Injection Valves

The TC is available in the following configurations:

TC Description	Part Number
TC with one 2-position, 6-port high-pressure injection valve	064660
TC with two 2-position, 6-port high-pressure injection valves	064661
TC with one 2-position, 6-port high-pressure injection valve and one 2-position, 10-port high-pressure injection valve (The 6-port valve is installed on the left side of the module.)	064651
TC with no injection valves	064659

2.22.1 Injection Valve Operation

The 6-port injection valve has two operating positions: Load and Inject. Liquid flows through either the Load or Inject path, depending on the valve position.

<u>Figure 2-55</u> shows flow schematics for the 6-port valve.

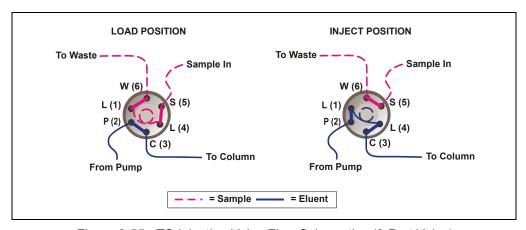


Figure 2-55. TC Injection Valve Flow Schematics (6-Port Valve)

• In the Load position, sample is loaded into the sample loop, where it is held until injection. Eluent flows from the pump, through the valve, and to the column, bypassing the sample loop. Sample flows from the syringe or autosampler line (if installed), through the valve, and into the sample loop. Excess sample flows out to waste.

• In the Inject position, sample is swept to the column for analysis. Eluent flows from the pump, through the sample loop, and on to the column, carrying the contents of the sample loop with it. Section 5.2 describes how to inject samples.

The 10-port injection valve has two operating positions: 10_1 and 1_2. Liquid flows through the path between either ports 10 and 1 or ports 1 and 2, depending on the valve position.

<u>Figure 2-56</u> shows example flow schematics for the 10-port valve when it is connected for an application that uses a concentrator column.

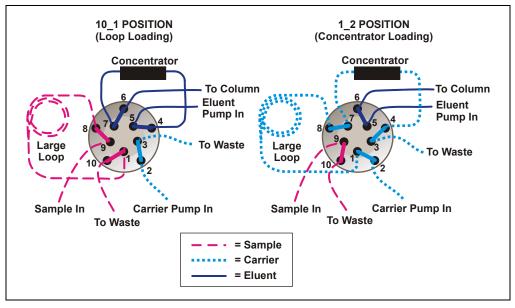


Figure 2-56. TC Injection Valve Flow Schematics (10-Port Valve)
Example Connections: Large Loop to Concentrator

- In the 10_1 position, sample flows from the syringe or autosampler line, through the valve, and into the sample loop. Excess sample flows out to waste. Eluent flows from the eluent pump, through the valve, through the concentrator column, and to the separator column. If sample was previously loaded onto the concentrator column, it is swept to the separator column for analysis.
- In the 1_2 position, carrier liquid flows through the sample loop and on to the concentrator column, carrying the contents of the sample

loop with it. Excess flows out to waste. Eluent flows from the pump, through the valve, and to the separator column, bypassing the concentrator column.

2.22.2 Injection Valve Plumbing

Figure 2-57 shows the injection valve connections for the 6-port valve. The injection valve is plumbed at the factory with all tubing and fittings for connection to the pump, sample loading port, column, and waste. A 25 μ L PEEK sample loop (P/N 042857) is installed between ports L (1) and L (4).

Dionex offers sample loops in various sizes. If needed, the pre-installed 25 μL loop can be replaced with a loop that has a different sample injection volume.

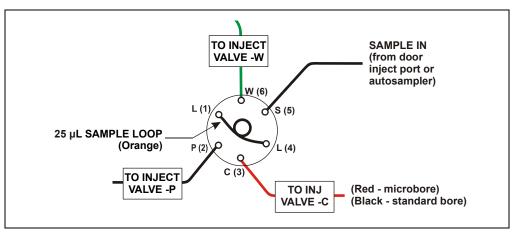


Figure 2-57. Injection Valve Plumbing (6-Port Valve)

2.23 Column Identification (ID) System

The electronic column ID system stores column-specific information on a column ID chip card (P/N 5710.1500) that is connected to a column for the column life cycle (see Figure 2-58).

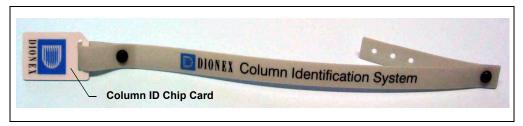


Figure 2-58. Column ID (Identification) System

The columns to be monitored are selected in the TC Properties dialog box in the Server Configuration program. Use the TC Control panel (or the Commands dialog box) in Chromeleon or Chromeleon Xpress to select column properties (see below) for storage and to review the stored information at any time. Information on the column ID chip card is continuously updated, as long as the column and the chip card are installed in the TC.

Column Properties

Two types of information can be stored on the column ID chip card:

- Primary column properties are entered once by the column manufacturer or the user and are never changed during the lifetime of the column. The primary properties are the product ID, serial number, batch/lot number, date of manufacture, and custom information.
- Secondary column properties are entered by the user and can be changed
 whenever required by any Chromeleon or Chromeleon Xpress user with
 Expert privileges. Secondary properties include the column manufacturer,
 diameter, length, particle size, maximum number of injections, pH limits, and
 upper pressure limit. For a complete list of secondary properties, refer to the
 Chromeleon or Chromeleon Xpress Help.

2.24 Gas and Humidity Sensors

The TC contains two sensors to detect any gas or humidity that may accumulate inside the TC. When a certain concentration of gas or humidity is reached (while the door is closed), the following events occur:

- The corresponding sensor is activated.
- The **ALARM** LED on the front of the TC lights (red), and remains red as long as the sensor is exposed to gases or humidity.
- The error is logged in the Chromeleon or Chromeleon Xpress Audit Trail.
- A beep sounds.

What to do when an alarm occurs:

- 1. Find and eliminate the leak.
- 2. On the TC Control panel, click the **Clear Alarm** button.
- 3. Ventilate the interior of the TC thoroughly before closing the door and resuming operation.

Adjusting the Sensor Sensitivity

Follow these steps to change the sensitivity setting of the gas or humidity sensor, or to turn off a sensor:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- 2. Under TC_Temp, select the GasLeakSensor or HumidityLeakSensor command.
- 3. Select the new sensitivity setting and click **Execute**.

To disable the sensor, select **Off** and click **Execute**.

2.25 Theory of Operation

The fundamental requirement for a thermal compartment intended for chromatography applications is the ability to maintain the preset temperature as precisely as possible. The TC contains advanced electronic circuitry that maintains the selected temperature with a precision of ± 0.1 °C.

The TC can be heated from 5 to 85 °C in 0.1 °C increments. An increase in temperature from 20 to 50 °C (or a decrease from 50 to 20 °C) is typically realized in less than 20 minutes. Columns can be warmed up to 85 °C. If the ambient temperature changes during the analysis, increased heating or cooling ensures that the column temperature remains constant.

IMPORTANT

Operating IC columns at temperatures above 70 °C (158 °F) may affect column performance. Before selecting the TC temperature, refer to the column manual for the recommended operating conditions. Column manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891).

The thermo-optimized design of the TC reduces the time required to equilibrate the temperature between the column and the eluent. Peltier elements in the TC heat up and cool down the columns. They also help to bring the air and all of the installed components (including the injection valves) to the desired temperature.

If necessary, a temperature stabilizer (standard bore, P/N 064548; microbore, P/N 064650) can be installed in the TC. Installation of a temperature stabilizer ensures that:

- The stationary phase temperature remains constant over the total column length.
- The column and the eluent have the same temperature during the analysis.

Under these conditions, the separation is performed at the nominal temperature. This minimizes fronting and tailing of peaks, as well as retention time variations.

The temperature achieved may vary from the control range, depending on the ambient temperature (see the table below).

Control Range	ontrol Range Achieved Temperatures (Based on Ambient)	
5 to 85 °C	Minimum temperature 18 °C below ambient	
	Maximum temperature 70 °C above ambient	

For example, if the ambient air temperature in the lab is 30 °C and you set the temperature to 5 °C, the actual temperature achieved will be about 12 °C.



Do not touch any metal parts inside the TC while it is heating up or after it reaches the set point temperature. Wait for the compartment to cool down before servicing any parts.



Ne touchez à aucune des pièces métalliques à l'intérieur du TC pendant qu'il chauffe ou après qu'il ait atteint la température de consigne. Attendez que le compartiment soit refroidi avant de réparer les pièces.



Berühren Sie im TC keine metallischen Teile, während dieses aufgeheizt wird oder nachdem die Solltemperatur erreicht ist. Warten Sie, bis die Kammer abgekühlt ist, ehe Sie Wartungsarbeiten durchführen.

2.25.1 Predictive Performance

The Predictive Performance feature of the TC provides various functions for estimating the lifetime of replaceable parts and for planning and recording service and qualification procedures.

A partial list of the TC Predictive Performance functions is provided below. Refer to the Chromeleon or Chromeleon Xpress Help for details.

- Monitoring of the total cooling and heating workload performed by the compartment and the total operating hours since initial operation
- Monitoring of the injection valve usage
- Reminders for service and qualification periods, including display of warning messages when limits are approaching or overdue

Predictive Performance commands and parameters are available in the Commands dialog box.

3.1 Overview

This chapter provides some example component and plumbing drawings for the following ICS-3000 system configurations:

ICS-3000 Configuration	See
IC System (CD)	page 106
Reagent-Free IC (RFIC) (single system)	page 107
RFIC Dual System with two CDs	page 108
RFIC Dual System with CD and ED	page 109
RFIC with Matrix Elimination Using a DP and a 6-Port Valve	page 110
RFIC with Matrix Elimination Using an SP and a 10-Port Valve	page 111
RFIC with Post-Column Reagent Addition (Bromate)	page 112

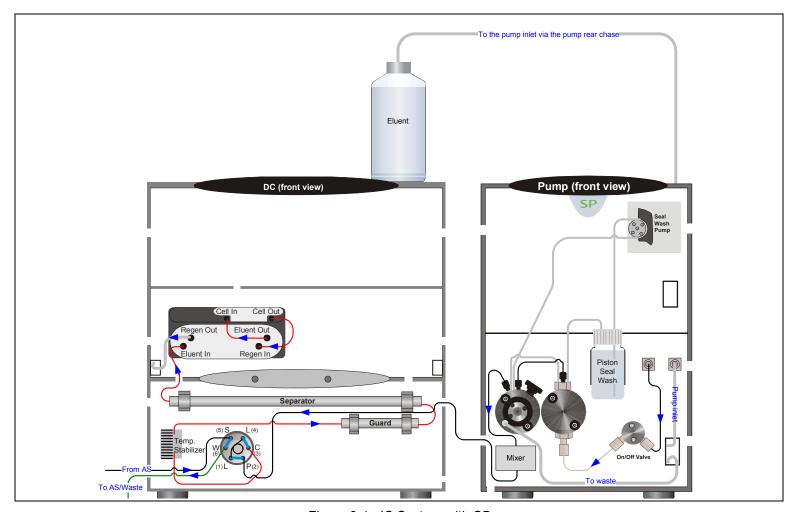


Figure 3-1. IC System with CD

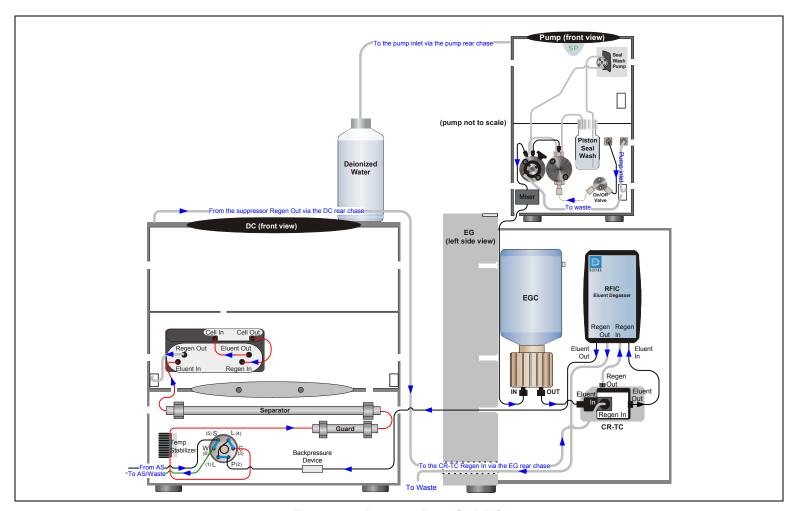


Figure 3-2. Reagent-Free IC (RFIC)

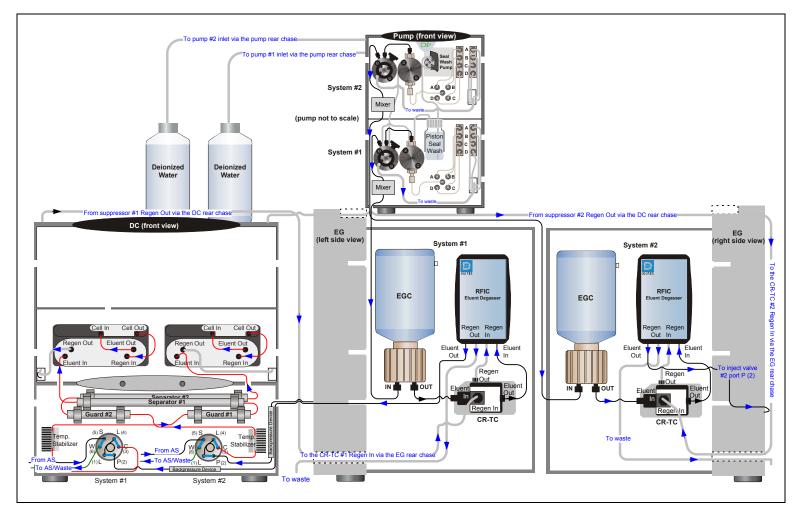


Figure 3-3. RFIC Dual System (with two CDs)

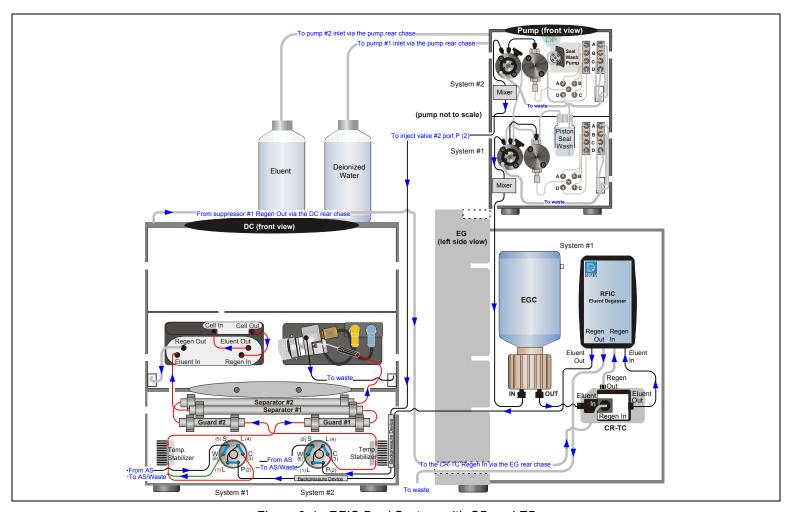


Figure 3-4. RFIC Dual System with CD and ED

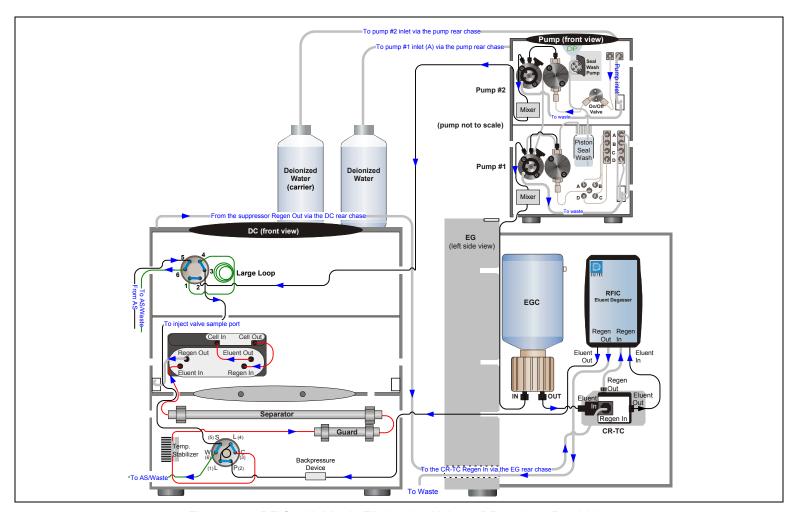


Figure 3-5. RFIC with Matrix Elimination Using a DP and a 6-Port Valve

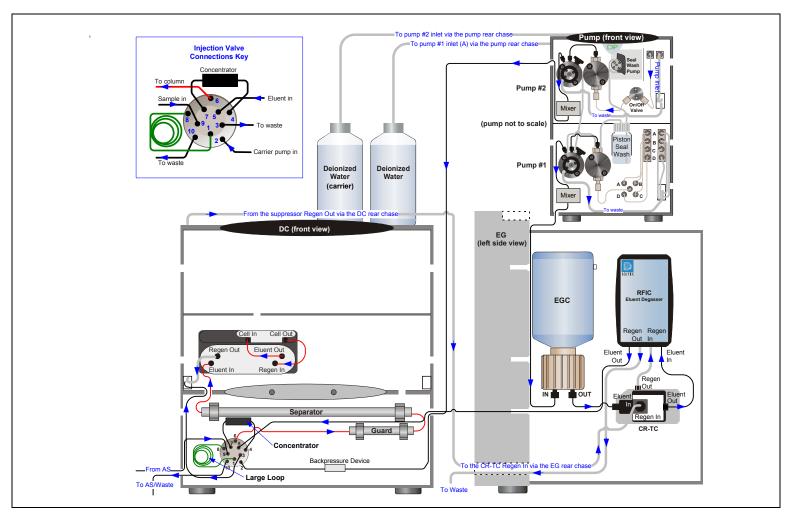


Figure 3-6. RFIC with Matrix Elimination Using a DP and a 10-Port Valve

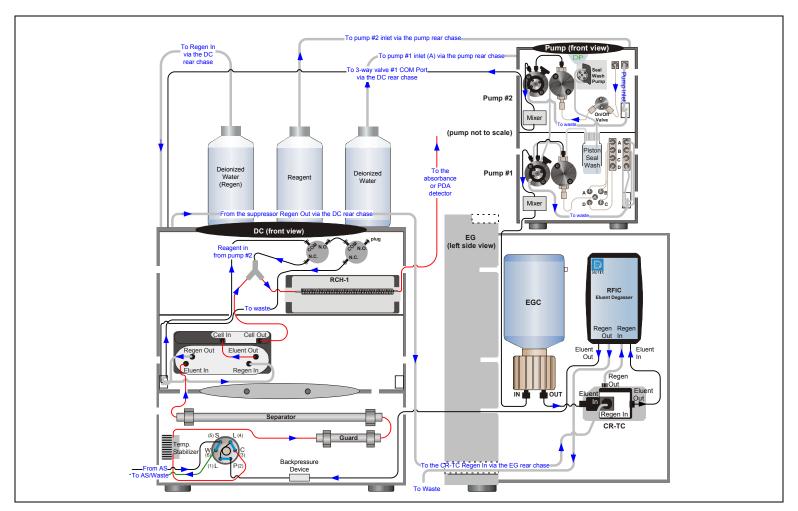


Figure 3-7. RFIC with Post-Column Reagent Addition (Bromate)

This section is an overview of the steps required to start up the ICS-3000 system components and prepare the system to run samples. The operating parameters (flow rate, compartment temperature, suppressor current, etc.) depend on the application you plan to run. Refer to the column manual, as well as the schematics in Chapter 3 of this manual, for the required operating parameters.

NOTE Before beginning operation, be sure to perform any special startup procedures required for the columns, suppressors, etc. Startup procedures are described in the quick start guides and manuals provided on the Dionex Reference Library CD-ROM (P/N 053891).

4.1 Operating Precautions

4.1.1 EG Operating Precautions

• The EluGen cartridge requires at least 14 MPa (2000 psi) of system backpressure. This ensures optimal removal of electrolysis gas from the eluent produced by the cartridge.

During system equilibration, monitor the **Current Pressure** reading on the pump Control panel in Chromeleon or Chromeleon Xpress; the pressure should remain between 14 and 21 MPa (2000 and 3000 psi). If necessary, increase the system backpressure by installing a backpressure coil between the injection valve and the **ELUENT OUT** port on the EluGen cartridge. For instructions, refer to Section 9.15.

IMPORTANT

The recommended maximum operating pressure for the EG is 21 MPa (3000 psi). Excessive backpressure may rupture the tubing inside the RFIC Eluent Degasser.



DO NOT CAP THE WASTE CONTAINER: The ICS-3000 Eluent Generator (EG), Atlas Electrolytic Suppressor, and Self-Regenerating Suppressor (SRS) use an electrolytic process that results in the production of small amounts of oxygen or hydrogen gas. To ensure that the gas is not trapped in a closed container and allowed to concentrate, install a 1.3-cm (0.52-in) ID black gas separator waste tube (P/N 045460) in an uncapped waste container. Connect the Waste, Gas Separator line to the waste tube.



NE FERMEZ PAS LE CONTENEUR DE GAZ RÉSIDUEL: Le ICS-3000 Eluent Generator (EG), le Atlas Electrolytic Suppressor, et le Self-Regenerating Suppressor (SRS) travaillent avec un proces d'électrolyse, qui produit des petites quantités de gaz d'oxygène ou d'hydrogène. Afin de garantir que le gaz ne soit pas enfermé dans un conteneur fermé et puisse s'y concentrer, connectez un tube noir à gaz résiduel (diamètre intérieur = 1,3 cm; n° de commande 045460) à un conteneur ouvert (non fermé). Connectez le conteneur résiduel au tube résiduel/gaz séparateur (désigné: « Waste, Gas Separator»).



VERSCHLIESSEN SIE DEN ABFALLBEHÄLTER NICHT: Der ICS-3000 Eluent Generator (EG), Atlas Electrolytic Suppressor und Self-Regenerating Suppressor (SRS) verwenden einen Elektrolyseprozess, wodurch kleine Mengen an Sauerstoff und Wasserstoff entstehen. Führen Sie einen schwarzen Gasabscheiderschlauch (ID = 1,3 cm; Bestell-Nr. 045460) in einen offenen (unverschlossenen) Abfallbehälter, damit sich das Gas nicht in einem geschlossenen Behälter sammelt und aufkonzentriert. Verbinden Sie die mit Waste, Gas Separator bezeichnete Leitung mit dem Abfallschlauch.

- If you select a low flow rate that allows the system pressure to drop below 14 MPa (2000 psi) and you do not turn off the EG, the EG will continue to generate eluent at the concentration set for the last step of the last Chromeleon or Chromeleon Xpress program.
 - If this occurs, the eluent concentration will increase in proportion to the decrease in the flow rate. In extreme cases, excessive heat buildup can occur and damage the EluGen cartridge.
- For a complete list of operating precautions for EluGen cartridges and CR-TC trap columns, refer to the product manuals. The manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891).

4.1.2 ED Amperometry Cell Operating Precautions

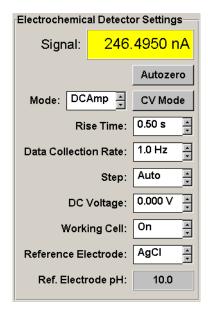
- To prevent electrode contamination:
 - Run only clean, filtered samples.
 - Prepare all eluents with high purity ASTM Type I (18 megohm-cm) filtered and deionized water.
 - Avoid contamination of the cell with incompatible eluents.
- Never apply potential to the electrode unless a stream of eluent or water is flowing through the cell.
- Do not allow the reference electrode to dry out. Make sure that eluent is 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, see Section 6.4.
- Be careful to keep the polished surface of the amperometry 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 Section 9.24.3.
- 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 with coarse and fine polishing compounds as instructed in Section 9.24.3.

- To help determine when the reference electrode needs regenerating or replacing, monitor the pH value displayed on the Control panel in Chromeleon or Chromeleon Xpress.
- To have an alarm displayed in the Audit Trail if the pH exceeds certain values, set pH limits in Chromeleon or Chromeleon Xpress.

To monitor the cell pH:

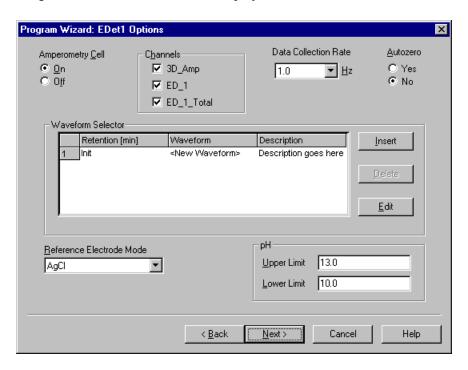
- 1. At installation, calibrate the pH electrode (see Section 9.24.5).
- 2. When you run your first chromatographic program, note the pH value displayed on the Chromeleon or Chromeleon Xpress Control panel.
- 3. Thereafter, monitor the pH value to determine whether there is a shift in the pH. A shift in the pH reading indicates a change in the Ag/AgCl reference potential.

If the pH value shifts by 0.5 pH unit from the value first observed, check the reference electrode (see Section 8.28.6).



To set amperometry cell pH limits:

You can set upper and lower pH limits in the Chromeleon or Chromeleon Xpress Program Wizard. The Audit Trail displays an alarm if the limits are exceeded.



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

4.2 System Startup Checklist

Set up the eluent reservoirs (see <u>page 123</u>)
Set up the piston seal wash system (see page 124)
Start the pump (see page 127)
Set the pressure limits (see <u>page 130</u>)
If an EG is installed, start the EG and set the eluent concentration (see page 131)
Start the DC (see page 135) or TC (see page 137)
Equilibrate the system and verify operational readiness (DC: see <u>page 136</u> ; TC: see <u>page 138</u>)
Prepare the samples (see page 119)
Fill the autosampler vials and load the sample tray (see page 120)

Notes:

- If the system is controlled by Chromeleon, you can use the SmartStartup Wizard to create a startup program and sequence. SmartStartup allows automatic system startup and equilibration.
- The **Start up** button on the **Home** tab on the ICS-3000 panel tabset starts the system components (pump, suppressor, eluent generator, CR-TC, detector, etc.). The flow rate, suppressor current, and other settings that were in effect when the system was shut down are restored.

4.3 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.

4.3.1 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. 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 (39° F) 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.

4.3.2 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 High Pressure Inline Filter (P/N 044105) 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 located on the Dionex Reference Library CD-ROM (P/N 053891).

4.3.3 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.

Use eluent or ASTM Type I (18 megohm-cm) filtered and deionized water to dilute the sample. 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.

4.3.4 Fill the Autosampler Vials and Load the Sample Tray

- Follow the instructions in the autosampler manual to fill the vials and load them into the sample tray or cassette. The autosampler manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).
- 2. Load the tray or cassette into the autosampler and close the autosampler door.

Tips for Filling and Loading Vials

- For an AS, fill the vials no higher than the vial shoulder. Make sure the needle vent is not immersed in liquid during sampling.
- For an AS40, fill the vials to the level marked on the vials.

- Install a cap on each vial. For an AS, make sure the septum is pushed fully into the cap and the cap is securely tightened. For an AS40, use the cap insertion tool (P/N 037987) to prevent contamination and ensure the cap is inserted to the proper depth.
- For an AS with the sample temperature option: After loading the vials, position tray covers (P/N 061010) over any empty tray rows.

DP/SP Startup

4.4 Set Up the Eluent Reservoirs

- 1. Rinse the eluent reservoirs with ASTM Type I (18 megohm-cm) filtered and deionized water.
 - NOTE Always use ASTM Type I (18 megohm-cm) filtered and deionized water to prepare eluent and regenerant or to rinse eluent reservoirs.
- 2. If necessary, prepare the eluent for the application. For instructions, refer to the manual for the column. Column manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891).
- 3. Fill the reservoirs with prepared eluent or deionized water.
- 4. Locate the end-line filters (P/N 045987) provided in the ship kit (DP Ship Kit, P/N 062463; SP Ship Kit, P/N 063342).
- 5. Before installation, thoroughly rinse the end-line filter with ASTM Type I (18 megohm-cm) filtered and deionized water to remove any loose particles.
- 6. Install an end-line filter on the end of each reservoir's eluent line.
- 7. Install the reservoir caps, making sure the end of each line extends to the bottom of the reservoir, and that each filter is submerged in liquid. This prevents air from being drawn through the eluent lines. Hand-tighten the caps.

NOTE A Dionex High Pressure Inline Filter (P/N 044105) can be used to remove particulates down to 0.45 micron from eluent. Connect the inline filter between the pump outlet and the eluent inlet port on the injection valve. For details, see the instructions provided with the inline filter.

4.5 Set Up the Piston Seal Wash System

Replumbing the Seal Wash System (DP only) (Optional)

The standard piston seal wash system is designed for use with only one of the two pumps in a DP module. When the DP is shipped from Dionex, the seal wash system is connected to pump 1 (the bottom pump). If you want to connect the seal wash system to pump 2 (the top pump), follow the instructions in this section.

NOTE Dionex offers an External Seal Wash Kit (P/N 063518) for users who need to operate a piston seal wash system for both pumps in the DP. Installation instructions are provided in the kit.

- 1. Note the tubing connected from the peristaltic pump to the seal wash tube on the secondary pump head on pump 1 (see <u>Figure 4-1</u>). Disconnect this tubing from the pump head and connect it to the seal wash tube on the secondary pump head on pump 2.
- 2. Disconnect the seal wash reservoir inlet line from the primary pump head on pump 1 (see <u>Figure 4-1</u>). Connect the inlet line to the primary pump head on pump 2.
- 3. Disconnect the short piece of tubing between the primary and secondary pump heads on pump 1 (see <u>Figure 4-1</u>). Connect this tubing to the pump heads on pump 2.
- 4. When you finish these connections, go to the next section to continue setting up the seal wash system.

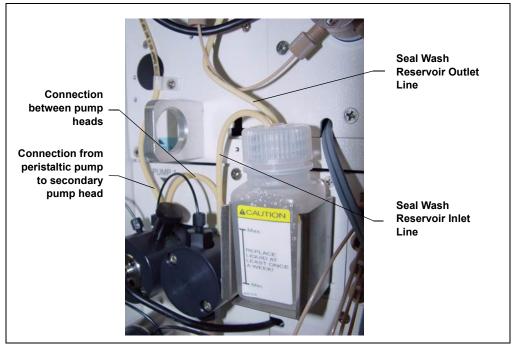


Figure 4-1. Piston Seal Wash Connections to Pump 1

Setting Up the Seal Wash System (All pumps)

- 1. Add ASTM Type I (18 megohm-cm) filtered and deionized water to the seal wash reservoir (P/N 064155).
 - The liquid level should be between the **Min.** and **Max.** markers on the reservoir label. **Do not overfill the reservoir.**
- 2. Place the cap on the reservoir and tighten fingertight.
- 3. Insert the reservoir into the holder on the component panel.
- 4. Check that the tubing is engaged in the peristaltic pump (see <u>Figure 4-2</u>). If it is not, lift the lever on the peristaltic pump up and to the right, insert the tubing neatly between the lever and the rotor, and release the lever to secure the tubing.

