

Dionex ICS-1100 Ion Chromatography System Operator's Manual

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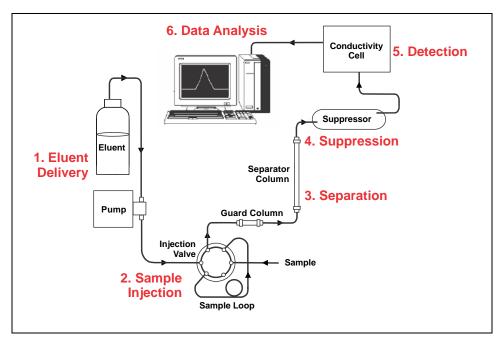
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1.1 Introduction to Ion Chromatography (IC)

The Thermo Scientific Dionex[™] ICS-1100 Ion Chromatography System (Dionex ICS-1100) performs ion analyses using suppressed or non-suppressed conductivity detection. An ion chromatography system typically consists of a liquid eluent, a high-pressure pump, a sample injector, a guard and separator column, a chemical suppressor, a conductivity cell, and a data collection system.

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



A typical IC analysis consists of six stages (see <u>Figure 1-1</u>).

Figure 1-1. Ion Analysis Process

1. Eluent Delivery

• Eluent, a liquid that helps to separate the sample ions, carries the sample through the ion chromatography system. The Dionex ICS-1100 is an isocratic delivery system. This means that the eluent composition and concentration remain constant throughout the run.

2. Sample Injection

- The liquid sample is loaded into a sample loop either manually or automatically (if an automated sampler is installed). When triggered, the Dionex ICS-1100 injects the sample into the eluent stream.
- The pump pushes the eluent and sample through the guard and separator columns (chemically-inert tubes packed with a polymeric resin). The guard column removes contaminants that might poison the separator column.

3. Separation

 As the eluent and sample are pumped through the separator column, the sample ions are separated. In the Dionex ICS-1100, the mode of separation is called ion exchange. This is based on the premise that different sample ions migrate through the IC column at different rates, depending upon their interactions with the ion exchange sites.

4. Suppression

 After the eluent and sample ions leave the column, they flow through a suppressor that selectively enhances detection of the sample ions while suppressing the conductivity of the eluent.

5. Detection

 A conductivity cell measures the electrical conductance of the sample ions as they emerge from the suppressor and produces a signal based on a chemical or physical property of the analyte.

6. Data Analysis

- The conductivity cell transmits the signal to a data collection system.
- The data collection system (for the Dionex ICS-1100, this is the Chromeleon [™] Chromatography Management System) identifies the ions based on retention time, and quantifies each analyte by integrating the peak area or peak height. The data is quantitated by comparing the sample peaks in a chromatogram to those produced from a standard solution. The results are displayed as a chromatogram and the concentrations of ionic analytes can be automatically determined and tabulated.

1.2 Overview of the Dionex ICS-1100

The Dionex ICS-1100 is an integrated ion chromatography system containing a pump, injection valve, and conductivity detector. Other system components, including a guard column, separator column, and suppressor vary, depending on the analyses to be performed.

The Dionex ICS-1100 can optionally be configured with a column heater for temperature control of the column. An optional second high-pressure valve (6-port or 10-port) can be installed for sample preparation applications.

The Dionex ICS-1100 contains a built-in ER Controller and pre-plumbed tubing for operation in the RFIC-ER (Reagent-Free In Chromatography with Eluent Regeneration) mode. RFIC-ER technology uses the suppressor to regenerate returning eluent, allowing a single batch of eluent to be used for up to four weeks.

Dionex ICS-1100 operation is controlled remotely by a personal computer running Windows® Vista or Windows XP and Chromeleon software (version 6.80 SR6a or later). Chromeleon also provides data acquisition and data processing functions.

For communication between the Dionex ICS-1100 and Chromeleon, the Dionex ICS-1100 is connected to a USB (Universal Serial Bus) port on the computer or a USB hub. For details, see the Dionex ICS-1100 installation instructions. Also refer to *Installing the Chromeleon Chromatography Management System with a Dionex Ion Chromatograph (IC)* (Document No. 031883).

1.3 **About This Manual**

The electronic version (i.e., PDF file) of this 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	Introduces ion analysis and the Dionex ICS-1100; explains the conventions used in this manual, including safety-related information.	
Chapter 2 Features	Provides an overview of Dionex ICS-1100 operating features and system components; introduces the Chromeleon user interface.	
Chapter 3 Operation and Maintenance	Provides operating instructions and describes routine preventive maintenance procedures.	
Chapter 4 Troubleshooting	Lists problems, and presents step-by-step procedures for how to isolate and eliminate the cause of each problem.	
Chapter 5 Service	Provides step-by-step instructions for routine service and parts replacement procedures that the user can perform.	
Appendix A Specifications	Lists the Dionex ICS-1100 specifications and installation site requirements.	
Appendix B TTL and Relay Control	Describes the Dionex ICS-1100 TTL and relay control features.	
Appendix C Reordering Information	Lists spare parts for the Dionex ICS-1100.	
Appendix D FAQ	Provides answers to frequently asked questions about Dionex ICS-1100 operation.	

Appendix E Glossary

Defines terms commonly used in ion analysis.

1.3.1 Safety Messages and Notes

This manual contains warnings and precautionary statements that, when properly followed, can prevent personal injury and/or damage to the instrument. 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 Dionex ICS-1100 was manufactured by Thermo Fisher Scientific at the following location: 527 Lakeside Drive, Sunnyvale, CA 94088-3603 U.S.A. The Dionex ICS-1100 is designed for IC (ion chromatography) applications and should not be used for any other purpose. Operation of a Dionex ICS-1100 in a manner not specified by Thermo Fisher Scientific may result in personal injury. If there is a question regarding appropriate usage, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

1.4.1 Safety Labels

The TUV T-Mark and cTUVus Mark safety labels and the CE Mark label on the system indicate that it is in compliance with the following standards:

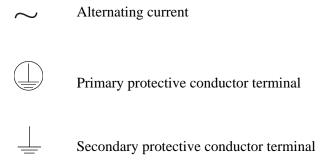
EMC Susceptibility and Emissions

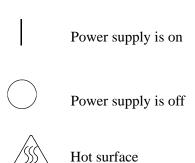
• EN 61326-1:2006

Safety

- CAN/CSA-C22.2 61010-1:2004
- UL 61010-1:2004
- EN 61010-1:2001

The symbols below appear on the Dionex ICS-1100 or on labels affixed to the Dionex ICS-1100.







Indicates a potential hazard. Refer to the operator's manual for an explanation of the hazard and how to proceed.

Dionex ICS-1100 Ion Chromatography System

This chapter describes key Dionex ICS-1100 features and introduces the Chromeleon user interface.

2.1 Operating Features

2.1.1 Front Panel

Figure 2-1 illustrates the front panel of the Dionex ICS-1100.

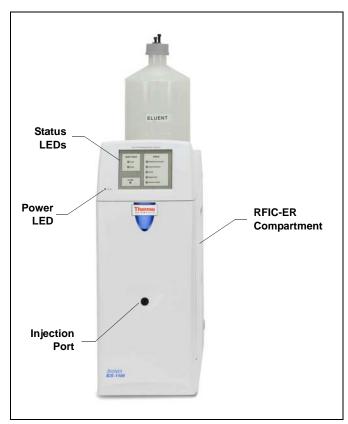


Figure 2-1. Dionex ICS-1100 Front Panel

Injection Port

The sample to be analyzed can be injected manually into the injection port, using a syringe. For automated sample injection, the Dionex ICS-1100 must be connected to an autosampler. For more information about sample injection, see Section 3.10.

LEDs

The status LEDs (see Figure 2-2) indicate the status of various system functions. See Table 2-1 for a description of each LED's function. The power LED indicates whether the Dionex ICS-1100 power is on.

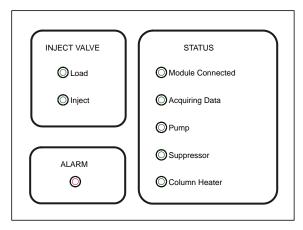


Figure 2-2. Status LEDs

LED Label	If On (Green)	If Flashing		
Load	Injection valve is in Load position.	Valve error.		
Inject	Injection valve is in Inject position.	Valve error.		
Alarm	This LED does not have an "on" (green) state.	Error detected. Check the Chromeleon Audit Trail for the cause.		
Module Connected	Dionex ICS-1100 is connected to a Chromeleon timebase.	Does not flash.		
Acquiring Data	Sequence or manual data acquisition is in progress.	Sequence has stopped due to an error.		
Pump	Pump is on.	High or low pressure limit is exceeded. The pump is turned off.		
Suppressor	Suppressor is on and current is being applied to it.	Continuity check failed or suppressor is over the voltage, current, or power limit. The suppressor is turned off.		
Column Heater	Column heater is at set temperature.	Column heater is transitioning to a new temperature.		
Table 2-1. Dionex ICS-1100 Status LED States				

2.1.2 Top Cover

Figure 2-3 illustrates the top cover of the Dionex ICS-1100.

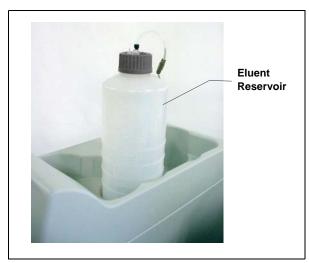


Figure 2-3. Dionex ICS-1100 (Top View)

Reservoir Storage

The Dionex ICS-1100 top cover has room for up to three 2-L plastic reservoirs (P/N 044129) *or* one 4-L plastic reservoir (P/N 039164).

Tubing Chase

The tubing chase under the top cover routes tubing to the front of the Dionex ICS-1100.

2.1.3 Component Panel

<u>Figure 2-4</u> shows the user-accessible components installed on the component panel behind the Dionex ICS-1100 front door.

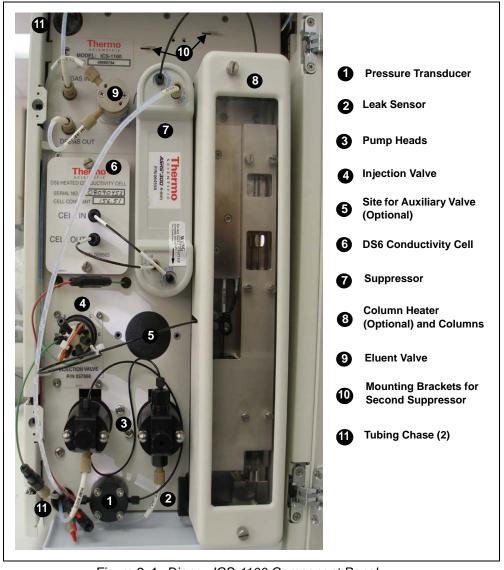


Figure 2-4. Dionex ICS-1100 Component Panel

Pressure Transducer

The pressure transducer measures the system backpressure.

Leak Sensor

The leak sensor is installed in the drip tray at the bottom of the component panel. If liquid accumulates in the tray, an error message is logged in the Chromeleon Audit Trail and the **Alarm** LED flashes.

Pump Heads

The Dionex ICS-1100 includes a dual-piston serial pump. The flow rate can be set to 0.00 mL/min or to between 0.05 and 5.00 mL/min. However, for optimum performance, set the flow rate to between 0.40 and 2.00 mL/min. Setting the flow rate to 0.00 mL/min turns off the pump. See Section 2.4.3 for details about the pump.

Injection Valve

The injection valve is a six-port, electrically-activated Rheodyne valve. A 25-µL sample loop (P/N 042857) is installed on the valve at the factory. See Section 2.4.4 for details about valve operation.

Auxiliary Valve (Optional)

The auxiliary valve is a two-position, electrically-activated Rheodyne valve (6-Port Valve Kit, P/N 069472; 10-Port Valve Kit, P/N 069473). See Section 2.4.4 for details about valve operation.

DS6 Heated Conductivity Cell

The flow-through conductivity cell measures the electrical conductance of analyte ions as they pass through the cell. A heat exchanger inside the cell regulates the temperature, which can be set to between 30 and 55 °C. For optimum performance, set the temperature to at least 7 °C above the ambient temperature and 5 °C above the column oven temperature. See Section 2.4.8 for details about the cell.

Suppressor

The suppressor reduces the eluent conductivity and enhances the conductivity of the sample ions, thereby increasing detection sensitivity. Either a Dionex AESTM Atlas Electrolytic Suppressor, Dionex SRSTM Self-Regenerating Suppressor, or Dionex MMSTM MicroMembraneTM Suppressor can be used with the Dionex ICS-1100. See Section 2.4.7 for details about the suppressor.

Separator and Guard Columns

Both the separator and guard columns are packed with resin and perform the separation of the sample ions. The main function of the guard column is to trap contaminants and remove particulates that might damage the separator column.

Column Heater (Optional)

The column heater controls the temperature of the separator and guard columns. The temperature can be set to between 30 and 60 °C; however, it must be set to at least 5 °C above the ambient temperature. See Section 2.4.6 for details about the column heater.

Eluent Valve

The eluent valve controls the flow from the eluent reservoir. The eluent valve opens automatically when the pump is started and closes when the pump is turned off.

Tubing Chases

The upper tubing chase routes tubing from the top cover to the component panel. The lower tubing chase routes tubing from the component panel, through the interior of the Dionex ICS-1100, to the rear panel. It also routes tubing from the RFIC-ER compartment, through the interior of the Dionex ICS-1100, to the component panel.

2.1.4 Right-Side Panel

<u>Figure 2-5</u> illustrates the RFIC-ER compartment on the right side of the Dionex ICS-1100.



Figure 2-5. RFIC-ER Compartment (Cover Removed)

The compartment houses the ER Controller (ERC 10) and various columns required for operation in the RFIC-ER mode. The ERC 10 electronics monitor the volume of electrolysis gases (such as hydrogen and oxygen) in the eluent stream. If the predefined safety limit is exceeded, the ERC 10 shuts down the pump to prevent the buildup of gases in the eluent bottle.

If you plan to operate in the RFIC-ER mode, you must order one of these kits:

- RFIC-ER Anion Startup Kit, ICS-1100/ICS-1600 (P/N 069570)
- RFIC-ER Cation Startup Kit, ICS-1100/ICS-1600 (P/N 069569)

Each Startup Kit includes one Installation Kit and one Consumables Kit. Together, these kits contain additional items (purification columns, a

special eluent bottle, etc.) required for RFIC-ER operation. For more information, refer to the manual shipped with the Installation Kit.

2.1.5 Rear Panel

Figure 2-6 illustrates the Dionex ICS-1100 rear panel.

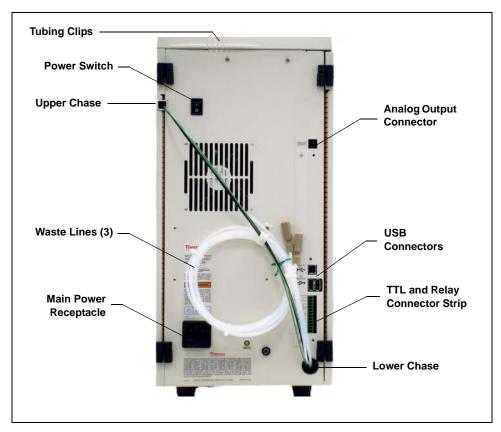


Figure 2-6. Dionex ICS-1100 Rear Panel

Analog Output Connector

The analog output connector outputs conductivity data (as a 0 to 1 V signal) to an integrator or recording device. For connection and setup information, refer to the Dionex ICS-1100 installation instructions.

USB Connectors

A USB receptacle is provided to allow connection to the Chromeleon computer. Two USB ports are provided for connecting to other USB devices. For connection instructions, refer to the Dionex ICS-1100 installation instructions.

TTL and Relay Connector

The TTL and Relay connector strip provides two TTL outputs, two relay outputs, and four TTL inputs. The outputs can be used to control functions in other TTL- or relay-controllable devices. The inputs can be used to switch the injection valve position, turn on the pump, perform an autozero command, and send an event mark to the analog output. See Section B.1 for connection instructions.

Tubing Chases

The upper tubing chase routes tubing from the RFIC-ER compartment to the Dionex ICS-1100 rear panel. The lower chase routes tubing from the rear panel to the component panel.

Tubing Clips

The tubing clips hold tubing routed from the top cover in place.

Power Switch

The power switch provides on/off control of power to the Dionex ICS-1100.

Main Power Receptacle

The power supply cord plugs into the AC power receptacle.







The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the Dionex ICS-1100 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.