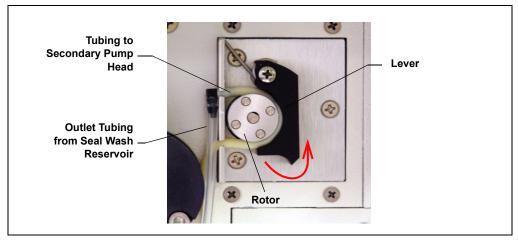


Figure 4-2. Peristaltic Pump

5. If a seal wash reservoir with a drop-sensor cap is installed, connect the outlet line from the reservoir cap to the secondary pump head (see Figure 4-3).

NOTE The drop-sensor cap is not installed on current pump models.

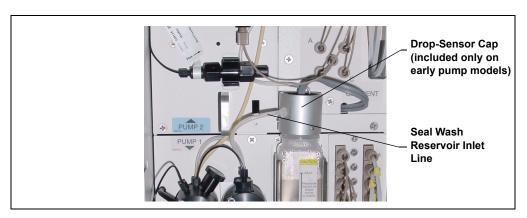


Figure 4-3. Optional Drop-Sensor Reservoir Cap Connections

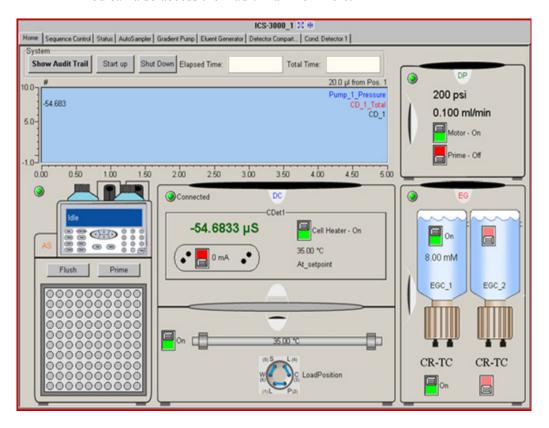
- 6. Follow these steps to activate the piston seal wash function:
 - a. In Chromeleon or Chromeleon Xpress, select **Command** on the **Control** menu or press the **F8** key.

- b. In the Commands dialog box, select the pump name.
- c. Select the **RearSealWashSystem** command and select an option:
 - Select **Internal** if the standard seal wash reservoir is installed.
 - Select **Automatic** if the drop-sensor cap reservoir is installed.
- d Click Execute

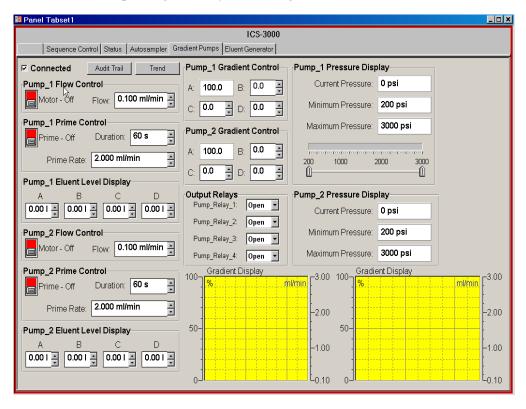
4.6 Start the Pump

- 1. Press the **POWER** button on the front of the DP/SP.
- 2. If any of the following conditions applies, prime the pump (see Section 9.3) before proceeding:
 - The eluent has been changed.
 - The eluent line is new (empty).
 - The eluent line contains air.
- 3. To display the ICS-3000 panel tabset, follow one of the steps below:
 - If Chromeleon is installed, launch the application and click the **Default**Panel Tabset toolbar button.
 - If Chromeleon Xpress is installed, launch the application; this automatically displays the ICS-3000 panel tabset.
- 4. The panel tabset opens to the **Home** panel by default. This panel displays basic status information for each instrument in the system. In addition, a

limited number of device functions can be controlled directly from this panel. You can also access the Audit Trail from here.



5. To display the pump Control panel, select the **Pump** tab. (The tab name varies, depending on the system configuration.)



- 6. Select the **Flow** rate required for your application under **Flow Control** on the Control panel.
- 7. Click the **Motor** switch to start the pump flow.

NOTE After starting the pump or changing the flow rate, wait at least 5 minutes (longer for flow rates below 1.0 mL/min) before beginning an analysis. This allows the DP/SP Moduleware to stabilize the flow rate.

8. Enter the **Minimum Pressure** and **Maximum Pressure** values under **Pressure Display** on the Control panel. Setting high and low pressure limits ensures that the DP/SP will automatically stop if a system malfunction occurs.

Notes

- When the system includes an EG, the high pressure limit for the DP/SP is 21 MPa (3000 psi) and the low pressure limit is 1.4 MPa (200 psi).
- The DP/SP is equipped with a pressure alarm limit that prevents operation above 34 MPa (5000 psi).

EG Startup

4.7 Setting the Eluent Concentration

1. In Chromeleon or Chromeleon Xpress, select the **Eluent Generator** tab on the ICS-3000 panel tabset. This displays the EG Control panel (see Figure 4-4).

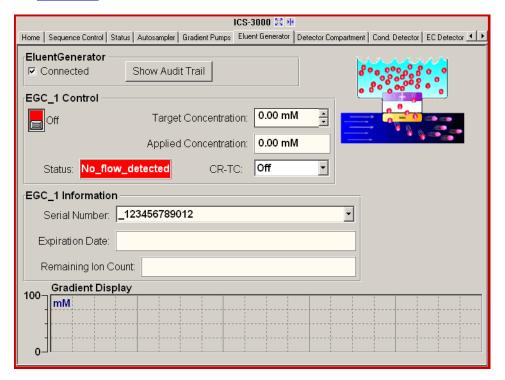


Figure 4-4. EG Control Panel

2. Under EGC Control, enter a value in the Target Concentration field.

The allowable eluent concentration depends on several factors: the flow rate, suppressor type, EluGen cartridge type, and cartridge configuration. For details, refer to Table 4-1 and Table 4-2.

Single-Cartridge or Independent Dual-Cartridge Configuration

In the single-cartridge configuration, the EG contains one EluGen cartridge. In the independent dual-cartridge configuration, the EG contains two EluGen cartridges operating independently on separate systems (each cartridge is linked to a different DP/SP). For details, refer to the EluGen cartridge manual. Cartridge manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

EluGen Cartridge	Eluent Concentration Range
K ₂ CO ₃	0.1 to 15 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 2.0 mL/min flow where X = 15/flow
КОН	0.1 to 100 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 3.0 mL/min flow where X = 100/flow
LiOH	0.1 to 80 mM at 0.1 to \leq 1.0 mL/min flow 0.1 to X mM at 1.0 to \leq 3.0 mL/min flow where X = 80/flow
MSA	0.1 to 100 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 3.0 mL/min flow where $X = 100/\text{flow}$
NaOH	0.1 to 100 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 3.0 mL/min flow where X = 100/flow

Table 4-1. Eluent Concentration Ranges for Single-Cartridge and Independent Dual-Cartridge Configurations

Linked Dual-Cartridge Configuration

In the linked dual-cartridge configuration, the EG contains two EluGen cartridges, linked to a single pump, that perform as one cartridge. Note that the allowable eluent concentration for a linked cartridge is less than when the cartridge is defined as independent. For details, refer to the EluGen cartridge manual. Cartridge manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

EluGen Cartridges	Eluent Concentration Range	Comment
K ₂ CO ₃ /EPM Electrolytic pH Modifier	0.1 to 15 mM at 0.1 to 1.0 ml/min flow 0.1 to X mM at 1.0 to 2.0 ml/min flow where X = 15/flow	The total of the eluent concentrations from both cartridges (K ₂ CO ₃ and EPM) must not exceed the specified range. The EPM concentration must not exceed 10 mM. See the notes below for additional information.
KOH/KOH KOH/MSA KOH/NaOH MSA/MSA MSA/NaOH NaOH/NaOH	0.1 to 50 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 3.0 mL/min flow where $X = 50/\text{flow}$	The eluent concentration range for each cartridge is 50% of the range for a single-cartridge or independent dual-cartridge configuration.
LiOH/LiOH	0.1 to 40 mM at 0.1 to 1.0 mL/min flow 0.1 to X mM at 1.0 to 3.0 mL/min flow where $X = 40/\text{flow}$	The eluent concentration range for each cartridge is 50% of the range for a single-cartridge or independent dual-cartridge configuration.

Table 4-2. Eluent Concentration Ranges for Linked Dual-Cartridge Configurations

Notes for Generating Carbonate/Bicarbonate Eluent

If a K_2CO_3 EluGen cartridge and an EPM Electrolytic pH Modifier are installed (EGC_1 and EGC_2, respectively), set **EGC_1 Target Concentration** to the concentration of K_2CO_3 required for your application and set **EGC_2 Target Concentration** to the concentration of KHCO₃ required. In order to achieve the

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desired K₂CO₃/KHCO₃ eluent mixture, the K₂CO₃ cartridge must generate the total of the two target concentrations.

For example, for a 3.50 mM $\rm K_2CO_3/1.00$ mM KHCO $_3$ eluent, set EGC $_1$ to 3.50 mM and EGC $_2$ to 1.00 mM. When eluent is being generated, the $\rm K_2CO_3$ cartridge generates 4.50 mM $\rm K_2CO_3$. This is indicated in the **Applied Concentration** field for the $\rm K_2CO_3$ EluGen cartridge. The EPM modifies the 4.50 mM $\rm K_2CO_3$ eluent to achieve the desired 3.50 mM $\rm K_2CO_3/1.00$ mM KHCO $_3$ mixture.

DC Startup

4.8 Start the DC

- 1. Press the **POWER** button on the front of the DC.
- 2. To display the DC Control panel, select the **Detector Compartment** tab (see Figure 4-5).

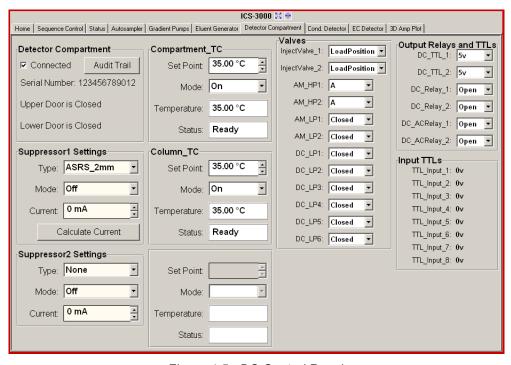


Figure 4-5. DC Control Panel

- 3. Turn on all temperature control devices installed in the DC and set the desired temperatures:
 - Compartment temperature control
 - Column temperature control
 - Reaction coil heater
 - Conductivity detector cell heater (select the **Cond. Detector** tab)

4.9 Equilibrate the System and Verify Operational Readiness

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

- 1. On the Chromeleon or Chromeleon Xpress Control panel, view the detector signal and monitor the background. Refer to the column manual for the appropriate background for your application. The column manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).
- 2. Offset the background and zero the reading by clicking the **Autozero** button.
- 3. Verify that the detector baseline is at the expected reading for your application and is stable. See <u>Section 8.11</u> for troubleshooting information if the reading is too high. See <u>Section 8.2</u> for troubleshooting information if the baseline is drifting or is excessively "noisy" (there are large fluctuations in readings).
- 4. Verify that all installed temperature control devices are at their set points and are stable.
- 5. Monitor the DP/SP pressure and make sure it is at the expected reading for the installed column and it is stable.

The system is now ready for operation.

TC Startup

4.10 Start the TC

- 1. Press the **POWER** button on the front of the TC.
- 2. To display the TC Control panel, select the TC tab (see Figure 4-6).

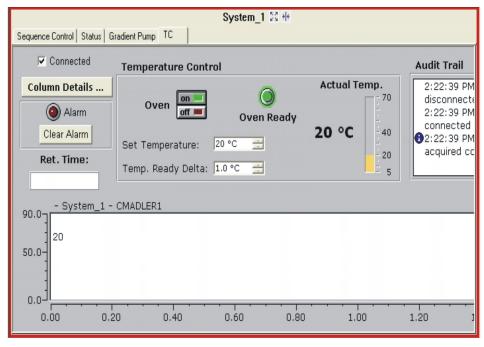


Figure 4-6. TC Control Panel

3. Click the **Oven on** button and set the temperature required for the application.

4.11 Equilibrate the System and Verify Operational Readiness

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

- 1. On the Chromeleon or Chromeleon Xpress Control panel, verify that the TC temperature is at its set point and is stable.
- 2. Monitor the pump pressure; make sure it is at the expected reading for the installed column and is stable.

The system is now ready for operation.

5.1 Overview

Samples can be run manually, one at a time, or else grouped in batches and run automatically. <u>Figure 5-1</u> compares the typical steps performed in manual and batch sample processing.

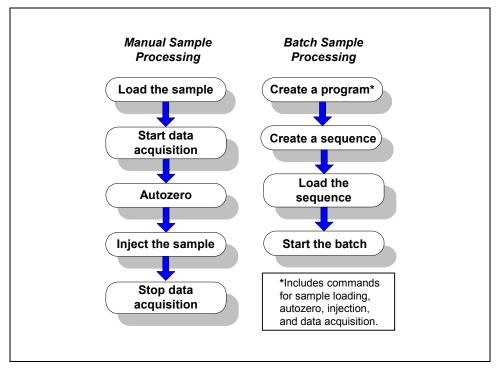


Figure 5-1. Sample Processing Overview

With either manual or batch sample processing, you can load samples into the sample loop with either an autosampler (see <u>Section 5.2</u>), or with a syringe through the sample loading ports on the DC front door (see <u>Section 5.3</u>).

5.2 Loading Samples with an Autosampler

- 1. Verify that the autosampler output line is connected to port **S** (5) on the DC (or TC) injection valve. Direct the waste line as required for this autosampler model.
- Prepare and fill the sample vials and place them in the autosampler tray or cassette. Refer to the autosampler manual for detailed instructions. Autosampler manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891)
- 3. Use one of the following methods to load sample into the injection valve sample loop:
 - Manually: Click the **Load/Inject** button on the **Autosampler** Control panel in Chromeleon or Chromeleon Xpress (see <u>Figure 5-2</u>).
 - Automatically: Include the Load command in a Chromeleon or Chromeleon Xpress program. See the examples in Section 5.4.

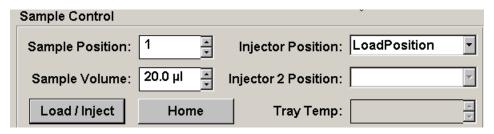


Figure 5-2. Control Panel Load/Inject Button

5.3 Loading Samples with a Syringe

This section describes two methods for using a syringe to load sample into the DC injection valve sample loop:

- Loading sample with a syringe through the sample loading port on the front of the DC (push method)
- Loading sample with a vacuum syringe through the sample loading port on the front of the DC (pull method)

NOTE For instructions on how to load and inject samples with an autosampler, see Section 5.2.

5.3.1 Loading Samples with a Syringe (Push Method)

Connecting a Sample Loading Port to the Injection Valve

- 1. For each sample loading port to be connected, locate the following items in the DC Ship Kit (P/N 062614, dual; P/N 063408, single):
 - One 1/4-28 fitting (P/N 052230) and ferrule (P/N 052231)
 - One 10-32 fitting (P/N 062980) and ferrule (P/N 062978)
 - 0.75-mm (0.030-in) ID green PEEK tubing (P/N 052304)
 - One luer adapter fitting (P/N 024305)
- 2. Press the metal release tab under the sample loading port on the front of the DC (see Figure 5-3) and pull the port out the front of the DC.

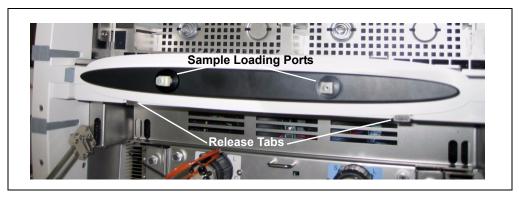


Figure 5-3. Sample Loading Ports and Release Tabs

- 3. Remove the fitting plug from the rear of the port.
- 4. Cut a length of green, 0.75-mm (0.030-in) ID PEEK tubing. The tubing will be used to connect the sample loading port and the sample port on the valve.
- 5. Attach a 1/4-28 fitting and ferrule to one end of this tubing and connect it to the rear of the sample loading port (see Figure 5-4).

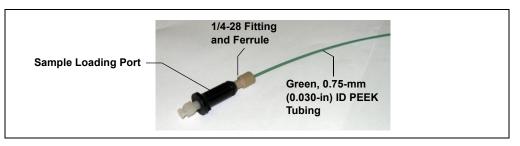


Figure 5-4. Sample Loading Port Tubing Connection

- 6. Thread the free end of the tubing through the sample loading port opening on the DC. Insert the port into the opening and turn it until it snaps into place.
- 7. Attach a 10-32 fitting and ferrule to the free end of the tubing and connect it to sample port **S** (5) on the injection valve.

Loading the Sample

- 1. Fill a syringe with a calibration standard or sample.
- 2. Insert the syringe into the sample loading port on the front of the DC.
- 3. Verify that the injection valve is in the Load position. If it is not, switch the valve by clicking the valve control on the Control panel or pressing the **VALVE** button on the front of the DC.
- 4. 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 port. This prevents the sample from exiting the loop before injection.
- 6. Switch the injection valve to the Inject position by clicking the valve control on the Control panel or pressing the **VALVE** button on the front of the DC.

5.3.2 Loading Samples with a Vacuum Syringe (Pull Method)

- Make sure the sample loading port on the DC front door is connected to sample port S (5) on the injection valve (see <u>Figure 5-5</u>). If the port is not already connected, see <u>Section 5.3.1</u> for connection instructions.
- 2. Disconnect the waste line from port **W** (6) on the injection valve (see <u>Figure 5-5</u>) and attach a shorter line: 25 to 30 cm (10 to 12 in) of PEEK or Teflon® tubing.
- 3. Place the free end of the line into the sample.
- 4. Verify that the injection valve is in the Load position. If it is not, switch the valve by clicking the valve control on the Control panel or pressing the **VALVE** button on the front of the DC.
- 5. Insert a 5 cc syringe into the sample loading port on the DC front door and pull out the plunger to draw the sample into the injection valve.
- 6. Switch the injection valve to the Inject position by clicking the valve control on the Control panel or pressing the **VALVE** button on the front of the DC.

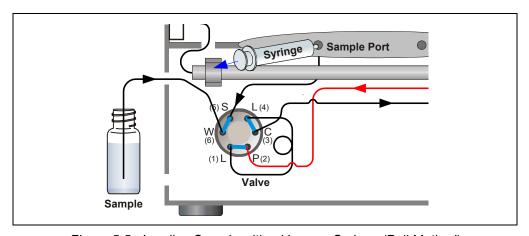


Figure 5-5. Loading Sample with a Vacuum Syringe (Pull Method)

5.4 Example Commands for Loading and Injecting Samples

The following examples show Chromeleon or Chromeleon Xpress program commands for loading and injecting samples.

5.4.1 Commands for an AS Autosampler

0.000	Load		;Switch the valve to Load.	
	Wait	CycleTimeState	;Wait for cycle time (if any).	
	Inject		;Switch the valve to Inject.	
	Wait	InjectState	;Wait for injection to finish.	
	CDet1.Autozero		;Zero the baseline.	
	CD_1.AcqOn		;Start data acquisition.	
14.000	CD_1.AcqOff		;Stop data acquisition.	

5.4.2 Commands for an AS40 Autosampler

A relay or TTL connection is required for Chromeleon or Chromeleon Xpress to control sample loading. Refer to *Installing the ICS-3000 Ion Chromatography System* (Document No. 065032) for relay connection instructions.

-2.300	Pump_Relay_1.Closed	Duration=138.00	;Note 1	
0.000	Autozero		;Note 2	
	AcqOn		;Note 3	
	InjectValve1.InjectPosition	Duration=30.00	;Note 4	
30.00	AcqOff		;Note 5	
	1. Close the RELAY OUT 1 , which is connected to the AS40 LOAD relay. This signals the AS40 to load the sample.			
	2. Zero the baseline.			
	3. Start data acquisition.			
	4. Switch the valve to Inject.			
	5. Stop data acquisition.			

5.4.3 Commands for a Remote Inject Device

If you manually load the sample using a syringe (see Section 5.3), you can configure a remote inject device in the same timebase as the DC. When Chromeleon or Chromeleon Xpress runs a program and a remote inject device is configured, it waits until an inject signal is received before starting data acquisition. To trigger the inject signal, switch the injection valve to inject by clicking the valve control on the Control panel or pressing the VALVE button on the front of the DC.

When Chromeleon or Chromeleon Xpress executes the program, it runs the commands that occur before the Inject command and then waits for the inject signal. During this waiting period, the following message is displayed in the Audit Trail: "Waiting for inject response on InjectValve." When the injection valve is switched to inject, the remote inject device signals Chromeleon or Chromeleon Xpress that injection occurred. A "Got inject response" message appears in the Audit Trail and program execution resumes.

```
:Commands before the
         Suppressor1. Type = ASRS 4mm
                                                     ;Inject command are
         CurrentSet =
                                 75 [mA]
                                                     :executed.
                                 1.000 [ml/min]
         Flow
0.000
                                                     The program pauses and
         Inject
                                                     ; waits for the inject signal.
                                                     When the inject signal is
         CDet1.Autozero
                                                     ;received, program
         CD 1.AcqOn
                                                     execution resumes.
14.000 CD 1.AcqOff
```

Notes

- When configuring the properties of the remote inject device in the Chromeleon Server Configuration program, select the name of the DC injection valve (InjectValve_Left or InjectValve_Right) in the Inject Port list on the General tab page.
- The VALVE 1 and VALVE 2 buttons on the front of the DC must be enabled in Chromeleon or Chromeleon Xpress in order to use them to manually switch the injection valves. To enable a button, open the Commands dialog box (press F8) and select the Valve1Button (or Valve2Button) command in the list of commands for the DC device.

5.5 Manual Sample Processing

To process samples manually, select operating parameters and commands from the panel tabset. Commands are executed as soon as they are entered.

- 1. Load and inject the sample, using a syringe (see Section 5.3.1), vacuum syringe (see Section 5.3.2), or autosampler (see Section 5.2).
- 2. On the **Sequence Control** panel, click **Acq On**.
- 3. On the **Detector** Control panel, click **Autozero**.
- 4. The signal plot is displayed on the **Detector** Control panel (see <u>Figure 5-6</u>). Monitor the chromatogram and when sample data has been collected, click **Acq Off**.

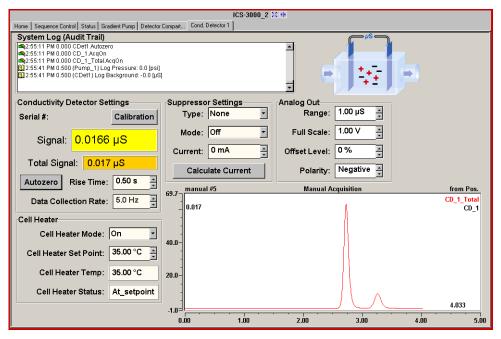


Figure 5-6. Manual Data Acquisition

5.5.1 Saving Manual Data

NOTE Chromeleon Xpress does not allow data to be saved.

If you are using Chromeleon, data from manual processing is saved in the **manual** sequence folder in the timebase folder of the local datasource. This data is overwritten each time a new manual sample is processed.

To save the data from a manual run:

- 1. Select the **manual** folder and select **Save As** from the **File** menu.
- 2. Enter a new name for the sequence and select the **Save raw data** check box.
- Click Save.

5.6 Automatic (Batch) Sample Processing

You can use Chromeleon or Chromeleon Xpress to create a list of samples (a sequence) to be processed automatically.

- For each sample, the sequence includes a program with commands and parameters for controlling ICS-3000 modules and acquiring sample data. To create a program, click **Create Program** on the **Sequence Control** panel.
- If you are using Chromeleon, the sequence also includes a quantification method for peak identification and area determination. To create a quantification method, select New on the File menu and select Method File from the list.
- You can also enter additional sample processing parameters in the sequence (for example, the sample name, sample type, injection volume, etc.).

After creating the sequence, you can start batch processing.

5.6.1 Creating a New Sequence

Two wizards are available to help you create a new sequence: Sequence Wizard and Application Wizard.

- If the program and quantification method have already been created, use the Sequence Wizard.
- If you want to create a new program and quantification method, in addition to the sequence, use the Application Wizard.

Using the Sequence Wizard

1. Click **Create Sequence** on the **Sequence Control** panel (see Figure 5-7).

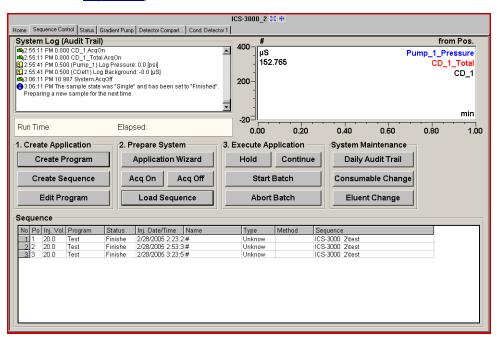


Figure 5-7. Sequence Control Panel

2. Complete the steps in the Sequence Wizard, adding the desired number of samples and standards to the list. For help at any time, click the **Help** button on the Sequence Wizard page.

Using the Application Wizard

- 1. Click **Application Wizard** on the **Sequence Control** panel.
- 2. Select a suppressor type (if used) and then select an application template from the list.

NOTE Instead of selecting an application template, you can use the Virtual Column Separation Simulator. Refer to the Chromeleon Help for details.

- 3. Click Next> and select the in a new sequence via Sequence Wizard option.
- 4. Click **Next>** to go to the Sequence Wizard.
- 5. Complete the steps in the Sequence Wizard, adding the desired number of samples and standards to the list. For help at any time, click the **Help** button on the Sequence Wizard page.

After you finish the Sequence Wizard, a sequence is created and a program appropriate for the selected application is copied to the sequence. If you are using Chromeleon, a quantification method is also copied to the sequence.

5.6.2 Starting Batch Sample Processing

- 1. Click Load Sequence on the Sequence Control panel.
 - A dialog box opens.
- 2. Select the sequence from the list and click **Open** (or **Load** if you are using Chromeleon Xpress).
- 3. Click **Start Batch** on the **Sequence Control** panel.



DP/SP Shutdown

If the ICS-3000 Dual Pump (DP) or ICS-3000 Single Pump (SP) will not be operated for a period of one week or more, follow the instructions below:

- Fill the pump with methanol (or a similar alcohol, such as 2-propanol or ethanol). If the eluents in the pump are not miscible with water, replace the eluents step-by-step.
- Fill the seal wash reservoir with wash solution (see Section 2.2.5).
- Rinse out buffers. This will reduce the time required for column equilibration when you resume operation.
- Disconnect the tubing from the peristaltic pump on the component panel: lift the lever up and to the right, remove the tubing, and release the lever (see Figure 6-1).

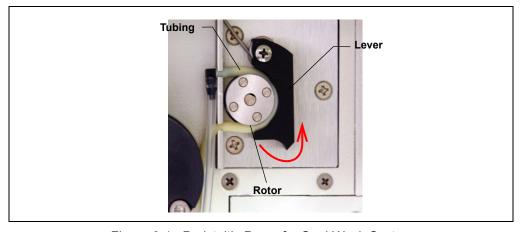


Figure 6-1. Peristaltic Pump for Seal Wash System

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Before shipping the pump:

- Empty the seal wash reservoir.
- Disconnect the tubing from the peristaltic pump on the component panel: press the lever to the right, remove the tubing, and release the lever (see Figure 6-1).

EG Shutdown

6.1 Short-term Shutdown

If the shutdown is for 3 months or less, follow the instructions below.

To prepare the EG for the shutdown:

- 1. Turn off the EG, including all consumables installed in the EG (the CR-TC, suppressor, etc.).
- 2. Verify that the current to the EluGen cartridge and the suppressor is off.



Supplying power to the EluGen cartridge when there is no eluent flow can severely damage the cartridge.

3. Store the EluGen cartridge in the EG during the shutdown.

To restart the EG:

- 1. Select the parameters for the analysis on the EG Control panel in Chromeleon or Chromeleon Xpress.
- 2. If the EG has been shut down for more than 3 to 4 days, hydrate the suppressor as instructed in the product manual. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).
- 3. Let the system equilibrate for 30 to 45 minutes before collecting data.

6.2 Long-term Shutdown

If the shutdown is for 3 months or more, follow the instructions below.

To prepare the EG for the shutdown:

- 1. Turn off the EG, including all consumables installed in the EG (the CR-TC, suppressor, etc.).
- 2. Verify that the current to the EluGen cartridge and the suppressor is off.

IMPORTANT

Supplying power to the EluGen cartridge when there is no eluent flow can severely damage the cartridge.

- 3. If you plan to store the EluGen cartridge in the EG, no further action is required.
 - If you plan to store the cartridge outside the EG (for example, in the original shipping container), follow the instructions in <u>Section 9.12</u>, <u>Step 4</u> through <u>Step 9</u>, to disconnect the EluGen cartridge cable, cap the EluGen cartridge vent opening, remove the EluGen cartridge from the holder, and disconnect the inlet and outlet tubing. Store the cartridge in an upright position (with the electrolyte reservoir at top) at 4 to 40 °C (39 to 104 °F) until its next use.
- 4. Cap both ends of the CR-TC securely, using the plugs supplied with the column

To restart the EG:

- 1. Select the parameters for the analysis on the EG Control panel in Chromeleon or Chromeleon Xpress.
- 2. Condition the EluGen cartridge (see <u>"Conditioning the New EluGen</u> Cartridge" on page 288).
- 3. Hydrate the CR-TC (see "Installing and Hydrating the New CR-TC" on page 291).
- 4. Hydrate the suppressor as instructed in the product manual. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).
- 5. Let the system equilibrate for 30 to 45 minutes before collecting data.

Before shipping the EG:

Follow the instructions in <u>Section 9.12</u>, <u>Step 4</u> through <u>Step 9</u>, to disconnect the EluGen cartridge cable, cap the EluGen cartridge vent opening, remove the EluGen cartridge from the holder, and disconnect the inlet and outlet tubing.

IMPORTANT

Make sure the EluGen cartridge packaging conforms to hazardous material shipping requirements. Refer to the Material Safety Data Sheet (MSDS) shipped with the EluGen cartridge for the chemical description.

DC Shutdown

6.3 Consumables Storage

The columns, suppressors, and other consumable items used with an ICS-3000 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 Dionex Reference Library CD-ROM (P/N 053891).

6.4 Amperometry Cell Storage

6.4.1 Short-term Storage of the Amperometry 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.

NOTE If the reference electrode remains in the cell but eluent is not being pumped through the cell, the 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.

6.4.2 Long-term Storage of the Amperometry Cell

If the cell will not be used for 2 days or more, remove the pH 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.
- 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 reference electrode from the cell.

- 4. Insert the electrode into the cap and screw on the cap (see Figure 6-2).
- 5. Make sure there is no air bubble in the cap. Add more KCl solution if needed.



Figure 6-2. Reference Electrode in Storage Cap

TC Shutdown

6.5 Consumables Storage

The columns and other consumable items used with an ICS-3000 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 Dionex Reference Library CD-ROM (P/N 053891).

6.6 Short-term Shutdown

Before a TC shutdown of less than one week, no special procedure is required.

6.7 Long-term Shutdown

Before interrupting TC operation for more than one week or before shipping the TC, rinse the fluid connections with deionized water.

This section describes routine maintenance procedures for the ICS-3000 system that users may perform. All other maintenance procedures must be performed by Dionex personnel.

For information about maintenance procedures for the EluGen cartridge, CR-TC trap column, or suppressor, refer to the appropriate product manual. These manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

7.1 **System Maintenance Checklists**

7.1.1	Daily M	Daily Maintenance			
	☐ Check for leaks.				
	☐ Wipe	e up liquid spills.			
	☐ Chec	☐ Check eluent reservoir levels and refill as needed.			
		ck eluent reservoirs for microbial growth; clean reservoirs and ace eluent as needed.			
	☐ Chec	ck waste containers and empty as needed.			
	For detai	iled daily maintenance instructions for each module, see:			
	DP/SP	page 161			
	EG	page 163			
	DC	page 165			
	TC	page 167			
7.1.2	Weekly	/ Maintenance			
	they bacte	ck end-line filters on eluent lines. When end-line filters are new are pure white. Replace filters if they become discolored, if erial buildup is evident, or if eluent flow is blocked. See ion 4.4 for instructions.			

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☐ Check for plugged or blocked tubing connections.

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For detailed	Weekly	maintenance	instructions	tor	each module, see:
i oi actanca	W CCIXI y	mamiculation	mon actions	101	cacii illoadic, sec.

DP/SP page 161
EG page 163
DC page 165
TC page 167

7.1.3 Periodic Maintenance

☐ Clean eluent reservoirs (when making fresh eluent or as needed) (see Section 9.2).

☐ (Optional) Have a Dionex Technical Support Representative perform the performance qualification (PQ) procedure for IC systems.

For detailed periodic maintenance instructions for each module, see:

DP/SP page 162
DC page 165
TC page 168

NOTE There are no periodic maintenance instructions for the EG.

7.1.4 Annual Maintenance

☐ Dionex recommends performing preventive maintenance on each module annually. Preventive Maintenance Kits contain all the required parts for these procedures.

DP Preventive Maintenance Kit (P/N 061794)

SP Preventive Maintenance Kit (P/N 061795)

DC/TC Preventive Maintenance Kit (P/N 061796)

NOTE Dionex does not offer a Preventive Maintenance Kit for the EG.



DP/SP Routine Maintenance

This section describes routine maintenance procedures for the ICS-3000 Dual Pump (DP) or ICS-3000 Single Pump (SP) that users may perform. All other maintenance procedures must be performed by Dionex personnel.

7.2 DP/SP Daily Maintenance

- Check for leaks at the following locations: the eluent proportioning valves (gradient pump only), vacuum degas chambers, and eluent reservoirs. Tighten or replace any leaking fittings.
- Wipe up liquid spills and rinse dried reagents off pump 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 Audit Trail.
- Check the liquid level in each eluent reservoir and refill as required.
- Check the liquid level in the seal wash reservoir. The liquid level should remain between the **Min.** and **Max.** markers on the reservoir label.
- Rinse each eluent reservoir thoroughly (inside and out) with ASTM filtered, Type I (18 megohm-cm) deionized water and dry with clean, particulate-free air. If a reservoir still appears dirty, or if there is a slimy film on the interior, clean the reservoir as instructed in Section 9.2.

7.3 DP/SP Weekly Maintenance

• The end-line filters (P/N 045987) on the eluent lines may need to be replaced weekly, depending on water quality and eluent conditions. For example, replace the filters weekly when using an EluGen cartridge to generate carbonate/bicarbonate eluent.

When the end-line filters are new, they are pure white. Replace the filters whenever they become discolored, if bacterial buildup is evident, or if eluent flow is blocked

NOTE It is especially important to regularly replace end-line filters when using aqueous eluents. Aqueous eluents may contaminate the filters with bacteria or algae; although the contamination may not be visible, it can cause flow restrictions to the pump.

End-line filters are provided in the ship kit (DP Ship Kit, P/N 062463; SP Ship Kit, P/N 063342). Before installation, thoroughly rinse the end-line filter with ASTM Type I (18 megohm-cm) filtered and deionized water to remove any loose particles.

- Replace the seal wash solution. ASTM Type I (18 megohm-cm) filtered and deionized water is appropriate for most applications.
- Check the tubing connected to the peristaltic pump. If the tubing is blocked or crimped, replace it (see Section 9.7).

7.4 DP/SP Periodic Maintenance

- Make fresh eluent as needed.
- Replace piston seals every 6 to 12 months (see Section 9.5).

7.5 DP/SP Annual Maintenance

• Dionex recommends performing preventive maintenance annually. The following kits are available:

DP Preventive Maintenance Kit (P/N 061794)

SP Preventive Maintenance Kit (P/N 061795)



EG Routine Maintenance

This section describes routine maintenance procedures for the ICS-3000 Eluent Generator (EG) that users may perform. All other maintenance procedures must be performed by Dionex personnel.

For information about maintenance procedures for the EluGen cartridge, CR-TC trap column, or suppressor, refer to the appropriate product manual. These manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

7.6 EG Daily Maintenance

- Locate and repair leaks inside the EG (see Section 9.11) and wipe up liquid. Rinse dried eluents off components with deionized water.
- Rinse the EG drip tray with deionized water (to prevent formation of salt
 crystals) and dry the drip tray thoroughly. Rinse and dry the leak sensor, also;
 if the sensor is not dry, it will remain activated and continue to report a leak to
 the Audit Trail.
- Check the waste container and empty as needed.

7.7 EG Weekly Maintenance

- Check all air and liquid lines for crimping. Move (or reroute) pinched lines and replace damaged lines (see <u>Section 9.10</u>). For help in isolating the cause of a tubing restriction, see <u>Section 9.11</u>.
- Monitor the system backpressure. A gradual increase in backpressure can indicate clogging of the EluGen cartridge inlet frit from particulates in the eluent water

7.8 EG Annual Maintenance

• Check the expiration date and remaining lifetime of the EluGen cartridge on the EG Control panel in Chromeleon or Chromeleon Xpress.



DC Routine Maintenance

This section describes routine maintenance procedures for the ICS-3000 Detector/Chromatography Module (DC) that users may perform. All other maintenance procedures must be performed by Dionex personnel.

7.9 DC Daily Maintenance

- Check the DC components for leaks or spills. Wipe up spills. Isolate and repair leaks (see <u>Section 8.26</u>). Rinse off any dried eluent with ASTM filtered, Type I (18 megohm-cm) deionized water.
- Check the waste container and empty as needed.

NOTE If you are using an electrochemical detector, also see the special maintenance considerations that are described in Section 4.1.2.

7.10 DC Weekly Maintenance

- Check liquid lines for crimping or discoloration. Relocate any pinched lines.
 Replace damaged lines.
- Check for excess condensation inside the DC. Condensation may build up
 when the DC is operated in a highly humid environment and the majority of
 the applications are run at cold temperatures. To remove condensation, run the
 DC at its maximum temperature for at least 48 hours for a dual-zone DC, or
 72 hours for a single-zone (shared) DC.

7.11 DC Periodic Maintenance

- Calibrate the CD cell (every 6 months).
- Inspect the door seal for signs of damage. A defective seal impairs the performance of the instrument. If the seal is defective, contact Dionex Technical Support.

7.12 DC Annual Maintenance

Dionex recommends performing preventive maintenance annually. A DC/TC Preventive Maintenance Kit (P/N 061796) is available.



TC Routine Maintenance

This section describes routine maintenance procedures for the ICS-3000 Thermal Compartment (TC) that users may perform. All other maintenance procedures must be performed by Dionex personnel.

7.13 TC Daily Maintenance

- Check the TC components for leaks or spills. Wipe up spills. Isolate and repair leaks (see <u>Section 8.31</u>). Rinse off any dried eluent with ASTM filtered, Type I (18 megohm-cm) deionized water.
- Check the liquid level in the waste container and empty as needed.



Neutralize acidic and caustic wastes before disposal. Dispose of all wastes in accordance with local regulations.



Neutralisez les déchets acides ou caustiques avant de les jeter. Jetez les déchets aux règlements locaux.



Neutralisieren Sie säurehaltige und ätzende Abfälle vor ihrer Entsorgung. Entsorgen Sie alle Abfälle entsprechend den lokalen Bestimmungen.

7.14 TC Weekly Maintenance

Check liquid lines for crimping or discoloration. Relocate any pinched lines.
 Replace damaged lines.

7.15 TC Periodic Maintenance

- Clean the interior of the TC, using a lint-free cloth. Use paper to absorb any
 liquid inside the module. The cleaner the module, the more effective the
 sensors are at detecting excessive gas or humidity.
- Check for excess condensation inside the TC. Condensation may build up
 when the TC is operated in a highly humid environment and the majority of
 applications are run at cold temperatures. To remove condensation, run the TC
 at its maximum temperature for at least 72 hours.

(IMPORTANT)

Some IC columns should not be operated at temperatures above 70 $^{\circ}$ C (158 $^{\circ}$ F). Remove all columns from the TC before beginning the maintenance procedure above.

• Inspect the door seal for signs of damage. A defective seal impairs the performance of the instrument. If the seal is defective, contact Dionex Technical Support.

7.16 TC Annual Maintenance

• Dionex recommends performing preventive maintenance annually. A DC/TC Preventive Maintenance Kit (P/N 061796) is available.

This chapter is a guide to troubleshooting minor issues that may arise during operation of the ICS-3000 system. Turn to the section of this chapter that best describes the operating problem or symptom that has been observed. Each section lists possible causes of the problem or symptom in order of probability. A systematic troubleshooting approach is the most effective way to determine the root cause.

If you are unable to resolve a problem by following the instructions here, contact Dionex Technical Support. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Dionex office. Please have this chapter at hand when talking with Technical Support personnel.

8.1 Audit Trail Error Messages

The Moduleware (the instrument control firmware installed in each ICS-3000 module) periodically checks the status of certain parameters. If a problem is detected, it is reported to Chromeleon or Chromeleon Xpress and logged in the Audit Trail. Each error message is preceded by an icon that identifies the seriousness of the underlying problem (see the table below). For most modules (except for the TC), you can change the severity level assigned to a problem whenever appropriate.

Icon	Default Severity Level	Description
!	Warning	A message is displayed in the Audit Trail, but the current run is not interrupted.
A	Error	A message is displayed in the Audit Trail, and the system attempts to correct the problem (sometimes by using an alternative parameter). An Error never interrupts the current analysis; however, if it occurs during the Ready Check, the analysis will not be started.
Stop	Abort	A message is displayed in the Audit Trail, and the running batch is aborted.

8.1.1 DP/SP Error Messages

The table below lists the most frequently observed DP/SP-related error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

DP/SP-Related Audit Trail Error Message	Default Severity Level	See
A program with this name already exists.	Abort	page 189
Abnormal drive current for x.x seconds.	Warning	page 189
Camshaft index too early.	Abort	page 189
Camshaft index too late.	Abort	page 189
Camshaft sensor always alight.	Abort	page 190
Camshaft sensor missing or dark.	Abort	page 190
Degasser malfunction.	Warning	page 190
Excessive drive current. Camshaft x.x.	Abort	page 190
Invalid flow value.	Abort	page 191
Leak detected. Flow stopped.	Abort	page 191
Left-hand pump block carryover pressure is too high.	Abort	page 192
Motor malfunction.	Abort	page 192
Motor position error. The motor is overloaded.	Abort	page 192
Pressure fallen below lower limit.	Abort	page 193
Relay 4 is configured for inject synchronization. Please change pump configuration.	Abort	page 194
Right-hand pump block carryover pressure is too high.	Abort	page 192
The maximum purge pressure was exceeded.	Abort	page 194
The pressure in the left-hand working cylinder exceeded the safety limit.	Abort	page 194
The pressure in the right-hand working cylinder exceeded the safety limit.	Abort	page 194
The rear-seal leak count is x.x (counted drops) and has exceeded the limit of y.y (leak detection threshold).	Warning	page 195
The rear-seal leak sensor is malfunctioning.	Warning	page 196
The system pressure exceeded the safety limit.	Abort	page 194

DP/SP-Related Audit Trail Error Message	Default Severity Level	See
This function cannot be adjusted by the user.	Abort	page 196
Upper pressure limit exceeded.	Abort	page 196

8.1.2 EG Error Messages

The table below lists the EG-related error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

EG-Related Audit Trail Error Message	Default Severity Level	See
CR-TC1 over current.	Abort	page 203
CR-TC2 over current.		
CR-TC1 stopped due to zero flow.	Abort	page 203
CR-TC2 stopped due to zero flow.		
EG1 cartridge disconnected.	Abort	page 204
EG2 cartridge disconnected.		
EG1 invalid concentration.	Abort	page 204
EG2 invalid concentration.		
EG1 invalid flow.	Abort	page 205
EG2 invalid flow.		
EG1 invalid flow rate-concentration.	Abort	page 205
EG2 invalid flow rate-concentration.		
EG1 over current.	Abort	page 205
EG2 over current.		
EG1 over power.	Abort	page 206
EG2 over power.		
EG1 over voltage.	Abort	page 206
EG2 over voltage.		
Leak sensor wet.	Warning	page 207

8.1.3 DC Error Messages

The table below lists the DC-related error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

DC-Related Audit Trail Error Message	Default Severity Level	See
CD cell 1 option disconnected.	Abort	page 213
CD cell 2 option disconnected.		
CD cell 1 over safe temperature.	Abort	page 214
CD cell 2 over safe temperature.		
Column open circuit.	Abort	page 214
Column over safe temperature.	Abort	page 214
Compartment open circuit.	Abort	page 215
Compartment over safe temperature.	Abort	page 215
ED cell 1 option disconnected.	Abort	page 215
ED cell 2 option disconnected.		
ED cell 1 working electrode disconnected.	Abort	page 216
ED cell 2 working electrode disconnected.		
High pressure valve 1 error.	Abort	page 217
High pressure valve 2 error.		
High pressure valve 3 error.		
High pressure valve 4 error.		
Lower door opened.	Warning	page 218
Lower leak sensor wet.	Warning	page 218
Reaction coil open circuit.	Abort	page 219
Reaction coil over safe temperature.	Abort	page 219
Suppressor 1 over-current.	Abort	page 219
Suppressor 2 over-current.		
Suppressor 1 over-power.	Abort	page 220
Suppressor 2 over-power.		
Suppressor 1 over-voltage.	Abort	page 220
Suppressor 2 over-voltage.		

DC-Related Audit Trail Error Message	Default Severity Level	See
Suppressor 1 open circuit. Suppressor 2 open circuit.	Abort	page 221
Upper door opened.	Warning	page 221

8.1.4 TC Error Messages

The table below lists the TC-related error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

TC-Related Audit Trail Error Message	Default Severity Level	See
AcqOff without previous AcqOn.	Error	page 227
Acquisition is still on at program end.	Error	page 227
All samples scheduled for run on timebases x need to specify the same value for property (nominal) Temperature.	Error	page 228
All samples scheduled for run on timebases x need to specify the same value for property TempCtrl.	Error	page 228
All samples scheduled for run on timebases x need to specify the same value for property y.	Error	page 228
Can't execute "Disconnect" command during data acquisition.	Warning	page 228
Can't execute "Standby" command during data acquisition.	Warning	page 228
Can't find download image TC3000.HEX.	Abort	page 229
Can't start acquisition at negative retention times.	Error	page 229
Can't start acquisition: The server configuration does not fulfill the license conditions.	Abort	page 229
Can't start TC firmware. A firmware download may be necessary.	Abort	page 230
Can't use device "x" as a source of the column pressure. Please check the property "SystemPressure" for a valid device name. The device must have the "Pressure" property available.	Error	page 231

TC-Related Audit Trail Error Message	Default Severity Level	See
Can't use device "x" as a source of the column pressure. Please check the property "y" for a valid device name. The device must have the "Pressure" property available.	Error	page 231
Card data checksum error.	Error	page 231
Command is not available in demo mode.	Abort	page 232
Communication error: x.	Abort	page 232
Communication time-out.	Abort	page 232
Configuration doesn't match. X not installed.	Warning	page 233
Configuration mismatch (x — CM uses y, Column Compartment uses z). Please use the Server Configuration program to check the installation.	Abort	page 233
Current flow is out of limits defined for the column.	Warning	page 233
Current pressure is out of limits defined for the column.	Warning	page 233
Current temperature is out of limits defined for the column.	Warning	page 234
Data buffer overrun.	Abort	page 234
Device control feature is not available! Please check key code and CM Features (see "About Chromeleon" in the client's Help menu).	Abort	page 234
Device is not remote.	Abort	page 235
Device not found on the USB.	Abort	page 235
Either the current date is later than the expiration date for x or the program or batch will end later than 24 hours before the expiration time for y.	Warning	page 236
Error deleting flash memory.	Abort	page 236
Error during storing of the card data to the chip card.	Error	page 236
Error finishing download.	Abort	page 230
Error programming flash memory.	Abort	page 237
Executing this command will reset associated counter values stored in the module.	Warning	page 237
Firmware download failed.	Abort	page 237
Firmware download in progress. Please wait.	Error	page 237

TC-Related Audit Trail Error Message	Default Severity Level	See
Gas leak detected.	Error	page 238
Humidity leak detected.	Error	page 238
Illegal parameter.	Abort	page 238
Invalid date/time format. Either use "DD MMM YYYY" or "current [+ [Nyear[s]] [Nmonth[s]] [Nday[s]]]." Examples: "31 Dec 1999," "current + 1year 6months."	Error	page 239
Invalid enumerated value.	Warning	page 239
Next qualification of this module is due in x day(s) (due date is [date]).	Warning	page 239
Next qualification of this module is overdue (due date was [date]).	Warning	page 239
Next qualification of this module is overdue (due date was [date]). Module may no longer be used.	Error	page 239
Next qualification of this module was due on [date]. Allowing x more grace day(s).	Warning	page 239
Next service of this module is due in x day(s) (due date is [date]).	Warning	page 240
Next service of this module is overdue (due date was [date]).	Warning	page 240
No chip card in the reader for the column x.	Error	page 241
No response from x for x seconds.	Warning	page 241
No response from x for x seconds. Device disconnected.	Abort	page 241
Operating columns above 70 °C could affect column performance. Verify recommended column operating conditions before setting temperature above 70 °C.	Warning	page 242
Parameter x value exceeded its allowed range. Set to closest valid value y.	Warning	page 242
Primary property "x" is already assigned and cannot be changed.	Error	page 243
Properties cannot be changed. A chip card has not been inserted properly. Check the CardState property for all cards and reinsert those indicating "BusBlocked."	Error	page 243
Properties cannot be changed if the compartment door is open.	Error	page 244

TC-Related Audit Trail Error Message	Default Severity Level	See
Properties cannot be changed—there is no card in the card reader.	Error	page 244
Raw data file x cannot be created.	Warning	page 244
Raw data file x cannot be created. Continuing with network failure protection.	Warning	page 244
Raw data file x cannot be written.	Warning	page 244
Raw data file x cannot be written. Continuing with network failure protection.	Warning	page 244
Starting data acquisition manually will overwrite any data which has been acquired so far.	Warning	page 245
TC—Firmware download failed.	Abort	page 230
TC is not remote.	Abort	page 235
The command can format only chip cards with an invalid data structure.	Error	page 245
The communication interface is closed. Reconnect the instrument.	Error	page 245
The compartment door is open; a batch or a program may need to wait until the door has been closed. Please close the door.	Warning	page 246
The counter [name] (value: x) has exceeded its limit (y). Module should no longer be used. Replace the [name].	Warning	page 246
The data format stored on the chip card is unsupported (format version x).	Error	page 246
The injections limit for the column has been exceeded.	Warning	page 247
The injections limit will be exceeded for the column x.	Warning	page 247
The nominal temperature must be set within the current limits.	Error	page 247
The nominal temperature value is out of range defined by the active column ID.	Error	page 247
The oven is not fully operable yet. Check the values of "Standby," "Ready," "Door," and "CardState."	Error	page 248
The primary column ID property "x" will be changed. The property can't be changed in the future.	Warning	page 248

TC-Related Audit Trail Error Message	Default Severity Level	See
The upper limit must be higher than the lower limit.	Error	page 249
The Warning threshold must be higher than the Limit threshold.	Error	page 249
The Warning threshold must be lower than the Limit threshold.	Error	page 249
There was already an AcqOff command for this channel before.	Warning	page 250
There was already an AcqOn command for this channel before.	Warning	page 250
This function cannot be adjusted by the user.	Abort	page 250
This operation may affect the oven's operation on timebase x, which is currently running a batch.	Warning	page 250
Two AcqOn commands for the same channel. The second chromatogram will overwrite the first.	Error	page 250
Unknown error code.	Warning	page 251
Unknown format of the card data.	Error	page 251
USB/LAN communication error. Please check communication cable and instrument's power.	Abort	page 235
Value for [name] parameter is not within valid range. Value passed (x) will be substituted by y.	Warning	page 242
Value x replaced by valid table entry y.	Warning	page 242
X position error.	Abort	page 251
You do not have the privilege to approve the module's qualification.	Error	page 252
You do not have the privilege to change the performance limits.	Error	page 252
You do not have the privilege to change the qualification intervals.	Error	page 252

8.2 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 and 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 in Section 9.2.
- 2. Flush the system with ASTM Type I (18 megohm-cm) filtered and deionized water.
- 3. Replace all end-line filters (P/N 045987). See Section 4.4 for instructions.
- 4. Prepare new stock solution.
- 5. 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.

DP/SP not properly primed

Prime the pump (see Section 9.3).

• Piston seal is damaged

If the piston seal is damaged, it allows liquid leaks. Leaks are usually visible, and thus easily detected. If necessary, replace the piston seal (see Section 9.5).

Inadequate system or cell backpressure

Add backpressure tubing to the cell outlet (see <u>Section 9.23.4</u>) or to the pump (if no column is installed).

• (EG Only) System backpressure is below 14 MPa (2000 psi)

When the EG is installed, the optimal system backpressure is 16 MPa (2300 psi). Low system backpressure may cause high baseline noise as the eluent concentration increases in the gradient. To correct this, install a backpressure coil in the EG after the RFIC Eluent Degasser (see Section 9.15).

• Flow system leak ahead of cell

Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace all liquid line connections (see Section 9.17).

• Rapid changes in ambient temperature

Make sure the column is installed in a thermostatically controlled compartment and the compartment door is 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 Dionex Reference Library CD-ROM (P/N 053891).

• Cell above or below temperature

Contact Dionex for assistance.

Trapped gases in cell

Release any trapped gases in the cell by loosening the lines to and from the cell and then retightening them. Also loosen and retighten the fittings to and from the suppressor eluent ports.

• Detector electronics not functioning correctly

Run the detector dummy cell diagnostics from the Wellness panel (see Section 9.23).

Inappropriate suppressor operating conditions

Refer to the suppressor manual for the correct operating conditions. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

Temperature compensation setting not optimized

Optimize the selected setting (see <u>Section 2.15</u>).

• Inadequate system backpressure

Add backpressure tubing to the cell outlet (see Section 9.23.4).

Noisy Baseline: ED Only

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

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. Repeat 2 or 3 times. If the baseline does not improve, check the other causes of baseline instability described in this section.



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 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 ED cell is 690 kPa (100 psi). Do not exceed this limit.
- (DC Amperometry and Integrated Amperometry modes) Frequent, random spikes in the baseline

The reference electrode diaphragm is plugged. First, try regenerating the 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

Reconnect the short length of titanium tubing to the cell inlet (see Figure 2-22).

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

Polish the working electrode (see Section 9.24.3). 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. While wearing gloves and eye protection, generate a slight temporary backpressure by putting your finger over the end of the cell outlet tubing for 2 to 3 seconds. Repeat 2 or 3 times. 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 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 ED cell is 690 kPa (100 psi). Do not exceed this limit.

3. The water used to prepare the eluent contains trace contaminants. Remake the eluent, using ASTM Type I (18 megohm-cm) filtered and deionized water containing no contaminants.

8.3 Poor Retention Time Reproducibility

Liquid leaks

- 1. Check for leaks from the piston seals. Replace the piston seal on any head with a leak (see Section 9.5).
- 2. Check for leaks throughout the remainder of the system, including the check valves, injection valve, and columns. Tighten or replace fittings as needed

DP/SP not primed

Prime the pump (see Section 9.3).

Liquid lines incompletely flushed after an eluent change

Attach a 10 cc syringe (P/N 054578) to the waste port on the priming valve. Open the priming valve (turn the knob one-half turn counterclockwise). Draw at least 20 mL of the new eluent through the liquid lines before operation.

• Inoperative eluent proportioning valve (gradient pump only)

The proportioning valve assembly should be replaced. Contact Dionex for assistance.

• Inoperative check valves

Replace the check valve cartridges (see Section 9.4).

• Insufficient mixing (gradient pump only)

The static mixer may be dirty or contaminated. Flush with IPA (isopropyl alcohol). If this does not eliminate the problem, install a new mixer (GM-3, P/N 042126; GM-4, P/N 049135).

Eluent is contaminated

- 1. Clean all eluent reservoirs thoroughly (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water and 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 in Section 9.2.
- 2. Flush the system with ASTM Type I (18 megohm-cm) filtered and deionized water.
- 3. Replace all end-line filters (P/N 045987). See Section 4.4 for instructions.
- 4. Prepare new stock solution.
- 5. Prepare fresh eluent. To ensure eluent purity, prepare all eluents with electrochemical-grade chemicals, and ASTM Type I (18 megohm-cm) filtered and deionized water.

• Problem unrelated to the pump

- 1. The eluent concentration may be wrong, or the eluent may have been prepared with impure chemicals/eluents or water. To ensure eluent purity, prepare all eluents with spectro-grade eluents, reagent-grade chemicals, and ASTM Type I (or better) filtered and deionized water.
- 2. The column may be the source of the problem. Refer to the column manual for troubleshooting assistance. Column manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891).
- 3. The injection valve may be the source of the problem. Rebuild the injection valve (see Section 9.19).

8.4 Peak Retention Times Are Too Early

Eluent concentration setting is too high

The correct eluent concentration setting depends on several factors (the flow rate, EluGen cartridge type, etc.). Refer to Section 4.7 for a list of valid concentration ranges for the cartridges, and then edit the program as required.

• DP/SP flow rate is too low

Increase the DP/SP flow rate.

• Inoperative eluent proportioning valve (gradient pump only)

The proportioning valve assembly should be replaced. Contact Dionex for assistance.

8.5 Peak Retention Times Are Too Late

• Eluent concentration setting is too low

The correct eluent concentration setting depends on several factors (the flow rate, EluGen cartridge type, etc.). Refer to Section 4.7 for a list of valid concentration ranges for the cartridges, and then edit the program as required.

• DP/SP flow rate is too high

Decrease the DP/SP flow rate.

• Inoperative eluent proportioning valve (gradient pump only)

The proportioning valve assembly should be replaced. Contact Dionex for assistance.

8.6 No Peaks

EG power is not turned on

- 1. Check that the **POWER** button on the front of the EG is turned on.
- 2. Check that the EG main power switch (on the rear panel) is turned on.
- 3. Check that the main power cord is plugged into both the EG rear panel connector and the power source. Check that the wall outlet has power.

Injection valve is not actuating or the sample loop is plugged

- 1. Test the valve by manually switching the position from the **Detector/Chromatography t**ab on the panel tabset.
- 2. Check the sample loop for blockage. Clean or replace the loop if needed.

• Detector not properly installed

CD: An electronics connector on the back of the detector plugs into a receptacle on the detector compartment. Push on the detector to make certain the connector is securely connected (see Figure 8-1).

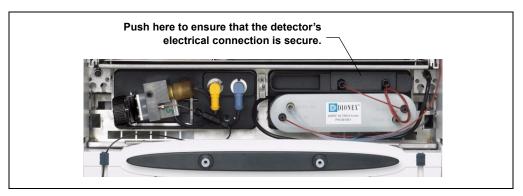


Figure 8-1. Conductivity Detector Connection

ED: Verify that the signal cables from the cell are connected to the detector block (see <u>Figure 8-2</u>). In addition, an electronics connector on the back of the detector plugs into a receptacle on the detector compartment. Push on the detector to make certain the connector is securely connected.

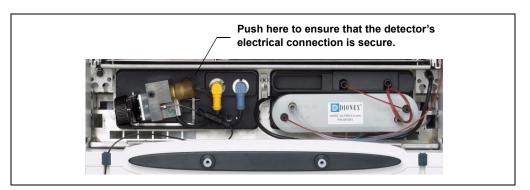


Figure 8-2. Electrochemical Detector Connection

ED Only

Cell is off

Turn on the cell from the Control panel in Chromeleon or Chromeleon Xpress.

8.7 Tailing Peaks

• Excess tubing void volumes

Check tubing connections for void volumes

• (CD) Long tubing lengths connecting

Minimize all tubing lengths between the injection valve and the detector.

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

Clean the working electrode with water and dry with pressurized air or nitrogen.

Polish the working electrode (see Section 9.24.3). If you are using a disposable electrode, replace it.

8.8 Low System Backpressure

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.

• RFIC Eluent Degasser tubing is ruptured

If flow from the EG waste line is normal but there is no flow through the columns, the tubing assembly inside the RFIC Eluent Degasser has ruptured. Replace the degasser (see Section 9.14).

• Internal EluGen cartridge leak (from membrane barrier)

Leakage from the membrane barrier may trip the DP/SP low pressure limit and shut down the pump. If all other causes of the low system pressure have been eliminated, replace the EluGen cartridge (see Section 9.12).

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

8.9 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. 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. If the EluGen cartridge is the source of the high backpressure, replace the outlet frit as instructed in the EluGen cartridge manual. The manual is on the Dionex Reference Library CD-ROM (P/N 053891).
- 3. 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.

• TC temperature stabilizer is blocked

Rinse the temperature stabilizer and the temperature stabilizer inlet line, using an appropriate solvent. If necessary, replace the temperature stabilizer (standard bore, P/N 064548; microbore, P/N 064650).

8.10 Low Detector Output

• Insufficient sample injected

Increase the injection size or concentration.

ED Only

Working electrode fouled

- 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. Dry

- with pressurized air or nitrogen. If the electrode is pitted, polish the electrode (see Section 9.24.3).
- 3. Check the value of the reference potential shift. See "Using a Digital Voltmeter to Determine Reference Potential Shift" on page 226. If the value fluctuates by more than 30 mV, electrode passivation may occur because potentials that are too high are being applied. Recalibrate the reference electrode (see Section 9.24.5). If the problem persists, replace the reference electrode (see Section 9.24.4).

Analog Output Option

Analog output range set too high

Select a more sensitive analog output range.

8.11 High Background

CR-TC is contaminated

Clean the trap column as instructed in the CR-TC manual. The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).

• Wrong eluent

Check that you are using the correct eluent for your application. For ED detectors, verify that the pH readout is correct for your eluent.

• Background signal not offset from detector signal

Before injecting sample, allow the background signal to equilibrate, and then press **Autozero** on the Control panel in Chromeleon or Chromeleon Xpress.

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 Dionex Reference Library CD-ROM (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 Dionex Reference Library CD-ROM (P/N 053891).