2.2 Flow Schematics

The following figures illustrate the liquid flow path through a Dionex ICS-1100 when using suppression in autorecycle mode. Two configurations are shown:

- <u>Figure 2-7</u> illustrates the liquid flow path through components when performing conventional ion chromatography.
- <u>Figure 2-8</u> illustrates the liquid flow path through components when operating in the RFIC-ER mode.

Flow Description for Standard Configuration

- Eluent from the eluent reservoir **1** flows first through the pump degas assembly (if it is installed) and then through the eluent valve **2** to the pump **3**. The eluent is then pushed through the pressure transducer **4**, which measures the system pressure.
- From there, the eluent is pushed through a pulse damper **3**, which smooths minor pressure variations from the pump to minimize baseline noise. The eluent then flows into the injection valve **3**.
- After sample is loaded into the sample loop **3** and the injection valve is toggled to the Inject position, eluent passes through the sample loop.
- If the optional column heater is installed, the eluent/sample mixture is pumped through the heat exchanger ③, which heats the mixture to the column heater temperature.
- The mixture then goes to the guard and separator columns $\mathbf{9}$ and through the suppressor $\mathbf{0}$.
- From the suppressor, the mixture flows through the cell **①**, where the analytes are detected. A digital signal is sent to Chromeleon software. Analog output can be collected simultaneously.
- 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 ②.

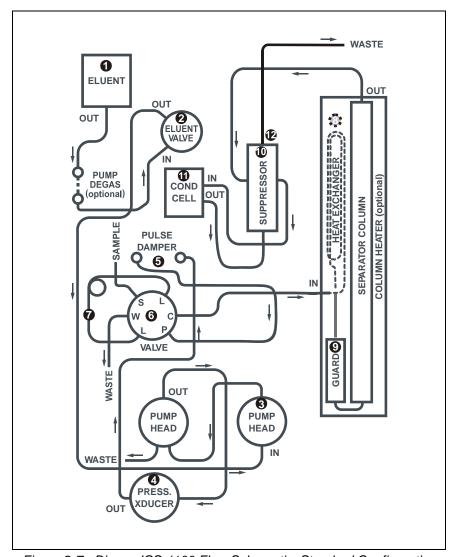


Figure 2-7. Dionex ICS-1100 Flow Schematic: Standard Configuration

Flow Description for RFIC-ER Mode

- Eluent from the eluent reservoir ① flows first through the pump degas assembly (if it is installed) and then through the eluent valve ② to the pump ③. The eluent is then pushed through the pressure transducer ④, which measures the system pressure.
- From there, the eluent is pushed through a pulse damper **3**, which smooths minor pressure variations from the pump to minimize baseline noise. The eluent then flows through the ER1 column **3** and into the injection valve **7**.
- After sample is loaded into the sample loop ③ and the injection valve is toggled to the Inject position, eluent passes through the sample loop.
- If the optional column heater is installed, the eluent/sample mixture is pumped through the heat exchanger **9**, which heats the mixture to the column heater temperature.
- The mixture then goes to the guard and separator columns **①** and through the suppressor **①**.
- From the suppressor, the mixture flows through the cell •, where the analytes are detected. A digital signal is sent to Chromeleon software. Analog output can be collected simultaneously.
- Finally, the mixture flows out of the cell, through the ER2 column ③, and is then recycled back into the suppressor.
- From the suppressor, flow moves to the ER3a and ER3b columns, which remove contaminants from the eluent.
- The ERC 10 monitors the eluent for gas and shuts down the pump if the gas exceeds a pre-defined limit.
- Finally, the output from the ERC 10 is recycled back to the eluent bottle.

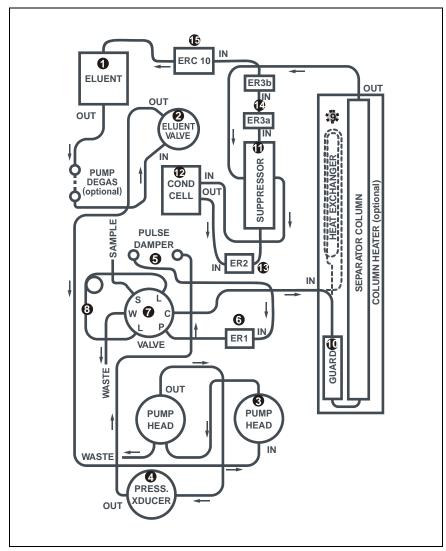


Figure 2-8. Dionex ICS-1100 Flow Schematic: RFIC-ER Mode

2.3 Chromeleon and Chromeleon Xpress

The Dionex ICS-1100 is controlled by a PC configured with Chromeleon Chromatography Management System or Chromeleon Xpress. Chromeleon Chromatography Management System provides complete instrument control, data acquisition, and data management. Chromeleon Xpress provides real-time control and monitoring of instruments, but does not include data management capabilities.

2.3.1 The Panel Tabset

The Chromeleon and Chromeleon Xpress *panel tabset* provides a centralized location for controlling system functions. A panel tabset for a Dionex ICS-1100 system typically includes the following Control panels:

 A Dionex ICS-1100 Control panel (see <u>Figure 2-9</u>) provides access to Dionex ICS-1100 functions. The label on the tab for this panel is the name of the timebase in which the Dionex ICS-1100 is configured.

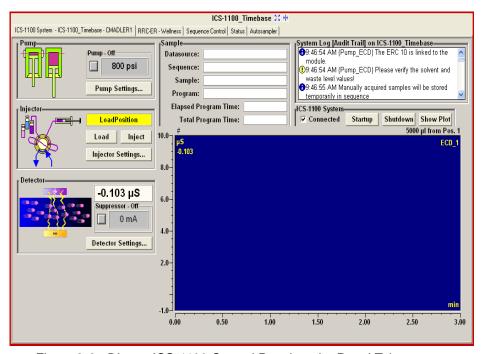
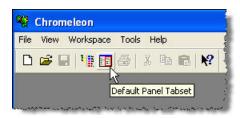


Figure 2-9. Dionex ICS-1100 Control Panel on the Panel Tabset

- A Sequence Control panel lets you define and run sequences (groups of sample injections to be analyzed in the order in which they are listed).
- A **Status** panel shows the overall system status.
- An Autosampler panel (included if the Dionex ICS-1100 is connected to a Dionex AS, Dionex AS-AP, Dionex AS-DV, or Dionex AS-HV Autosampler) provides access to autosampler functions.

To open the panel tabset, use one of the methods below:

 If Chromeleon is installed, start Chromeleon and click the **Default Panel Tabset** toolbar button, or select **View > Default Panel Tabset**.



 If Chromeleon Xpress is installed, start the application; this automatically displays the Dionex ICS-1100 panel tabset.

2.3.2 Software Control Modes

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

- With direct control, you select operating parameters and commands from the Control panels. Direct control commands are executed as soon as they are entered.
- With programmed control, you create a list of control commands to be executed in chronological order. Programs can be created automatically (with the help of a software wizard).

2.3.3 System Wellness

System Wellness monitors the overall "health" of a chromatographic system. It provides built-in diagnostic and calibration features that help prevent unscheduled system shutdowns and assure reliable operation of system devices. For details about System Wellness, see Section 5.1.

2.4 System Component Details

This section provides details about Dionex ICS-1100 system components, including the vacuum degas assembly (optional), RFIC-ER components, eluent valve, pump, injection valve, column heater (optional), suppressor, and conductivity cell.

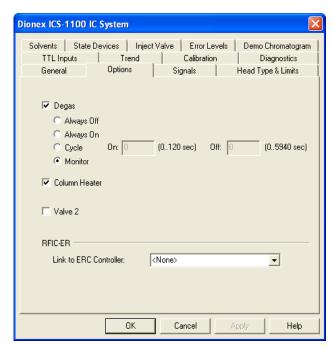
2.4.1 Vacuum Degas Assembly (Optional)

The vacuum degas assembly provides online eluent degassing at the time and duration specified by the user. The assembly, which must be installed in the Dionex ICS-1100 at the factory, consists of:

- A single-channel degas chamber (with degas membranes) with internal capacity of 17 mL
- A dual-stage diaphragm vacuum pump
- A solenoid valve
- An on-board vacuum sensor
- The electronics required to operate the vacuum pump
- Tubing, fittings, and other accessories

By default, the Dionex ICS-1100 monitors the degas pressure reading and turns the degas pump on and off as needed. Different degas operating modes can be selected from Chromeleon.

To select the degas operating options, open the Chromeleon Server Configuration program, right-click the Dionex ICS-1100 device in the timebase, and select **Properties**.



Select the **Options** tab (see Figure 2-10).

Figure 2-10. Server Configuration Properties: Degas Options

Degas Options

- Always Off: The degas pump is always off.
- Always On: The degas pump is always on. This setting is for test purposes by a Thermo Fisher Scientific Service Representative. Do not use this setting for routine operation.
- Cycle: The degas pump cycles on and off according to the times specified in the Cycle On and Off fields. Cycle On specifies for how long the degas pump runs during a cycle. Cycle Off specifies the time between cycles.
- **Monitor**: (default mode) The Dionex ICS-1100 monitors the degas pressure reading and turns the degas pump on and off as required.

2.4.2 Eluent Valve



the eluent reservoir. The valve opens automatically when the pump is started and closes when the pump is turned off. The valve can also be opened and closed manually from the Chromeleon Control panel (see Section 2.3.1). This lets you perform service procedures on pump components without eluent leaks occurring.

The eluent valve controls the flow from

Figure 2-11. Eluent Valve

2.4.3 Pump

The Dionex ICS-1100 pump is a microprocessor-based isocratic eluent delivery system. Its variable speed, dual-piston series design ensures pulse-free pumping for the most demanding applications.

Primary Pump Head

The primary pump head pumps eluent into the secondary head (see <u>Figure 2-12</u>). The check valves, which prevent reverse flow through the pump, are located on the bottom (inlet) and top (outlet) of the primary pump head. The priming valve is on the front of the pump head.

To open the priming valve, turn the knob one-quarter to one-half turn counterclockwise. When the priming valve is open, liquid can flow into and out of the primary pump head via the port in the front of the valve.

NOTE The priming valve must be open when the pump is being primed with a syringe or with isopropyl alcohol. For detailed priming instructions, see Section 5.16.

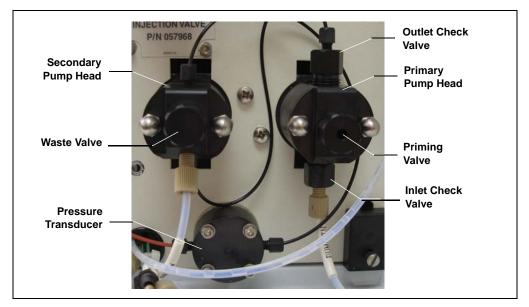


Figure 2-12. Dionex ICS-1100 Pump Components

Secondary Pump Head

The secondary pump head delivers eluent to the remainder of the chromatography system (the injection valve, column, and detector). The waste valve is located on the front of the secondary pump head (see Figure 2-12).

To open the waste valve, turn the knob one-quarter to one-half turn counterclockwise. When the waste valve is in the open position, all pump flow is directed to waste.

NOTE The waste valve must be open when the pump is being primed using the Prime button. For detailed priming instructions, see Section 5.16.

Pressure Transducer

Flow exiting the secondary pump head is directed to the pressure transducer (see Figure 2-12), which measures the system pressure.

Pressure readings indicate that the pumping system is delivering smooth, accurate flow. Pressure readings can be monitored from the Chromeleon Control panel.

The system pressure should remain consistent (no more than a 3% difference from one pressure reading to the next). High and low pressure limits can be used to stop the pump flow if a limit is exceeded. The pressure limits can be set from Chromeleon (in the Server Configuration or in the control program). See Section 4.2 for troubleshooting information if a pressure limit is exceeded.

Pulse Damper

Flow output from the pressure transducer continues to the pulse damper, which smooths minor pressure variations. From there, flow is directed to the injection valve and then to the remainder of the chromatography system.

Piston Seal Wash

The pump includes a piston seal wash assembly that can be set up to continuously rinse the back of the piston seals to remove salt crystals and prolong the life of the seals. To use this feature, an external water source must be connected. For connection instructions, refer to the Dionex ICS-1100 installation instructions.

For continued protection of the pump, replace the piston rinse seals (see Section 5.7) and O-rings in the seal wash assembly every 6 months, or whenever you replace the main piston seals for the Dionex ICS-1100 pump.

2.4.4 Injection Valve

The injection valve (P/N 057968) is a six-port, electrically-activated valve. A 25- μ L sample loop (P/N 042857) is installed on the valve at the factory.

The valve has two operating positions: Load and Inject (see Figure 2-13).

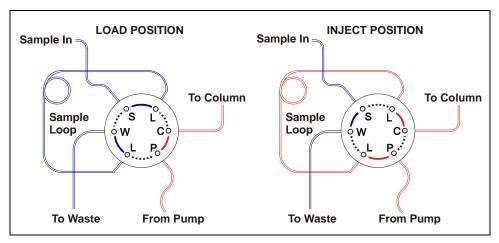


Figure 2-13. Injection Valve Flow Schematics

Eluent flows through either the Load or Inject path, depending on the valve position.

- 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 automated sampler 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 3.10 describes how to inject samples.

<u>Figure 2-14</u> shows the injection valve connections. The injection valve is plumbed at the factory with all tubing and fittings for connection to the pump, injection port, column, and waste. A 25- μ L PEEK sample loop (P/N 042857) is installed between ports L (1) and L (4). Thermo Fisher Scientific offers sample loops in various sizes. If necessary, the pre-installed 25- μ L loop can be replaced with a loop that has a different sample injection volume.

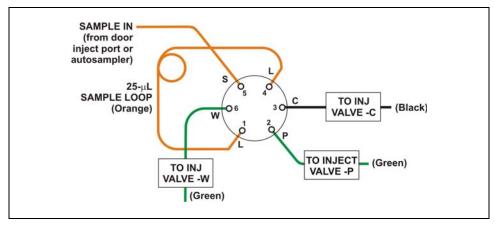


Figure 2-14. Injection Valve Plumbing

2.4.5 Auxiliary Valve (Optional)

The auxiliary valve is a high-pressure Rheodyne valve. The electrically-activated, 2-position PEEK valve is offered in both 6-port and 10-port models (6-Port Valve Kit, P/N 069472; 10-Port Valve Kit, P/N 069473). The auxiliary valve enables a variety of sample preparation activities, including:

- Online filtration
- Matrix elimination (for example, the removal of high backgrounds of chloride or organic material)
- Concentrator-based techniques
- Conditional injections (large loop/small loop applications where the data system monitors sample concentration and reinjects the sample, using the smaller loop, if the concentration is too high)
- AutoNeutralization TM
- Matrix diversion prior to MS (mass spectrometry) detection

For more information, refer to *Installing the ICS-1100/ICS-1600/ICS-2100 Auxiliary Valve* (Document No. 065288). The manual is provided in the Auxiliary Valve Kit.

2.4.6 Column Heater (Optional)



The column heater provides temperature control for the separator and guard column.

The heater temperature can be set to between 30 °C and 60 °C. However, the set temperature must be at least 5 °C above the ambient temperature. A thermistor mounted in the heater block monitors the temperature. Setting the temperature to 0 °C turns off the column heater.

If the temperature exceeds 65 °C, the column heater is shut off and the error message "Column heater exceeds safe temperature." is displayed in the Chromeleon Audit Trail. See Section 4.2 for troubleshooting information.

The column heater can either be installed at the factory or ordered separately (P/N 069564) and installed on-site. For best results with 2-mm columns, a microbore heat exchanger (P/N 060943) should be installed in the column heater (see Section 5.13). The tubing connections between the injection valve and column heat exchanger, guard column and separator column, and separator column to detector cell should all use red 0.125-mm, 0.005-in ID PEEK tubing (P/N 044221), rather than the standard black tubing.

Figure 2-15. Column Heater

2.4.7 Suppressor

The suppressor reduces the eluent conductivity and enhances the conductivity of the sample ions, thereby increasing detection sensitivity. Either a Dionex Atlas Electrolytic Suppressor, Dionex Self-Regenerating Suppressor, or Dionex MicroMembrane Suppressor can be used with the Dionex ICS-1100.

For details about any of the suppressors or for information about selecting a suppressor for your application, refer to the suppressor manuals. The manuals are on the Thermo Scientific Reference Library DVD (P/N 053891).

2.4.8 DS6 Heated Conductivity Cell

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

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.

In ion analysis, the effect of temperature variation is minimized by suppressing eluent conductivity. To further reduce the effect of temperature variation, a heater inside the cell regulates the temperature. The cell heater can be set to between 30 °C and 55 °C. The set temperature must be at least 7 °C above the ambient temperature. Setting the cell temperature to 0 °C turns off the cell heater.

Temperature Compensation

Built-in preset temperature compensation of 1.7% per °C helps minimize changes in the baseline or in peak heights when the operating temperature is different from the temperature at which the cell was calibrated.

DS6 Heated Conductivity Cell Components

The cell front cover provides **CELL IN** and **CELL OUT** fittings for connecting the cell to the suppressor (see <u>Figure 2-4</u>). The remaining cell components are mounted behind the component panel. To replace, the cell remove the screws on the cell front cover and pull the entire cell assembly out through the component panel. See <u>Section 5.10</u> for cell replacement instructions.

2.4.9 ER Controller

The ER Controller (ERC 10) is installed in the RFIC-ER compartment at the factory. During operation in the RFIC-ER mode, the ERC 10 electronics monitor the volume of electrolysis gases (such as hydrogen and oxygen) in the eluent stream. If the predefined safety limit is exceeded, the ERC 10 shuts down the Dionex ICS-1100 pump to prevent the buildup of gases in the eluent bottle.

Dionex ICS-1100 Ion Chromatography System

3 • Operation and Maintenance

This chapter describes routine operating and maintenance procedures for the Dionex ICS-1100.

The Dionex ICS-1100 is designed for IC (ion chromatography) applications and should not be used for any other purpose. Operation of the Dionex ICS-1100 in a manner not specified by Thermo Fisher Scientific may result in personal injury.

3.1 Operation Overview

Figure 3-1 illustrates the basic steps for routine operation of the Dionex ICS-1100.

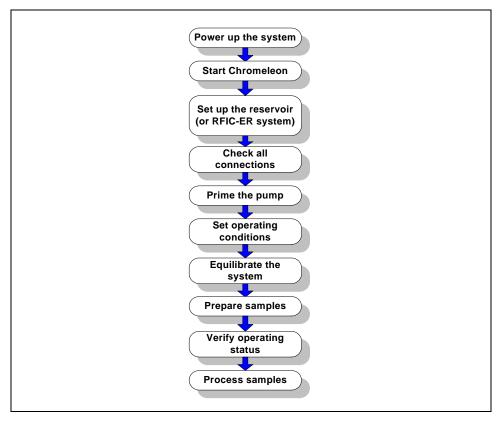


Figure 3-1. Operation Flow Chart

Sample Processing Overview

Samples can be run manually (one at a time), or they can be grouped and run automatically in batches. <u>Figure 3-2</u> shows the typical steps for manual and batch sample processing.

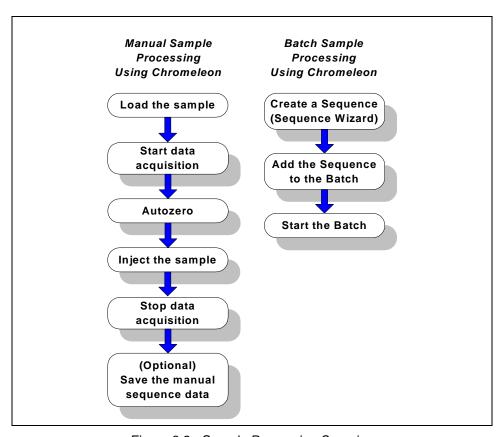
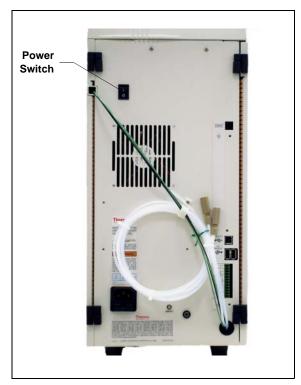


Figure 3-2. Sample Processing Overview

3.2 Turning On the System Power



Press the power switch on the Dionex ICS-1100 rear panel (see Figure 3-3) to turn on the system power. Table 3-2 shows the Dionex ICS-1100 conditions at power-up.

Also turn on the power to the computer and the autosampler (if used).

Figure 3-3. Dionex ICS-1100 Rear Panel

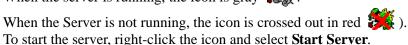
Feature	Power-Up Condition	
Pump	Off	
Injection valve	Load position	
Cell	Reading current value	
Suppressor	Off*	
Cell heater	Set to the last value used. The default when the Dionex ICS-1100 is turned on for the first time is 35 °C.	
Column oven temperature (Optional)	Set to the last value used. The default when the Dionex ICS-1100 is turned on for the first time is 30 °C.	
* When you start the suppressor, the value used last is restored.		

Table 3-2. Dionex ICS-1100 Power-Up Conditions

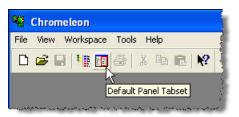
3.3 Connecting to Chromeleon

NOTE If you are beginning operation of a Dionex ICS-1100 that has not been configured in a timebase, refer to the Dionex ICS-1100 installation instructions for Chromeleon setup instructions.

- 1. Turn on the PC.
- 2. Start the Chromeleon Server, if it is not already running.
 - a. Check the Chromeleon Server Monitor icon on the Windows taskbar.
 - When the server is running, the icon is gray



- b. If the Server Monitor icon is not on the Windows taskbar, click **Start** > **All Programs** > **Chromeleon** > **Server Monitor**. The Server Monitor opens. Click **Start** to start the server.
- 3. To start the Chromeleon client, click **Start** and select **All Programs** > **Chromeleon** > **Chromeleon**.
- If Chromeleon is installed, the main window and Browser appear.
 Display the panel tabset by selecting View > Default Panel Tabset or by clicking the Default Panel Tabset toolbar button.



If Chromeleon Xpress is installed, starting the application automatically displays the panel tabset.

5. To display the Dionex ICS-1100 Control panel, click the tab labeled with the Dionex ICS-1100 timebase name (see Figure 3-4).

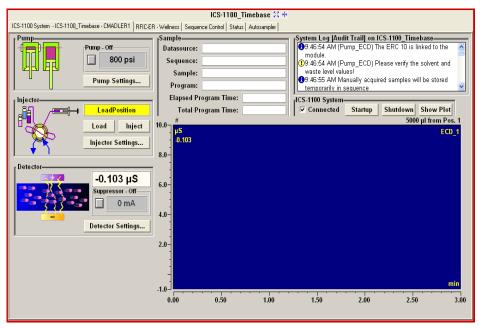


Figure 3-4. Dionex ICS-1100 Control Panel on the Chromeleon Panel Tabset

3.4 Set Up the Eluent Reservoir

NOTE If you plan to operate the system in the RFIC-ER mode, disregard the instructions here. Follow the instructions provided with your RFIC-ER Installation Kit to set up the RFIC-ER eluent reservoir.

3.4.1 Prepare the Eluent

Prepare the eluent according to the instructions in the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 053891).

3.4.2 Degas the Eluent

Eluent quality significantly affects the performance of the Dionex ICS-1100. To ensure optimal pump performance, observe the following precautions:

- Dionex strongly recommends vacuum degassing all eluents. This helps prevent bubbles, caused by outgassing, from forming in the pump heads and the detector flow cell.
- The optional vacuum degas assembly provides programmable, online degassing. The degas assembly must be installed in the Dionex ICS-1100 at the factory. If the Dionex ICS-1100 does not contain a degas assembly, manually degas eluent daily (following the instructions below) and store it in pressurized reservoirs.

Manual Degassing of Eluent

- 1. Prepare the eluent required for the application and pour it into a vacuum flask.
- 2. Attach the flask to a vacuum pump or water aspirator.
- 3. Vacuum degas the eluent for 5 to 10 minutes by shaking or sonication.
- 4. Remove the flask from the vacuum. **Do not allow eluent to flow** from the aspirator back into the flask.
- 5. Pour the degassed eluent into a pressurizable reservoir. **Do not shake** the eluent.

- 6. Install an end-line filter on the eluent line (see Section 3.4.3).
- 7. (Optional) Pressurize the reservoir.

The Dionex ICS-1100 does not require pressurized reservoirs. However, if eluent is manually degassed or is sensitive to contamination, Dionex recommends pressurizing the reservoir with helium or nitrogen.

The air regulator accessory bracket and other items needed for pressurizing the eluent reservoir must be ordered separately (P/N 060054). For more information, refer to the Dionex ICS-1100 installation instructions.

3.4.3 Filter the Eluent

Filtering removes small particulates in the eluent that may contaminate the pump check valves and cause erratic flow rates or loss of prime. An end-line filter (P/N 045987) is provided in the Dionex ICS-1100 Ship Kit (P/N 057905) for this purpose.

Install the end-line filter on the end of the eluent line, inside the reservoir. Verify that the end of the filter extends to the bottom of the reservoir and that the filter is submerged in eluent. This prevents air from being drawn through the lines.

3.4.4 Fill the Reservoir

Fill the reservoir with the prepared eluent.

3.4.5 Set the Eluent Level

After filling the reservoir, enter the volume of liquid in the reservoir on the Dionex ICS-1100 Control panel on the Chromeleon panel tabset (see <u>Figure 3-5</u>).

The Dionex ICS-1100 determines the eluent usage by monitoring the flow rate and the length of time the pump is on, and updates the **Eluent Bottle** volume as the eluent is depleted. A warning appears if the level falls below 200 mL. Warnings are repeated at 100 mL and 0 mL.

IMPORTANT

In order for the level displayed in the Eluent bottle box and gauge to be accurate, you must enter the level when the reservoir is filled. The Dionex ICS-1100 does not automatically detect when the reservoir is filled, nor when it is empty.

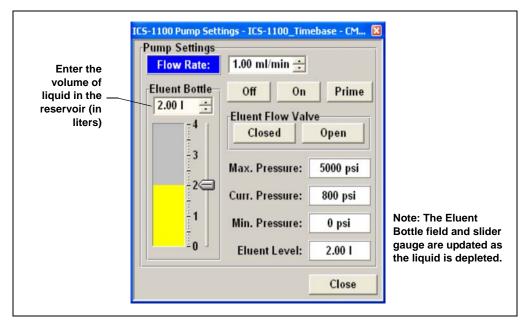


Figure 3-5. Pump Settings Window: Setting the Eluent Level

3.4.6 Connect the Reservoir

If it is not already connected, connect the **ELUENT BOTTLE OUT** line from the reservoir cap to the **ELUENT IN** line, which extends from the plumbing and cable chase on the top of the Dionex ICS-1100.

3.5 Check All Connections

Make sure the eluent reservoir is filled and the tubing connecting the reservoir to the Dionex ICS-1100 tubing is securely connected.

3.6 Prime the Pump

IMPORTANT

If you changed eluent or if the eluent lines are dry, prime the lines first with a syringe before following the procedure below to prime the pump. See Section 5.16.1 for instructions on priming the lines with a syringe.

- 1. Verify that the priming valve on the primary pump head (see <u>Figure 3-6</u>) is closed (turned all the way clockwise).
- Open the waste valve on the secondary pump head by turning the knob onequarter to one-half turn counterclockwise. Opening the valve directs the eluent flow path to waste and eliminates backpressure.

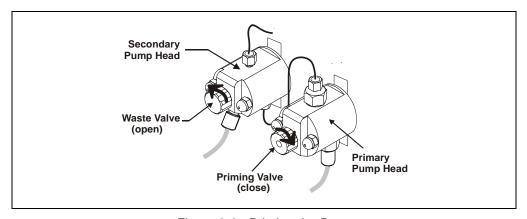


Figure 3-6. Priming the Pump

- 3. Press **Prime** on the Chromeleon Control panel. The pump will begin pumping at approximately 3 mL/min.
- 4. Continue priming the Dionex ICS-1100 until all air and previous eluent are purged and no air bubbles are exiting the waste line.
- 5. Press Pump Off.
- 6. Close the waste valve. **Do not overtighten.** The pump is now ready for operation.

3.7 Set System Operating Conditions

NOTE This section is an overview of the steps needed to start up the system and begin running samples. Actual operating parameters (flow rate, cell heater temperature, suppressor current, etc.) depend on the application to be run. Refer to the column manual for the required parameters for your application.

Set or verify system operating parameters from the Chromeleon Control panel. Operating parameters can also be set automatically by loading a Chromeleon sequence.

NOTE Clicking the System Startup button on the Chromeleon Control panel starts the pump and suppressor. The flow rate and suppressor current settings that were in effect when the system was shut down are restored.

- 1. Verify that the pump is on and set to the correct flow rate.
- 2. Verify that the suppressor current is on and that the setting is correct.
- 3. Verify that the cell heater is set to the correct value.
- 4. Verify that the column heater is set to the correct value.

3.8 Equilibrate the System and Verify Operational Status

- 1. Allow the system to equilibrate. During equilibration, the Chromeleon Control panel displays the background conductivity (the conductivity of the eluent before sample injection) and the system backpressure.
- 2. Monitor the background conductivity. Refer to the column manual for the appropriate background conductivity for your application.
- 3. Offset the background and zero the reading by clicking the **Autozero** button on the Chromeleon Control panel (see Figure 3-4).
- 4. Monitor the system pressure from the Control panel to make sure it is at the expected pressure for the installed column (refer to the column manual for details) and is stable.

- If the pressure is less than the expected amount, gas may be trapped in the system. To release the gas, remove the pump fitting on the injection valve port, labeled **P** (2). Allow the air to escape and then reconnect the fitting.
- If the pressure fluctuates by more than about 0.13 MPa (20 psi), prime the pump. See Section 4.4 for additional troubleshooting information.
- If the pressure is too high, there may be a restriction in the system plumbing. See Section 4.8 for troubleshooting information.
- 5. Verify that the baseline conductivity is at the expected reading for your application and is stable. In general, it should be $<30~\mu S$ for a system set up for anion analyses and $<2~\mu S$ for a system set up for cation analyses. See Section 4.13 for troubleshooting information if the conductivity is too high. See Section 4.14 if the baseline is drifting or has excessive "noise" (large fluctuations in readings).
- 6. Verify that the cell heater is at the set point and is stable. The temperature is at equilibrium when the **Set Temperature** and **Current Temperature** readings on the Control panel are the same.
- 7. Verify that the column heater temperature (if installed) is at the set point and stable. The temperature is at equilibrium when the **Set Temperature** and **Current Temperature** readings on the Control panel are the same.

The system is now ready for sample processing.

3.9 Prepare Samples

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

3.9.1 Collecting and Storing Samples

Collect samples in high density polyethylene containers that have been thoroughly cleaned with deionized (DI) 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 $0.45~\mu m$ 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 changes in anion concentrations.

3.9.2 Pretreating Samples

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

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

Before injection, pretreat samples that may contain high concentrations of interfering substances by putting them through Dionex OnGuard cartridges. Refer to *Installation and Troubleshooting Guide for OnGuard Cartridges* (Document No. 032943) for instructions.

3.9.3 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 filtered, Type I (18-megohm) 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 standards 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.

3.10 Loading and Injecting Samples

NOTE Samples can be injected using either the standard injection valve or the optional auxiliary valve, if installed. For more information, see the Chromeleon Help or user's manual.

The two techniques for loading samples into the sample loop are: (1) with an autosampler or (2) with a syringe or vacuum syringe through the injection port on the Dionex ICS-1100 front door.

For autosampler injections, the injection port tubing is disconnected from the Dionex ICS-1100 injection valve and replaced by the autosampler outlet tubing. Other setup requirements depend upon the model of autosampler.

Setup for a Dionex AS Autosampler

To use a Dionex AS Autosampler with the Dionex ICS-1100, the following requirements must be met:

- The Dionex ICS-1100 injection valve (or the auxiliary valve, if installed) must be specified as the injection valve and must be linked to the Dionex AS in the Chromeleon timebase.
- An injection valve should not be installed in the AS.

See the Dionex ICS-1100 installation instructions for Dionex AS connection instructions.

Setup for a Dionex AS-DV Autosampler

To use a Dionex AS-DV Autosampler with the Dionex ICS-1100, the following requirements must be met:

• The Dionex ICS-1100 injection valve must be linked to the Dionex AS-DV in the Chromeleon timebase.

Setup for a Dionex AS-HV Autosampler

For setup information for the AS-HV Autosampler, see the *AS-HV Autosampler Operator's Manual* (Document No. 065125). The manual is provided on the Thermo Scientific Reference Library DVD (P/N 053891).

3.10.1 Loading Samples with a Syringe

Make sure the injection port on the Dionex ICS-1100 front door (see <u>Figure 2-1</u>) is connected to sample port **S** (5) on the injection valve (see Figure 3-7).