• (DC Amperometry and Integrated Amperometry modes) Amperometry 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 Section 9.24.2).

Turn the PEEK yoke knob (see <u>Figure 9-33</u>) a full 360 degrees to completely engage the knob.

DP/SP Troubleshooting

8.12 Troubleshooting DP/SP Error Messages

If any of the following alarm conditions occurs, a message is displayed in the Chromeleon or Chromeleon Xpress Audit Trail.

A program with this name already exists.

This error occurs if you attempt to save a modified Chromeleon or Chromeleon Xpress program under the name of an existing program.

To troubleshoot:

Enter a new name for the modified program *or* select **Save** to save your changes under the existing program name.

Abnormal drive current for x.x seconds.

If this error occurs, the tubing between the pump heads may be blocked or the fitting may be overtightened.

To troubleshoot:

Inspect the tubing for blockage or crimping and replace if needed. Be careful to not overtighten the fitting. If the message appears again, contact Dionex for assistance.

Camshaft index too early.

-or-

Camshaft index too late.

This error is caused by an internal error in the pump drive mechanism.

To troubleshoot:

Turn off the DP/SP power for 30 seconds and then turn it on again. If the error message appears again, contact Dionex for assistance.



-or-



This error is caused by an internal error in the pump drive mechanism.

To troubleshoot:

Turn off the DP/SP power for 30 seconds and then turn it on again. If the error message appears again, contact Dionex for assistance.

! Degasser malfunction.

The vacuum degassing module monitors the vacuum system continuously. If a a problem is detected, this error occurs.

To troubleshoot:

- 1. Check all tubing connections to the vacuum degassing module for leakage; tighten loose fitting connections.
- 2. Turn off the DP/SP power for 30 seconds and then restart the pump.
- 3. If the message appears again, the vacuum degassing module should be replaced. Contact Dionex for assistance.

Excessive drive current. Camshaft x.x.

This error occurs if the drive current is above the allowed value. When this message appears, the DP/SP stops running.

To troubleshoot:

One or more tubing connections may be plugged, blocked, or crimped. Check all tubing connections and replace as needed (see Section 9.1). Be careful not to overtighten fittings.

Stop

Invalid flow value.

This error occurs if you enter an invalid value for the DP/SP flow rate in Chromeleon or Chromeleon Xpress.

To troubleshoot:

Select a flow rate within the DP/SP flow rate range (0.001 to 10.0 mL/min).

Stop

Leak detected.

This error occurs if the DP/SP leak sensor detects a liquid leak inside the enclosure. When this error occurs, the pump stops running.

To troubleshoot:

To find and eliminate the source of the leak, see <u>Section 8.15</u>.

Left-hand pump block carryover pressure is too high.

-or-

Right-hand pump block carryover pressure is too high.

This error occurs if the pressure in the primary pump head exceeds the maximum allowed. ("Right-hand" refers to the bottom pump; "left-hand" refers to the top pump in a dual-pump module.)

To troubleshoot:

- 1. Make sure that the tubing between the pump heads and the tubing leading to the priming/outlet block is not plugged or blocked. Replace tubing connections as needed.
- 2. Inspect the outlet check valve for blockage. Replace the check valve cartridge if needed (see Section 9.4).

Motor malfunction.

This error occurs if an internal error in the pump drive mechanism occurs.

To troubleshoot:

Turn off the DP/SP power for 30 seconds, and then turn on the power again. If the error message appears again, contact Dionex for assistance.

Motor position error. The motor is overloaded.

This error occurs if the pump motor is overloaded.

To troubleshoot:

- 1. One or more tubing connections may be plugged or blocked. Check all tubing connections and replace as needed (see Section 9.1).
- 2. If the "Upper pressure limit exceeded message" is also displayed, refer to page 196 for additional troubleshooting steps.



Pressure fallen below lower limit.

This error occurs if the DP/SP pressure falls below the low pressure limit specified in Chromeleon or Chromeleon Xpress.

To troubleshoot:

- 1. The eluent supply may be depleted. Refill any empty eluent reservoirs.
- 2. Check all eluent lines for air bubbles. If air is trapped in a line, replace the end-line filter (P/N 045987). See <u>Section 4.4</u> for instructions. Verify that the end of each filter extends to the bottom of the reservoir and is submerged in eluent. Prime the pump (see <u>Section 9.3</u>).
- 3. Eluents may be insufficiently degassed. Check the vacuum degassing module.
- 4. Check all tubing connections for leaks; tighten loose fitting connections.
- 5. A check valve may be defective. Replace the check valve cartridges (see Section 9.4) and then prime the pump (see Section 9.3).

Rear-seal wash system has run out of wash solution.

This error occurs only when the cap on the seal wash reservoir is equipped with a drop sensor and the sensor does not detect any wash solution when piston seal washing is activated.

To troubleshoot:

- 1. Refill the reservoir with wash solution, if necessary. ASTM Type I (18 megohm-cm) filtered and deionized water is usually appropriate.
- 2. Verify that the tubing is engaged in the peristaltic pump (see <u>Section 4.5</u>). The tubing should not be plugged or crimped. Replace the tubing as needed (see Section 9.1).
- 3. Clean the sensor on the reservoir with water or solvent.
- Check for leaks at connections throughout the pump module; tighten or replace any leaking fittings. Replace any tubing that is plugged or damaged.

Relay 4 is configured for inject synchronization. Please change pump configuration.

This error occurs if you attempt to use relay 4 as a standard relay in a Chromeleon or Chromeleon Xpress program, although the relay has been set to synchronize the gradient with the autosampler sample injection.

To troubleshoot:

Define the relay as a standard relay *or* specify a different relay for use in the program.

The maximum purge pressure was exceeded.

If the DP/SP pressure exceeds 5 MPa (725 psi) during priming, this error occurs and the priming process is aborted.

To troubleshoot:

- 1. Verify that the priming valve is open and then reprime the pump. (To open the valve, turn the knob one-half turn counterclockwise.)
- 2. Check fittings for overtightening. Check tubing for crimping and replace it as necessary. Be careful not to overtighten fittings.
- The pressure in the left-hand working cylinder exceeded the safety limit.

-or-

The pressure in the right-hand working cylinder exceeded the safety limit.

-or-

The system pressure exceeded the safety limit.

This error may be caused by a blockage in the system or by a problem with the column. ("Right-hand" refers to the bottom pump; "left-hand" refers to the top pump in a dual-pump module.)

To troubleshoot:

- 1. Check all tubing connections for signs of blockage and replace it as needed. Be careful not to overtighten fittings.
- 2. 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 Dionex Reference Library CD-ROM (P/N 053891) for instructions.) If this does not eliminate the problem, replace the column.
- 3. 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 Dionex for assistance.

! The rear-seal leak count is x.x (counted drops) and has exceeded the limit of y.y (leak detection threshold).

This error occurs when the cap on the seal wash reservoir is equipped with a drop sensor and the number of drops of wash solution detected by the sensor while the peristaltic pump is idle (55 minutes per hour) exceeds the leak detection threshold.

To troubleshoot:

- 1. Inspect the piston seals for leakage. Replace the piston seals and O-rings as needed (see Section 9.5).
- 2. Increase the value for the rear-seal leak threshold:
 - a. In Chromeleon or Chromeleon Xpress, select **Command** on the **Control** menu or press the **F8** key.
 - b. In the Commands dialog box, select the pump name.
 - c. Select the **RearSealLeakCounter** command.
 - d. Enter a new value and click **Execute**.

! The rear-seal leak sensor is malfunctioning.

This error occurs only when the cap on the seal wash reservoir is equipped with a drop sensor and the sensor is very dirty.

To troubleshoot:

- 1. Clean the sensor electrodes with water or solvent.
- 2. If the message appears again, the leak sensor may be defective. Contact Dionex for assistance.

This function cannot be adjusted by the user.

This error occurs if you attempt to change a parameter that users are not allowed to adjust.

To troubleshoot:

Only Dionex Service Representatives can change this parameter.

Upper pressure limit exceeded.

This error occurs if the upper pressure limit specified in the program is exceeded. The running batch is aborted (default) and this message appears.

To troubleshoot:

- 1. Check all tubing connections for signs of blockage; replace as needed (see Section 9.1).
- 2. 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 Dionex Reference Library CD-ROM (P/N 053891) for instructions.) If this does not eliminate the problem, replace the column.
- 3. 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 Dionex for assistance.

8.13 DP/SP Does Not Start

Power is off

Check that the main power switch on the DP/SP rear panel is turned on.

Press the **POWER** button on the front of the DP/SP to turn on the pump.

Flow rate is set to zero

Select a **Flow** rate on the Chromeleon or Chromeleon Xpress Control panel.

While being primed, pump starts briefly and an alarm sounds

If the high pressure limit was tripped, check that the priming valve on the secondary pump head is opened (see <u>Figure 2-3</u>). To open the valve, turn the knob one-half turn counterclockwise.

If the low pressure limit was tripped:

- Verify that the Minimum Pressure setting is several hundred psi below the Current Pressure reading on the Chromeleon or Chromeleon Xpress Control panel.
- 2. Make sure there are no liquid leaks in the flow system.
- 3. Make sure the priming valve is closed (see <u>Figure 2-3</u>). To close the valve, turn the knob clockwise until closed. 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 down.

8.14 DP/SP Stops

Program (or other remote input) instructed the pump to stop

If no error message is displayed in the Audit Trail, the DP/SP was probably instructed to stop by the Chromeleon or Chromeleon Xpress program, the computer, or other remote signal source. If you do not want the pump to stop, take the appropriate preventive action (edit the program, etc.).

Low pressure limit was tripped

- 1. Verify that the **Minimum Pressure** setting is several hundred psi below the **Current Pressure** reading on the Chromeleon or Chromeleon Xpress Control panel.
- 2. Make sure there are no liquid leaks in the flow system.
- 3. Check the **Eluent Level Display** on the Chromeleon or Chromeleon Xpress Control panel to verify that eluent is present in the selected channel. If the eluent reservoir is empty, refill it (or select a channel that does have eluent). Prime the pump before resuming operation (see Section 9.3).
- 4. Make sure the priming valve is closed (see <u>Figure 2-3</u>). To close the valve, turn the knob clockwise until closed. 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 down.

High pressure limit was tripped

- 1. Make sure that no tubing is plugged or overtightened. 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. Press the **POWER** button on the front of the DP/SP to turn on the pump.
 - c. Set the **Flow** rate to 1.0 mL/min on the Chromeleon or Chromeleon Xpress Control panel and record the backpressure.
 - 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.

- 2. 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 Dionex Reference Library CD-ROM (P/N 053891) for instructions.) If this does not eliminate the problem, replace the column.
- 3. Observe a run to see whether the high pressure limit is triggered when sample is injected; if it is, the injection valve may be causing a blockage (i.e., the rotor seal or stator may need to be replaced). Contact Dionex for assistance.

Electrical connections incorrectly installed

The electrical cables may not be properly installed. Contact Dionex for assistance

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

8.15 DP/SP 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.

Defective piston seal

Check the pump heads for leaks. If there is any leakage, tighten the fitting connections just enough to stop the leak. Dry the components. If the pump head continues to leak, replace the piston seal (see Section 9.5).

• Leaking check valve

If the leaking check valve is securely tightened but allows leaks despite this, the valve is defective. Replace both check valve cartridges (see Section 9.4).

If the leaking check valve is loose, follow these steps to tighten it:

- 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 valve cartridges (see Section 9.4).

Eluent proportioning valve leaks (gradient pump only)

Tighten loose fittings. If there are no loose fittings, the proportioning valve assembly should be replaced. Contact Dionex for assistance.

• Priming valve knob leaks

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 in the priming valve knob (see Section 9.8).

If leaks occurs when the priming valve knob is closed, either the pump head or the knob is damaged and should be replaced. Contact Dionex for assistance.

• Excessive system backpressure

If system backpressure is substantially higher than the normal operating backpressure for the currently configured system (including the column), tubing may be plugged or overtightened. Follow the procedure below to isolate segments of the flow path and determine the sources of the high backpressure.

- 1. Remove the pump outlet tubing at the injection valve.
- 2. Press the **POWER** button on the front of the DP/SP to turn on the pump.
- 3. Set the **Flow** rate to 1.0 mL/min on the Chromeleon or Chromeleon Xpress Control panel and record the backpressure.
- 4. One at a time, reconnect each component (tubing, fittings, etc.) 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.

8.16 Vacuum Degassing Module Low Vacuum

• Leak in the vacuum degassing module

Check all tubing connections for leakage; tighten loose fitting connections.

8.17 Vacuum Degassing Module Does Not Run

Electrical connections incorrectly installed

There may be a problem with the connections from the vacuum degassing module to the CPU board. Contact Dionex for assistance.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

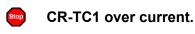
8.18 DP/SP Digital I/O Port Inoperative

- TTL input-related error
 - 1. The TTL input mode must match the signal type output by the device connected to the pump. Check the user's manual for the device to verify that the correct signal type is selected.
 - 2. A programming error occurred in the device sending the signal. Refer to the user's manual for the device for troubleshooting assistance.
- TTL/Relay output-related error
 - 1. A programming error occurred in the device sending the signal. Refer to the user's manual for the device for troubleshooting assistance.
 - 2. The device being triggered may require a TTL input, not a relay. Connect the device to a pump TTL output.

EG Troubleshooting

8.19 Troubleshooting EG Error Messages

If any of the following alarm conditions occurs, a message is displayed in the Chromeleon or Chromeleon Xpress Audit Trail.



-or-

CR-TC2 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. Check the CR-TC cable connection to the electrical bulkhead (see Figure 2-8).
- 2. Refer to Section 8.23 to determine why liquid flow stopped.
- 3. If the error message appears again, contact Dionex for assistance. The CR-TC control electronics may have malfunctioned.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

CR-TC1 stopped due to zero flow.

-or-

CR-TC2 stopped due to zero flow.

This message appears if you turn off the pump flow while the EluGen cartridge 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 Section 8.23.

EG1 cartridge disconnected.

-or-

EG2 cartridge disconnected.

This error occurs if Chromeleon or Chromeleon Xpress sends a command to set an EG parameter when the EluGen cartridge is disconnected.

To troubleshoot:

- 1. Connect the EluGen cartridge cable connection to the electrical bulkhead (see Figure 2-8).
- 2. If the error message appears again, contact Dionex for assistance. The EluGen cartridge control electronics may have malfunctioned.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

EG1 invalid concentration.

-or-

EG2 invalid concentration.

This error occurs if the eluent concentration is outside 0.10 to 100 mM, the range allowed by the EG. This may indicate corrupted memory or a problem in the EG Moduleware (the instrument control firmware installed in the EG).

To troubleshoot:

Contact Dionex for assistance.

NOTE The ICS-3000 Ion Chromatography System electronics components and Moduleware cannot be serviced by the user.

EG1 invalid flow.

-or-

EG2 invalid flow.

This error occurs if the flow rate is set to a value the EG does not support. The DP/SP flow rate range is 0.001 to 10.0 mL/min; however, when an EG is installed, the allowed range is 0.01 to 3.00 mL/min. The recommended operating range is 0.4 to 2.00 mL/min.

To troubleshoot:

Set the flow rate to a value within the allowed range.

EG1 invalid flow rate-concentration.

-or-

EG2 invalid flow rate-concentration.

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

To troubleshoot:

Set the flow rate to a value within the allowed range. The allowable eluent concentration for a particular application depends on several factors: the flow rate, suppressor type, EluGen cartridge type, and cartridge configuration. For details, refer to Section 4.7.

EG1 over current.

-or-

EG2 over current.

This error occurs when the current applied to the EluGen cartridge exceeds the maximum current allowed. (The EluGen 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. Connect the EluGen cartridge cable connection to the electrical bulkhead (see Figure 2-8).
- 2. Refer to Section 8.23 to determine why there is no flow.
- 3. If the error message appears again, contact Dionex for assistance. The cartridge control electronics may have malfunctioned.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

EG1 over power.

-or-

EG2 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 EluGen cartridge can support.

To troubleshoot:

Replace the EluGen cartridge (see Section 9.12).

EG1 over voltage.

-or-

EG2 over voltage.

This error occurs when the current applied to the EluGen cartridge exceeds the maximum current allowed. (The EluGen 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. Connect the EluGen cartridge cable connection to the electrical bulkhead (see Figure 2-8).
- 2. Refer to Section 8.23 to determine why there is no flow.

3. If the error message appears again, contact Dionex for assistance. The EluGen cartridge control electronics may have malfunctioned.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

! Leak sensor wet.

This error occurs when liquid accumulates in the drip tray in the bottom of the EG.

To troubleshoot:

- 1. Locate the source of the leak by visually inspecting the tubing, fittings, and components in the EG.
- Tighten fittings (or replace tubing and fittings) as required (see <u>Section 9.10</u>). Refer to <u>Section 8.22</u> for detailed troubleshooting of various types of leaks.
- 3. After fixing the leak, dry the drip tray and leak sensor thoroughly to prevent the leak sensor from triggering additional error messages.

8.20 EG ALARM LED Is Lighted

Leaking fitting

Locate the source of the leak. Tighten or replace liquid line connections as needed (see Section 9.10).

Blocked or improperly installed waste line

Check the EG waste lines to be sure they are not crimped or otherwise blocked. Make sure the lines are not elevated at any point after they exit the EG.

EluGen cartridge leaks

Replace the EluGen cartridge (see Section 9.12).

• RFIC Eluent Degasser leaks

Replace the RFIC Eluent Degasser (see Section 9.14).

• EluGen cartridge electrical connection is open

- 1. Tug gently on the EluGen cartridge electrical cable; the locking connector should hold the cable in place (see Figure 2-8).
- 2. If the electrical cable is fully seated but the problem persists, the cartridge is defective and must be replaced (see Section 9.12).

• EluGen cartridge input electrical connection has shorted out

Replace the EluGen cartridge (see Section 9.12).

• Electrical error

The EG current and/or voltage may have become unstable. Contact Dionex for assistance

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

8.21 EG POWER LED Fails to Light

No power

Check that the **POWER** button on the front of the EG is turned on.

Check that the EG main power switch (on the rear panel) is turned on.

Check that the main power cord is plugged into both the EG rear panel connector and the power source. Check that the wall outlet has power.

If the **POWER** LED still fails to light, contact Dionex for assistance.

8.22 Liquid Leaks in the EG

• Leaking fitting

Locate the source of the leak. Tighten or replace liquid line connections as needed (see Section 9.10).

• Blocked or improperly installed waste line

Check the EG waste lines to be sure they are not crimped or otherwise blocked. Make sure the lines are not elevated at any point after they exit the EG.

EluGen cartridge leaks

Replace the EluGen cartridge (see Section 9.12).

• RFIC Eluent Degasser leaks

Replace the RFIC Eluent Degasser (see Section 9.14).

8.23 No Flow

• DP/SP power is off

Turning off the DP/SP 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.

Check that the power to the DP/SP is turned on. Prime the pump (see Section 9.3) and resume operation.

DP/SP pressure limit tripped

When a system includes an EG, the high pressure limit for the DP/SP is 21 MPa (3000 psi) and the low pressure limit is 1.4 MPa (200 psi). Verify that the **Current Pressure** reading on the Control panel is within this range.

NOTE The EluGen cartridge requires at least 14 MPa (2000 psi) of backpressure for optimal removal of electrolysis gas from the eluent produced by the cartridge. A system backpressure of 16 MPa (2300 psi) is ideal.

RFIC Eluent Degasser tubing is ruptured

If flow from the EG waste line is normal but there is no flow through the columns, the tubing assembly inside the RFIC Eluent Degasser has ruptured. Replace the degasser (see Section 9.14).

8.24 EG Stops Operation

• DP/SP power is off

Turning off the DP/SP 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

Check that the power to the DP/SP is turned on. Prime the pump (see Section 9.3) and resume operation.

• DP/SP pressure limit tripped

When a system includes an EG, the high pressure limit for the DP/SP is 21 MPa (3000 psi) and the low pressure limit is 1.4 MPa (200 psi). Check that the system pressure is within this range.

NOTE The EluGen cartridge requires at least 14 MPa (2000 psi) of backpressure for optimal removal of electrolysis gas from the eluent produced by the cartridge. A system backpressure of 16 MPa (2300 psi) is ideal.

• DP/SP flow rate is too low or too high

Select a flow rate between 0.1 and 3.0 mL/min.

• Electrical error detected (ALARM LED is lighted)

To prevent damage to the EluGen cartridges, the DP/SP automatically turns off electrical power to the cartridge when excessive current or voltage is detected. Contact Dionex for assistance.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

• EluGen cartridge is expended

Replace the cartridge (see Section 9.12).

• No communication with Chromeleon or Chromeleon Xpress

- 1. Check that the **POWER** button on the front of the EG is turned on.
- 2. Check that the EG main power switch (on the rear panel) is turned on.

- 3. Check that the main power cord is plugged into both the EG rear panel connector and the power source. Check that the wall outlet has power.
- 4. Check the USB connections. The EG should be connected to the DP/SP (or other ICS-3000 module) via a USB cable (P/N 960777). In addition, one module in the system must be connected to the PC on which Chromeleon or Chromeleon Xpress is installed.
- 5. Check that the EG is configured in Chromeleon or Chromeleon Xpress and is assigned to a timebase.

DIONEX

DC Troubleshooting

8.25 Troubleshooting DC Error Messages

If any of the following alarm conditions occurs, a message is displayed in the Chromeleon or Chromeleon Xpress Audit Trail.

CD cell 1 option disconnected.

© CD cell 1 option disconnected.

This error occurs when the conductivity cell is disconnected.

To troubleshoot:

- 1. Check the detector connection: An electronics connector on the back of the detector plugs into a receptacle on the detector compartment. Push on the upper part of the detector (see <u>Figure 8-3</u>) to make certain the connector is securely connected.
- 2. If the error persists, there may be a problem in the detector electronics. Contact Dionex for assistance.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

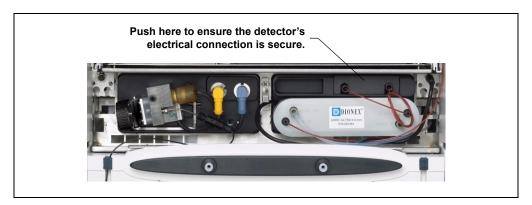


Figure 8-3. Conductivity Detector Connection

Stop

CD cell 1 over safe temperature.

-or-

Stop

CD cell 2 over safe temperature.

This error occurs when the temperature of the specified conductivity detector is higher than the maximum allowed. This error may occur if the ICS-3000 Ion Chromatography System is operating in an environment in which the temperature is greater than $40 \, ^{\circ}\text{C} (104 \, ^{\circ}\text{F})$.

To troubleshoot:

- 1. Refer to Section A.12 for environmental specifications.
- 2. Verify that the compartment setpoint is at least 5 °C less than the CD cell setpoint.
- 3. Check the suppressor current setting. Running the suppressor at a higher current than is recommended for the application can cause heat up of the CD cell

Column open circuit.

This error may indicate a problem in the detector electronics. Contact Dionex for assistance.

Column over safe temperature.

This error occurs when the temperature of the column compartment exceeds the maximum allowed. This error may occur if the ICS-3000 Ion Chromatography System is operating in an environment in which the temperature is greater than 40 °C (104 °F).

To troubleshoot:

Refer to Section A.12 for environmental specifications.

Compartment open circuit.

This error may indicate a problem in the detector electronics. Contact Dionex for assistance.

Compartment over safe temperature.

This error occurs when the temperature of the DC lower compartment is higher than the maximum allowed. This error may occur if the ICS-3000 Ion Chromatography System is operating in an environment in which the temperature is greater than $40 \, ^{\circ}\text{C} (104 \, ^{\circ}\text{F})$.

To troubleshoot:

Refer to Section A.12 for environmental specifications.

ED cell 1 option disconnected.

-or-

ED cell 2 option disconnected.

This error indicates that the electrochemical detector is unplugged.

To troubleshoot:

Check the detector connections: Verify that the signal cable from the cell is connected to the appropriate connector on the detector block (see <u>Figure 8-4</u>). Also, push firmly on the detector to ensure the electronics connector on the back of the detector is securely connected to the receptacle on the detector compartment.

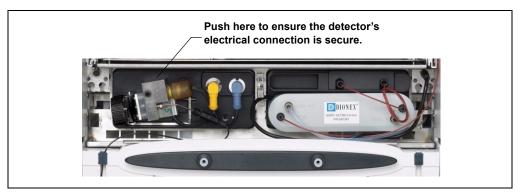


Figure 8-4. Electrochemical Detector Connection

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

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

ED cell 1 working electrode disconnected.

ED cell 2 working electrode disconnected.

This error occurs when the cable on the working electrode is disconnected from the cell electronics.

To troubleshoot:

Check the cable connection: Verify that the signal cable from the working electrode is connected to the detector block (see Figure 8-4).

High pressure valve 1 error.

-or-

High pressure valve 2 error.

High pressure valve 3 error.

High pressure valve 4 error.

This error occurs if a high-pressure valve fails to switch position within 1 second of being toggled. The high-pressure valves are the 6- or 10-port valves installed in either the DC lower (see <u>Figure 8-5</u>) or upper compartment (see <u>Figure 8-6</u>).

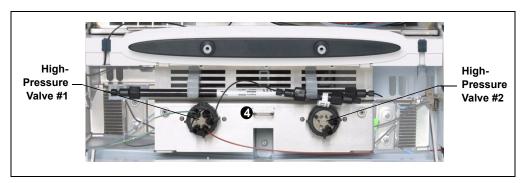


Figure 8-5. High-Pressure Valves #1 and #2



Figure 8-6. High-Pressure Valves #3 and #4

To troubleshoot:

- 1. If a sequence is being executed, terminate the sequence by selecting **Stop** on the Chromeleon or Chromeleon Xpress Control panel.
- 2. Turn off the ICS-3000 Ion Chromatography System power briefly by pressing the **POWER** button on the front of each module. Then press each button again to restart the system.
- 3. Try to toggle the valve from Load to Inject by pressing the **Load** and **Inject** buttons on the front of the DC.
- 4. If the problem persists, contact Dionex for assistance.

! Lower door opened.

This message occurs when the door to the lower compartment is opened during a run.

To troubleshoot:

- 1. Verify that the door is fully closed.
- 2. Check for—and remove—any obstruction.
- 3. If the door is fully closed and the error persists, contact Dionex for assistance

! Lower leak sensor wet.

The leak sensor is installed in the drip tray at the bottom of the column compartment (see <u>Figure 9-21</u>). If liquid accumulates in the 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. Refer to Section 8.26 for detailed troubleshooting of various types of leaks.
- 3. After fixing the leak, dry the drip tray thoroughly to prevent the leak sensor from triggering additional error messages.

Reaction coil open circuit.

This error occurs when the reaction coil heater is unplugged from the DC.

To troubleshoot:

- 1. Check that the reaction coil heater is correctly plugged into the component panel.
- 2. If the error persists, the heater may be faulty. Replace the heater (P/N 061746).

Reaction coil over safe temperature.

This error occurs when the temperature of the DC upper compartment is higher than the maximum allowed. This error may occur if the ICS-3000 Ion Chromatography System is operating in an environment in which the temperature is greater than $40 \, ^{\circ}\text{C}$ ($104 \, ^{\circ}\text{F}$).

To troubleshoot:

Refer to Section A.12 for environmental specifications.

Suppressor 1 over-current.

-or-

Suppressor 2 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 Dionex Reference Library CD-ROM (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 Dionex for assistance.

NOTE The ICS-3000 Ion Chromatography System electronics components cannot be serviced by the user.

Suppressor 1 over-power.

-or-

Suppressor 2 over-power.

This error appears when, in order to maintain the selected current, the ICS-3000 Ion Chromatography System 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 Dionex Reference Library CD-ROM (P/N 053891).
- 3. If the error persists, replace the suppressor (see Section 9.23.2).
- Suppressor 1 over-voltage.

-or-

Suppressor 2 over-voltage.

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

To troubleshoot:

- 1. Check the suppressor cable connection.
- 2. If the error persists, replace the suppressor (see <u>Section 9.23.2</u>).

Suppressor 1 open circuit.

-or-

Suppressor 2 open circuit.

To troubleshoot:

1. Verify that the suppressor cable is connected correctly.

! Upper door opened.

This message occurs when the door to the upper compartment is opened during a run.

To troubleshoot:

- 1. Verify that the door is fully closed.
- 2. Check for—and remove—any obstruction.
- 3. If the door is fully closed and the error persists, contact Dionex for assistance

8.26 Liquid Leaks from DC Components

• Leaking fitting

Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection (see Section 9.17).

Broken liquid line

Replace the line and fittings with the same length and internal diameter tubing (see Section 9.17).

Blocked or improperly installed line

Make sure the lines are not crimped or otherwise blocked. Also, if the blocked line is a waste line, make sure it is not elevated at any point after it exits the DC. If a line is blocked, replace it (see Section 9.17).

• Leaking injection valve

- 1. Make sure the liquid line connections to the transducer are tight. Replace any damaged fittings (see Section 9.17).
- 2. If the leak is from behind the valve stator, the rotor seal may be scratched. Rebuild the injection valve (see Section 9.19).

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 Dionex for assistance.

Leaking suppressor

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

8.27 VALVE Button Not Working

The **VALVE 1** and **VALVE 2** buttons on the front of the DC must be enabled in Chromeleon or Chromeleon Xpress in order to use them to manually switch the inject valves. To enable a button, open the Commands dialog box and select the **Valve1Button** (or **Valve2Button**) command in the list of commands for the DC.

8.28 Amperometry Cell Troubleshooting

8.28.1 Amperometry Cell pH Readout Always 7.0

The pH reading is displayed on the Chromeleon or Chromeleon Xpress EC Detector Control panel.

Disconnected reference electrode

Verify that the reference electrode cable is securely connected (see Figure 8-7).



Figure 8-7. Amperometry Cell Electrical Connections

- Reference electrode short circuit
 Replace the reference electrode (see Section 9.24.4).
- Reference electrode glass membrane broken or cracked
 Replace the reference electrode (see <u>Section 9.24.4</u>).

8.28.2 Cannot Set Amperometry Cell pH Readout to 7.0

The pH reading is displayed on the Chromeleon or Chromeleon Xpress **EC Detector** Control panel.

Inaccurate calibration buffer

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

• Dry reference electrode

- Soak the 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 reference electrode potential, see "Using a Digital Voltmeter to Determine Reference Potential Shift" on page 226.
- 2. Replace the reference electrode (see Section 9.24.4).

To prevent a 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 install fitting plugs. For longer shutdowns, remove the electrode from the cell and store it in its storage bottle filled with saturated KCl solution. See Section 4.1.2 for detailed instructions.

8.28.3 Shift in Amperometry 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 reference electrode was new.

Faulty reference electrode

- 1. Check the electrode by following the instructions in Section 8.28.6.
- Regenerate the electrode by soaking 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 reference electrode potential, see "Using a Digital Voltmeter to Determine Reference Potential Shift" on page 226.

3. If soaking does not fix the problem, replace the electrode (see Section 9.24.4).

8.28.4 No Amperometry Cell pH Readout or Intermittent Readout

The pH reading is displayed on the Chromeleon or Chromeleon Xpress **EC Detector** Control panel.

Disconnected reference electrode

Verify that the reference electrode cable is securely connected (see Figure 8-7).