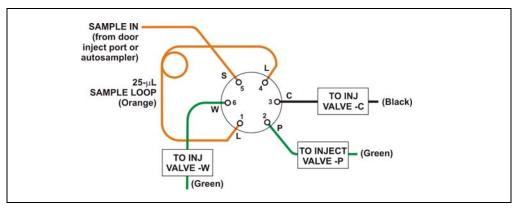


Figure 3-7. Injection Valve Connections

- 2. Fill the 1 cc syringe (P/N 016388) provided in the Dionex ICS-1100 Ship Kit (P/N 057905) with a calibration standard or sample.
- 3. Insert the syringe into the injection port on the Dionex ICS-1100 front door (see Figure 2-1).
- 4. Verify that the injection valve is in the Load position.
- 5. Overfill the sample loop with several sample loop volumes. Excess sample will exit through the injection valve waste line.
- 6. Leave the syringe in the port.
- 7. Switch the injection valve to the Inject position (see <u>Section 3.10.4</u>).

3.10.2 Loading Samples with a Vacuum Syringe

- 1. Disconnect the waste line from port **W** (6) of the injection valve (see <u>Figure 3-7</u>) and attach a shorter line: 25 to 30 cm (10 to 12 inches) of PEEK or PTFE (polytetrafluoroethylene) tubing.
- 2. Place the free end of the line into the sample.
- 3. Verify that the injection valve is in the Load position.
- 4. Insert the 1 cc syringe (P/N 016388) provided in the Dionex ICS-1100 Ship Kit (P/N 057905) into the injection port on the Dionex ICS-1100 front door (see <u>Figure 2-1</u>) and pull out the plunger to draw the sample into the injection valve.
- 5. Switch the injection valve to the Inject position (see <u>Section 3.10.4</u>).

3.10.3 Loading Samples with an Autosampler

- 1. Verify that the autosampler output line is connected to port **S** (5) of the Dionex ICS-1100 injection valve.
- 2. Prepare and fill the sample vials and place them in the autosampler tray or cassette. Refer to the autosampler manual for detailed instructions.
- 3. The sample loading process depends on the autosampler. Refer to the autosampler manual for detailed instructions. In general, use one of the following methods:
 - Include the commands for controlling sample loading in a Chromeleon program. (If necessary, refer to the Chromeleon Help or user manual for assistance.)
 - Enter the commands for loading the sample on the autosampler front panel.
- 4. Switch the injection valve to the Inject position (see Section 3.10.4).

3.10.4 Injecting Samples

After loading the sample in the sample loop, use one of the following methods to switch the injection valve to the Inject position:

- Manually: Click the **Inject** button on the Chromeleon Control panel (see <u>Figure 3-4</u>).
- Automatically: Include the Inject command in a Chromeleon program. (If necessary, refer to the Chromeleon Help or user manual for assistance.)

3.11 Processing Samples

Samples can be run manually (one at a time) or else they can be grouped and run automatically in batches.

3.11.1 Manual Sample Processing

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

Summary of Manual Sample Processing

- 1. Complete the instructions in <u>Section 3.2</u> through <u>Section 3.8</u> to prepare the Dionex ICS-1100 for operation and to prepare the sample for processing.
- 2. Load the sample, using a syringe, vacuum syringe, or autosampler (see Section 3.10).
- 3. On the Dionex ICS-1100 Control panel, click the **Autozero** button.
- 4. On the **Sequence Control** panel, click the **Acq On** button (or click the **Acquisition On/Off** button on the Chromeleon toolbar).
- 5. Inject the sample (see <u>Section 3.10.4</u>).
- 6. The signal plot is displayed on the Control panel. Monitor the chromatogram. When sample data has been collected, click the Acq Off button on the Sequence Control panel (or click the Acquisition On/Off button on the Chromeleon toolbar).

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 under the timebase folder in the local datasource.

To save the data from a manual run:

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

3.11.2 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 the following:

- A program with commands and parameters for controlling the Dionex ICS-1100 and autosampler (if used), and for acquiring sample data.
- A quantification method for peak identification and area determination.
- Additional sample processing parameters (sample name, sample type, injection volume, etc.).

After creating the sequence, you can start batch processing.

Summary of Automatic Sample Processing

- 1. Complete the instructions in <u>Section 3.2</u> through <u>Section 3.8</u> to prepare the Dionex ICS-1100 for operation and to prepare the sample for processing.
- 2. If an autosampler is installed, 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 Thermo Scientific Reference Library DVD (P/N 053891).
- 3. If an autosampler is not installed, load the sample into the injection valve sample loop through the sample port on the Dionex ICS-1100 front door (see Section 3.10.1).
- 4. Use the Application Wizard to specify a program and quantitation method, and create a sequence:
 - a. On the **Sequence Control** panel, click **Application Wizard**.
 - b. Select an application template from the list (see <u>Figure 3-8</u>).

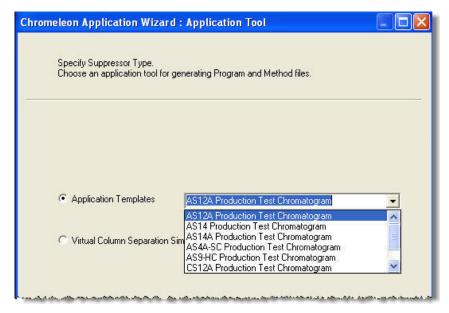


Figure 3-8. Application Wizard

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

After you click **Finish**, 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. Load the sequence and start batch processing:
 - a. On the **Sequence Control** panel, click **Load Sequence**.
 - b. Select the sequence created in <u>Step 4</u> and click **Open**.
 - c. Click Start Batch.

3.12 Maintenance

This section describes routine maintenance procedures that users may perform. All other maintenance procedures must be performed by Thermo Fisher Scientific personnel.

As Needed

- Regularly check the eluent reservoir and refill when needed.
- Make fresh eluent as needed.
- RFIC-ER systems only: The Chromeleon RFIC-ER Wellness feature tracks
 the usage of the RFIC-ER eluent and columns. An Audit Trail message will
 inform you when an item is approaching the end of its useful life span. You
 can also check the life expectancy of these components on the RFIC-ER
 Wellness panel.

Daily

 Check the Dionex ICS-1100 component panel (see <u>Figure 2-4</u>) for leaks or spills. Wipe up spills. Isolate and repair leaks (see <u>Section 4.3</u>). Rinse off any dried eluent with deionized water.

• Check the waste container daily and empty when needed.

Weekly

- Once a week, check fluid lines for crimping or discoloration. Replace any pinched lines. Replace damaged lines.
- Check the junctions between the pump heads and the pump casting for evidence of liquid leaks. If the piston seal wash tubing is not connected, check the drain tubes at the rear of the pump heads for evidence of moisture. Normal friction and wear may gradually result in small liquid leaks around the piston seal. If unchecked, these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks occur, replace the piston seals (see Section 5.7).
- Check the end-line filter (P/N 045987) and change if needed. When new, end-line filters are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent does not contain solvent.
 - NOTE It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Every Six Months

- Calibrate the cell (see Section 5.1.2).
- Calibrate the vacuum degas assembly (see <u>Section 5.1.4</u>).
- Replace the pump piston rinse seals and piston seals (see <u>Section 5.7</u>).

Yearly

- Thermo Fisher Scientific recommends performing preventive maintenance annually, as well as before scheduled Performance Qualification tests. A Dionex ICS-1100 Preventive Maintenance Kit (P/N 057954) is available for this purpose.
- Rebuild the auxiliary valve, if installed (see Section 5.4).
- If a Dionex AS Autosampler is installed, perform the preventive maintenance procedure. The Dionex AS Preventive Maintenance Kit (P/N 060581) is available for this purpose.

• If a Dionex AS-DV Autosampler is installed, replace the tip and tubing. The Dionex AS-DV Preventive Maintenance Kit (P/N 055647) contains all of the components required to replace the sampling tip and the tubing between the tip and the injection valve. Instructions are included with the kit.

Dionex ICS-1100 Ion Chromatography System

4 • Troubleshooting

This chapter is a guide to troubleshooting minor issues that may arise during operation of the Dionex ICS-1100. 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 Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office. Please have this chapter at hand when talking with Technical Support personnel.

4.1 Error Messages

The Moduleware (the instrument control firmware installed in each Dionex ICS-1100 module) periodically checks the status of certain parameters. If a problem is detected, it is reported to Chromeleon 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). You can change the severity level assigned to a problem whenever appropriate.

Icon	Severity Level	Description
1	Warning	A message is displayed in the Audit Trail, but the current run is not interrupted.
Δ	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.

Dionex ICS-1100 Ion Chromatography System

The table below lists the most frequently observed Dionex ICS-1100 error messages. For troubleshooting assistance, refer to the page indicated in the table.

Alarms and Error Conditions	See
Column heater exceeds safe temperature	page 61
Column heater open circuit	page 61
Column heater short circuit	page 61
Degas calibration failed	page 61
Hardware not present	page 62
Leak sensor wet	page 62
Load/Inject valve error	page 62
Option not installed	page 63
Pressure slope calibration error	page 63
Pump motor lost control	page 63
Pump over pressure	page 64
Pump pressure hardware error	page 64
Pump stopped due to lost USB communication error	page 64
Pump under pressure	page 65
Second valve error	page 65
Suppressor not connected	page 66
Suppressor over current	page 66
Suppressor over power	page 67
Suppressor over voltage	page 67
Suppressor stopped for zero flow rate	page 67

Table 4-3. Alarms and Error Messages Summary

4.2 **Troubleshooting Error Messages**



COLUMN HEATER EXCEEDS SAFE TEMPERATURE

This error occurs when the column heater temperature is higher than the maximum allowed. This error may occur if the Dionex ICS-1100 is operating in an extreme environment (greater than 40 °C (104 °F)).

To troubleshoot:

Refer to Section A.3 for environmental specifications.

COLUMN HEATER OPEN CIRCUIT

This error occurs when the column heater is unplugged from the component panel.

To troubleshoot:

- 1. Check that the column heater is plugged into the component panel.
- 2. If the error persists, the column heater may be faulty. Replace the column heater (see Section 5.12).

COLUMN HEATER SHORT CIRCUIT

This error occurs when there is a short circuit on the thermistor input used to measure the column heater temperature. This error indicates a faulty column heater.

To troubleshoot:

Replace the column heater (see <u>Section 5.12</u>).

① DEGAS CALIBRATION FAILED

This error occurs when the degas vacuum does not reach the expected level during calibration.

To troubleshoot:

- 1. Retry the calibration.
- 2. If calibration still fails, the vacuum degas assembly may need to be replaced. Contact Thermo Fisher Scientific for assistance.

Δ

HARDWARE NOT PRESENT

This error indicates a problem in the Dionex ICS-1100 electronics.

To troubleshoot:

Contact Thermo Fisher Scientific for assistance. The Dionex ICS-1100 electronics components cannot be serviced by the user.

! LEAK SENSOR WET

The leak sensor is installed in the drip tray at the bottom of the component panel (see <u>Figure 2-4</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 on the component panel. Refer to Section 4.3.
- 2. Tighten fittings or replace tubing and fittings as required. Refer to Section 4.3 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.

LOAD/INJECT VALVE ERROR

If the injection valve fails to switch position within 1 second of being toggled, the Dionex ICS-1100 Moduleware reports an error to Chromeleon and this error message appears.

To troubleshoot:

- 1. If a sequence is being executed, terminate the sequence by selecting **Batch** > **Stop** in Chromeleon.
- 2. Try to toggle the valve from Load to Inject by clicking the **Load** and **Inject** buttons on the Dionex ICS-1100 Control panel in Chromeleon.
- 3. Turn off the Dionex ICS-1100 power briefly and then restart.
- 4. If the problem persists, repeat Step 2.
- 5. If the problem persists, contact Thermo Fisher Scientific for assistance.

Λ

OPTION NOT INSTALLED

This error occurs if a command is issued to control an option that is not installed.

To troubleshoot:

Check the Chromeleon Server Configuration program to verify that the option is enabled.

PRESSURE SLOPE CALIBRATION ERROR

This error occurs if you try to calibrate the pressure slope when the pressure is less than 3 MPa (500 psi).

To troubleshoot:

Contact Thermo Fisher Scientific for assistance.

PUMP MOTOR LOST CONTROL

This error indicates a problem in the pump controller electronics.

To troubleshoot:

Contact Thermo Fisher Scientific for assistance. The Dionex ICS-1100 electronics components cannot be serviced by the user.

Stop

PUMP PRESSURE HARDWARE ERROR

This error indicates a problem in the pump controller electronics.

To troubleshoot:

Contact Thermo Fisher Scientific for assistance. The Dionex ICS-1100 electronics components cannot be serviced by the user.

Stop

PUMP OVER PRESSURE

If the system pressure exceeds the set limit for at least 0.5 second, this error message appears and the pump stops. Pressure limits can be set in the Chromeleon Server Configuration or in the control program.

To troubleshoot:

- 1. Check for blockages in the liquid lines by working your way backward from the cell to the pump. See Section 2.2 for the system flow schematics.
- 2. Check that the flow rate is set to the correct value.
- 3. Check that the high pressure limit is set to the correct value.
- 4. Restart the pump.

Stop

PUMP STOPPED DUE TO LOST USB COMMUNICATION ERROR

This error occurs if the Dionex ICS-1100 loses communication with Chromeleon.

To troubleshoot:

Verify that the USB cable is connected correctly from the Dionex ICS-1100 rear panel to the Chromeleon PC.



PUMP UNDER PRESSURE

If the system pressure falls below the low pressure limit, the pump stops and this error message appears. Pressure limits can be set in the Chromeleon Server Configuration or in the control program.

To troubleshoot:

- 1. Make sure the eluent reservoir is full.
- 2. Check for liquid leaks (see <u>Section 4.3</u>).
- 3. Check that the waste valve is closed.
- 4. Prime the pump (see <u>Section 5.16</u>).
- 5. Restart the pump.



SECOND VALVE ERROR

If the auxiliary valve fails to switch position within 1 second of being toggled, the Dionex ICS-1100 Moduleware reports an error to Chromeleon and this error message appears.

To troubleshoot:

- If a sequence is being executed, terminate the sequence by selecting Batch > Stop in Chromeleon.
- Try to toggle the valve from position A to position B by clicking the corresponding buttons on the Dionex ICS-1100 Control panel in Chromeleon (or by clicking the Valve_2 commands under Pump_ECD in the Commands dialog box).
- 3. Turn off the Dionex ICS-1100 power briefly and then restart.
- 4. If the problem persists, repeat <u>Step 2</u>.
- 5. If the problem persists, contact Thermo Fisher Scientific for assistance.

SUPPRESSOR NOT CONNECTED

This error occurs if you turn on the suppressor and the Dionex ICS-1100 cannot establish a connection with the suppressor.

To troubleshoot:

- 1. Check the suppressor cable connection (see Section 5.11).
- 2. If the error persists, contact Thermo Fisher Scientific for assistance. There may be a problem in the suppressor controller electronics. The Dionex ICS-1100 electronics components cannot be serviced by the user.

SUPPRESSOR OVER CURRENT

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

To troubleshoot:

- 1. Follow the instructions in the suppressor manual to regenerate the suppressor. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
- 2. Follow the instructions in the suppressor manual to clean the suppressor.
- 3. If a malfunction in the suppressor controller is suspected, contact Thermo Fisher Scientific for assistance. The Dionex ICS-1100 electronics components cannot be serviced by the user.

Stop

SUPPRESSOR OVER POWER

This error occurs when, in order to maintain the selected current, the Dionex ICS-1100 is required to apply a higher voltage than the suppressor can support.

To troubleshoot:

- 1. Reduce the flow rate.
- 2. Rehydrate the suppressor. Refer to the suppressor manual for instructions. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
- 3. If the error persists, replace the suppressor (see Section 5.11).

Stop

SUPPRESSOR OVER VOLTAGE

This error occurs if you turn on the suppressor but the Dionex ICS-1100 cannot establish a connection with the suppressor.

To troubleshoot:

- 1. Check the suppressor cable connection (see <u>Section 5.11</u>).
- 2. If the error persists, replace the suppressor (see Section 5.11).

SUPPRESSOR STOPPED FOR ZERO FLOW RATE

This error message appears when you click the **Shutdown** button on the Dionex ICS-1100 Control panel in Chromeleon. The message also appears anytime you turn off the pump flow while the suppressor is on. The suppressor is automatically turned off to prevent damage to the suppressor.

To troubleshoot:

• If the pump stopped unexpectedly, see <u>Section 4.6</u> for pump troubleshooting information.