Uncalibrated reference electrode

Calibrate the reference electrode (see Section 9.24.5).

• Dry reference electrode

- 1. Soak the 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 226.
- 2. If soaking the reference electrode does not fix the problem, replace the electrode (see Section 9.24.4).

To prevent a 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 install fitting plugs. For longer shutdowns, remove the electrode from the cell and store it in its storage bottle filled with saturated KCl solution. See Section 4.1.2 for detailed instructions

• Contaminated pH reference electrode

Replace the reference electrode (see <u>Section 9.24.4</u>).

8.28.5 Leak in pH Reference Electrode Compartment

Defective pH reference electrode O-ring

Replace the pH reference electrode O-ring (see Section 9.24.6).

8.28.6 Shift in Ag/AgCl Reference Potential

Faulty 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 reference electrode with no shift.

Following the steps below, measure the Ag/AgCl reference electrode potential shift by comparing it to the potential shift of an unexposed electrode. A spare 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. Using the reference electrode adapter cable, connect the voltmeter's test probes to the center sockets of the unexposed electrode and the reference electrode being tested.
- 2. Immerse both reference 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 50 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 Section 9.24.6).



TC Troubleshooting

8.29 Troubleshooting TC Error Messages

If any of the following alarm conditions occurs, a message is displayed in the Chromeleon or Chromeleon Xpress Audit Trail.

Λ

AcqOff without previous AcqOn.

This error occurs if the Chromeleon or Chromeleon Xpress program contains an **AcqOff** command, but no **AcqOn** command.

To troubleshoot:

Every program should include one **AcqOn** command. Enter the **AcqOn** command after the first injection.

\triangle

Acquisition is still on at program end.

This error occurs if a Chromeleon or Chromeleon Xpress program does not include a command that ends data acquisition.

To troubleshoot:

The **AcqOn** command must be paired with an **AcqOff** command. Enter the **AcqOff** command at the appropriate point in the program.



All samples scheduled for run on timebases x need to specify the same value for property (nominal) Temperature.

-or-



All samples scheduled for run on timebases x need to specify the same value for property TempCtrl.

-or-



All samples scheduled for run on timebases x need to specify the same value for property y.

When a TC is shared by two timebases, the same values must be selected for certain parameters. If the Chromeleon Ready Check detects a discrepancy, this error occurs and batch processing cannot be started.

To troubleshoot:

Find and eliminate any discrepancies in the parameter settings. For example, all samples must turn on **TempCtrl** or all samples must turn off **TempCtrl**.

! Can't execute "Disconnect" command during data acquisition.

-or-

(!)

Can't execute "Standby" command during data acquisition.

Selecting the **Disconnect** or **Standby** command during data acquisition will stop data transmission and cause data acquisition to stall. If you attempt to issue one of these commands during data acquisition, this error occurs.

To troubleshoot:

Wait until data acquisition is completed before attempting to issue either of these commands.



Can't find download image TC3000.HEX.

TC3000.HEX is the version of the TC firmware (Moduleware) file that should be available in the \Bin directory of your Chromeleon or Chromeleon Xpress installation. This error occurs if the file is missing.

To troubleshoot:

Select **Qualification > Chromeleon IQ** to run the Chromeleon IQ and diagnose the problem. When you eliminate the source of the error, rerun the Setup program.



Can't start acquisition at negative retention times.

This error occurs if you attempt to execute the **AcqOn** command while retention times are negative.

To troubleshoot:

Wait until the retention time value is positive.



Can't start acquisition: The server configuration does not fulfill the license conditions.

Operation of the TC requires the appropriate version of Chromeleon or Chromeleon Xpress plus a Timebase Class 1 license. This error occurs if you attempt to start data acquisition when these requirements are not met.

To troubleshoot:

Contact Dionex to obtain the appropriate software version and/license.

Can't start TC firmware. A firmware download may be necessary.

-or-

Error finishing download.

-or-

TC—Firmware download failed.

If the TC firmware (Moduleware) cannot be started after a firmware download or if Chromeleon or Chromeleon Xpress is unable to successfully download firmware to the TC, the appropriate message (shown above) is displayed.

To troubleshoot:

- 1. Press the **POWER** button on the front of the TC to turn off the power; after 30 seconds, press the button again to turn on the power.
- 2. Download new firmware to the TC as follows:
 - a. Insert the CD containing the new firmware version into the CD drive.
 - b. Start the Server Configuration program.
 - c. In the timebase, right-click the TC for which you want to update the firmware and select **Properties** on the context menu.
 - d. On the **General** tab page in the Properties dialog box, click **Firmware Download** to display a file open dialog box.
 - e. Select the file (the extension is .fmw) containing the new firmware version and click **Open** to begin the download. Audit Trail messages will inform you of the status of the download.
- 3. If the error occurs again, contact Dionex for assistance. The firmware cannot be serviced by the user.



Can't use device "x" as a source of the column pressure. Please check the property "SystemPressure" for a valid device name. The device must have the "Pressure" property available.

-or-



Can't use device "x" as a source of the column pressure. Please check the property "y" for a valid device name. The device must have the "Pressure" property available.

This error occurs if the device selected to monitor the system pressure is ineligible to do so.

To troubleshoot:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- 2. Click the plus sign beside the column name and select the **SystemPressure** command.
- 3. Enter the name of the pump currently delivering flow for this column and click **Execute**.

Λ

Card data checksum error.

This error indicates that the column ID chip card memory is corrupted.

To troubleshoot:

Replace the column ID chip card (see Section 9.26).

Command is not available in demo mode.

This error occurs if you attempt to issue a command that is not available when the TC is in demo (or virtual) mode.

To troubleshoot:

If a TC is present and connected, disable the demo mode:

- 1. Start the Server Configuration program.
- 2. In the timebase, right-click the TC and select **Properties** on the context menu
- 3. On the **General** tab page in the Properties dialog box, clear the **Demo Mode** check box and click **OK**.

Communication error: x.

This error occurs if there is a communication failure between the TC and the PC on which Chromeleon or Chromeleon Xpress is installed.

To troubleshoot:

- 1. Check the USB connections.
- 2. Check the connection from the TC to the power supply.
- 3. If you cannot identify the cause of the error, contact Dionex for assistance

Communication time-out.

This error occurs if a TC firmware (Moduleware) command is not executed within the time allotted for its completion.

To troubleshoot:

- 1. Check the USB connections.
- 2. Check the connection from the TC to the power supply.
- 3. If you cannot identify the cause of the error, contact Dionex for assistance



Configuration doesn't match. X not installed.

-or-



Configuration mismatch (x - CM uses y, Column Compartment uses z). Please use the Server Configuration program to check the installation.

This error occurs if there is a discrepancy between the TC properties and the actual configuration.

To troubleshoot:

- 1. Start the Server Configuration program.
- 2. In the timebase, right-click the TC and select **Properties** on the context menu.
- 3. Correct any discrepancies in the Properties dialog box.

(!) Current flow is out of limits defined for the column.

This error occurs if the current flow rate exceeds the value specified by the Column_x.FlowRate_UpperLimit command.

To troubleshoot:

Select a valid flow rate.

• Current pressure is out of limits defined for the column.

This error occurs if the current pressure reading is outside the range specified by the Column_x.Pressure_UpperLimit and Column_x.Pressure_LowerLimit commands.

To troubleshoot:

Find and eliminate any liquid leaks or blockages in tubing connections (see Section 8.31). Adjust the flow rate accordingly.

•

Current temperature is out of limits defined for the column.

This error occurs if the current temperature reading is outside the range specified by the Column_x.Temp_UpperLimit and Column_x.Temp_LowerLimit commands.

To troubleshoot:

Select a valid temperature setting.

Stop

Data buffer overrun.

This error occurs if the system cannot write incoming data to disk fast enough.

To troubleshoot:

Increase the buffer size:

- 1. In the Server Configuration program, right-click the system name and select **Properties** on the context menu.
- 2. On the **Advanced** tab page in the Server Configuration dialog box, enter a new value (in seconds) in the **Buffer** field and click **OK**.

If the error occurs again, contact Dionex for assistance.



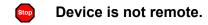
Device control feature is not available! Please check key code and CM Features (see "About Chromeleon..." on the client's Help menu).

This error occurs if you attempt to select or define a feature that is not available for your TC.

To troubleshoot:

Select **Help > About Chromeleon...** to open the About Chromeleon Client screen. On the screen, all license features you purchased (devices controlled, number of timebases, and other software options) are labeled **On**.

If necessary, upgrade your software by acquiring the corresponding licenses. Contact Dionex for assistance.



-or-



TC is not remote.

If you try to issue certain commands (for example, Column_x and InjectValve_x commands) when the TC is not under Chromeleon control, the appropriate message (shown above) is displayed.

To troubleshoot:

Select the **Connect** command to connect the TC to the Chromeleon server. Check the Audit Trail for messages indicating whether the module was successfully connected.

Device not found on the USB.

-or-



USB/LAN communication error. Please check communication cable and instrument's power.

This error occurs if there is a problem with the connection between the TC and the Chromeleon server or with the power supply.

To troubleshoot:

- 1. Check the USB connections.
- 2. Check the connection from the TC to the power supply.
- 3. If you cannot identify the cause of the error, contact Dionex for assistance.



Either the current date is later than the expiration date for x or the program or batch will end later than 24 hours before the expiration time for x.

Chromeleon and Chromeleon Xpress compare the current date with the column expiration date during injection or before beginning batch processing. This error occurs if the expiration date has already passed or if batch processing is scheduled to start less than 24 hours before the expiration date.

To troubleshoot:

Replace the column.

Stop

Error deleting flash memory.

This error occurs if, during a firmware (Moduleware) download, the existing firmware cannot be erased from memory.

To troubleshoot:

Flash memory cannot be serviced by the user. Contact Dionex for assistance.

Λ

Error during storing of the card data to the chip card.

This error occurs if the column ID chip card is installed incorrectly, or is defective.

To troubleshoot:

When the chip card is properly installed, the Dionex logo on the card faces up and the LED on the memory slot is green. If this error occurs when the chip card is properly installed, it indicates that the card is defective and should be replaced (for instructions, refer to Section 9.26).



Error programming flash memory.

This error occurs if, during a firmware (Moduleware) download, the new firmware cannot be programmed into memory.

To troubleshoot:

Flash memory cannot be serviced by the user. Contact Dionex for assistance.

! Executing this command will reset associated counter values stored in the module.

This error occurs when you select a command (**RotorSealChanged**, for example) that updates information stored in the TC. To prevent information from being inadvertently changed, Chromeleon or Chromeleon Xpress requests confirmation before executing the command.

To troubleshoot:

If you want to execute this command, click **Execute**; otherwise, click **Cancel**.

Λ

Firmware download in progress. Please wait.

This error occurs if you attempt to issue a command while firmware (Moduleware) is being downloaded to the TC.

To troubleshoot:

Wait until the firmware download is complete. Audit Trail messages will inform you of the status of the download.

lack

Gas leak detected.

This error occurs if the gas sensor detects an increased concentration of gas inside the TC.

To troubleshoot:

- 1. Find and eliminate the leak.
- 2. On the TC Control panel, click the **Clear Alarm** button.
- 3. Ventilate the interior of the TC thoroughly before closing the door and resuming operation.

Λ

Humidity leak detected.

This error occurs if the humidity sensor detects an increased concentration of humidity inside the TC.

To troubleshoot:

- 1. Find and eliminate the leak.
- 2. On the TC Control panel, click the **Clear Alarm** button.
- 3. Ventilate the interior of the TC thoroughly before closing the door and resuming operation.

Stop

Illegal parameter.

This error occurs if you enter a parameter that is not valid for the TC.

To troubleshoot:

Select a valid parameter.



Invalid date/time format. Either use "DD MMM YYYY" or "current [+ [Nyear[s]] [Nmonth[s]] [Nday[s]]]." Examples: "31 Dec 1999," "current + 1 year 6 months."

This error occurs if you enter the date associated with an event in an invalid format.

To troubleshoot:

Enter the date in the format specified in the message.

! Invalid enumerated value.

This error occurs if you attempt to enter an invalid value for a property, rather than selecting a value from the drop-down list box in the Commands dialog box.

To troubleshoot:

Select a valid option from the drop-down list box available for this property.

• Next qualification of this module is due in x day(s) (due date is [date]).

-or-

• Next qualification of this module is overdue (due date was [date]).

-or-

Next qualification of this module was due on [date]. Allowing x more grace day(s).

-or-



Next qualification of this module is overdue (due date was [date]). Module may no longer be used.

Chromeleon and Chromeleon Xpress monitor the date on which qualification was last performed and display an appropriate message (shown above) at the following times: when the due date for the next qualification is approaching; when the due date is overdue, but the TC can still be used; and when the due date is overdue and the TC can no longer be used.

The **Qualification.WarningPeriod** property determines the number of days before the due date that the warning is first displayed. The **Qualification.GracePeriod** property determines for how many days after the due date you can continue using the TC without performing the qualification.

To troubleshoot:

Contact Dionex to schedule an Operational Qualification and Performance Qualification.

Next service of this module is due in x day(s) (due date is [date]).

-or-

Next service of this module is overdue (due date was [date]).

Chromeleon and Chromeleon Xpress monitor the date on which service was last performed and display an appropriate message (shown above) at the following times: when the due date for the next service is approaching and when the due date is overdue. The **Service.WarningPeriod** property determines the number of days before the due date that the warning is first displayed.

To troubleshoot:

Contact Dionex for information about performing annual preventive maintenance. The DC/TC Preventive Maintenance Kit (P/N 061796) contains all required replacement parts.

lack

No chip card in the reader for the column x.

This error occurs if a column ID chip card is not installed for the column identified in the message.

To troubleshoot:

Install a column ID chip card for the column (see <u>Section 9.26</u>). Or, if you do not want to store information about this column, follow the steps below to disable this option.

- 1. In the Server Configuration program, right-click the TC and select **Properties** on the context menu.
- 2. Select the **Components** tab in the Properties dialog box. Clear the check mark beside the column name and click **OK**.
- No response from x for x seconds.

-or-



No response from x for x seconds. Device disconnected.

This error occurs if there is a communication failure between the TC and the PC on which Chromeleon or Chromeleon Xpress is installed.

To troubleshoot:

- 1. Check the USB connections.
- 2. Check the connection from the TC to the power supply.
- 3. If you cannot identify the cause of the error, contact Dionex for assistance.

Operating columns above 70 °C could affect column performance. Verify recommended column operating conditions before setting temperature above 70 °C.

This error occurs if you select a TC temperature setting above 70 °C (158 °F).

To troubleshoot:

Refer to the column manual for the recommended operating conditions. Column manuals are provided on the Dionex Reference Library CD-ROM (P/N 053891).

Parameter x value exceeded its allowed range. Set to closest valid value x.

-or-

! Value for [name] parameter is not within valid range. Value passed (x) will be substituted by y.

-or-

Ualue x replaced by valid table entry y.

If you select a value outside the allowable range for certain parameters, Chromeleon ignores the setting, replaces it with the closest valid setting, and this error occurs.

To troubleshoot:

No immediate action is required. In future, specify a valid setting for the parameter.

(!)

Primary property "x" is already assigned and cannot be changed.

This error occurs if you attempt to change one of the primary column properties (batch or lot number, date of manufacture, etc.) used for column identification.

To troubleshoot:

Once a primary column property has been assigned, it cannot be changed. As an alternative, consider changing a secondary column property. For a list of primary and secondary properties, refer to the Chromeleon or Chromeleon Xpress Help.



Properties cannot be changed. A chip card has not been inserted properly. Check the CardState property for all cards and reinsert those indicating "BusBlocked."

This error occurs if you attempt to change column ID properties when at least one column ID chip card is inserted incorrectly.

To troubleshoot:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- One at a time, click the plus sign beside each column name and select the CardState command. If the status is BusBlocked, remove the card from the card slot and reinsert it.

When the card is properly installed, the Dionex logo on the card faces up and the LED on the memory slot is green.

\triangle

Properties cannot be changed if the compartment door is open.

This error occurs if you attempt to change column ID properties when the TC door is open (or is not fully closed).

To troubleshoot:

- 1. Verify that the door is fully closed.
- 2. If the door is not fully closed, check for—and remove—any obstruction.
- 3. If the door is fully closed but the error persists, contact Dionex for assistance.



Properties cannot be changed—there is no card in the card reader.

This error occurs if you attempt to change column ID properties for a column for which no chip card is installed.

To troubleshoot:

Insert a column ID chip card into the appropriate slot (see Section 9.26).



Raw data file x cannot be created.

-or-



Raw data file x cannot be created. Continuing with network failure protection.

-or-



Raw data file x cannot be written.

-or



Raw data file x cannot be written. Continuing with network failure protection.

If a network failure prevents Chromeleon from creating or writing to a raw data file, the corresponding error occurs.

To troubleshoot:

Wait until batch processing is complete; then, reboot the PC and restart the Chromeleon server.



Starting data acquisition manually will overwrite any data which has been acquired so far.

This error occurs if you attempt to start data acquisition manually while a batch is already running.

To troubleshoot:

To retain the data already acquired, cancel manual data acquisition; otherwise, go ahead and start data acquisition.



The command can format only chip cards with an invalid data structure.

This error occurs if you select the **FormatChipCard** command for a column ID chip card that is not defective.

To troubleshoot:

Cancel the command. Do not attempt to reinitialize a chip card unless it contains invalid or corrupted data.



The communication interface is closed. Reconnect the instrument.

This error occurs if there is a communication failure between the TC and the PC on which Chromeleon or Chromeleon Xpress is installed.

To troubleshoot:

- Check the USB connections.
- 2. Check the connection from the TC to the power supply.
- 3. If you cannot identify the cause of the error, contact Dionex for assistance.



The compartment door is open; a batch or a program may need to wait until the door has been closed. Please close the door.

This error occurs if you attempt to operate the TC when the door is open (or not fully closed).

To troubleshoot:

- 1. Verify that the door is fully closed.
- 2. If the door is not fully closed, check for—and remove—any obstruction.
- 3. If the door is fully closed but the error persists, contact Dionex for assistance.

The counter [name] (value: x) has exceeded its limit (y). Module should no longer be used. Replace the [name].

This error occurs if the number of hours a TC part has been in use exceeds the lifetime set by the **OperationTime.Limit** property.

To troubleshoot:

Replace the part identified in the message before resuming operation.

lack

The data format stored on the chip card is unsupported (format version x).

This error occurs if the column ID chip card data is inadvertently saved in an invalid format.

To troubleshoot:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- 2. Click the plus sign beside the column name.
- 3. Select FormatChipCard and click Execute to reinitialize the chip card.

! The injections limit for the column has been exceeded.

-or-

The injections limit will be exceeded for the column x.

Chromeleon and Chromeleon Xpress monitor the number of injections done with each column and display an appropriate message (shown above) at the following times: when the injections limit is about to be exceeded or has been exceeded

To troubleshoot:

Replace the column.



The nominal temperature must be set within the current limits.

This error occurs if the selected TC temperature setting is outside the range specified by the **Temperature.LowerLimit** and **Temperature.UpperLimit** commands.

To troubleshoot:

Select a valid temperature setting.



The nominal temperature value is out of range defined by the active column ID.

This error occurs if the selected TC temperature setting is outside the range specified by the Column_x.Temp_LowerLimit and Column_x.Temp_UpperLimit commands.

To troubleshoot:

Select a valid temperature setting.



The oven is not fully operable yet. Check the values of "Standby," "Ready," "Door," and "CardState."

Each time the TC power is turned on, the firmware (Moduleware) runs a series of self-diagnostics in which the status of the main components is checked. This error occurs if a component is not ready for operation.

To troubleshoot:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- 2. Check the status of each command listed in the error message. The table below indicates the status required for operation.

Command	Status
CardState	OK
Door	Closed
Ready	Ready
Standby	NoStandby

3. When you identify the component that is not ready for operation, find and eliminate the cause of the problem.



The primary column ID property "x" will be changed. The property can't be changed in the future.

This error occurs when you modify a primary column property (batch or lot number, date of manufacture, etc.). To prevent any unintended changes, Chromeleon or Chromeleon Xpress requests confirmation before saving the new property.

To troubleshoot:

If you are sure that the proposed change is correct, go ahead and save it.

\triangle

The upper limit must be higher than the lower limit.

This error occurs if the value entered for a parameter's upper limit (for example, **TC_Temp.Signal.UpperLimit**) is less than the lower limit for the parameter.

To troubleshoot:

Increase the value of the upper limit setting.

lack

The Warning threshold must be higher than the Limit threshold.

This error occurs if the **Warning** value selected for a Predictive Performance command (for example, **LeftRotorSealSwitches**) is lower than the **Limit** value. Chromeleon or Chromeleon Xpress issues a warning message in the Audit Trail when the **Warning** value is reached and an error message when the **Limit** value is reached.

To troubleshoot:

Select a Warning value that is higher than the Limit value.

Λ

The Warning threshold must be lower than the Limit threshold.

This error occurs if the **Warning** value selected for a parameter is higher than the **Limit** value. Chromeleon or Chromeleon Xpress issues a warning message in the Audit Trail when the **Warning** value is reached and an error message when the **Limit** value is reached.

To troubleshoot:

Select a Warning value that is lower than the Limit value.

! There was already an AcqOff command for this channel before.

-or-

! There was already an AcqOn command for this channel before.

-or-

lack

Two AcqOn commands for the same channel. The second chromatogram will overwrite the first.

A Chromeleon or Chromeleon Xpress program can include no more than one command to start or terminate data acquisition for a channel; if multiple commands are present, the appropriate message (shown above) appears.

To troubleshoot:

Remove the duplicate commands from the program.

This function cannot be adjusted by the user.

This error occurs if you attempt to change the setting for a read-only parameter.

To troubleshoot:

Do not attempt to reset read-only parameters.

This operation may affect the oven's operation on timebase x, which is currently running a batch.

If batch processing is in progress when you attempt to change a property (such as **Temperature** or **TempCtrl**) that affects the entire TC, this error occurs.

To troubleshoot:

To avoid disrupting operation, cancel the command.

! Unknown error code.

This error occurs if Chromeleon or Chromeleon Xpress does not recognize error code sent by the TC firmware (Moduleware).

To troubleshoot:

The firmware cannot be serviced by the user. Contact Dionex for assistance.

\triangle

Unknown format of the card data.

This error occurs if Chromeleon or Chromeleon Xpress does not recognize the format of data stored on the column ID chip card.

To troubleshoot:

- 1. In the Commands dialog box in Chromeleon or Chromeleon Xpress, click the plus sign beside **TC** to display the items underneath.
- 2. Click the plus sign beside the column name.
- 3. Select FormatChipCard and click Execute.
- 4. If the error occurs again, insert a new column ID chip card (see Section 9.26).

Stop

X position error.

This error occurs if a TC injection valve fails to switch position within the time allotted.

To troubleshoot:

- 1. If a sequence is being executed, terminate the sequence by clicking **Abort Batch** on the **Sequence Control** panel.
- 2. Press the **POWER** button on the front of the TC to turn off the power; after 30 seconds, press the button again to turn on the power.
- 3. Try to toggle the injection valve from Load to Inject by clicking the **Load** and **Inject** buttons on the Control panel.
- 4. If the error occurs again, contact Dionex for assistance.

- lack
- You do not have the privilege to approve the module's qualification.
- Λ
- You do not have the privilege to change the performance limits.
- lack

You do not have the privilege to change the qualification intervals.

In controlled environments, users are not allowed to perform certain operations unless they have been granted the appropriate privileges. If you attempt to do so, the appropriate message (shown above) is displayed.

To troubleshoot:

The Chromeleon administrator assigns various privileges to individual users or to user groups. Contact the administrator if you need one or more of the above privileges assigned to you.

8.30 TC ALARM LED Is Lighted

If the **ALARM** LED lights (red), check the Chromeleon or Chromeleon Xpress Audit Trail for the cause.

Gas leak inside the TC

- 1. Find and eliminate the leak.
- 2. On the TC Control panel, click the **Clear Alarm** button.
- 3. Ventilate the interior of the TC thoroughly before closing the door and resuming operation.

• Humidity leak inside the TC

- 1. Find and eliminate the leak.
- 2. On the TC Control panel, click the **Clear Alarm** button.
- 3. Ventilate the interior of the TC thoroughly before closing the door and resuming operation.

• Failure of a mechanical component

If a **VALVE** LED is flashing, a mechanical component in the TC injection valve has failed. Replace the injection valve "pod" (see <u>Section 9.28</u>) and then click the **Clear Alarm** button on the TC Control panel.

If the **OVEN** LED is flashing, a mechanical component in the TC has failed. These parts are not user-serviceable. Contact Dionex for assistance.

8.31 Liquid Leaks from TC Components

• Leaking fitting

Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection (see Section 9.25).

Broken liquid line

Replace the line with tubing of the same length and internal diameter (see Section 9.25).

• Blocked or improperly installed line

Make sure the lines are not crimped or otherwise blocked. Also, make sure the waste line is not elevated at any point after it exits the TC. If a line is blocked, replace it (see Section 9.25).

• Leaking injection valve

- 1. Make sure the liquid line connections to the valve are tight. Replace any damaged fittings (see Section 9.25).
- 2. Liquid leaks from behind the valve stator may indicate a scratched rotor seal. Rebuild the injection valve (see Section 9.27).

• Leaking temperature stabilizer

- 1. Tighten the fittings on the temperature stabilizer inlet and outlet.
- 2. If tightening the fittings does not eliminate the leak, excessive system backpressure may be the source of the problem. Follow the troubleshooting steps in Section 8.9 to reduce the backpressure.

8.32 TC Temperature Does Not Increase

If the temperature fails to increase as expected, although the temperature set point has not been reached, follow the troubleshooting steps below.

Front door not closed

Verify that the TC door is fully closed. If it is, check the door seal for signs of damage. If the seal is damaged, contact Dionex for assistance.

• Column tubing connections incorrectly oriented

Column tubing connections that are incorrectly oriented can open a small path for ambient air into the TC. Check that the tubing connections are at a 90-degree angle in relation to the door seal.

Ventilation slots on TC are obstructed

Make sure the ventilation slots are not obstructed in any way. Allow at least 6 cm (2.4 in) behind the TC for ventilation.

Ambient temperature is too high

Reduce the ambient temperature (for example, ventilate the lab in which the TC is installed).

8.33 TC Temperature Stabilizer Not Operating Properly

Temperature stabilizer installed incorrectly

Remove the temperature stabilizer and reinstall it correctly. Make sure there is good thermal contact between the temperature stabilizer and the thermal element. For detailed instructions, refer to the *ICS-3000 Ion Chromatography System Installation Instructions* (Document No. 065032).

• TC calibration is incorrect

The TC cannot be calibrated by the user. Contact Dionex for assistance.

• Defective temperature stabilizer

Replace the temperature stabilizer (standard bore, P/N 064548; microbore, P/N 064650).

This chapter describes ICS-3000 Ion Chromatography System service and repair procedures that users may perform. All procedures not included here, including electronics-related repair procedures, must be performed by Dionex personnel. For assistance, contact Dionex Technical Support. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Dionex office.

Before replacing any part, refer to the troubleshooting information in <u>Chapter 8</u> to correctly identify the cause of the problem.



Substituting non-Dionex parts may impair a module's performance, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.



DP/SP Service

9.1 Replacing Tubing and Fittings

The DP/SP is plumbed with the tubing and fittings listed below.

Tubing Size and Type	Color	P/N	Used For
0.25-mm (0.010-in) ID PEEK	Black	042690	Connection between pump heads; connection from vacuum degassing assembly to proportioning valve or eluent valve
0.51-mm (0.020-in) ID PEEK, 13 cm (5 in)	Orange	042855	Microbore systems only: Connection from proportioning valve or eluent valve to inlet check valve
1.02-mm (0.040-in) ID PEEK, 13 cm (5 in)	Tan	054410	Standard bore systems only: Connection from proportioning valve or eluent valve to inlet check valve
0.159-cm (0.0625-in) ID PharMed®	Yellow	063268	Connection from peristaltic pump to primary pump head

Tubing Size and Type	Color	P/N	Used For
0.15-cm (0.060-in) ID Tygon® 2075 tubing	Colorless	064079	Connection from peristaltic pump to seal wash reservoir; connection between pump heads for seal wash
1.58-mm (0.062-in) ID Teflon	Colorless	014157	Connection to eluent reservoirs or vacuum degassing assembly
1.58-mm (0.062-in) ID Polyurethane	Colorless	047203	Connection from secondary pump head to waste
10-mm (0.39-in) ID Polyethylene	Colorless	055075	Drip tray waste line

- Use 1/16-in fittings (P/N 052230) and ferrules (P/N 062511) for connections between the vacuum degassing assembly and the injection valve.
- Use 1/8-in fittings (P/N 052267) and ferrules (P/N 048949) for connections to eluent reservoirs.
- Use double-cone 10-32 ferrule fittings (P/N 043276) and 10-32 bolts (P/N 043275) for all other tubing connections. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Dionex Reference Library CD-ROM (P/N 053891).

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 DC Ship Kit (P/N 062614, dual; P/N 063408, single). Refer to the instructions provided with the cutter.

9.2 Cleaning Eluent Reservoirs

Before preparing new eluent, all eluent reservoirs should be rinsed thoroughly (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water. If a reservoir still appears dirty, or has a slimy film on the inside, clean it as instructed below.

- 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.
- 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.

9.3 Priming the DP/SP

The pump should be primed if the eluent has been changed, the eluent line is new (empty), or the eluent line contains air. This section describes two procedures:

- For instructions on using the **PUMP PRIME** button to prime the pump, refer to Section 9.3.1.
- For instructions on priming the pump from the Control panel in Chromeleon or Chromeleon Xpress, refer to Section 9.3.2.

Although a 10 cc syringe (P/N 054578) can be used with either priming procedure, Dionex recommends using a syringe only if eluent lines are 100% empty or if the pump is dry.

9.3.1 Priming with the PUMP PRIME Button

- 1. **Gradient pump only:** Set the channel to be primed (A, B, C, or D) to 100% on the pump Control panel in Chromeleon or Chromeleon Xpress.
- 2. Disconnect the DP/SP from the software by clearing the **Connected** check box in the upper left corner of the Control panel.
 - NOTE When the pump is connected to Chromeleon or Chromeleon Xpress, the PUMP PRIME buttons are disabled.
- 3. Open the priming valve on the secondary pump head (see <u>Figure 9-1</u>) by turning it one-half turn counterclockwise.

NOTE If the priming valve is opened too much, air is drawn through the valve and air bubbles can be seen exiting the waste line.

- 4. Press **PUMP PRIME 1** (or **PUMP PRIME 2**) on the front of the DP/SP. The pump will begin pumping at approximately 6.0 mL/min.
- 5. Continue priming the pump until all air and previous eluents are purged and no air bubbles can be seen exiting the waste line.

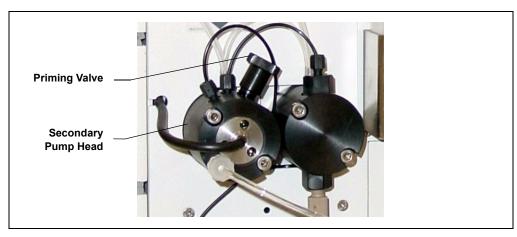


Figure 9-1. DP/SP Priming Valve

6. Gradient pump only:

- a. If all eluent lines have not been primed, select another eluent channel and repeat the priming procedure.
- b. After priming all eluent lines, press **PUMP PRIME 1** (or **PUMP PRIME 2**) to stop priming and return to the flow rate last selected.
- 7. Close the priming valve by turning it clockwise. **Do not overtighten** the priming valve.

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 down.

8. Click the **Connected** check box on the Control panel to reconnect the pump to Chromeleon or Chromeleon Xpress.

9.3.2 Priming from the Control Panel

- Gradient pump only: Set the channel to be primed (A, B, C, or D) to 100% on the pump Control panel in Chromeleon or Chromeleon Xpress.
- 2. Under **Prime Control** on the Control panel:
 - a. Set the **Duration** to 300 sec.
 - b. Set the **Prime Rate** to 6.0 mL/min.
- 3. Open the priming valve on the secondary pump head (see <u>Figure 9-1</u>) by turning it one-half turn counterclockwise.

NOTE If the priming valve is opened too much, air is drawn through the valve and air bubbles can be seen exiting the waste line.

- 4. To enable priming, click the **Prime** button on the Control panel.
- 5. Continue priming the pump until all air and previous eluents are purged and no air bubbles can be seen exiting the waste line.

6. Gradient pump only:

- a. If all eluent lines have not been primed, select another eluent channel and repeat the priming procedure.
- b. After priming all eluent lines, click the **Prime** button to stop priming.
- c. Close the priming valve by turning it clockwise. **Do not overtighten the priming valve.**

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 down.

- 7. Enter the **Flow** rate required for your application on the Control panel.
- 8. **Gradient pump only:** Enter the required proportions of eluents A, B, C, and D on the Control panel (under **Gradient Control**).
- 9. Click **Motor** on the Control panel to turn on the pump flow.

NOTE After starting the pump, wait at least 5 minutes (longer for flow rates below 1.0 mL/min) before beginning an analysis. This allows the DP/SP Moduleware to stabilize the flow rate.

9.4 Replacing the 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.

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Begin monitoring the **Current Pressure** reading on the Control panel. When the system pressure reaches zero, do the following:
 - Disconnect the pump from the software by clearing the **Connected** check box on the Control panel.
 - Press the **POWER** button on the front of the DP/SP for 2 seconds to turn off the pump.
- 3. Open the DP/SP door to access the mechanical components.
- 4. Use the handles on the component mounting panel to pull the panel forward until it reaches the stop.
 - NOTE On newer pumps, three red Phillips screws are installed on the component mounting panel before shipment from the factory. If the mounting panel does not move freely, make sure these screws have been removed.
- 5. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

IMPORTANT

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.



6. The check valves are installed in the primary pump head (see <u>Figure 9-2</u>).

Figure 9-2. DP/SP Check Valve Connections

- 7. Disconnect the tubing connections from the inlet and outlet check valves.
- 8. Using the 1/2-inch wrench (P/N 062336) provided in the Ship Kit (DP Ship Kit, P/N 062463; SP Ship Kit, P/N 063342), loosen both check valve assemblies.
- 9. Remove both check valve assemblies from the pump head.

NOTE The *inlet* check valve assembly housing has a 1/4-28 port; the *outlet* check valve assembly housing has a smaller, 10-32 port (see Figure 9-3).

10. Inspect the new inlet check valve assembly (P/N 045722) to verify that the double-hole end of the cartridge (see Figure 9-3) is visible.

If the double-hole end is not visible, remove the cartridge from the housing and install it correctly.

11. Inspect the new outlet check valve assembly (P/N 045721) to verify that the single-hole end of the cartridge is visible (see Figure 9-3).

If the single-hole end is not visible, remove the cartridge from the housing and install it correctly.

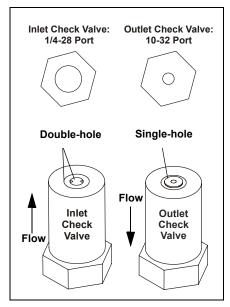


Figure 9-3. Check Valve Assemblies

NOTE The pump cannot operate properly unless the cartridges are 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.

12. 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.

NOTE The opening for the outlet check valve assembly is slightly closer to the back of the pump head than the opening for the inlet check valve assembly.

13. 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.

- 14. Push the component mounting panel back into the enclosure. Close the DP/SP door
- 15. Press the **POWER** button on the front of the DP/SP to turn on the pump.

- 16. Select the **Connected** check box on the Control panel to reconnect the pump to the software.
- 17. Click **Motor** on the Control panel to start the pump flow.

9.5 Replacing a Piston Seal

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 (see Section 9.5.1)
- Cleaning the piston (see Section 9.5.2)
- Removing the main piston seal (see <u>Section 9.5.3</u>)
- Removing the piston seal wash seal (see <u>Section 9.5.4</u>)
- Installing new seals (see <u>Section 9.5.5</u>)
- Reinstalling the piston and pump head (see Section 9.5.6)

9.5.1 Removing the Pump Head and Piston

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Begin monitoring the **Current Pressure** reading on the Control panel. When the system pressure reaches zero, do the following:
 - Disconnect the pump from the software by clearing the **Connected** check box on the Control panel.
 - Press the **POWER** button on the front of the DP/SP for 2 seconds to turn off the pump.
- 3. Open the DP/SP door to access the mechanical components.

- 4. Use the handles on the component mounting panel to pull the panel forward until it reaches the stop.
 - NOTE On newer pumps, three red Phillips screws are installed on the component mounting panel before shipment from the factory. If the mounting panel does not move freely, make sure these screws have been removed.
- 5. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

IMPORTANT

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.

- 6. Disconnect all tubing connections to the pump head with the defective piston seal.
- 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.
 - Refer to <u>Figure 9-4</u> or <u>Figure 9-5</u> when disassembling and reassembling the primary pump head or secondary pump head, respectively.
- 8. Remove the seal wash body (P/N 062064) from the pump head. Or, if the seal wash body was not removed with the pump head in <u>Step 7</u>, pull it straight out of the pump mechanism now.
- 9. Pull the piston (P/N 062082) 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.

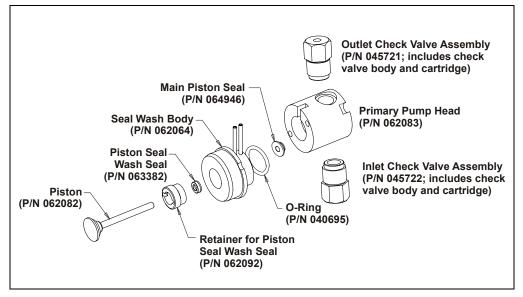


Figure 9-4. DP/SP Primary Pump Head Assembly

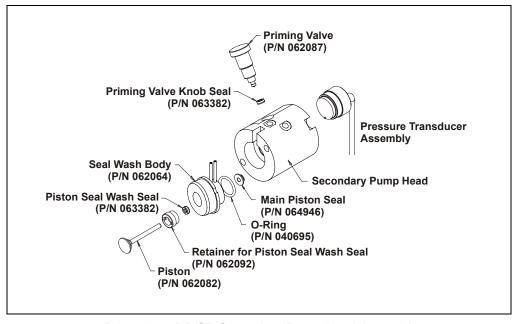


Figure 9-5. DP/SP Secondary Pump Head Assembly

9.5.2 Cleaning the Piston

- 1. Place the piston in a beaker containing ASTM Type I (18 megohm-cm) filtered and deionized water or methanol and sonicate for several minutes.
- 2. After cleaning, rinse the piston thoroughly with ASTM Type I (18 megohm-cm) filtered and deionized water and 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 Section 9.6).

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.

9.5.3 Removing 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.
- 2. 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.
- 3. Using a 10 cc syringe (P/N 054578), inject a few drops of ASTM Type I (18 megohm-cm) filtered and deionized water through the main piston seal (P/N 064946) and into the piston cavity.
- 4. Reinsert the piston (P/N 062082) 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 piston seal. This will scratch the inside of the pump housing; these scratches will prevent a proper seal and cause leakage.

- 5. If the piston seal was not removed in <u>Step 4</u>, 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 <u>Step 4</u>.

9.5.4 Removing the Piston Seal Wash Seal

- 1. Remove the O-ring (P/N 040695) from the seal wash body.
- 2. Follow these steps to remove the piston seal wash seal (P/N 063382) from the seal wash body:
 - a. Using a large flat-blade screwdriver, remove the retainer (P/N 062092) for the seal 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.

9.5.5 Installing the Piston Seals

- 1. Follow these steps to reassemble the seal wash body:
 - a. Place the seal wash body on a clean work surface.
 - b. Slide the seal wash seal (P/N 063382), with the open side of the seal facing upward, onto the seal insertion tool (P/N 063675) (see Figure 9-6).

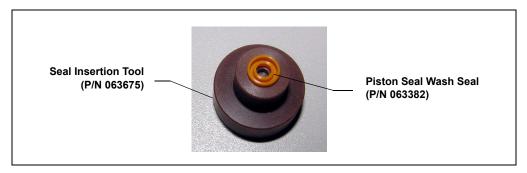


Figure 9-6. Piston Seal Wash Seal and Seal Insertion Tool

- 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.

- 2. Remove the 10-32 fitting plugs from the pump head.
- 3. Insert the piston through the seal wash assembly, and then through the new piston seal (P/N 064946).
- 4. Make sure the piston seal is centered.

IMPORTANT

If the piston seal is not centered, applying pressure to it in <u>Step 7</u> will damage the seal and make it unusable.

- 5. Rinse the piston seal with isopropyl alcohol (IPA) or dip it into a container of IPA. (The seal is easier to install when it is moist.)
- 6. 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.
- 7. Place the components on the pump head and *gently* press the housing until the piston seal snaps into place.

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.

9.5.6 Reinstalling 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 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. Push the component mounting panel back into the enclosure. Close the DP/SP door
- 6. Press the **POWER** button on the front of the DP/SP to turn on the pump.
- 7. Select the **Connected** check box on the Control panel to reconnect the pump to the software.
- 8. Click **Motor** on the Control panel to turn on the pump flow.

9.6 Replacing the 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 (see <u>Section 9.6.1</u>)
- Installing a new piston (see <u>Section 9.6.2</u>)
- Reinstalling the pump head (see <u>Section 9.6.3</u>)

9.6.1 Removing the Pump Head and Piston

Refer to <u>Figure 9-4</u> or <u>Figure 9-5</u> when disassembling and reassembling the primary pump head or secondary pump head, respectively.

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Begin monitoring the **Current Pressure** reading on the Control panel. When the system pressure reaches zero, do the following:
 - Disconnect the pump from the software by clearing the **Connected** check box on the Control panel.

- Press the **POWER** button on the front of the DP/SP for 2 seconds to turn off the pump.
- 3. Open the DP/SP door to access the mechanical components.
- 4. Use the handles on the component mounting panel to pull the panel forward until it reaches the stop.

NOTE On newer pumps, three red Phillips screws are installed on the component mounting panel before shipment from the factory. If the mounting panel does not move freely, make sure these screws have been removed.

5. To prevent contamination of pump parts, wear cleanroom gloves while disassembling the pump head.

IMPORTANT

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.

- 6. Disconnect all tubing connections to the pump head with the damaged piston.
- 7. Using a 3.0 mm hex key (P/N 062338), loosen the Allen screws on the pump head with the damaged piston. Remove the Allen screws, and then carefully remove the head and place it on a clean surface.
- 8. If the piston was not removed with the pump head in <u>Step 7</u>, 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.

9. 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 remove all debris.

9.6.2 Installing the New Piston

Slide the new piston (P/N 062082) *partway* into the pump head; approximately 6 mm (1/4 in) of the sapphire part of the piston should extend from the head.

9.6.3 Reinstalling the Pump Head

- 1. Place the pump head back on the pump.
- 2. 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.
- 3. Reconnect all tubing connections to the pump head. Tighten connections fingertight, and then tighten an additional one-quarter turn only.
- 4. Push the component mounting panel back into the enclosure, using the handle in the center of the panel. Close the DP/SP door.
- 5. Press the **POWER** button on the front of the DP/SP to turn on the pump.
- 6. Select the **Connected** check box on the Control panel to reconnect the pump to the software.
- 7. Click **Motor** on the Control panel to turn on the pump flow.

9.7 Replacing the Piston Seal Wash Tubing

Check the piston seal wash tubing weekly for crimping or blockage and replace as needed.

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Open the DP/SP door to access the mechanical components.
- 3. Remove the old tubing from the peristaltic pump (see Figure 9-7) as follows:
 - a. Lift the lever up and to the right and hold it in that position with one hand.
 - b. With your other hand, pull the PharMed tubing away from the rotor and out of the lower notch on the left side of the mounting plate.
 - c Release the lever
 - d. Pull on the fitting slightly to remove the PharMed tubing from the upper notch on the pump mounting plate.

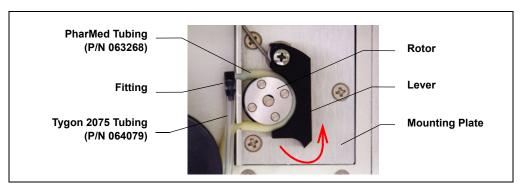


Figure 9-7. Peristaltic Pump for Seal Wash System

- 4. Pull off the PharMed and Tygon 2075 tubing from the fitting. Save the fitting.
- 5. Pull off all seal wash tubing from the seal wash reservoir and the primary and secondary pump heads.
- 6. Cut three new pieces of Tygon 2075 tubing (0.15-cm (0.060-in) ID; P/N 064079) and one piece of PharMed tubing (0.159-cm (0.0625-in) ID; P/N 063268) to the same lengths as the tubing just removed.

The Ship Kit (DP Ship Kit, P/N 062463; SP Ship Kit, P/N 063342) includes a coil of PharMed tubing. The Preventive Maintenance Kit (DP, P/N 061794; SP, P/N 061795) includes a coil of Tygon tubing.

- 7. Connect the new tubing pieces to the seal wash reservoir and pump heads (see Figure 9-8).
- 8. Push the tubing onto the fitting that was removed in Step 4.
- 9. Connect the PharMed tubing to the peristaltic pump as follows:
 - a. Push the tubing into the lower notch on the pump mounting plate.
 - b. Lift the lever to the right and hold it in that position with one hand.
 - c. With your other hand, wind the tubing around the rotor.
 - d. Release the lever.
 - e. Pull on the fitting slightly to slide the tubing into the upper notch.

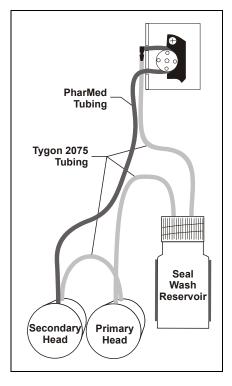


Figure 9-8. Piston Seal Wash Tubing Connections

- f. Make sure there is no slack in the tubing. If necessary, lift the lever again, adjust the tubing around the rotor and through the lower notch to remove any slack, and release the lever.
- 10. Push the component mounting panel back into the enclosure. Close the DP/SP door.
- 11. Click **Motor** on the Control panel to turn on the pump flow.

9.8 Replacing the DP/SP Priming Valve Knob Seal

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.

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Begin monitoring the **Current Pressure** reading on the Control panel. When the system pressure reaches zero, do the following:
 - Disconnect the pump from the software by clearing the **Connected** check box on the Control panel.
 - Press the **POWER** button on the front of the DP/SP for 2 seconds to turn off the pump.
- 3. Open the front door of the DP/SP to access the mechanical components.
- 4. Use the handles on the component mounting panel to pull the panel forward until it reaches the stop.

NOTE On newer pumps, three red Phillips screws are installed on the component mounting panel before shipment from the factory. If the mounting panel does not move freely, make sure these screws have been removed.

5. The priming valve knob is located on the secondary pump head (see <u>Figure 9-9</u>). To remove the knob, turn it counterclockwise all the way and then pull it straight off the pump head.



Figure 9-9. DP/SP Priming Valve

- 6. Pull the old seal off the end of the priming valve knob.
- 7. When correctly installed, the groove in the priming valve knob seal faces away from the knob (see <u>Figure 9-10</u>). Hold the new seal (P/N 063382) carefully, to avoid scratching or nicking the sides, and then slide the seal onto the knob.

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.

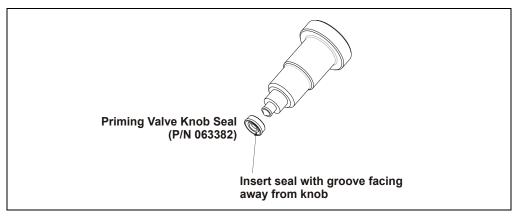


Figure 9-10. Replacing the Priming Valve Knob Seal

- 8. Insert the priming valve knob into the secondary pump head, turn the knob clockwise, and tighten fingertight.
- 9. Push the component mounting panel back into the enclosure. Close the DP/SP door.
- 10. Press the **POWER** button on the front of the DP/SP to turn on the pump.
- 11. Select the **Connected** check box on the Control panel to reconnect the pump to the software.
- 12. Click **Motor** on the Control panel to turn on the pump flow.

9.9 Changing the DP/SP Main Power Fuses

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow.
- 2. Begin monitoring the **Current Pressure** reading on the Control panel. When the system pressure reaches zero, do the following:
 - Disconnect the pump from the software by clearing the **Connected** check box on the Control panel.
 - Press the **POWER** button on the front of the DP/SP for 2 seconds to turn off the pump.
- 3. Turn off the main power switch on the rear panel of the pump (see Figure 2-6).
- 4. Disconnect the main power cord from both its source and from the rear panel of the pump.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the pump.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du pump.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite der Pumpe.

- 5. The fuse cartridge is located next to the main power switch (see <u>Figure 9-11</u>). Use a small screwdriver to remove the fuse cartridge.
- 6. Replace the two fuses with new 2 A IEC 60127-2 slow-blow fuses (P/N 954773). Dionex recommends always replacing *both* fuses.
- 7. Reinstall the fuse cartridge.
- 8. Reconnect the main power cord to its source and to the DP/SP.



Figure 9-11. DP/SP Fuse Cartridge

- 9. Turn on the main power switch.
- 10. Press the **POWER** button on the front of the DP/SP to turn on the pump.
- 11. Select the **Connected** check box on the Control panel to reconnect the pump to the software.
- 12. Click **Motor** on the Control panel to turn on the pump flow.

EG Service

9.10 Replacing Tubing and Fittings

The EG is plumbed with the tubing and fittings listed below.

Tubing Size and Type	Color	P/N	Used For
0.25-mm (0.010-in) ID PEEK	Black	042690	Connections from the EluGen cartridge to the pump, from the EluGen cartridge to the CR-TC, from the RFIC Eluent Degasser to the injection valve, from the RFIC Eluent Degasser to the eluent reservoir
3-mm (0.125-in) OD Teflon tubing	Clear	014157	Connections from the RFIC Eluent Degasser REGEN IN to the CR-TC, from the RFIC Eluent Degasser REGEN OUT to waste
1.58-mm (0.062-in) ID Polyurethane	Clear	047203	EluGen cartridge gas vent line
10-mm (0.39-in) ID Polyethylene	Clear	055075	Drip tray waste line

- Use 1/8-in fittings (P/N 052267) and ferrules (P/N 048949) for connections to the suppressor **REGEN OUT** port and the eluent reservoir.
- Use double-cone 10-32 ferrule fittings (P/N 043276) and 10-32 bolts (P/N 043275) for all other tubing connections. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Dionex Reference Library CD-ROM (P/N 053891).

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 DC Ship Kit (P/N 062614, dual; P/N 063408, single). Refer to the instructions provided with the cutter.

9.11 Isolating a Restriction in the Liquid Lines

A restriction in the liquid plumbing will cause excessive system backpressure.

- 1. Begin pumping eluent through the system (including the columns) at the flow rate normally used.
- 2. 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. If the EluGen cartridge is the source of the high backpressure, replace the outlet frit as instructed in the EluGen cartridge manual. The manual is included on the Dionex Reference Library CD-ROM (P/N 053891).
- 3. 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.

9.12 Replacing the EluGen Cartridge

Replace the EluGen cartridge when the cartridge is expended or when it leaks.

The EluGen cartridge replacement procedure consists of:

- Removing the old EluGen cartridge (see <u>Section 9.12.2</u>)
- Disposing of the old EluGen cartridge (see Section 9.12.3)
- Installing the new EluGen cartridge (see <u>Section 9.12.4</u>)
- Conditioning the new EluGen cartridge (see <u>Section 9.12.5</u>)



The EluGen cartridge 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.



La cartouche d'EluGen 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.



Die EluGen-Kassette 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 Kassette eine Schutzbrille und Handschuhe.

9.12.1 EG Flow Schematic

Refer to <u>Figure 9-12</u> when performing EG and CR-TC service procedures.

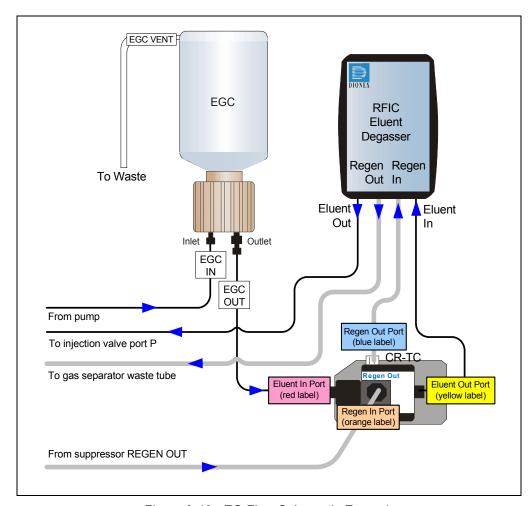


Figure 9-12. EG Flow Schematic Example

9.12.2 Removing the Old EluGen Cartridge

- 1. Click **Motor** on the pump Control panel in Chromeleon or Chromeleon Xpress to turn off the pump flow. (This turns off the power to the EluGen cartridge and the suppressor, also.)
- 2. Open the front door of the EG.
- 3. Push down on the slide release latch and pull the tray forward until it reaches the stop.
- 4. Disconnect the EluGen cartridge electrical cable from the **EGC 1** (or **EGC 2**) bulkhead connector (see <u>Figure 2-8</u>). Twist the ring on the cable counterclockwise to loosen it, and then pull the cable straight out of the connector.
- 5. Remove the **VENT** line from the EluGen cartridge by unscrewing the 10-32 luer adapter (see Figure 9-13).

Tip: If necessary, use a wrench to hold the luer adapter in place while unscrewing the vent line.

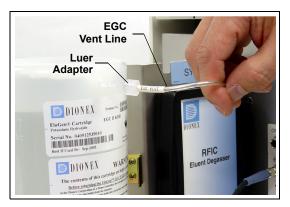


Figure 9-13. Detaching the EluGen Cartridge Vent Line

- 6. Locate the 10-32 plug (P/N 053981) removed from the EluGen cartridge vent opening during installation. Install the plug in the vent opening. (This will prevent leakage from the vent opening when you turn over the cartridge in Step 8.)
- 7. Lift the EluGen cartridge straight up and off the cartridge holder.
- 8. Turn the EluGen cartridge upside down and place it on the EG drip tray (see Figure 9-14). This is the "service position" for the cartridge.

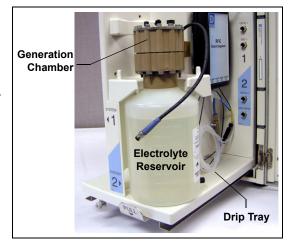


Figure 9-14. EluGen Cartridge in the Service Position

Disconnect the EGC IN and EGC OUT lines from the INLET and OUTLET ports on the EluGen cartridge (see Figure 9-15).

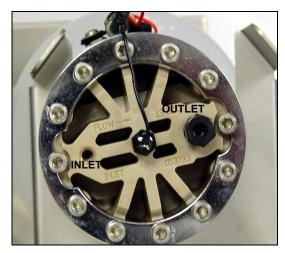


Figure 9-15. EluGen Cartridge Inlet and Outlet Fittings (Shown with fittings removed)

9.12.3 Disposing of the Old EluGen Cartridge

If the EluGen cartridge is expended, follow these steps to dispose of it:

1. Hold the cartridge upright. 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 EluGen cartridge for the chemical description.

2. Rinse the electrolyte reservoir and membranes with ASTM Type I (18 megohm-cm) filtered and deionized water *three times*. Rinsing should render the reservoir and membranes nonhazardous; however, check your local, state, and federal regulatory agency regulations for proper disposal.

If the EluGen cartridge is not expended, it can be stored for up to two years. Follow these steps to prepare the cartridge for storage:

- 1. Plug all fittings. Cap the vent port (this will prevent the evaporation of water in the electrolyte reservoir).
- 2. Store the cartridge in an upright position (with the electrolyte reservoir at 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 "Conditioning the New EluGen Cartridge" on page 288.

9.12.4 Installing the New EluGen Cartridge

1. Remove the new EluGen cartridge from the shipping container.

NOTE Save the shipping container; it may be required for storage or disposal of the EluGen cartridge.

- 2. Verify that a 10-32 plug is installed in the EluGen cartridge vent opening. (This will prevent leakage from the vent opening when you turn over the cartridge.)
- 3. Turn the cartridge upside down and place it on the EG drip tray in the service position (see Figure 9-14).

- 4. Remove the 10-32 plugs from the **INLET** and **OUTLET** fittings on the eluent generation chamber (see Figure 9-15).
- 5. Locate the yellow 0.5 mL/min, 7 MPa (1000 psi) backpressure coil (P/N 053765) in the EG Ship Kit (P/N 062453).
- 6. Connect one end of the backpressure coil to the EluGen cartridge **OUTLET** port; leave the other end of the coil unconnected (see Figure 9-16).

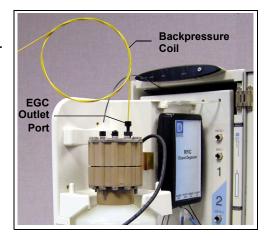


Figure 9-16. EluGen Cartridge Outlet Connection for Conditioning Procedure

NOTE The backpressure coil connection is temporary; it is used only for the EluGen cartridge conditioning procedure.

- 7. Connect the **EGC IN** line from the pump to the EluGen cartridge **INLET** port.
- 8. While holding the EluGen cartridge right-side up (with the electrolyte reservoir on top), shake the cartridge vigorously and tap it with the palm of your hand 10 to 15 times. Make sure any bubbles trapped in the eluent generation chamber are dislodged.
- 9. Slide the EluGen cartridge (with the electrolyte reservoir on top) into the cartridge holder in the EG.

10. Push the cartridge's blue electrical cable connector onto the EGC 1 (or EGC 2) bulkhead connector (see Figure 9-17). Twist the ring on the cable connector fingertight to secure it.

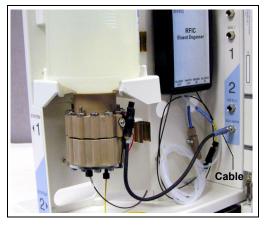


Figure 9-17. Connecting the EluGen Cartridge Electrical Cable

- 11. Remove the 10-32 plug from the EluGen cartridge vent opening. Install a 10-32 luer adapter in the vent opening.
- 12. Connect the luer adapter and **VENT** line removed from the old cartridge to the vent opening in the new cartridge (see <u>Figure 9-13</u>).

IMPORTANT

The vent line must be connected to the EluGen cartridge to ensure proper ventilation.

NOTE Place the 10-32 plug removed from the vent opening in a safe place for storage. The 10-32 plug is required for shipping or storing the cartridge.

9.12.5 Conditioning the New EluGen Cartridge

Always condition a new EluGen cartridge before initial use. This requires directing a backpressure coil connected to the cartridge outlet to waste, and then generating 50 mM of eluent at 1.0 mL/min for 30 minutes.

- 1. Start the Chromeleon Server Configuration program and open the EG Properties dialog box. Click the **Cartridges** tab and enter the serial number of the EluGen cartridge.
- 2. Set a small temporary waste container (for example, a beaker) next to the EG. Direct the yellow backpressure coil from the EluGen cartridge **OUTLET** port to the waste container. The backpressure must be at least 1.4 MPa (200 psi).

- 3. On the panel tabset, select the following settings:
 - a. **Detector Compartment** tab page: Under **Suppressor Settings**, verify that **Mode** is set to off.
 - b. **Eluent Generator** tab page: Verify that the CR-TC is off.

IMPORTANT

To avoid damaging the suppressor and CR-TC, always turn them off before conditioning the EluGen cartridge. The pump flow is on during conditioning; however, no flow reaches the suppressor or CR-TC.

c. **Pump** tab page:

Gradient pump only: Under **Gradient Control**, select the correct eluent channel (A, B, C, or D).

All pumps: Under **Flow Control**, set **Flow** to 1.0 mL/min. This turns on the pump flow, also.

- d. **Eluent Generator** tab page: Enter 50 mM in the **Target Concentration** field. This turns on the power to the EluGen cartridge, also.
- 4. Run at the selected settings (1.0 mL/min at 50 mM) for 30 minutes.
- 5. On the **Pump** tab page, click the **Motor** switch to turn off the pump flow. This turns off the power to the EluGen cartridge, also.

9.12.6 Completing the EluGen Cartridge Installation

- Disconnect the EluGen cartridge electrical cable from the EGC 1 (or EGC 2) bulkhead connector. Twist the ring on the cable counterclockwise to loosen it and then pull the cable straight out of the connector.
- 2. Remove the backpressure tubing from the waste container and remove the waste container.
- 3. Replace the 10-32 plug in the EluGen cartridge vent opening.
- 4. Lift the EluGen cartridge out of the holder, turn it upside down, and place it on the drip tray in the service position (see Figure 9-14).
- 5. Disconnect the backpressure coil from the EluGen cartridge **OUTLET** port.
- 6. Connect the **EGC OUT** line to the **OUTLET** port.

- 7. Turn the EluGen cartridge right-side up, and check for bubbles in the eluent generation chamber. If necessary, shake and tap the cartridge to remove bubbles.
- 8. Reinstall the EluGen cartridge in the holder. Remove the 10-32 plug from the vent opening and reinstall the luer adapter and vent line.
- Push the cartridge's electrical cable connector onto the EGC 1 (or EGC
 bulkhead connector. Twist the ring on the cable connector fingertight to secure it.

9.13 Replacing the CR-TC

Replace the CR-TC when the background and drift are not acceptable.

The CR-TC replacement procedure consists of:

- Removing the old CR-TC (see Section 9.12.2)
- Installing and hydrating the new CR-TC (see <u>Section 9.13.2</u>)
- Completing the plumbing for the new CR-TC (see Section 9.13.3)

Refer to Figure 9-12 for a flow schematic of the EG and CR-TC.

9.13.1 Removing the Old CR-TC

- 1. On the **Pump** tab page on the panel tabset, click the **Motor** switch to turn off the pump flow. This turns off the power to the EluGen cartridge and the suppressor, also.
- 2. Open the front door of the EG.
- 3. Push down on the slide release latch and pull the tray forward until it reaches the stop.
- 4. Follow the instructions in Section 9.12, Step 4 through Step 9, to disconnect the EluGen cartridge electrical cable from the bulkhead connector, cap the EluGen cartridge vent opening, remove the EluGen cartridge, and disconnect the inlet and outlet tubing.
- 5. Pull the CR-TC off the ball studs on the component mounting panel.
- 6. Disconnect the liquid lines from the four ports on the CR-TC.