4.3 Liquid Leaks

Leaking fitting

Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection (see <u>Section 5.3</u>). Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

• Broken liquid line

Replace the line and fittings with the same length and internal diameter tubing (see Section 5.3).

• 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 Dionex ICS-1100. If a line is blocked, replace it (see Section 5.2).

Loose pump check valve

- Make sure the check valves are firmly seated in the pump head. If they are not, tighten them carefully with an open-end wrench just until the leak stops.
- 2. If the leak persists, replace the check valve (see <u>Section 5.6</u>).

Leaking seal wash port

Leaking from the seal wash port indicates a leaking piston seal. Replace the seal (see Section 5.7).

Damaged pump piston seal

- 1. Replace the piston seal (see <u>Section 5.7</u>).
- 2. If the leak persists, replace the piston (see Section 5.8).

• Pump head not tight against casting

Carefully tighten the pump head mounting nuts just until the leak stops. **DO NOT OVERTIGHTEN!**

• Leaking pressure transducer

- Make sure the liquid line connections into the pressure transducer are tight. Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891). Replace any damaged fittings.
- 2. If the pressure transducer continues to leak, contact Thermo Fisher Scientific for assistance. The pressure transducer cannot be serviced by the user.

Leaking pump head waste valve

Make sure the waste valve is closed. To close the valve, turn the knob clockwise, and tighten fingertight. **DO NOT OVERTIGHTEN!**Overtightening may damage the valve and the pump head.

If the leak persists, replace the waste valve O-ring (see Section 5.9).

Leaking suppressor

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

• Leaking injection valve or auxiliary valve

- Make sure the liquid line connections to the transducer are tight. Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891). Replace any damaged fittings.
- 2. Liquid leaks from behind the valve stator may indicate a scratched rotor seal. Rebuild the valve (see Section 5.4).

Leaking detector 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, causing it to leak. If the problem continues, contact Thermo Fisher Scientific for assistance.

4.4 Pump Difficult to Prime or Loses Prime

Excessive pressure fluctuations (more than 3% difference from one pressure reading to the next) indicate that the pump is out of prime.

• Empty eluent reservoir and/or no eluent connected

Fill the reservoir. Make sure all connections are secure.

• Eluent improperly or insufficiently degassed

If the Dionex ICS-1100 does not contain a vacuum degas assembly, degas the eluent manually (see Section 3.4.4).

If the Dionex ICS-1100 contains a vacuum degas assembly, check the degas settings:

- 1. Open the Chromeleon Server Configuration program. Right-click the Dionex ICS-1100 device in the timebase and select **Properties**.
- 2. Select the **Options** tab (see Figure 4-1).

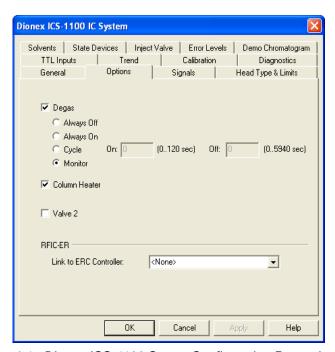


Figure 4-1. Dionex ICS-1100 Server Configuration Properties: Options

- 3. Verify that the **Degas** check box is selected.
- 4. Check the **Degas** settings. Select either the **Monitor** option or the **Cycle** option. If you select **Cycle**, set the time **On** to 30 seconds and the time **Off** to 600 seconds. If **Cycle** is already selected, try increasing the time on or decreasing the time off.

End-line filter is dirty or clogged

When new, end-line filters (P/N 045987) are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent does not contain solvent.

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

Blockages in inlet tubing

Kinked or clogged tubing causes the pump to be "starved" for eluent. Replace the tubing and fittings (see Section 5.3).

Dirty check valve

Clean or replace the pump check valve (see <u>Section 5.6</u>).

• Liquid leaks at junction between pump head and pump casting

Use the open-end wrench (P/N 014605) provided in the Dionex ICS-1100 Ship Kit (P/N 057905) to tighten the two acorn nuts that attach the pump head to the pump housing (see <u>Figure 5-7</u>). Tighten the nuts evenly (12 in-lb torque).

If the leak persists, replace the piston seal (see <u>Section 5.7</u>).

• Liquid leaks from the seal wash port

Replace the piston seal (see <u>Section 5.7</u>).

• Scratched pump piston

Check the pump pistons for scratches and replace if necessary (see Section 5.8).

4.5 Pump Does Not Start

• No power (front Power LED indicator is not lit)

Check that the power cord is plugged in.

Check the main power fuses and replace, if needed (see Section 5.18).

No communication between Dionex ICS-1100 and Chromeleon

Verify that the USB cable is connected correctly. For connection and setup information, refer to the Dionex ICS-1100 installation instructions.

Pump is turned off

Turn on the pump from the Dionex ICS-1100 Control panel in Chromeleon.

• Flow rate is set to 0

Set the flow rate from the Dionex ICS-1100 Control panel in Chromeleon.

4.6 No Flow

• Pump waste or priming valve open (see Figure 4-2)

Close the valves by turning the knobs clockwise until fingertight. **DO NOT OVERTIGHTEN!** Overtightening may damage the valve and the pump head.

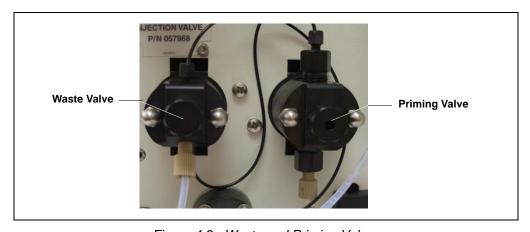


Figure 4-2. Waste and Priming Valves

• Flow rate is set to 0

Set the flow rate from the Dionex ICS-1100 Control panel in Chromeleon.

Eluent valve is closed

Open the eluent valve from the Dionex ICS-1100 Control panel in Chromeleon.

Pump not primed

Prime the pump (see Section 5.17).

Broken pump piston

Replace the piston (see <u>Section 5.8</u>).

4.7 Erratic Flow/Pressure Reading

• Pump needs priming

Prime the pump (see Section 5.17).

• Damaged piston seal

Replace the piston seal (see Section 5.7).

• Dirty pump check valve

Clean or replace the check valve (see <u>Section 5.6</u>).

• Leaking liquid lines or fittings

Check the liquid lines and fittings for small leaks. Tighten or, if necessary, replace the liquid line connection (see Section 5.3). Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

4.8 Excessive System Backpressure

• Restriction in the system plumbing

Check all liquid lines for crimping or blockage. Make sure the ferrule fittings are not overtightened onto tubing. Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for details. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

If you have trouble isolating the restriction, refer to <u>Section 5.2</u>.

Plugged or damaged fitting

Isolate the faulty fitting by loosening fittings, one by one, until the pressure returns to normal. Repair or replace the fitting (see Section 5.3).

• Flow rate through the columns too high

Set the correct rate for your application. If an incorrect flow rate calibration is suspected, calibrate the flow (see <u>Section 5.1.3</u>).

Clogged column bed supports

Refer to the instructions in the column manual for troubleshooting guidance. Column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

Contaminated columns

Clean the columns as instructed in the column manual or replace the guard column.

• Plugged injection valve or auxiliary valve passages

Rebuild the valve (see Section 5.4).

4.9 Peak "Ghosting"

"Ghosting" is the appearance of extraneous peaks in a chromatogram. These may be late-eluting peaks from a previous injection. They may also be the result of a contaminated standard or eluent, or a malfunctioning injection valve. These peaks may co-elute with peaks of interest, resulting in nonreproducible peak heights/areas.

• Insufficient time between sample injections

Wait until the previous sample has been completely eluted before making another injection.

Insufficient flush between samples

Flush the sample loop with at least 10 loop volumes of deionized water or sample between sample injections.

Incorrect or contaminated standards

Remake standards.

Incorrect or contaminated eluent

Remake the eluent. If the system is not operating in the RFIC-ER mode, refer to the instructions in the column manual. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

If this is an RFIC-ER system, refer to the instructions in the manual provided in your RFIC-ER Installation Kit.

Install or replace the end-line filter on the end of the deionized water line (see Section 3.4.3).

• Malfunctioning injection valve or auxiliary valve

- 1. Rebuild the valve (see <u>Section 5.4</u>).
- 2. If the valve leak persists, contact Thermo Fisher Scientific for assistance.

4.10 Nonreproducible Peak Height or Retention Time

Column overloading

Dilute the sample (see Section 3.9.3).

• Liquid leaks

Locate and eliminate the leaks (see <u>Section 4.3</u>).

• Incomplete or imprecise filling of the sample loop

- 1. Fill the sample loop until excess sample exits the waste line.
- 2. Inspect the 1-cc syringe (P/N 016388) and replace if damaged.

Pump not primed properly

Prime the pump (see Section 5.17).

4.11 Abnormal Retention Time or Selectivity

Incorrect or contaminated eluent

Remake the eluent, using concentrated eluent and ASTM filtered, Type I (18-megohm) deionized water.

Contaminated or degraded sample

Take appropriate precautions when preparing and storing samples to prevent contamination and degradation (see Section 3.9).

Contaminated column

- Clean the column as instructed in the column manual. Column manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).
- 2. If cleaning is unsuccessful, replace the column.

4.12 No Cell Response

• Cell not properly installed

Verify that the cell front cover is flush against the component panel. If necessary, tighten the two mounting screws. When the cell is correctly installed and the screws tightened, an electronics connector on the cell plugs into a receptacle inside the Dionex ICS-1100.

No flow from pump

This condition has several possible causes; see <u>Section 4.5</u> and <u>Section 4.6</u> for details.

• Cell electronics malfunctioning

Use the Chromeleon Wellness Panel diagnostics to test the electronics (see Section 5.1 and the Chromeleon Help for instructions). If the variance reading is outside the tolerance range (less than 1 μ S), the electronics are malfunctioning. Contact Thermo Fisher Scientific for assistance. The Dionex ICS-1100 electronics cannot be serviced by the user.

4.13 High Cell Output

Background not suppressed by suppressor

Verify that the suppressor is turned on and the current is set to the correct value. Refer to the suppressor manual for additional troubleshooting

guidance. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

• Sample concentration too high

Dilute the sample (see Section 3.9.3).

Wrong eluent

Check that you are using the correct eluent for your application.

RFIC-ER systems only: Replace the RFIC-ER columns, if necessary. An Audit Trail message will inform you when a column is approaching the end of its useful life span. You can also check the life expectancy of the columns on the RFIC-ER Wellness panel.

Background conductivity not offset from conductivity reading

Before making an injection, allow the background conductivity to equilibrate, and then click **Autozero** on the Dionex ICS-1100 Control panel in Chromeleon.

Cell out of calibration

Recalibrate the cell (see <u>Section 5.1.2</u>).

4.14 Baseline Noise or Drift

Flow system leak; erratic baseline

Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace all liquid line connections. Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

Trapped gases

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.

• Pump not properly primed

Prime the pump (see <u>Section 5.17</u>).

• Contaminated or incorrect eluent

Remake the eluent, using concentrated eluent and ASTM filtered, Type I (18-megohm) deionized water.

RFIC-ER systems only: Replace the RFIC-ER columns, if necessary. An Audit Trail message will inform you when a column is approaching the end of its useful life span. You can also check the life expectancy of the columns on the RFIC-ER Wellness panel.

• Rapid changes in ambient temperature

Make sure the ambient temperature is between 4 and 40 $^{\circ}$ C (40 and 104 $^{\circ}$ F).

Make sure air conditioning and heating vents are directed away from the Dionex ICS-1100 and the Dionex ICS-1100 front 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.

• Incorrect suppressor operating conditions

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

• Cell above or below temperature

Contact Thermo Fisher Scientific for assistance.

• Damaged piston seal

Replace the piston seal (see <u>Section 5.7</u>).

4.15 Vacuum Degas Assembly Does Not Run

- Degas option not enabled in Chromeleon
 - 1. Open the Chromeleon Server Configuration program. Right-click the Dionex ICS-1100 device in the timebase and select **Properties**.
 - 2. Select the **Options** tab (see Figure 4-3).

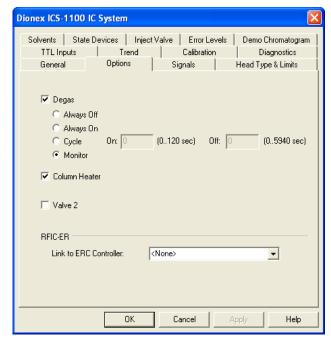


Figure 4-3. Dionex ICS-1100 Server Configuration Properties: Options

3. Verify that the **Degas** check box is selected.

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- 4. Under **Degas**, verify that the **Always Off** option is *not* selected. If it is, select one of the following settings instead:
 - Cycle; also select On: 30 seconds and Off: 10 minutes
 - Monitor
- 5. If the **Degas** settings are correct, but the degas assembly still does not run, test the assembly by selecting the **Always On** option. The pump should turn on immediately. If it does not, the vacuum degas assembly may need to be replaced. Contact Thermo Fisher Scientific for assistance.

IMPORTANT

Select the Degas, Always On option only when testing the vacuum degas assembly. Do not select the Always On option for routine use.

6. If the degas pump runs when you select **Always On**, but does not run when **Monitor** is selected, recalibrate the degas assembly (see Section 5.1.4).

This chapter describes Dionex ICS-1100 service and repair procedures that the user can perform. All procedures not included here, including electronics-related repair procedures, must be performed by Thermo Fisher Scientific personnel. For assistance, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

Before replacing any part, refer to the troubleshooting information in <u>Chapter 4</u> to isolate the cause of the problem.



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

5.1 Diagnostic and Calibration Procedures

Diagnostic and calibration procedures are performed from the Chromeleon Wellness Panel (see Figure 5-1).

For an overview of the Wellness Panel features, see <u>Section 5.1.1</u>. For instructions on performing calibration and diagnostic procedures, see the following sections:

- Calibrating the Conductivity Cell (Section 5.1.2)
- Calibrating the Flow Rate (Section 5.1.3)
- Calibrating the Vacuum Degas Assembly (Section 5.1.4)

5.1.1 Chromeleon Wellness Panel Overview

NOTE Do not open a System Wellness Control panel if the name includes "Service.pan." These Wellness panels are reserved for use by Thermo Fisher Scientific Service Representatives.

To Open the Wellness Panel

- In the Chromeleon Browser, expand the **Dionex** Templates\Panels\Wellness folder.
- 2. Double-click ICS-1100_1600_2100_Wellness_user.pan.

The Wellness Panel opens (see <u>Figure 5-1</u>). If the controls on the Wellness panel are disabled, select **Control** > **Connect to timebase** and then select the Dionex ICS-1100 timebase.

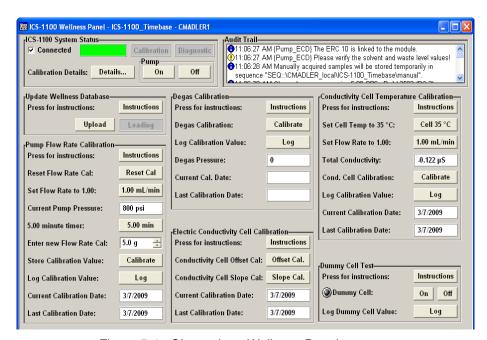


Figure 5-1. Chromeleon Wellness Panel

Use the Wellness Panel to perform the following functions:

- Calibrate the pump flow rate
- Calibrate the vacuum degas assembly
- Calibrate the conductivity cell
- Test the conductivity cell electronics, using a dummy cell
- Upload calibration values from the Dionex ICS-1100 to the Chromeleon Wellness database

5.1.2 Calibrating the Conductivity Cell

When to Calibrate

• After every 6 months of use

NOTE Do not use this procedure to calibrate a new cell. If you are replacing a cell, see the instructions in Section 5.10 to install the new cell and calibrate it.

Items Needed	Description
1.0 mM KCl solution	Prepare by dissolving 0.07456 g of reagent-grade KCl in 1 liter of 18-megohm DI water.
Backpressure tubing to provide at least 7 MPa (1000 psi)	Use 0.076-mm (0.003-in) ID yellow PEEK tubing (P/N 049715).

- 1. Open the Dionex ICS-1100 Wellness Panel in Chromeleon (see Section 5.1.1).
- 2. On the Wellness Panel under Electric Conductivity Cell Calibration, click Offset Cal.
- 3. When the offset calibration is complete, click **Slope Cal** on the Wellness Panel.
- 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. Disconnect the line from the suppressor **REGEN IN** port to the cell outlet. Direct the cell outlet line to a waste container.
- 7. Verify that the backpressure is at least 7 MPa (1000 psi).
- 8. On the Wellness Panel under Conductivity Cell Calibration, click Cell 35 °C. Allow the cell to reach this temperature, and then wait an additional 5 minutes to let it stabilize.
- 9. Select **1.00 mL/min** to begin pumping 1.00 mM KCl through the cell at 1.0 mL/min.
- 10. Wait until the total conductivity reading stabilizes (in about 15 minutes) and then select **Calibrate** on the Wellness Panel.

After calibration, the conductivity reading should be 147.00 \pm 2 μS and the cell constant should be between 120 and 180. If this is not the case, contact Thermo Fisher Scientific for help.

- 11. Click **Log** to record the new calibration values in the Audit Trail.
- 12. Flush the KCl solution from the system by pumping deionized water through the cell. When the conductivity drops to less than 1 μ S/cm, stop the pump.
- 13. Reconnect the pump to the injection valve and reconnect the line from the suppressor **ELUENT OUT** port to the cell inlet.
- 14. Reconnect the cell outlet to the suppressor **REGEN IN** port.

5.1.3 Calibrating the Flow Rate

When to Calibrate

If you run the Dionex ICS-1100 Operational Qualification or Performance Oualification and it fails.

Items Needed

- 0.076-mm (0.003-in) ID yellow PEEK tubing (P/N 049715) to create 14 ± 2 MPa (2000 \pm 300 psi) of backpressure (if needed)
- High purity ASTM filtered, Type I (18 megohm-cm) deionized water
- Balance capable of weighing more than 10 g with 0.001 g readability
- Tared beaker

To Calibrate

- 1. Open the Dionex ICS-1100 Wellness Panel in Chromeleon (see Section 5.1.1).
- 2. Verify that there is 14 ± 1.4 MPa (2000 ± 200 psi) of backpressure.
- 3. On the Chromeleon Wellness Panel under Pump Flow Rate Calibration, select Reset Cal.
- 4. Select **1 mL/min** to set the flow rate and begin pumping deionized water.
- 5. Allow the pump to stabilize for 20 minutes.
- 6. Click **5.00 min** to start the timer. Immediately start collecting water into the tared beaker.
- 7. Collect the water for exactly 5.00 minutes.
- 8. Enter the weight of the water (in grams).
- 9. Click **Calibrate** to download the value to the Dionex ICS-1100.
- 10. To recheck the value, wait at least 15 minutes, and then repeat the calibration.
- 11. Click **Log** to record the new calibration value in the Audit Trail.

5.1.4 Calibrating the Vacuum Degas Assembly

When to Calibrate

- After every 6 months of use
- 1. Open the Dionex ICS-1100 Wellness Panel in Chromeleon (see Section 5.1.1).
- 2. On the Wellness Panel under **Degas Calibration**, select **Calibrate**. The degas pump runs for 90 seconds to allow a vacuum to be created. After 90 seconds, the vacuum degas pressure reading is recorded. When the vacuum degas assembly is running in monitor mode, this calibration value is used to determine when to turn on the degas pump.
- 3. Click **Log** to record the new calibration value in the Audit Trail.

5.2 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).
- 2. Follow the flow schematics in <u>Figure 5-2</u> or <u>Figure 5-3</u> and work backward through the system, beginning at the suppressor **Regen Out** port. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops abnormally indicates the point of restriction.

NOTE The numbers on the flow schematics indicate the order in which liquid flows through the system components.

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

3. If the restriction is in the tubing or fitting, remove the restriction by back flushing or by replacing the tubing or fitting (see Section 5.3).

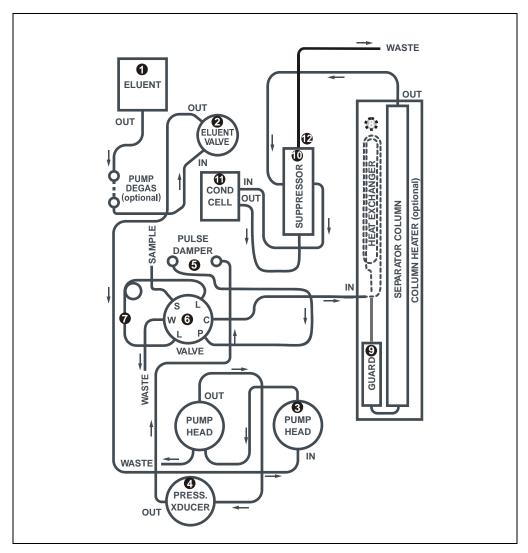


Figure 5-2. Dionex ICS-1100 Flow Schematic: Standard Configuration

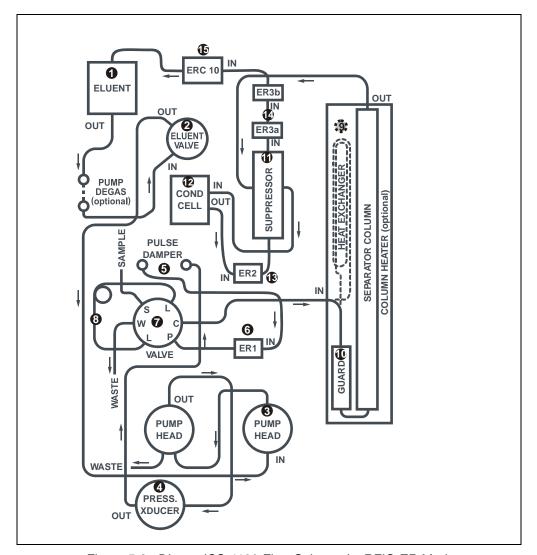


Figure 5-3. Dionex ICS-1100 Flow Schematic: RFIC-ER Mode

5.3 Replacing Tubing and Fittings

The Dionex ICS-1100 is plumbed with the tubing and tubing assemblies listed below.

Tubing Size and Type	Color	Used For
0.125-mm (0.005-in) ID PEEK (P/N 044221)	Red	Connection from pump pulse damper to pressure transducer
0.25-mm (0.010-in) ID PEEK (P/N 042690)	Black	Connections between other system components
0.50-mm (0.020-in) ID PEEK (P/N 042855)	Orange	Connection from injection port to injection valve
0.75-mm (0.030-in) ID PEEK (P/N 044777)	Green	Connection from injection valve to waste
1.58-mm (0.062-in) ID PTFE (P/N 014157)	Clear	Connection from pump to degas or eluent reservoir; pump waste
25 μL sample loop (P/N 042857)	Orange	Connection between ports L (1) and L (4) on the injection valve

- The ER Tubing Kit (P/N 070061) contains the four pieces of tubing required for connections from the RFIC-ER columns to the component panel. This tubing is initially installed in the Dionex ICS-1100 at the factory.
- 10-32 fittings (P/N 043275) and ferrules (P/N 043276) are used for most tubing connections. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).
- 1/8-in fittings (P/N 052267) and ferrules (P/N 048949) are used for connections to the suppressor **REGEN OUT** port and the eluent reservoir.

5.4 Rebuilding the Injection Valve or Auxiliary Valve

Thermo Fisher Scientific recommends rebuilding the injection valve and the auxiliary valve (if installed) annually. The Injection Valve Rebuild Kit (P/N 057896) contains all required replacement parts for one valve.

NOTE If you prefer, you can replace the auxiliary valve "pod," instead of rebuilding the valve. Replacing the pod is easier and faster than rebuilding the auxiliary valve. For instructions, see Section 5.5.

IMPORTANT

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

- 1. Turn off the pump from the Dionex ICS-1100 Control panel in Chromeleon.
- 2. Open the Dionex ICS-1100 front door.
- 3. Disconnect each liquid line connected to the valve.
- 4. Follow the instructions provided in the Rebuild Kit to replace the rotor seal, isolation seal, and stator face.
- 5. Reconnect all liquid lines to the injection valve (see <u>Figure 5-4</u>) or auxiliary valve.
- 6. Close the door.
- 7. Turn on the pump.

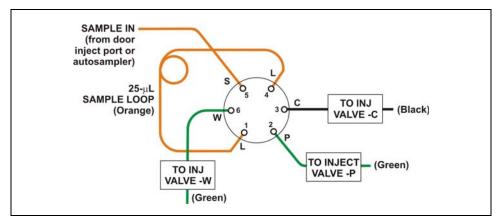


Figure 5-4. Injection Valve Plumbing

5.5 Replacing an Auxiliary Valve Pod

This procedure describes how to replace the mechanical part (the "pod") of the optional auxiliary valve. This procedure is an alternative to rebuilding the valve (see Section 5.4).

NOTE If the valve electronics require service, contact Thermo Fisher Scientific. Electronics-related repair procedures must be performed by Thermo Fisher Scientific personnel.

IMPORTANT

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

- 1. Turn off the pump flow from the Dionex ICS-1100 Control panel in Chromeleon.
- 2. Open the Dionex ICS-1100 front door.
- 3. Disconnect each liquid line connected to the valve.

- Unscrew the black locking ring on the front of the valve (see <u>Figure 5-5</u>) and remove the ring.
- 5. Grasp the front of the valve pod and pull firmly to remove it from the Dionex ICS-1100.

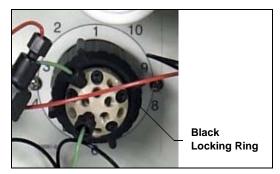


Figure 5-5. Unscrew the Valve Locking Ring

- 6. Check that the new pod (6-port, P/N 061947; 10-port, P/N 061948) has the correct number of ports for the valve being serviced.
- 7. Align the slots in the new pod with the runner in the valve holder on the Dionex ICS-1100 (see <u>Figure 5-6</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 5-6</u>). If necessary, twist the end of the pod to adjust the position of the splines.

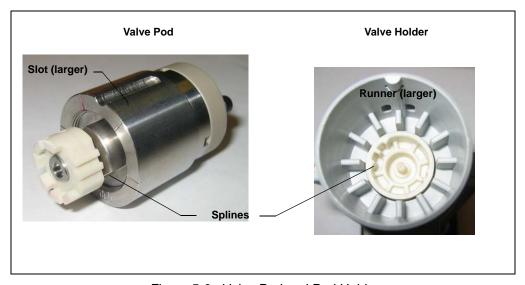


Figure 5-6. Valve Pod and Pod Holder

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- 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 5.1.1).
- 12. Close the door.

5.6 Cleaning and Replacing the Pump Check Valves

A dirty check valve causes erratic flow rates and pressures. In addition, it may cause the pump to lose prime and/or be difficult to reprime. If possible, replace dirty check valves. If new check valves are not available, follow the instructions for cleaning.

Replacing Check Valves

- 1. Close the eluent valve from the Dionex ICS-1100 Control panel in Chromeleon.
- 2. Turn off the main power switch, to ensure that you do not unintentionally start the Dionex ICS-1100.
- 3. To prevent contamination of pump parts, put on a pair of rubber gloves before disassembling the pump head.
- 4. Disconnect the tube fittings from the inlet and outlet check valve assemblies on the primary pump head (see <u>Figure 5-7</u>).
- 5. Use a 1/2-inch wrench to loosen both check valve assemblies. Remove the check valve assemblies from the pump head.

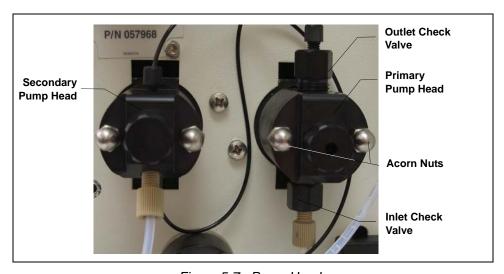


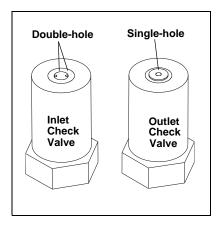
Figure 5-7. Pump Heads

NOTE The *inlet* check valve assembly housing has a 1/4-28 port. The *outlet* check valve assembly housing has a 10-32 port.

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6. If you are installing new cartridges (P/N 045994) in the existing check valve housings, place the cartridge in the *inlet* check valve housing with the double-hole end of the cartridge visible and place the cartridge in the *outlet* housing with the single-hole end visible.

NOTE The pump will not operate properly unless the cartridge is installed in the housing in the correct orientation. Liquid flows through the check valve in the large single hole and out the small double holes.



7. Install the inlet check valve assembly (P/N 045722) on the bottom of the primary pump head. Install the outlet check valve assembly (P/N 045721) on the top of the head. Tighten the check valves fingertight, and then use a 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.

- 8. Reconnect the liquid lines. Turn on the Dionex ICS-1100 main power.
- 9. Open the eluent valve from the Dionex ICS-1100 Control panel in Chromeleon.
- 10. Prime the pump (see Section 5.17).
- 11. When the Dionex ICS-1100 is at operating pressure, check for leaks from the check valves. Tighten a check valve a *little more* only if it leaks.

Cleaning Check Valves

- 1. Carefully remove the check valve cartridges from the valve housings.
- 2. Place the check valve housings and cartridges in a beaker with methanol. Sonicate or agitate the parts for several minutes.
- 3. Rinse each check valve housing and cartridge thoroughly with filtered deionized water.
- 4. To reinstall the check valves and complete the procedure, see <u>Step 6</u> through <u>Step 11</u> above.

5.7 Replacing a Pump Piston Seal and Piston Rinse Seal

A damaged seal allows leakage past the piston, as well as leakage from the piston seal wash housing. The pump may be difficult to prime, flow rates may be unstable, and there may be baseline noise.

Preparation

- 1. Rinse the pump flow path with deionized water. Direct the flow to waste by opening the waste valve on the secondary pump head (see <u>Figure 5-10</u>). To open the valve, turn the knob one-quarter to one-half turn counterclockwise.
- 2. After rinsing, close the waste valve.
- 3. Close the eluent valve from the Dionex ICS-1100 Control panel in Chromeleon.
- 4. To prevent contamination of pump parts, put on a pair of rubber gloves before disassembling the pump head.

Removing the Head and Piston

- 1. Turn off the main power switch, to ensure that you do not unintentionally start the Dionex ICS-1100.
- 2. Disconnect all tubing connections to the pump head.
- 3. Locate the open-end wrench (P/N 014605) provided in the Dionex ICS-1100 Ship Kit (P/N 057905). Use the wrench to remove the two acorn nuts (see Figure 5-7) from the pump head.
- 4. Slowly pull the head and allow it to separate from the housing. Carefully disengage the head from the piston by pulling the head straight off and away from its mounting studs.



Lateral motion while disengaging the pump head from the piston may break the piston.

Un mouvement latéral pendant la séparation de la tête et du piston peut casser le piston.



Vermeiden Sie Seitwärtsbewegungen, wenn Sie den Kopf vom Kolben lösen. Andernfalls kann der Kolben brechen.

5. Place the head (front end down) on a clean work surface and lift off the spacer to expose the piston seal (see Figure 5-8 or Figure 5-9).

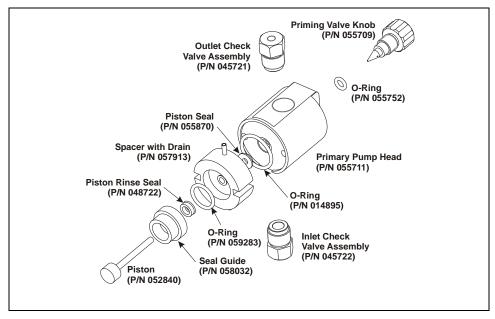


Figure 5-8. Primary Pump Head

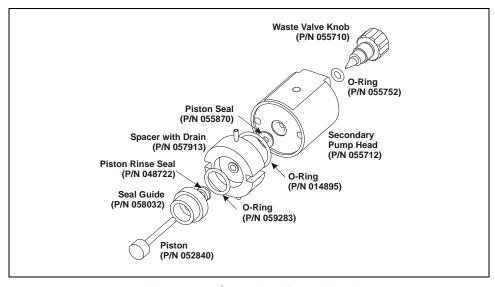
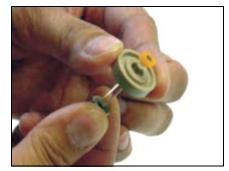


Figure 5-9. Secondary Pump Head

6. The piston does not come off as part of the pump head assembly because it is captured by a magnetic retention system. After removing the pump head, hold the shaft of the piston (near the base), tilt the piston slightly, and pull the piston away from the pump.

Installing the New Piston Rinse Seal

- 1. Remove the guide from the spacer to expose the piston rinse seal and O-ring. Remove the O-ring.
- 2. Remove the old piston rinse seal from the guide as follows:
 - a. Hold the guide with the flat side facing up.
 - To dislodge the piston rinse seal, gently insert the shaft of the piston through the small hole in the center of the guide (see photo).
 - Pull the seal off the end of the piston shaft and remove the piston from the guide.
- 3. Hold the new piston rinse seal (P/N 048722) with the grooved side facing up.