7. Twist the ring on the CR-TC electrical cable counterclockwise to loosen it and then pull the cable straight out of the CR-TC 1 (or CR-TC 2) bulkhead connector. Remove the CR-TC from the EG.

9.13.2 Installing and Hydrating the New CR-TC

Always hydrate a new CR-TC before initial operation. This requires pumping ASTM Type I (18 megohm-cm) filtered and deionized water through the CR-TC for 10 minutes while bypassing the analytical columns and the suppressor.

- 1. Remove the plugs from the ports on the new CR-TC (CR-ATC, P/N 060477; CR-CTC, P/N 060478).
- 2. Locate the CR-TC Hydration Tubing Kit (P/N 063487) provided in the EG Ship Kit.
- 3. Refer to Figure 9-18 to plumb the four ports on the CR-TC for the hydration procedure.

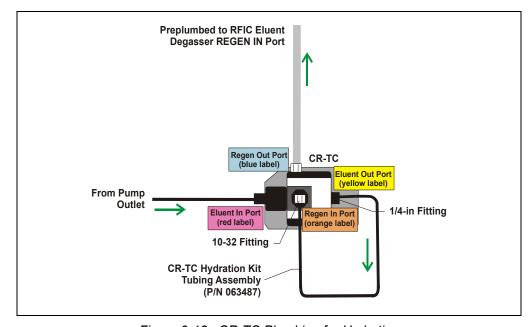


Figure 9-18. CR-TC Plumbing for Hydration

4. On the panel tabset, select the **Detector Compartment** tab. Under **Suppressor Settings**, verify that **Mode** is set to **Off**.

IMPORTANT

To avoid damaging the suppressor, always turn off the suppressor before hydrating the CR-TC. The pump flow is on during conditioning; however, no flow reaches the suppressor.

- 5. Select the **Pump** tab and select the following parameters:
 - a. Under **Flow Control**, set **Flow** to the flow rate recommended for your application.
 - b. Click the **Motor** switch to start the pump flow.
 - c. Gradient pump only: Under Gradient Control, enter 100% for the reservoir containing the ASTM Type I (18 megohm-cm) filtered and deionized water.
- 6. Allow the pump to run for at least 10 minutes.
- 7. Click **Motor** to turn off the pump flow.
- 8. Disconnect the hydration tubing assembly from the **ELUENT OUT** and **REGEN IN** ports on the CR-TC.
- 9. Disconnect the pump outlet line from the **ELUENT IN** port on the CRTC.

9.13.3 Completing the New CR-TC Plumbing

Reinstall the EluGen Cartridge

- 1. Reconnect the EluGen cartridge inlet and outlet lines.
- 2. Turn the EluGen cartridge right-side up, and check for bubbles in the eluent generation chamber. If necessary, shake and tap the cartridge to remove bubbles.
- 3. Reinstall the EluGen cartridge in the holder. Remove the 10-32 plug from the vent opening and reinstall the luer adapter and vent line.
- 4. Push the cartridge's electrical cable connector onto the EGC 1 (or EGC 2) bulkhead connector. Twist the ring on the cable connector fingertight to secure it.
- 5. Refer to Figure 9-19 to plumb the four ports on the new CR-TC.

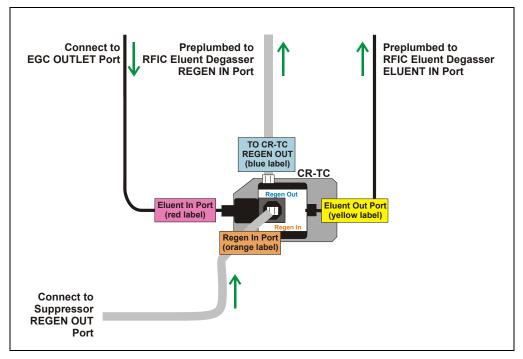


Figure 9-19. CR-TC Plumbing Diagram: Installation Completed

- 6. Check that no liquid lines are caught under the CR-TC. Align the two mounting holes on the CR-TC back plate with the ball studs on the mounting plate, and then push the CR-TC firmly onto the mounting ball studs. The CR-TC clicks into place when properly installed.
- 7. Connect the CR-TC electrical cable to the CR-TC 1 (or CR-TC 2) bulkhead connector. Twist the ring on the cable connector fingertight to secure it.
- 8. Push the tray into the module until it locks into place. Close the EG door.
- 9. Turn on the power and resume operation.

9.14 Replacing the RFIC Eluent Degasser

The RFIC Eluent Degasser contains a tubing assembly that may rupture if subjected to excessive pressure.

- 1. Press the **POWER** button on the front of the EG to turn off the power.
- 2. Open the front door of the EG.
- 3. Push down on the slide release latch and pull the tray forward until it reaches the stop.
- 4. Grasp the degasser by the sides and pull it straight off the ball studs that secure it to the component mounting panel. Dispose of the degasser.
- 5. Disconnect the four lines connected to the RFIC Eluent Degasser fittings (ELUENT OUT, REGEN OUT, REGEN IN, and ELUENT IN).
- 6. Connect the four lines that were disconnected in <u>Step 5</u> to the new RFIC Eluent Degasser (P/N 062137).
- 7. Align the two mounting holes on the back plate of the new RFIC Eluent Degasser with the ball studs on the component mounting panel. Push the degasser firmly onto the mounting ball studs. The degasser clicks into place when properly installed.
- 8. Push the tray into the module until it locks into place. Close the EG door.
- 9. Turn on the power and resume operation.

9.15 Installing a Backpressure Coil

The EluGen cartridge requires at least 14 MPa (2000 psi) of system backpressure; this ensures optimal removal of electrolysis gas from the eluent produced by the cartridge.

During system equilibration, monitor the pressure and verify that it is between 14 and 21 MPa (2000 and 3000 psi). A system backpressure of 16 MPa (2300 psi) is optimal.

If the backpressure is too low, install a backpressure coil between the injection valve and the **OUTLET** port on the EluGen cartridge. Connect one end of the backpressure coil to Port P (2) on the injection valve; connect the other end to the **TO INJ VALVE IN - P** line.

The EG Ship Kit (P/N 062453) includes four backpressure coils (see Table 9-1).

Part Number	Backpressure Coil Description	Flow Rate	Approximate Backpressure Added
053762	4-mm ID	2.0 mL/min	3.5 MPa (500 psi)
		1.0 mL/min	1.75 MPa (250 psi)
053763	4-mm ID	2.0 mL/min	7 MPa (1000 psi)
		1.0 mL/min	3.5 MPa (500 psi)
053764	2-mm ID	0.50 mL/min	3.5 MPa (500 psi)
		0.25 mL/min	1.75 MPa (250 psi)
053765	2-mm ID	0.50 mL/min	7 MPa (1000 psi)
		0.25 mL/min	3.5 MPa (500 psi)

Table 9-1. Backpressure Coils

9.16 Changing the EG Main Power Fuses

- 1. Press the **POWER** button on the front of the EG for 2 seconds to turn off the power.
- 2. Turn off the main power switch on the EG rear panel (see Figure 2-9).
- 3. Disconnect the main power cord from both its source and from the EG rear panel.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the EG.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du EG.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des EG.

- 4. The fuse drawer is located above the main power switch (see <u>Figure 9-20</u>). A small tab locks the fuse drawer in place. Using a small screwdriver, press the tab *in* and *then up* to release the fuse drawer
- 5. Pull the fuse drawer out of the rear panel and remove the old fuses.
- 6. Replace the two fuses with new 2A IEC 60127-2 slow-blow fuses (P/N 954773). Dionex recommends always replacing *both* fuses



Figure 9-20. EG Fuse Drawer

- 7. Insert the fuse drawer into the rear panel and press until the drawer snaps into place.
- 8. Reconnect the main power cord and turn on the power.

DC Service

9.17 Replacing Tubing and Fittings

The DC is plumbed with the tubing and fittings listed below.

Tubing Size and Type	Color	P/N	Used For
0.75-mm (0.030-in) ID PEEK	Green	044777	Connection from injection valve to waste
AS needle seal assembly, 46 cm (18 in), with 10-32 ferrule fitting	Pink	057301	AS normal and concentrate modes only: Connection from the injection valve to the AS inject port
0.33-mm (0.013-in) ID, PEEK	Light Blue	049714	AS sequential modes only: Connection from injection valves to AS diverter valve
0.51-mm (0.020-in) ID, PEEK	Orange	042855	AS simultaneous mode only: Connection from each injection valve to the AS simultaneous injection tee
1.58-mm (0.062-in) ID Teflon	Colorless	014157	Connection from REGEN OUT port on the suppressor
0.125-mm (0.005-in) ID PEEK	Red	044221	Microbore systems: Connections between other system components
0.25-mm (0.010-in) ID PEEK	Black	042690	Standard bore systems: Connections between other system components

- Use 1/8-in fittings (P/N 052267) and ferrules (P/N 048949) for connections to the suppressor **REGEN OUT** port and the eluent reservoir.
- Use double-cone 10-32 ferrule fittings (P/N 043276) and 10-32 bolts (P/N 043275) for all other tubing connections. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Dionex Reference Library CD-ROM (P/N 053891).

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 DC Ship Kit (P/N 062614, dual; P/N 063408, single). Refer to the instructions provided with the cutter.

9.18 Replacing the Leak Sensor

- 1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 2. Press the **POWER** button on the front of the DC to turn off the DC.



To avoid injury, wait for the DC to cool down before opening the door or performing any maintenance or service procedures.



Afin d'éviter toute brûlure, laisser refroidir le DC avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der DC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

3. Open the DC lower door.

4. Loosen the screw on the front of the leak sensor (P/N 062437) (see Figure 9-21). **Note:** The screw remains attached to the sensor.

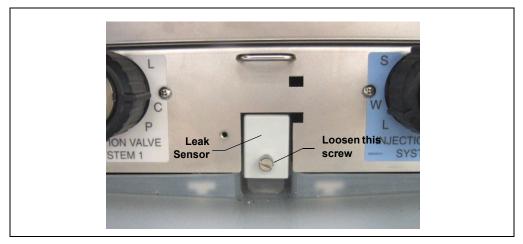


Figure 9-21. Leak Sensor

- 5. Pull out the leak sensor from the component panel and continue pulling until the attached cable is also out of the opening.
- 6. The cable attached to the leak sensor connects to a cable inside the DC. Continue pulling the cable until the connectors for the two cables are outside the panel (see Figure 9-22).
- 7. Disconnect the two cables.

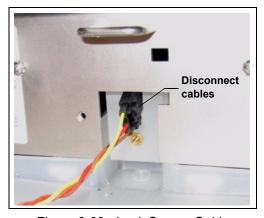


Figure 9-22. Leak Sensor Cable

- 8. Connect the cable from the new leak sensor to the DC cable.
- 9. Feed the cables back inside the DC. Align the leak sensor with the component panel opening and fingertighten the screw.
- 10. Make sure the leak sensor does not touch the bottom of the drip tray.

9.19 Rebuilding a High-Pressure (Injection) Valve

Dionex recommends rebuilding the injection valve annually. The Injection Valve Rebuild Kit (P/N 057896, 6-port valve; P/N 061759, 10-port valve) contains all required replacement parts. This procedure also applies to high-pressure valves installed in an ICS-3000 Automation Manager.

NOTE Substitution of non-Dionex 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.

- 1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 2. Press the **POWER** button on the front of the DC to turn off the DC.



To avoid injury, wait for the DC to cool down before opening the door or performing any maintenance or service procedures.



Afin d'éviter toute brûlure, laisser refroidir le DC avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der DC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

- 3. Open the DC 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.

SAMPLE IN (from door inject port or autosampler)

10 µL SAMPLE LOOP (Black) W 06 1 2 (Red - microbore) (Black - standard bore)

6. Reconnect all liquid lines to the injection valve (see Figure 9-23).

Figure 9-23. Injection Valve Plumbing (6-Port Valve)

VALVE -P

7. Turn on the power to the DC.

(Green)

- 8. Turn on the pump flow. Check for leaks from the valve. Tighten fittings as required (see Section 9.17).
- 9. Close the door.

9.20 Replacing a High-Pressure (Injection) Valve Pod

This procedure describes how to replace the mechanical parts (the "pod") of a high-pressure valve (P/N 061947, 6-port; P/N 061948, 10-port).

NOTE If the valve electronics require service, contact Dionex. Electronics-related repair procedures must be performed by Dionex personnel.

NOTE Substitution of non-Dionex parts may impair valve performance and void the product warranty.

1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.



To avoid injury, wait for the DC to cool down before opening the door or performing any maintenance or service procedures.



Afin d'éviter toute brûlure, laisser refroidir le DC avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der DC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

- Open the DC door.
- 3. Disconnect each liquid line connected to the valve.

4. Unscrew the black locking ring on the front of the valve (see <u>Figure 9-24</u>) and remove the ring.



Figure 9-24. Unscrew the Locking Ring

- 5. Grasp the front of the valve pod and pull out firmly to remove it from the DC or AM.
- 6. Align the slots in the new pod with the runner in the valve holder on the DC or AM (see <u>Figure 9-25</u>). 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.

7. Also verify that the two splines on the pod align with the matching splines inside the valve holder (see <u>Figure 9-25</u>). If necessary, twist the end of the pod to adjust the position of the splines.

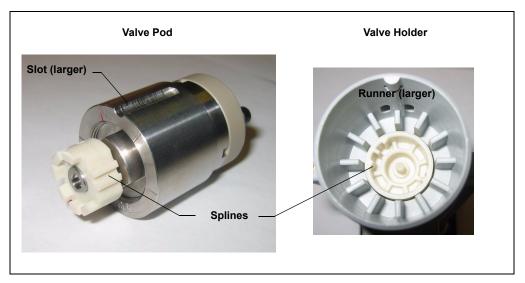


Figure 9-25. High-Pressure Valve Pod and Pod Holder

- 8. Push the Pod into the holder until it clicks into place. Replace the black locking ring.
- 9. Reconnect all liquid lines to the valve.
- 10. Turn on the pump flow. Check for leaks from the valve. Tighten fittings as required (see Section 9.17).
- 11. Close the door.

9.21 Installing or Replacing an I/O Option Board

- 1. Press the **POWER** button on the front of the DC to turn off the power.
- 2. Turn off the main power switch on the DC rear panel (see Figure 2-15).
- 3. Disconnect the main power cord from both its source and from the DC rear panel.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the DC.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du DC.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des DC.

4. On the DC rear panel, remove the two screws that attach the I/O option cover plate or the existing I/O option board to the rear panel (see <u>Figure 9-26</u>). Save the screws.

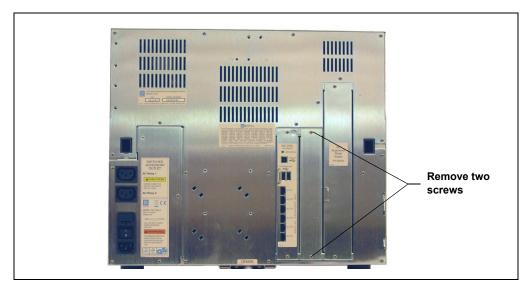


Figure 9-26. DC Rear Panel: Removing the I/O Option Cover Plate

- 5. Slide the new I/O option board (P/N 062201) into the rear panel opening and press firmly to connect the board to the DC motherboard.
- 6. Replace the two screws.

Figure 9-27 shows the I/O option installed.



Figure 9-27. DC Rear Panel: I/O Option Installed

7. Reconnect the main power cord and turn on the power.

9.22 Changing the DC Main Power Fuses

- 1. Press the **POWER** button on the front of the DC for 2 seconds to turn off the power.
- 2. Turn off the main power switch on the DC rear panel (see Figure 2-15).
- 3. Disconnect the main power cord from both its source and from the DC rear panel.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the DC.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du DC.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des DC.

- 4. The fuse drawer is located above the main power switch (see <u>Figure 9-28</u>). A small tab locks the fuse drawer in place. Using a small screwdriver, press the tab *in* and *then up* to release the fuse drawer.
- 5. Pull the fuse drawer out of the rear panel and remove the old fuses.
- Replace the two fuses with new 10 A IEC 60127-2 slow-blow fuse (P/N 954772).
 Dionex recommends always replacing both fuses.



Figure 9-28. DC Fuse Drawer

- 7. Insert the fuse drawer into the rear panel and press until the drawer snaps into place.
- 8. Reconnect the main power cord and turn on the power.

9.23 CD Service Procedures

9.23.1 Calibrating the CD Cell

When to Calibrate

• After every 6 months of use

NOTE Do not use this procedure to calibrate a new cell. New cells are calibrated at the factory.

Items Needed	Description
1.0 mM KCl solution	Prepare by dissolving 0.07456 g of reagent-grade KCl in 1 liter of 18 megohm-cm DI water.
Backpressure tubing to provide at least 7 MPa (1000 psi)	Use 0.25-mm (0.010-in) ID black PEEK tubing (P/N 042690).

- 1. Click the **Cond. Detector** tab in Chromeleon Xpress or on the panel tabset in Chromeleon.
- 2. Click the **Calibration** button under **Conductivity Detector Settings**. The Wellness panel opens (see <u>Figure 9-29</u>).

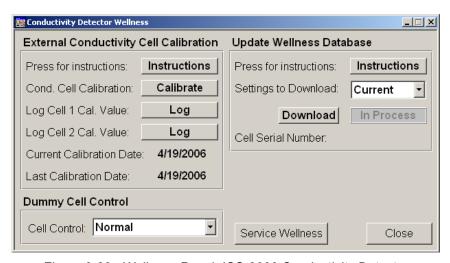


Figure 9-29. Wellness Panel: ICS-3000 Conductivity Detector

- 3. On the Wellness panel under **External Conductivity Cell Calibration**, click **Instructions** for a summary of the procedure (described in detail in this section).
- 4. Disconnect the pump output line from the injection valve.
- 5. Disconnect the line from the suppressor **ELUENT OUT** port to the cell inlet and connect the pump output line directly to the cell inlet.
- 6. Verify that there is a minimum of 7 MPa (1000 psi) of backpressure.
- 7. Set the cell heater to 35 °C. Allow the cell to reach this temperature and then wait an additional 5 minutes to let it stabilize.
- 8. Begin pumping 1.0 mM KCl through the cell at 1.0 mL/min.
- 9. Wait until the total conductivity reading stabilizes (in about 15 minutes). Open the Wellness panel again and click **Calibrate**.
 - After calibration, the conductivity reading should be $147.00 \pm 2 \mu S$. If this is not the case, contact Dionex for help.
- 10. To record the new calibration value in the Audit Trail, click the **Log** button for the cell that was calibrated.
- 11. 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 μS/cm, stop the pump.
- 12. Reconnect the pump to the injection valve and reconnect the line from the suppressor to the cell inlet.

9.23.2 Replacing a Suppressor

Refer to the suppressor manual for guidance about when to replace a suppressor. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

- 1. Refer to the suppressor quick start guide (shipped with the suppressor) for instructions on preparing the suppressor before initial use.
- 2. Press the **POWER** button on the front of the DP/SP to turn off the pump.
- 3. Press the **POWER** button on the front of the DC to turn off the DC.

- 4. Open the DC upper door.
- 5. Disconnect the two eluent and the two regenerant lines from the suppressor (see Figure 9-30).
- 6. Unplug the suppressor cable from the detector.



Figure 9-30. Disconnecting the Suppressor Liquid Lines and Cable

- 7. Remove the suppressor from the detector by sliding it to the left a few millimeters to detach it from the mounting tabs on the detector. Then, pull the suppressor toward you.
- 8. Orient the new suppressor with the **ELUENT IN** fitting on the bottom (see <u>Figure 9-30</u>). Press the suppressor against the back of the detector and then slide it to the right to secure it onto the mounting tabs. Pull out slightly on the center of the suppressor to verify that it is securely fastened.
- 9. Connect the two eluent and the two regenerant lines to the new suppressor.
- 10. Plug in the cable.
- 11. Close the DC door.
- 12. Turn on the DC power and restart the pump flow.

9.23.3 Replacing a CD

- 1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 2. Press the **POWER** button on the front of the DC to turn off the DC.
- 3. Open the DC upper door and disconnect the tubing from the conductivity detector **CELL IN** and **CELL OUT** fittings (see Figure 9-31).



Figure 9-31. Disconnecting the Conductivity Detector Liquid Lines

- 4. Remove the suppressor from the detector (see Section 9.23.2).
- 5. Press the release lever located below the detector. The detector pops out slightly from the DC compartment. Grasp the top of the detector and pull outward to remove the detector from the compartment. **Do not pull from the CELL IN and CELL OUT fittings**.
- 6. Note the electrical plug on the back of the detector and the receptacle on the inside of the DC.
- 7. Push the new detector into the opening in the DC and press firmly to ensure the electrical connection is secure.
- 8. Reconnect the cell inlet and outlet lines.
- 9. Reinstall the suppressor (see Section 9.23.2).
- 10. Turn on the DC power and restart the pump flow.

9.23.4 Removing Trapped Air from the Conductivity Cell

Air bubbles in the cell can cause pulsations of the baseline, random noise, and low readings. Air may result from outgassing of the eluent.

 Connect enough backpressure tubing to the cell outlet to shrink bubbles and allow them to pass more easily through the cell. Refer to <u>Table 9-2</u> for the appropriate type and number of backpressure tubing coils required.

Suppressor Type	Column Format	Flow Rate (mL/min)	Backpressure Coils
AES	2-mm	0.25 to 0.50	2 red*
	3-mm	0.50 to 1.00	2 red
	4-mm	1.00 to 2.00	1 red
SRS or MMS	2-mm or 3-mm	< 0.25	2 red
	2-mm or 3-mm	0.25 to 0.50	1 red
	4-mm	0.50 to 1.50	2 black**
	4-mm	1.50 to 3.00	1 black

^{*}The red coil (P/N 045878) consists of 0.125-mm (0.005-in) ID PEEK tubing with fittings.

Table 9-2. Backpressure Coil Requirements

Table 9-3 lists the correct pressure range for each type of suppressor.

Suppressor Type	Pressure Range	
AES	0.14 to 0.70 MPa (20 to 100 psi)	
SRS or MMS	≤ 0.28 MPa (40 psi)	

Table 9-3. Suppressor Operating Pressure Ranges

2. To verify that the required amount of backpressure is being generated, follow the instructions in the flier, *Backpressure Coil Pressure Test for Dionex Suppressors* (Document No. 031759), which is shipped with the suppressor. The instructions are also in the suppressor manual on the Dionex Reference Library CD-ROM (P/N 053891).

^{**}The black coil (P/N 045877) consists of 0.25-mm (0.010-in) ID PEEK tubing with fittings.

- 3. If a bubble is still trapped in the cell, try these techniques to remove it:
 - Loosen and then tighten the cell outlet fitting.
 - Temporarily disconnect the backpressure coil from the suppressor **REGEN IN** port and then plug and unplug the end of the tubing with your fingertip 2 or 3 times to create a pressure difference.

9.24 ED Service Procedures

9.24.1 Disconnecting the Amperometry Cell

Before performing an amperometry cell service procedure, follow these instructions to disconnect the cell.

- 1. Turn off the cell voltage from the Control panel in Chromeleon or Chromeleon Xpress.
- 2. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 3. Disconnect the inlet and outlet lines from the amperometry cell and disconnect the two electrical cables (see Figure 9-32).
- 4. Grasp the cell by the cell body and pull straight out to remove it from the detector.

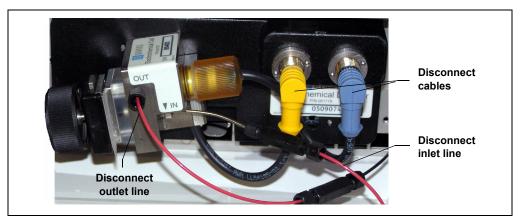


Figure 9-32. Disconnecting the Amperometry Cell (Titanium Cell Shown)

9.24.2 Replacing an Amperometry Cell Gasket

These instructions are for conventional (nondisposable) working electrodes only. To install a disposable working electrode, refer to the manual, which is shipped with the electrode.

When to Replace the Gasket

Replace the gasket if there is a leak between the gasket and electrode, or between the gasket and cell body.

Items Needed

- Amperometry cell gasket for nondisposable working electrodes (P/N 045972)
- Gloves
- Blunt-end tweezers

Amperometry Cell Gasket Replacement Procedure

NOTE To avoid electrode fouling, always wear gloves when handling electrodes.

- 1. Turn off the cell voltage from the Control panel in Chromeleon or Chromeleon Xpress.
- 2. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 3. Disconnect the cell (see Section 9.24.1).
- 4. Loosen the PEEK yoke knob holding the working electrode to the cell body by unscrewing it two to three turns.
- 5. Squeeze the tabs on the sides of the yoke block and pull the block and knob off the working electrode (see Figure 9-33).



Figure 9-33. Disassembling the Amperometry Cell

NOTE Handle the cell gasket and the inside surfaces of the cell carefully to prevent scratches which may subsequently cause leaks.

6. Carefully separate the parts (see Figure 9-34).

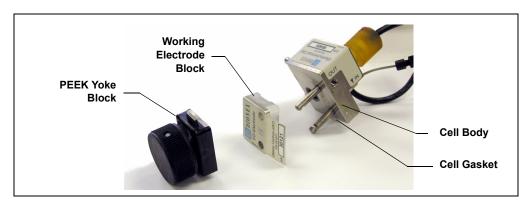


Figure 9-34. Amperometry Cell Components

7. Use tweezers to remove the old cell gasket (see <u>Figure 9-35</u>) from the cell body.

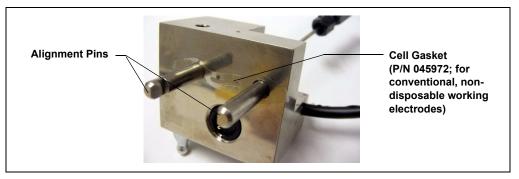


Figure 9-35. Amperometry Cell Gasket

- 8. Rinse surface of the cell with ASTM Type I (18 megohm-cm) filtered and deionized water.
- 9. Clean the polished surface of the cell with a damp soft cloth or tissue.
- 10. Install the new gasket (P/N 045972) over the alignment pins on the cell body. When correctly installed, one end of the gasket extends beyond the cell body, to facilitate gasket installation and removal.
- 11. Verify that the gasket is flat against the cell body and has no wrinkles.
- 12. Reinstall the working electrode block and the PEEK yoke knob and block. Fingertighten the PEEK yoke knob by turning it a full 360 degrees.

NOTE It is not possible to overtighten the PEEK yoke knob. Once the knob clicks into place, it does not tighten any further. The PEEK yoke knob ensures constant pressure is applied to the cell.

- 13. Orient the cell assembly with the PEEK yoke knob on the left and push the cell onto its mounting location in the DC compartment.
- 14. Reconnect the cell's inlet and outlet lines.
- 15. Reconnect the electrical cables.
- 16. Start the pump flow.
- 17. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.

9.24.3 Polishing an Amperometry Cell Working Electrode

These instructions are for conventional (nondisposable) working electrodes only. **Do not polish disposable electrodes**.

When to Polish the Working Electrode

- Polish carbohydrate gold, platinum, silver, and glassy carbon working electrodes before initial installation in the amperometry cell. Do not polish new AAA gold electrodes before installation.
- 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).
- Pen that writes with indelible ink
- Gloves
- Tweezers

Working Electrode Polishing Procedure

NOTE To avoid electrode fouling, always wear gloves when handling electrodes.

- 1. Follow the instructions in <u>Section 9.24.2</u>, <u>Step 1</u> through <u>Step 6</u> to remove the working electrode from the cell.
- 2. Prepare the polishing pads:
 - a. Using indelible ink, label the plastic side of a pad to designate it for use with the coarse polishing compound.
 - b. Label 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:

- 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 one minute. If the pad dries out while polishing, add water sparingly. However, **never** allow the polishing compound to dry on the cell body.
- 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 <u>Step 3</u>, 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 <u>Step 5</u>, 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.
- 7. Replace the working electrode block and PEEK yoke knob. Fingertighten the knob by turning it a full 360 degrees.
 - NOTE It is not possible to overtighten the PEEK yoke knob. Once the knob clicks into place, it does not tighten any further. The PEEK yoke knob ensures constant pressure is applied to the cell.
- 8. Orient the cell assembly with the PEEK yoke knob on the left and push the cell onto its mounting location in the DC compartment.
- 9. Reconnect the cell's inlet and outlet lines.
- 10. Reconnect the electrical cables.
- 11. Start the pump flow.
- 12. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.
- 13. 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

9.24.4 Replacing an Amperometry Cell Reference Electrode

When to Replace the Reference Electrode

Replace the reference electrode 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 reference electrode typically lasts from 3 months to 1 year, depending on use.

NOTE To regenerate a reference electrode, soak it in a solution of 1 M KCl and 1 M HCl.

Items Needed

pH-Ag/Ag/Cl reference electrode (P/N 061879)

Reference Electrode Replacement Procedure

- 1. Turn off the cell voltage from the Control panel in Chromeleon or Chromeleon Xpress.
- 2. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 3. Disconnect the cell (see Section 9.24.1).

4. Unscrew the reference electrode and remove it from the cell body (see Figure 9-36).

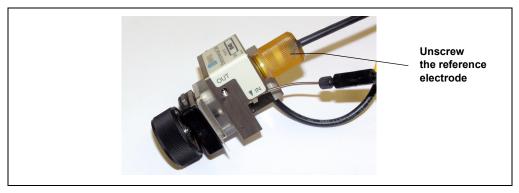


Figure 9-36. Removing the Reference Electrode

5. Unscrew the storage cap from the new reference electrode (see <u>Figure 9-37</u>). Save the 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 reference electrode membrane from drying out and damaging the electrode. See Section 6.4 for storage instructions.



Figure 9-37. Reference Electrode in Storage Cap

- 6. Rinse the electrode thoroughly in ASTM Type I (18 megohm-cm) filtered and deionized water to remove any precipitated salt.
- 7. Calibrate the reference electrode (see Section 9.24.5).

9.24.5 Calibrating the Reference Electrode

When to Calibrate

• Calibrate after installing a new reference electrode.

Items Needed

- 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, either pH 10 or pH 4 in most cases)

Reference Electrode Calibration Procedure

- 1. Complete the following if you are calibrating an existing electrode:
 - a. Turn off the cell voltage from the Control panel in Chromeleon or Chromeleon Xpress.
 - Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press PUMP FLOW on the front of the pump.
 - c. Disconnect the cell (see Section 9.24.1).
 - d. Unscrew the reference electrode and remove it from the cell body.
 - e. Rinse the reference electrode thoroughly in ASTM Type I (18 megohm-cm) filtered and deionized water to remove any precipitated salt.
- 2. With the reference electrode removed from the cell, connect the electrical cables to the cell and electrode.
- 3. Click the **EC Detector** tab in Chromeleon Xpress or on the panel tabset in Chromeleon

Electrochemical Detector Wellness pH Electrode Calibration Current and Previous Values Current pH Electrode Offset: 0.00 Instructions Press for instructions: 59.17 Current pH Electrode Slope: 10.00 Current pH Reading: Current Calibration Date: 4/19/2006 Offset Cal. pH 7 Offset Calibration: 0.00 Previous pH Electrode Offset: pH Slope Buffer Value: 10.00 Previous pH Electrode Slope: 59.17 Slope Cal. pH Slope Calibration: Previous Calibration Date: 4/19/2006 Update Wellness Database Instructions Press for instructions: Settings to Download: Current ▼ Download Service Wellness Close

4. Click the **Calibration** button under **Electrochemical Detector Settings**. The Wellness panel opens (see Figure 9-38).