4. Using your fingertip, gently press the piston rinse seal into the guide until the edge of the seal is below the surface of the guide.

IMPORTANT

The piston rinse seal is made of soft plastic. Do not press on the seal with anything hard or sharp, including your fingernail. If the seal is nicked or gouged, it will not seal properly and may result in leaks.

- 5. Place the new O-ring (P/N 059283) into the groove in the guide.
- 6. Remove the O-ring from the groove in the flat side of the spacer and replace it with the new O-ring (P/N 014895).
- 7. In one hand, hold the guide with the O-ring and piston rinse seal facing up (this prevents the O-ring from falling out). In the other hand, hold the spacer with the cavity facing down.
- 8. Gently press the guide into the cavity in the spacer until it is fully seated.

Removing the Piston Seal from the Head

- 1. Fill the head cavity with deionized water by injecting the liquid through either the piston opening or the inlet check valve.
- 2. Reinsert the piston approximately 3 mm (0.125) inch into the seal.
- 3. If this is the *primary* pump head, install a 10-32 fitting plug (P/N 042772) on the outlet check valve. Tighten the plug.
- 4. If this is the *secondary* pump head, install a 10-32 fitting plug (P/N 042772) in both the inlet and outlet ports. Tighten the plugs.
- 5. Push the piston into the head. (This should hydraulically unseat the seal from the head.) Remove the piston and pull off the seal.
 - NOTE If the piston seal is not removed, make sure the 10-32 fitting plug(s) are tight and add more water. Make sure the head contains no air bubbles, and then repeat Step 2 and Step 5.
- 6. Remove the 10-32 fitting plug(s).

Installing the New Piston Seal

- 1. Open the priming valve knob (primary pump head) or waste valve knob (secondary pump head) by turning the knob one-quarter to one-half turn counterclockwise.
- 2. Push the piston through the spacer and then through the new seal. Insert the piston and seal into the pump head until the seal makes contact with the bottom of the counterbore.

NOTE If necessary, lubricate the seal with a small amount of isopropyl alcohol to facilitate insertion.

- 3. To seat the seal, push down on the spacer until it is flush with the head. A clicking sound indicates that the seal is correctly seated.
- 4. Close the priming valve knob or waste valve knob.

Reinstalling the Head and Piston

Thermo Fisher Scientific recommends reinstalling the head and piston as a single assembly, so that the piston centers itself onto the magnetic follower.

- 1. Hold the assembled spacer and guide with the drain tubes aligned vertically and press the spacer into the head until it is flush with the indented surface of the head.
- 2. Insert the piston so that 1/4 inch of the shaft is exposed. This ensures that the magnet in the follower picks up the piston. (The follower is the cylinder that holds the piston in place as it moves in and out of the pump head assembly.)
- 3. Reinstall the head and piston assembly; use a wrench to tighten the nuts evenly (12 in-lb torque).

Completing the Procedure

- 1. Reconnect all liquid lines to the pump head.
- 2. Turn on the main power switch.
- 3. Open the eluent valve.
- 4. Prime the pump (see Section 5.17).

5.8 Replacing a Pump Piston

Continued leaking of the piston seal after installation of a new seal (assuming the pump head is tight) indicates a dirty, scratched, or broken piston.

Follow the instructions in <u>Section 5.7</u> to install a new piston (P/N 052840) and piston seal (P/N 055870).

5.9 Replacing the Waste Valve or Priming Valve O-Ring

A damaged O-ring causes leakage around the base of the waste valve or priming valve knob.

- 1. Close the eluent valve from the Dionex ICS-1100 Control panel in Chromeleon.
- 2. Turn off the main power switch, to ensure that you do not unintentionally start the Dionex ICS-1100.
- 3. To remove the waste valve or priming valve from the pump head (see <u>Figure 5-10</u>), turn the knob counterclockwise until it is loose, and then pull the knob straight out of the cavity in the pump head.

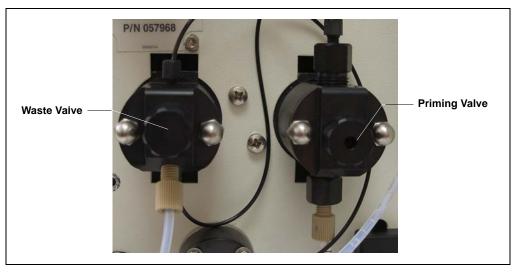


Figure 5-10. Waste and Priming Valves

4. If the O-ring is removed with the valve knob in Step 3, pull the O-ring off the end of the knob (see Figure 5-11). If the O-ring is not removed with the valve knob, insert a thin object (for example, the bent end of a paper clip) into the cavity in the pump head and carefully pull out the O-ring. **Do not scratch the cavity.**

IMPORTANT

Scratches in the cavity will cause leaks around the base of the knob while the pump is being primed.

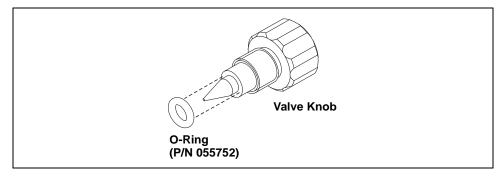


Figure 5-11. Waste Valve or Priming Valve O-Ring Replacement

- 5. Slide a new O-ring (P/N 055752) over the end of the valve.
- 6. To reinstall the valve containing the new O-ring, turn the knob clockwise and then tighten fingertight.

NOTE It is normal to encounter resistance after several rotations of the knob; the O-ring is being pushed into the cavity of the pump head.

- 7. Turn on the main power switch.
- 8. Open the eluent valve.
- 9. Prime the pump (see <u>Section 5.17</u>).

5.10 Replacing the Conductivity Cell

- 1. Turn off the Dionex ICS-1100 power.
- 2. Open the front door and disconnect the tubing from the **CELL IN** and **CELL OUT** fittings (see <u>Figure 5-12</u>, View A). Loosen the two screws on the cell front cover. The screws remain attached (see <u>Figure 5-12</u>, View B).

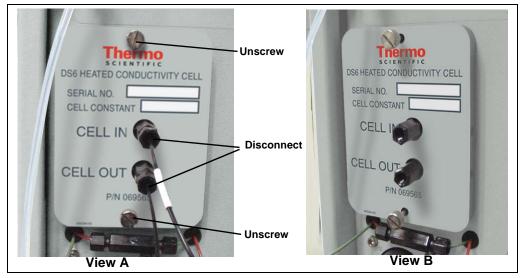


Figure 5-12. Removing the Cell Front Cover

- 3. Grip the two loosened screws and pull the cell cover toward you to remove it from the component panel. **Do not pull from the CELL IN and CELL OUT fittings.**
- 4. Continue pulling on the cover until the attached cell assembly slides out the front of the Dionex ICS-1100 (see Figure 5-13).



Figure 5-13. Removing the Cell Assembly

- 5. Slide the new cell assembly straight into the opening in the component panel until it stops and the cell cover is flush against the component panel. The cell's electronic connector automatically plugs into a connector inside the Dionex ICS-1100 when you push the assembly into the opening.
- 6. Tighten the screws on the cell front cover fingertight.
- 7. Close the side panel door and tighten the two mounting screws.
- 8. Reconnect the inlet and outlet lines.
- 9. Turn on the Dionex ICS-1100 power.
- 10. Calibrate the new cell in Chromeleon. Calibration of a new cell consists of three parts:
 - Running the offset calibration, which measures the output of the cell electronics alone and determines an offset value to be applied to raw conductivity readings.
 - Running the slope calibration, which measures the output of the cell electronics when a built-in dummy cell, which provides a constant conductivity reading of 21 μS, is automatically enabled. The slope value is applied to raw conductivity readings.
 - Entering the cell constant measured at the factory. The Dionex ICS-1100 uses the cell constant to determine the measured conductivity.
 - a. Open the Wellness Panel (see <u>Section 5.1.1</u>)
 - b. Under Electric Conductivity Cell Calibration, click Offset Cal.
 - c. When the offset calibration is complete, click **Slope Cal**.
 - d. Click **Calibration Details**. Under **Conductivity Cell Cal Details**, enter the **Cell Constant** value printed on the front of the cell.
 - e. In the field next to the **Download** button, verify that **Current** is selected and then click **Download** to download the new value to the Dionex ICS-1100.
 - f. To ensure that the new values are recorded in the Dionex ICS-1100 memory, do not turn off the Dionex ICS-1100 power for at least 1 minute after downloading.

5.11 Replacing the Suppressor

Refer to the suppressor manual for guidance about when to replace a suppressor. Suppressor manuals are included on the Thermo Scientific Reference Library DVD (P/N 053891).

- 1. Refer to the suppressor quick start guide (shipped with the suppressor) for instructions on preparing the suppressor before initial use and for additional installation details.
- 2. Turn off the pump from the Dionex ICS-1100 Control panel in Chromeleon.
- 3. Open the front door of the Dionex ICS-1100.
- 4. Disconnect the two eluent and the two regenerant lines from the suppressor.
- 5. To remove the suppressor from the component panel, slide it up to detach it from the mounting tabs on the panel and then pull it toward you.
- 6. Continue pulling the suppressor out until the yellow connector on the cable inside the Dionex ICS-1100 is outside the panel (see Figure 5-14).
- 7. Disconnect the suppressor cable (twist the ring on the yellow connector and pull the two connectors apart).

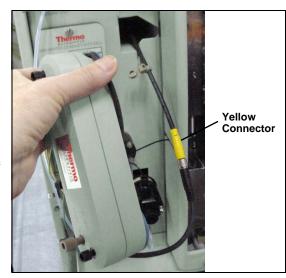
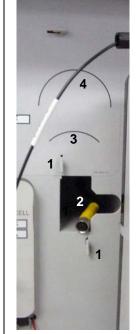


Figure 5-14. Removing the Suppressor

- 8. Connect the cable from the new suppressor to the Dionex ICS-1100 cable (align the pins on the two connectors and push them together).
- 9. Orient the suppressor with the **REGEN OUT** fitting on top and the cables to the right.
- 10. Push the cables into the opening in the component panel.

- 11. Align the top of the suppressor with the guide line printed on the component panel (see Figure 5-15) and align the slots on the rear of the suppressor with the tabs on the panel.
- 12. Press the suppressor onto the tabs and then slide it down until it locks into place. Pull slightly on the center of the suppressor to verify that it is securely fastened.

 Note: Some suppressors require more force to secure them onto the tabs.
- 13. Connect the two eluent and two regenerant lines to the new suppressor.
- 14. Close the Dionex ICS-1100 front door.



- 1 Suppressor Mounting Tabs
- 2 Suppressor Cable Connector
- 3 Guideline for AES
- 4 Guideline for SRS

Figure 5-15. Suppressor Guidelines and Mounting Tabs on Component Panel

5.12 Replacing the Column Heater

- 1. Turn off the Dionex ICS-1100 power.
- 2. Open the front door of the Dionex ICS-1100.
- 3. Remove the existing column heater:
 - a. Unscrew the two thumbscrews on the heater cover (they remain attached to the cover).
 - b. Pull the heater cover straight out to remove it.
 - c. Unscrew the six thumbscrews on the top metal plate (see <u>Figure 5-16</u>) and remove the plate.

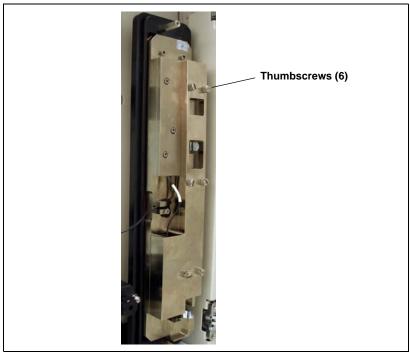


Figure 5-16. Dionex ICS-1100 Column Heater Cover Removed

- d. Remove the columns from the column heater.
- e. Pull the column heater straight toward you to remove it from the component panel.
- 4. Before installing the new column heater, write down the two calibration values (offset and slope) recorded on the back of the heater.
- 5. Align the connector on the back of the new column heater (P/N 069564) with the connector on the component panel (see <u>Figure 2-4</u>) and push the column heater onto the component panel.
- 6. Remove the heater cover and the metal plate on the new heater.
- 7. Install the columns in the new heater and replace the top metal plate and heater cover.
- 8. Turn on the Dionex ICS-1100 power.
- 9. Enter the column heater calibration values in Chromeleon as follows.

- a. Open the Wellness Panel (see <u>Section 5.1.1</u>) and click **Calibration Details**.
- b. Under Column Heater Calibration Values, enter the Column Heater Offset and Column Heater Slope values recorded in Step 4.
- c. In the field next to the **Download** button, verify that **Current** is selected and then click **Download** to download the new calibration values to the Dionex ICS-1100.
- d. To ensure the new calibration values are recorded in the Dionex ICS-1100 memory, do not turn off the Dionex ICS-1100 power for at least 1 minute after downloading the values.

5.13 Replacing the Column Heater Heat Exchanger

- 1. Turn off the Dionex ICS-1100 power.
- 2. Open the front door of the Dionex ICS-1100.
- 3. Unscrew the two thumbscrews on the column heater cover (they remain attached to the cover).
- 4. Pull the heater cover straight out to remove it.
- 5. Unscrew the six thumbscrews on the top metal plate (see <u>Figure 5-17</u>) and remove the plate.
- 6. Disconnect the heat exchanger inlet line from port C on the injection valve.

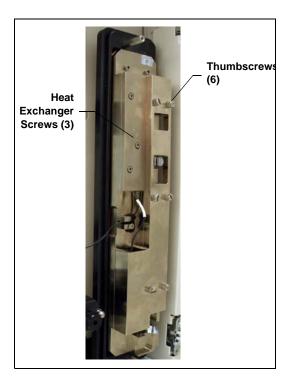


Figure 5-17. Dionex ICS-1100 Column Heater Cover Removed

- 7. Disconnect the heat exchanger outlet line from the guard column inlet.
- 8. Use an Allen wrench to remove the three screws from the heat exchanger metal plate and remove the plate (with the tubing) from the heater.
- 9. Remove the plastic backing from the new heat exchanger (P/N 059979, with 0.25-mm (0.010-in) ID tubing; P/N 060943, with 0.125-mm (0.005-in) ID tubing) and press the heat exchanger onto the heater. Replace the screws removed in Step 8.
- 10. Connect the new heat exchanger inlet line to port C on the injection valve and connect the outlet line to the guard column inlet.
- 11. Reinstall the top metal plate and the heater cover.

5.14 Replacing the Eluent Valve

- 1. Turn off the power to the Dionex ICS-1100.
- 2. Open the Dionex ICS-1100 front door.
- 3. To prevent an eluent leak during the valve replacement procedure, disconnect the eluent line from the reservoir cap *or* plug the line on the left side of the valve with a coupler (P/N 039056) and a plug (P/N 037268) after you disconnect the liquid line from the valve in Step 4.
- 4. Disconnect the two liquid lines connected to the eluent valve (see Figure 5-18).
- 5. Unscrew and remove the two screws that attach the valve to the component panel (see Figure 5-18).

 Save the screws.

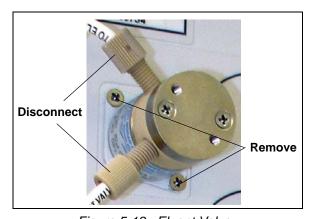


Figure 5-18. Eluent Valve

- 6. Pull the valve straight out from the component panel and begin pulling the attached cable out of the opening in the panel.
- 7. The cable is connected to a matching cable inside the Dionex ICS-1100. Continue pulling until the connectors for the two cables are outside the panel (see Figure 5-19). Disconnect the cables.

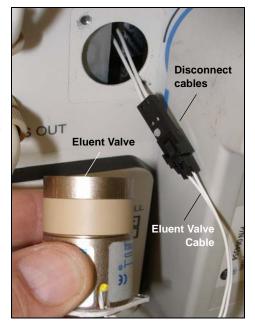


Figure 5-19. Eluent Valve Cable

- 8. Remove the mounting plate on the old valve by unscrewing and removing the two screws. Save the screws.
- 9. Thread the cable from the new eluent valve through the mounting plate.
- Attach the mounting plate to the new valve using the screws removed in Step 8.



Figure 5-20. Removing the Eluent Valve Mounting Plate

11. Connect the cable from the new valve (P/N 057945) to the Dionex ICS-1100 cable. Feed the cables back inside the Dionex ICS-1100.