Figure 9-38. Wellness Panel: ICS-3000 Conductivity Detector

- 5. Place the electrode in the pH 7.00 buffer. Allow the pH to stabilize (about 1 minute) until the reading fluctuates no more than 0.05, and then click the **Offset Cal.** button.
- 6. Remove the electrode from the first buffer, rinse, and then dry it. Place the electrode in the second buffer solution. Allow the pH to stabilize until the reading fluctuates no more than 0.05.
- 7. On the Wellness panel, enter the pH of the second buffer in the pH Slope Buffer Value field and then click the Slope Cal. button.
- 8. 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.
- 9. Verify that the reference electrode O-ring is present (see <u>Figure 9-39</u>) and then screw the reference electrode into the reference electrode cavity.



Figure 9-39. Reference Electrode O-Ring

- 10. Orient the cell assembly with the PEEK yoke knob (see <u>Figure 9-34</u>) on the left and push the cell onto its mounting location in the DC compartment.
- 11. Reconnect the cell's inlet and outlet lines.
- 12. Start the pump flow.
- 13. Turn on the DC power.
- 14. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.

9.24.6 Replacing the Reference Electrode O-Ring

- 1. Turn off the cell voltage from the Control panel in Chromeleon or Chromeleon Xpress.
- 2. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 3. Disconnect the cell (see Section 9.24.1).
- 4. Unscrew the reference electrode and remove it from the cell body (see Figure 9-40).

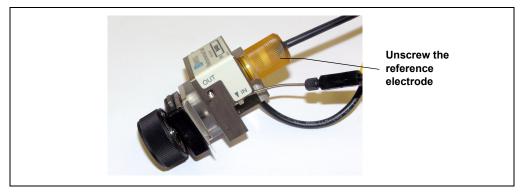


Figure 9-40. Removing the Reference Electrode

- 5. To remove any particulate matter (such as salt crystals), rinse and dry the reference electrode cavity.
- 6. Remove the O-ring from the bottom of the reference electrode (see Figure 9-41).



Figure 9-41. Removing the Reference Electrode O-Ring

- 7. Slide the new O-ring (P/N 014067) onto the electrode.
- 8. To avoid any hydraulic pressure buildup when inserting the reference electrode, make sure that fitting plugs are not installed on the cell inlet and outlet fittings.
- 9. Screw the reference electrode into the cell body and tighten firmly with your fingers.

- 10. Orient the cell assembly with the PEEK yoke knob (see <u>Figure 9-34</u>) on the left and push the cell onto its mounting location in the DC compartment.
- 11. Reconnect the two cables and the liquid lines.
- 12. Start the pump flow.
- 13. Wait until the pump pressure has stabilized (30 to 60 seconds) and then turn on the cell voltage.

9.24.7 Replacing an ED Detector

- 1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 2. Disconnect the ED from the Control panel in Chromeleon or Chromeleon Xpress.
- 3. Press the **POWER** button on the front of the DC to turn off the DC.
- 4. Open the DC upper door and disconnect the cell (see Section 9.24.1).
- 5. Remove the cell from the detector.
- 6. Press the release lever located below the detector. The detector pops out slightly from the DC compartment. Grasp the detector and pull outward to remove the detector from the compartment.
- 7. Note the electrical plug on the back of the detector and the receptacle on the inside of the DC.
- 8. Push the new detector into the opening in the DC and press firmly to ensure the electrical connection is secure.
- 9. Reinstall the cell and reconnect the cell inlet and outlet lines and the cables.
- 10. Turn on the DC power and restart the pump flow.





To avoid injury, wait for the TC to cool down before opening the door or performing any maintenance or service procedures.



Afin d'éviter toute brûlure, laisser refroidir le compartiment thermique avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der TC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

9.25 Replacing Tubing and Fittings

The TC is plumbed with the tubing and fittings listed below.

Tubing Size and Type	Color	P/N	Used For
0.125-mm (0.005-in) ID PEEK	Black	052310	Standard bore system connections
0.25-mm (0.010-in) ID PEEK	Red	052306	Microbore system connections
10-mm (0.39-in) ID polyethylene	Colorless	055075	Waste line

Use double-cone 10-32 ferrule fittings (P/N 043276) and 10-32 bolts (P/N 043275) for all tubing connections. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Dionex Reference Library CD-ROM (P/N 053891).

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 DC Ship Kit (P/N 062614, dual; P/N 063408, single). Refer to the instructions provided with the cutter.

9.26 Setting Up the Column ID System

The TC Ship Kit (P/N 064789) includes two column ID chip cards (P/N 5710. 1500). The optional Column ID Kit (P/N 066353) includes five chip cards.

These are the main steps required to implement the column ID system:

- Specify the column you want to monitor.
- Install a column ID chip card for the column.
- Close the TC door and select the column properties to be monitored.

To select the column to be monitored:

- 1. Launch the Chromeleon Server Configuration program.
- 2. In the timebase, right-click the TC and select **Properties** on the context menu.
- 3. In the TC Properties dialog box, select the **Components** tab to display the **Components** tab page (see Figure 9-42).

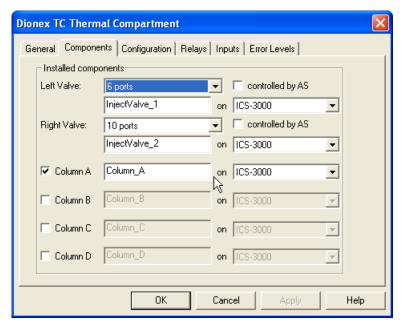


Figure 9-42. TC Properties Dialog Box: Components Tab Page

4. Select the check box for the column to be monitored (**A**, **B**, **C**, or **D**) and click **OK**.

To install a column ID chip card:

1. To attach a column ID chip card to the column, wrap the strap around the column, insert the rivet into one of the holes on the strap, and press to secure the strap to the column (see Figure 9-43).



Figure 9-43. Attaching the Column ID System to the Column

2. Insert the chip card (with the Dionex logo facing up) into the appropriate card reader (A, B, C, or D) along the top of the compartment (see Figure 9-44).

When the chip card is installed correctly, the LED next to the slot is green.



Figure 9-44. Inserting a Column ID Chip Card

3. Install a chip card for any other columns to be monitored.

To select the column properties to be monitored:

- 1. Close the TC door, if it is open. (You cannot select column properties unless the door is closed.)
- 2. On the TC Control panel, click the **Column Details...** button. This opens the Column Details screen.
- 3. For each column, specify the properties the column ID system will monitor. When you finish, click **Close**.

9.27 Rebuilding an Injection Valve

Dionex recommends rebuilding the TC injection valve annually. The Injection Valve Rebuild Kit (P/N 057896, 6-port valve; P/N 061759, 10-port valve) contains all required replacement parts.

- NOTE Substitution of non-Dionex parts may impair valve performance and void the product warranty.
- NOTE If you prefer, you can replace the injection valve "pod," instead of rebuilding the valve. Replacing the pod is easier and faster than rebuilding the valve.
- 1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.
- 2. Press the **POWER** button on the front of the TC to turn off the power.



To avoid injury, wait for the TC to cool down before opening the door or performing any maintenance or service procedures.

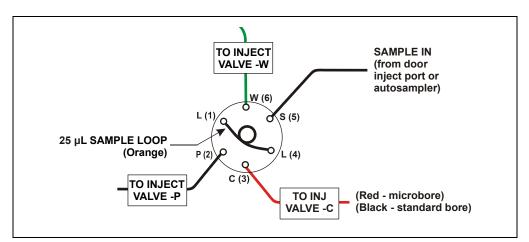


Afin d'éviter toute brûlure, laisser refroidir le compartiment thermique avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der TC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

- 3. Open the TC door.
- 4. Disconnect each liquid line connected to the injection 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 (see <u>Figure 9-45</u>).

Figure 9-45. Injection Valve Plumbing (6-Port Valve)

- 7. Turn on the power to the TC.
- 8. Turn on the pump flow. Check for leaks from the valve. Tighten fittings as required (see Section 9.25).
- 9. Close the TC door.

9.28 Replacing an Injection Valve Pod

This procedure describes how to replace the mechanical part (the "pod") of the TC injection valve.

NOTE If the valve electronics require service, contact Dionex. Electronics-related repair procedures must be performed by Dionex personnel.

NOTE Substitution of non-Dionex parts may impair valve performance and void the product warranty.

1. Turn off the pump flow from the Control panel in Chromeleon or Chromeleon Xpress or press **PUMP FLOW** on the front of the pump.



To avoid injury, wait for the TC to cool down before opening the door or performing any maintenance or service procedures.



Afin d'éviter toute brûlure, laisser refroidir le compartiment thermique avant d'ouvrir la porte ou d'effectuer des opérations de maintenance.



Um Verletzungen zu vermeiden, warten Sie bitte, bis der TC abgekühlt ist, ehe Sie die Tür öffnen bzw. Wartungs oder Servicearbeiten durchführen.

- 2. Open the TC door.
- 3. Disconnect each liquid line connected to the valve.
- 4. Unscrew the black locking ring on the front of the valve (see <u>Figure 9-46</u>) and remove the ring.
- 5. Grasp the front of the valve pod and pull out firmly to remove it from the TC.

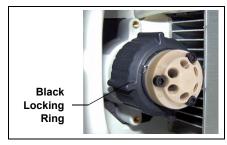


Figure 9-46. Unscrewing the Locking Ring

- 6. Check that the new pod (P/N 061961, 6-port; P/N 061962, 10-port) has the correct number of ports for the injection valve being serviced.
- 7. Align the slots in the new pod with the runner in the valve holder in the TC (see <u>Figure 9-47</u>). 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 <u>Figure 9-47</u>). If necessary, twist the end of the pod to adjust the position of the splines.

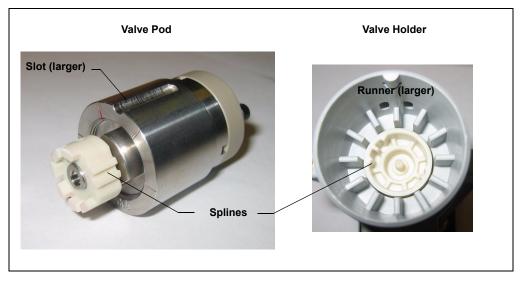


Figure 9-47. Injection Valve Pod and Pod Holder

- 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 pump flow. Check for leaks from the valve. Tighten fittings as required (see Section 9.25).
- 12. Close the TC door.

9.29 Changing the Main Power Fuses

- 1. Press the **POWER** button on the front of the TC for 2 seconds to turn off the power.
- 2. Disconnect the main power cord from both its source and from the TC rear panel.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the TC.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du TC.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des TC.

3. The fuse cartridge is located next to the main power switch (see <u>Figure 9-48</u>). Use a small screwdriver to remove the fuse cartridge.

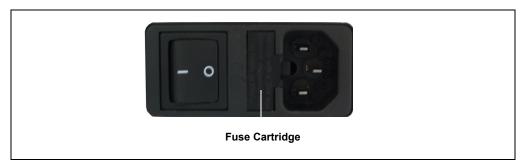


Figure 9-48. TC Fuse Cartridge

- 4. Replace the two fuses with new 2 A IEC 60127-2 time lag fuses (P/N 954776). Dionex recommends always replacing *both* fuses.
- 5. Reinstall the fuse cartridge.
- 6. Reconnect the main power cord and turn on the power.

A • Specifications



DP/SPSpecifications

A.1 Electrical

Main Power 90 to 120 VAC, 47 to 63 Hz **Requirements** 200 to 240 VAC, 47 to 63 Hz

(Auto-sensing power supply; no manual voltage or frequency

adjustment required)

Typical input power: 28 W

Maximum line draw: 1.2 A at 110 VAC

Fuse Two IEC 60127-2 slow-blow fuses (P/N 954773) rated at 2 A,

Requirements 250 V

A.2 Environmental

Operating 4 to 40 °C (40 to 104 °F)

Temperature

Operating 5% to 95% relative humidity (noncondensing)

Humidity

A.3 Physical

Dimensions Height: 40.6 cm (16 in)

Width: 22 cm (8.75 in) Depth: 55.9 cm (22 in)

Minimum required clearance behind the pump: 6 cm (2.5 in)

Weight DP: 25 kg (55 lbs)

SP: 20 kg (45 lbs)

A.4 Hydraulics

Feature	Isocratic Pump	Gradient Pump
Pump	Dual-piston (in series), variable speed, 80 μL per revolution	Dual-piston (in series), variable speed, 80 μL per revolution
Flow Rate Range	0.001 to 10.0 mL/min in increments of 0.001 mL/min	0.001 to 10.0 mL/min in increments of 0.001 mL/min
Flow Rate Accuracy and Reproducibility	±0.1% at 1.0 mL/min (typical)	±0.1% at 1.0 mL/min (typical)
Operating Pressure	0.1 to 35 MPa (15 to 5000 psi)	0.1 to 35 MPa (15 to 5000 psi)
Pressure Ripple	1% (typical)	1% (typical)
Gradient Proportioning Accuracy and Reproducibility	Not applicable	±0.5% at 2.0 mL/min (typical)
Number of Eluents	One	Four
Gradient Mixer Delay Volume	Not applicable	GM-3: $<$ 500 μ L, 3 to 9 mm ID columns GM-4: $<$ 290 μ L, 2 mm ID columns
High Pressure Limit	0 to 35 MPa (0 to 5000 psi) in increments of 0.1 MPa (14 psi); trips instantaneously	0 to 35 MPa (0 to 5000 psi) in increments of 0.1 MPa (14 psi); trips instantaneously
Low Pressure Limit	0 to 34 MPa (0 to 4900 psi) in increments of 0.1 MPa (14 psi)	0 to 34 MPa (0 to 4900 psi) in increments of 0.1 MPa (14 psi)
Vacuum Degassing Module	Built-in single-channel degas chamber (with degassing membranes) with internal volume of 670 µL and dual- stage diaphragm vacuum pump	Built-in four-channel degas chamber (with degassing membranes) with internal volume of 670 µL per channel and dual-stage diaphragm vacuum pump

A.5 Computer Connections

USB Three USB (Universal Serial Bus) ports provide connections to

other ICS-3000 modules

One USB receptacle provides a connection to the PC on which Chromeleon or Chromeleon Xpress software is installed

A.6 I/O Interfaces and Inputs/Outputs

Digital I/O Four programmable relays

Analog output for system pressure

Three digital inputs for Start, Stop, and Hold



A.7 Electrical

Main Power 90 to 264 VAC, 47 to 63 Hz (Auto-sensing power supply; no

Requirements manual voltage or frequency adjustment required)

Typical input power: 18 W

Maximum line draw: 1 A at 110 VAC

Fuse Two IEC 60127-2 slow-blow fuses (P/N 954773) rated at 2 A,

Requirements 250 V

A.8 Environmental

Operating 4 to 40 °C (40 to 104 °F)

Temperature

Operating 5% to 95% relative humidity, noncondensing

Humidity

Operating Pressure Minimum: 14 MPa (2000 psi)

Maximum: 21 MPa (3000 psi)

A.9 Physical

Dimensions Height: 40.6 cm (16 in)

Width: 22 cm (8.75 in) Depth: 55.9 cm (22 in)

Minimum required clearance behind the EG: 5 cm (2 in)

Weight 18 kg (40 lb)

A.10 EluGen Cartridge

Concentration 0.10 to 100 mM

Range Note: The allowable eluent concentration for any particular

application depends on several factors. For more information,

refer to Section 4.7.



A.11 Electrical

Main Power 90 to 265 VAC, 47 to 63 Hz (Auto-sensing power supply; no

Requirements manual voltage or frequency adjustment required)

Typical input power: 190 W

Maximum line draw: 9.2 A at 110 VAC

Fuse Two IEC 60127-2 slow-blow fuses (P/N 954772) rated at 10 A,

Requirements 250 V

A.12 Environmental

Operating 4 to 40 °C (40 to 104 °F)

Temperature (Ambient)

Humidity 5 to 95% relative humidity, noncondensing

(Ambient)

A.13 Physical

Dimensions Height: 40.8 cm (16 in)

Width: 44 cm (17.5 in) Depth: 54.9 cm (21.6 in)

Weight 38 kg (84 lb)

A.14 Lower Compartment Components

Column Capacity 2 sets

Column IDs 1 to 9 mm

Column Lengths Up to 250 mm

Injection Valves One or two 2-position electrically-activated Rheodyne PEEK

valves (6-port or 10-port)

A.15 Temperature Control

A.15.1 Upper Compartment

Temperature 15 to 40 °C in 1 °C increments; thermo-electric control

Control Range If the RCH-1 is off: Minimum temperature 10 °C below ambient

If the RCH-1 is at 80 °C or the CD cell is at 60 °C:

Minimum temperature 5 °C below ambient

Temperature ±0.5 °C

Accuracy

Temperature ±0.2 °C

Stability

Temperature ±0.2 °C

Precision

Heat-up/ $< 30 \text{ min from } 20 \text{ to } 40^{\circ} \text{ C}$

Cooldown Time < 40 min from 40 to 20° C

A.15.2 Lower Compartment (Optional)

Temperature 10 to 70 °C in 1 °C increments;

±0.5 °C

Control Range Minimum temperature \geq (ambient – 15 °C)

Maximum temperature \geq (ambient + 50 °C)

Temperature

Accuracy

Temperature ±0.2 °C

Stability

Temperature ±0.2 °C

Precision

Heat-up/ $< 30 \text{ min from } 20 \text{ to } 50^{\circ} \text{ C}$

Cooldown Time < 30 min from 50 to 20° C

A.16 ICS-3000 Automation Manager

A.16.1 RCH-1 Reaction Coil Heater

Temperature 5 °C above the compartment temperature in 1 °C increments;

Control Range maximum of 80 °C

Temperature ±1 °C

Accuracy

Temperature ±0.2 °C

Stability

Temperature ±0.2 °C

Precision

A.16.2 Valves

High-Pressure Electrically-activated Rheodyne PEEK valve: 2-position, 6-port

or 2-position, 10-port (up to two valves can be installed)

Low-Pressure 2-way or 3-way PEEK valve (up to two valves can be installed)

A.17 ICS-3000 Conductivity Detector

Electronics Microprocessor-controlled digital signal processing

Detection Types Single- or dual-conductivity detection. Dual-detection can be

controlled either simultaneously or independently.

Cell Drive 8 kHz square wave

Linearity 1% at 1000 μS

Resolution 0.10 nS

Signal Sampling Maximum of 100 Hz

Rate

Electronic Noise, S/N ratio = 200,000:1 (Example: $1000 \mu S:5 nS$)

Dry

Electronic Noise, At 23 μ S, noise <0.2 nS

Wet At 1 μ S, noise < 0.1 nS

Filter Rise times from 0 to 10 s

Temperature Factory set at 1.7% per °C; programmable from 0.0 to

Compensation 3.0% per °C

Cell Body PEEK

Cell Electrodes Passivated 316 stainless steel

Active Volume of $< 1.0 \mu L$

Cell

Cell Operating 5 °C above the DC detector compartment temperature;

Temperature maximum of 60 °C

Cell Maximum 3 MPa (500 psi)

Operating

Pressure

Signal Gain 0 to 180 μ S, 0 to 1000 μ S, and 0 to 15,000 μ S

Ranges

Optional Analog Three separate ranges from 0 to 15,000 μ S

Output Full-Scale

Ranges

Optional Analog Output Signal Voltage User-selectable full-scale recorder output of 10, 100, or 1000 mV

A.18 ICS-3000 Electrochemical Detector

Electronics Microprocessor-controlled digital signal processing

Detection Types Single- or dual-electrochemical detection. Dual-detection can be

controlled either simultaneously or independently.

Noise < 1 pA (DC Amperometry)

10 pC (Integrated Amperometry)

Cell Drive -2.00 to 2.00 V in 0.001 V increments

Potential Settings

Optional Analog 50 pC to 200 μC (Integrated Amperometry)

Output Range 5 pA to 74 μA (DC Amperometry)

Cell Working Disposable gold, silver, or platinum

Electrode Non-disposable platinum, gold, silver, or glassy carbon

Cell Counter Titanium

Electrode

Electrode

Cell Reference pH-Ag/AgCl combination

Wetted Materials PEEK, titanium, Kel-F, polyetherimide, EPR, glass, high density

polyethylene (HDPE), ULTEM, Teflon working electrode

materials

Cell Volume at Working Electrode

 $< 0.5 \,\mu L$

Cell Maximum
Operating

Pressure

0.7 MPa (100 psi)



A.19 Electrical

Main Power 90 to 264 VAC, 47 to 63 Hz (Auto-sensing power supply; no

Requirements manual voltage or frequency adjustment required)

Power limit: 150 W maximum

Fuse Two IEC 60127-2 time lag fuses (P/N 954776) rated at 2 A,

Requirements 250 VAC

A.20 Environmental

Operating 10 to 35 °C (50 to 90 °F)

Temperature (Ambient)

Humidity 5% to 80% relative humidity, noncondensing

(Ambient)

A.21 Physical

Dimensions Height: 17.4 cm (6.9 in)

Width: 44.4 cm (17.5 in) Depth: 50.3 cm (19.8 in)

Weight 15.4 kg (34 lb)

A.22 Thermal Compartment

Temperature 5 to 85 °C in 0.1 °C increments (41 to 185 °F in 0.18 °F

Control Range increments); thermo-electric control

Temperature ± 0.5 °C (0.9 °F)

Accuracy

Temperature ± 0.1 °C (0.18 °F)

Stability

Temperature ± 0.1 °C (0.18 °F)

Precision

Column Up to 3 columns; maximum length of 30 cm (11.8 in)

Capacity

Heat-up Time < 20 min from 20 to 50 °C (68 to 122 °F)

Cooldown Time < 20 min from 50 to 20 °C (122 to 68 °F)

Control Provided by Chromeleon or Chromeleon Xpress software; the TC

and the host computer are connected through USB (Universal

Serial Bus)

I/O Interfaces 2 digital inputs, 2 programmable relay outputs

Injection Valve Electrically-activated Rheodyne TitanHP valve: 2-position, 6-port

or 2-position, 10-port (up to two valves can be installed)

Safety Features Humidity sensor and gas sensor

Temperature control and temperature sensors

B • Reordering Information



DP/SP Reordering Information

Part Number	Item
	Primary Pump Head Components
062083	Primary pump head
045722	Inlet check valve assembly (includes inlet nut and 1/8-in cartridge)
045721	Outlet check valve assembly (includes outlet nut and 1/8-in cartridge)
062062	Inlet check valve nut (use only to replace an existing nut of the same type)
062063	Outlet check valve nut (use only to replace an existing nut of the same type)
062648	1/16-in cartridge (use only if the following check valve nuts are currently installed: inlet nut, P/N 062062; outlet nut, P/N 062063)
064946	Main piston seal
040695	O-ring
062064	Seal wash body
063382	Piston seal wash seal
062092	Retainer for piston seal wash seal
062082	Piston
	Secondary Pump Head Components
062079	Secondary pump head
062087	Priming valve
063382	Piston seal wash seal
064946	Main piston seal
040695	O-ring
062064	Seal wash body
062092	Retainer for piston seal wash seal
062082	Piston
063382	Priming valve knob seal

Part Number	Item	
	Piston Seal Wash System	
064155	Piston seal wash reservoir	
063268	PharMed tubing, 0.159-cm (0.0625-in) ID	
	Note: Order 12 in of tubing for replacement of piston seal wash tubing.	
064079	Tygon 2075 tubing, 0.15-cm (0.060-in) ID	
004079	Note: Order 12 in of tubing for replacement of piston seal	
	wash tubing.	
063518	External Seal Wash Kit	
	Eluent Storage and Delivery	
062628	ICS-3000 Eluent Organizer with 4 reservoirs	
062629	ICS-3000 Eluent Organizer with 4 reservoirs	
063291	Eluent reservoir (plastic), 1-liter	
062510	Eluent reservoir (plastic), 2-liter	
063292	Eluent reservoir (plastic), 4-liter	
063493	EO Regulator Kit	
064792	TC/VWD/PDA Regulator Bracket Kit	
062345	Regulator	
045987	Filter, end-line	
044105	Filter, high-pressure inline	
	Miscellaneous	
042126	GM-3 Gradient Mixer	
049135	GM-4 Gradient Mixer	
043276	Ferrule fitting, 10-32 double-cone	
0643275	Fitting bolt, 10-32 (for double-cone ferrule fitting)	
042772	Plug	
054578	Syringe, 10 cc	
954773	Fuse, IEC 60127-2 slow-blow; rated at 2A, 250V	
063246	USB cable, 1 m (3 ft) long	
005.9001A	Cinch connector, 2-pin	
	Maintenance Kits	
061794	DP Annual Preventive Maintenance Kit	
061795	SP Annual Preventive Maintenance Kit	



EG Reordering Information

5 (N)	16	
Part Number	Item	
	Consumables	
058904	EGC II K ₂ CO ₃ EluGen Cartridge	
058900	EGC II KOH EluGen Cartridge	
058906	EGC II LiOH EluGen Cartridge	
058902	EGC II MSA EluGen Cartridge	
058908	EGC II NaOH EluGen Cartridge	
060477	CR-ATC (Anion Continuously Regenerated Trap Column)	
060478	CR-CTC (Cation Continuously Regenerated Trap	
	Column)	
063175	EPM Electrolytic pH Modifier	
061686	EGC-CO ₃ Mixer	
Backpressure Coils		
053762	Backpressure coil, 2.0 mL/min, 500 psi, 4-mm	
053763	Backpressure coil, 2.0 mL/min, 1000 psi, 4-mm	
053764	Backpressure coil, 0.5 mL/min, 500 psi, 2-mm	
053765	Backpressure coil, 0.5 mL/min, 1000 psi, 2-mm	
Miscellaneous		
062137	RFIC Eluent Degasser	
954773	Fuse, IEC 60127-2 slow-blow; rated at 2A, 250V	
063246	USB cable, 1 m (3 ft) long	
043276	Ferrule fitting, 10-32 double-cone	
043275	Fitting bolt, 10-32 (for double-cone ferrule fitting)	
046985	Mini-screwdriver	



DC Reordering Information

Part Number	Item
	Valves and Accessories
061961	High-pressure (injection or switching valve), 6-port
061962	High-pressure (injection or switching valve), 10-port
061971	Low-pressure valve, 3-way
061745	Low-pressure valve, 2-way
042949	Sample loop, 10 μL
042857	Sample loop, 25 μL
044105	Filter, high-pressure inline (for sample filtering)
024305	Luer adapter fitting, 1/4-28 (for manual injections)
016388	Syringe, 1 cc (for manual injections)
057896	Valve rebuild kit, 6-port
061759	Valve rebuild kit, 10-port
	Electrochemical Detector and Accessories
061718	ICS-3000 Electrochemical Detector (ED) (without cell)
061756	ED titanium cell with reference electrode (no working
	electrode)
061879	pH-Ag/AgCl reference electrode
014067	pH-Ag/AgCl reference electrode O-ring
061749	ED gold working electrode, with gasket and polishing kit
061751	ED platinum working electrode, with gasket and polishing kit
061753	ED glassy carbon working electrode, with gasket and polishing kit
061755	ED silver working electrode, with gasket and polishing kit
063722	ED AAA gold working electrode, with gasket and polishing kit
060082	AAA-Direct disposable gold working electrodes (6 electrodes, with 2 gaskets)
060140	AAA-Direct disposable gold working electrodes (4 packages of 6 electrodes, with 8 gaskets)
060139	Carbohydrate disposable gold working electrodes (6 electrodes, with 2 gaskets)
060216	Carbohydrate disposable gold working electrodes (4 packages of 6 electrodes, with 8 gaskets)

Part Number	Item
064440	Disposable platinum working electrodes
	(6 electrodes, with 2 gaskets)
063003	Disposable silver working electrodes
	(6 electrodes, with 2 gaskets)
060141	Gaskets for disposable working electrodes (4 gaskets)
045972	Gasket for non-disposable working electrode
062158	ED cell polypropylene support block (for use with disposable electrodes)
036313	Polishing kit
036319	Coarse polishing compound
036318	Fine polishing compound
036321	Polishing pads
	Conductivity Detector and Accessories
061716	ICS-3000 Conductivity Detector
056116	AAES Anion Atlas Electrolytic Suppressor
056118	CAES Cation Atlas Electrolytic Suppressor
064555	ASRS 300 2-mm Anion Self-Regenerating Suppressor
064556	CSRS 300 4-mm Cation Self-Regenerating Suppressor
064557	CSRS 300 2-mm Cation Self-Regenerating Suppressor
045460	Suppressor gas separator waste tube
045877	Backpressure coil for 4 mm suppressor
045878	Backpressure coil for 2 mm suppressor
	Automation Manager
061738	ICS-3000 Automation Manager, two 10-port high-pressure valves, two low-pressure 3-way valves
061736	ICS-3000 Automation Manager, one 10-port high-pressure valve, one low-pressure 3-way valve
061740	ICS-3000 Automation Manager, one 6-port high-pressure
	valve, one low-pressure 3-way valve
061734	ICS-3000 Automation Manager, tray with no valves
061746	RCH-1 Reaction Coil Heater
062561	Temperature stabilizer, standard bore, 0.25-mm (0.010-in) ID
062562	Temperature stabilizer, microbore, 0.125-mm (0.005-in) ID

Part Number	Item	
Analog Output/Relay/TTL		
062201	I/O option	
923686	Connector plug, 12-position	
043598	Twisted pair of wires	
Miscellaneous		
954772	Fuse, IEC 60127-2 slow-blow; rated at 10A, 250V	
960777	USB cable, 1.8 m (6 ft) long	
062437	Leak sensor	
043276	Ferrule fitting, 10-32 double-cone	
043275	Fitting bolt, 10-32 (for double-cone ferrule fitting)	
044105	Filter, high-pressure inline	
	Maintenance Kit	
061796	DC/TC Annual Preventive Maintenance Kit	



TC Reordering Information

Part Number	Item
	Valves and Accessories
057896	Injection Valve Rebuild Kit for 6-port valve (includes valve seal, hexagon wrench, and instructions)
061759	Injection Valve Rebuild Kit for 10-port valve (includes valve seal, hexagon wrench, and instructions)
061961	Injection valve head (including seal) for 2-position, 6-port valve
061962	Injection valve head (including seal) for 2-position, 10-port valve
042857	Sample loop, 25 μ L
	Consumables
6710.1505	Column ID Kit (includes five column ID chip cards)
064786	Column Clips Kit (includes six column clips)
064548	Temperature stabilizer, standard bore, 0.25-mm (0.010-in) ID
064650	Temperature stabilizer, microbore, 0.125-mm (0.005-in) ID
	Miscellaneous
954776	Fuse, IEC 60127-2 time lag, rated at 2 A (two fuses required)
063246	USB cable, 1 m (3 ft)
6000.1004	6-pin mini-DIN signal cable, 5 m (16.4 ft)
064792	TC/VWD/PDA Regulator Bracket Kit
	Maintenance Kit
061796	DC/TC Annual Preventive Maintenance Kit

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