- 12. Align the new valve on the component panel with the liquid ports facing up and attach it with the two screws removed in Step 5. Reconnect the liquid lines.
- 13. Turn on the power to the Dionex ICS-1100.
- 14. Prime the pump (see Section 5.17).

5.15 Replacing the Leak Sensor

- 1. Turn off the power to the Dionex ICS-1100.
- 2. Open the Dionex ICS-1100 front door.
- 3. Loosen the screw on the front of the leak sensor (see <u>Figure 5-21</u>). **Note:** The screw remains attached to the sensor.

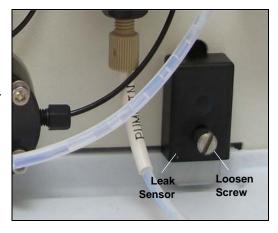


Figure 5-21. Leak Sensor

- 4. Remove the leak sensor from the component panel and pull the cable out of the opening in the panel.
- 5. The cable attached to the leak sensor is connected to a matching cable inside the Dionex ICS-1100. Continue pulling the cable until the connectors for the two cables are outside of the panel (see Figure 5-22).
- 6. Disconnect the two cables.

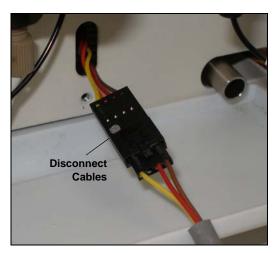


Figure 5-22. Leak Sensor Cable

- 7. Connect the cable from the new leak sensor (P/N 058053) to the Dionex ICS-1100 cable.
- 8. Feed the cables back inside the Dionex ICS-1100. Align the leak sensor with the component panel opening and fingertighten the screw.
- 9. Make sure the leak sensor does not touch the bottom of the drip tray.

5.16 Priming the Pump

- 1. Verify that the eluent reservoir is filled, the reservoir cap is installed and hand tightened, and the liquid line from the Dionex ICS-1100 to the reservoir cap is connected.
- 2. Verify that the waste lines are directed to a waste container.
- 3. The priming procedure consists of two parts:
 - Priming the eluent lines with a syringe (see Section 5.16.1). Perform this procedure at initial installation, after changing eluents, or when eluent lines are empty.
 - Priming the pump heads with the **Prime** button (see <u>Section 5.16.2</u>). Perform this procedure after the eluent lines are primed.

5.16.1 Priming the Eluent Lines with a Syringe

NOTE Prime the eluent lines after initial installation, after changing eluents, or when eluent lines are empty.

- 1. Verify that the pump is turned off.
- 2. Connect a 10 mL syringe (P/N 079803) to the priming valve port on the primary pump head (see Figure 5-23).

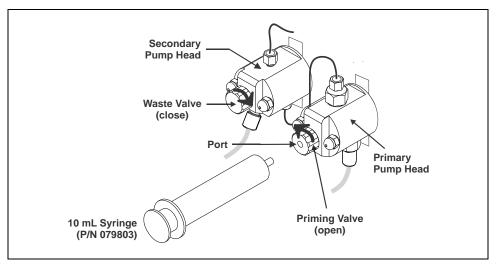


Figure 5-23. Priming the Eluent Lines

- 3. Open the priming valve by turning it one-quarter to one-half turn counterclockwise.
- 4. On the Dionex ICS-1100 Control panel in Chromeleon, click the **Pump Settings** button. The **Pump Settings** window opens (see Figure 3-5).
- 5. Under Eluent Flow Valve, click the Open button.
- 6. Draw the syringe back to begin pulling eluent through the flow path. It may take several syringe draws to remove all air or previous eluent from the tubing.

NOTE If the vacuum degas assembly is installed, draw out an additional 20 mL of eluent.

7. After priming the lines thoroughly, close the priming valve. **Do not overtighten**.

5.16.2 Priming with the Prime Button

NOTE Perform this procedure after priming the eluent lines (see Section 5.16.1).

1. Check that the priming valve on the primary pump head is closed (see Figure 5-24).

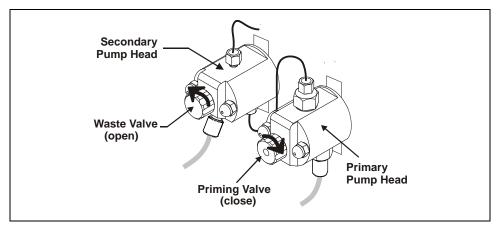


Figure 5-24. Priming the Pump

- Open the waste valve on the secondary pump head by turning the knob one-quarter to one-half turn counterclockwise (see <u>Figure 5-24</u>).
 Opening the valve directs the eluent flow path to waste and eliminates backpressure.
- 3. In the **Pump Settings** window (see <u>Figure 3-5</u>), click the **Prime** button. Confirm that the waste valve is open by clicking **OK** when the reminder message appears. The pump will begin pumping at about 3 mL/min.
- 4. Continue priming the Dionex ICS-1100 until no air bubbles are exiting the pump waste line.
- 5. In the **Pump Settings** window, click **Pump Off**.
- 6. Close the waste valve. **Do not overtighten the valve.**

5.17 Priming the Pump with Isopropyl Alcohol

NOTE Prime the pump heads with isopropyl alcohol only if the two standard priming procedures described in Section 5.16 are unsuccessful.

- 1. Connect a 10 mL syringe (P/N 079803) filled with isopropyl alcohol (IPA) to the port in the primary pump head (see Figure 5-25).
- 2. Open the waste valve on the secondary pump head (see <u>Figure 5-25</u>) by turning the knob one-quarter to one-half turn counterclockwise

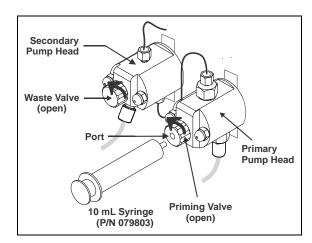


Figure 5-25. Priming the Pump Heads with IPA

- 3. In the **Pump Settings** window (see <u>Figure 3-5</u>), click **Pump On**.
- 4. Open the priming valve on the primary pump head by turning it one-quarter to one-half turn counterclockwise.
- 5. Use the syringe to slowly push alcohol through the pump.
 - NOTE Be careful not to push any air trapped in the syringe through the pump. Check the waste line from the secondary pump head to verify that there are no air bubbles.
- 6. Close the priming valve. **Do not overtighten.** Disconnect the syringe from the priming valve.
- 7. Let the pump run for several minutes, to purge alcohol from the pump heads. Then, click the **Prime** button in the **Pump Settings** window to flush the heads with the desired eluent.

IMPORTANT

Isopropyl alcohol may damage some columns. Be sure to thoroughly rinse the alcohol from the pump, using the process described in Step 7.

- 8. Close the waste valve. **Do not overtighten.**
- 9. Select the flow rate required for the analysis.
- 10. In the **Pump Settings** window, click **Pump Off**.

5.18 Changing Main Power Fuses

1. Turn off the main power switch.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the Dionex ICS-1100.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du Dionex ICS-1100.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des Dionex ICS-1100.

 The fuse holder is part of the main power receptacle (see <u>Figure 5-26</u>) on the Dionex ICS-1100 rear panel. To remove the fuse holder, squeeze the tab on the top of the holder to release it and pull the holder straight out of its compartment.

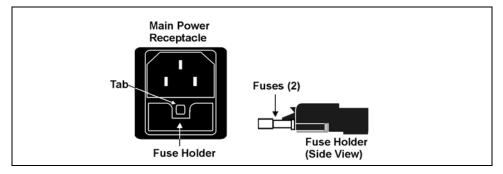


Figure 5-26. Main Power Fuse Holder

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- 3. Replace the two fuses in the holder with new IEC 127 fast-blow fuses rated 3.15 amps (P/N 954745). Thermo Fisher Scientific recommends always replacing both fuses.
- 4. Reinsert the fuse holder into its compartment and push in until the tab clicks in place.
- 5. Reconnect the main power cord and turn on the power.

A.1 Electrical

Main Power 100 to 240 Vac, 50 to 60 Hz (Auto-sensing power supply; no

manual voltage or frequency adjustment required)

Fuses Two 3.15 amp fast-blow IEC 127 fuses (P/N 954745)

A.2 Physical

Dimensions Height: 56.1 cm (22.1 in) Width: 22.4 cm (8.8 in)

reservoir) Depth: 53.3 cm (21.0 in)

Weight 24.5 kg (54 lb)

Decibel Level <52 dBA

Control Modes Full control through front panel and Chromeleon software;

alternative control through TTL or relay closure (two relay outputs,

two TTL outputs, four programmable inputs)

USB One USB input; one built-in USB hub with two outputs

Communication Protocol

A.3 Environmental

Operating 4 to 40 °C (40 to 104 °F); cold room-compatible (4 °C) as long

Temperature as system power remains on

Operating 5% to 95% relative humidity, noncondensing

Humidity

Operating Pressure 35 MPa (5000 psi) maximum liquid path (tubing, valve,

columns, etc.)

A.4 Front Panel

Power LED On when power is present; off when no power is present

Inject Valve Indicates the injection valve position.

LEDs

Alarm LED Indicates an alarm state.

Status LEDs Indicates the instrument status.

A.5 Analytical Pump and Fluidics

Type Serial dual-reciprocating pistons, microprocessor-controlled

constant stroke, variable speed

Construction Chemically inert, metal-free PEEK pump heads and flow paths

compatible with aqueous eluents of pH 0 to 14 and reversed-phase

solvents

Operating 0 to 35 MPa (0 to 5000 psi)

Pressure

Flow Rate 0.00 to 5.00 mL/min in 0.01 mL/min increments without changing

Range pump heads

Flow Precision <0.1% typical

Flow Accuracy <0.1% typical

Pressure Ripple <1% at 13.8 MPa (2000 psi) and 1.0 mL/min

Eluent On/Off Standard feature

Valve

Dual-pump head; wash can be continuous when connected to rinse **Piston Seal**

Wash solution supply

Pressure Alarm Upper limit: 0 to 35 MPa or 0 to 5000 psi in one unit (MPa or Limits

psi) increments

Lower limit: Up to one unit lower than upper limit

Vacuum Degas Optional feature; automatic control

Eluent Bottles Polypropylene

Eluent Bottle Not required

Pressure

Leak Detection Built-in optical sensor

A.6 Eluent Regeneration

Eluent Optional feature; RFIC-ER Startup Kit required

Regeneration

Eluents • Carbonate and carbonate/bicarbonate up to 20 mM

• MSA up to 34 mM

Flow Rates 0.01 to 2.00 mL/min

Continuous Up to 28 days or 2000 samples, typically

Operation

(4 L of Eluent)

Always On/ Standard feature

Always Ready

Capable

Remains Fully Standard feature; results are traceable to a single calibration

Calibrated for

Extended Periods

(**≤28 days**)

System Wellness Consumables usage monitoring for predictive maintenance

Maximum 21 MPa (3000 psi)

Operating

Pressure

Operating 4 to 40 °C (40 to 104 °F)

Temperature

Range

A.7 Detector Electronics

Type Microprocessor-controlled digital signal processor

Cell Drive 8 kHz square wave

Detector

1% up to 1 mS

Linearity

Resolution 0.00238 nS/cm

Full-Scale Output Ranges • **Digital signal range:** 0 to 15,000 μS

• Analog signal range: 0 to 15,000 µS

Electronic Noise

 ± 0.1 nS/cm when background conductivity is 0 to 150 $\mu S/cm$ ± 2 nS/cm when background conductivity is 151 to 3200 $\mu S/cm$

Filter Rise times from 0 to 10 s; user-selectable

Temperature Compensation

Fixed at 1.7% per 1 °C at cell temperature

Temperature

Range

Stability

Ambient +7 °C; 30 to 55 °C

Temperature

<0.01 °C

A.8 Conductivity Cell with Heat Exchanger

Cell Body Chemically inert polymeric material

Cell Electrodes Passivated 316 stainless steel; compatible with MSA

Cell Volume <1 μL

Maximum Cell

10 MPa (1500 psi)

Operating Pressure

Heat Exchanger Inert, tortuous path for low axial dispersion

Operating 30 to 55 °C (86 to 130 °F); must be at least 7 °C above the ambient

Temperature temperature

A.9 Injection Valve

Injection Valve 6-port, 2-position Rheodyne valve with PEEK wetted components;

electrically-activated

A.10 Auxiliary Valve (Optional)

Auxiliary Valve 6-port or 10-port, 2-position Rheodyne valve with PEEK wetted

components; electrically-activated

A.11 Vacuum Degas Assembly (Optional)

Channel Single-channel degas channel with degas membranes

Pump Dual-stage diaphragm vacuum pump

Materials Wetted materials, PEEK, PTFE

A.12 Column Heater (Optional)

Operating 30 to 60 °C (86 to 140 °F); minimum 5 °C above ambient **Temperature** temperature. Settable range is equal to working range.

Temperature ±0.5 °C at sensor

Stability

Temperature ± 0.5 °C at sensor, at 40 °C

Accuracy

Maximum 250 mm analytical column with 50 mm guard column

Column Lengths

A.13 Suppressors

Chemical Suppression

2 mm and 4 mm anion and cation, membrane suppression bed types

Displacement Chemical Regeneration

2 mm and 4 mm anion and cation, membrane suppression bed types

Electrolytic Suppression

• **Self-Regenerating:** 2 mm and 4 mm anion and cation; both membrane and MonoDiskTM suppression bed types available

• Self-Regenerating with External Water Mode: 2 mm and 4 mm anion and cation; both membrane and MonoDisk suppression bed types available

Current Control

SRS (4 mm): 0 to 300 mA in 1 mA increments SRS (2 mm): 0 to 100 mA in 1 mA increments

AES: 0 to 150 mA in 1 mA increments CMD: 0 to 500 mA in 1 mA increments SRN: 0 to 500 mA in 1 mA increments

Salt Converter

2 mm and 4 mm versions available

Dionex AMMS

2 mm and 4 mm versions available

 ICE^{TM}

Anions

Range

Carbonic Acid Removal for ASRS [™] 300 with CRD 200 for hydroxide eluents ASRS 300 with CRD 300 for carbonate eluents

Non-Suppressed

Supported

Suppressor Wear Parts None; peristaltic pump and inline filters not required

Suppression Capacity

Cation SRS 300 (4 mm): 110 μeq/min Anion SRS 300 (2 mm): 50 μeq/min Cation SRS 300 (2 mm): 37.5 μeq/min Anion MMS 300 (4 mm): 150 μeq/min Cation MMS 300 (4 mm): 150 μeq/min Anion MMS 300 (2 mm): 37.5 μeq/min Cation MMS 300 (2 mm): 37.5 μeq/min Cation MMS 300 (2 mm): 37.5 μeq/min

Anion SRS 300 (4 mm): 200 μeg/min

Anion AES: 25 μeq/min Cation AES: 25 μeq/min

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Void Volume SRS 300 (4 mm): <50 µL

> SRS 300 (2 mm): <15 µL MMS 300 (4 mm): $<50 \mu L$ MMS 300 (2 mm): $<15 \mu L$ AMMS ICE 300 (4 mm): <50 μL AMMS ICE 300 (2 mm): <15 μL

Anion AES: <35 µL Cation AES: <35 µL

A.14 Autosampler

Automation Dionex AS, Dionex AS-AP, Dionex AS-DV, or Dionex AS-HV

Using Autosampler (or third-party autosampler)

Autosampler

Sequential/ May be available; depends on autosampler capabilities **Simultaneous**

Injection

Automated Available with a Dionex AS Autosampler Dilution

Dilution Time: 15 seconds with sample overlap

Online Sample Optional; CRD 200/300 required

Degassing Online Filtration Available with a Dionex AS-DV Autosampler or an inline filter

Dilution Factor: 1:1 to 1:1000

High, using Chromeleon software and post-run features Automation

Flexibility

A.15 System Software

Chromeleon Chromatography Management System or Chromeleon Software

Xpress; validated for use with Windows Vista or Windows XP

Automated Standard feature

Procedure

System Wellness Standard feature

Wizards

Performance

and Predictive

Data Trending Numerical device parameters can be plotted **Plots**

Virtual Column • Evaluation mode: Standard feature

• Isocratic and gradient modes: Optional features

Application Standard feature
Templates

Automation Fully controls over 300 different instruments from more than 30

Support for manufacturers, including GC, HPLC, and MS

Third-Party Vendors

3D Software for Optional feature

and ED

Panels

PDA, MS,

Customizable Standard feature

System Control

System Status Standard feature

Virtual Channels

Power Failure Standard feature

Protection

Sequential Standard feature **Injection**

System Trigger Standard feature

Commands and Conditionals

Daily Audit Trail Standard feature

Sample Audit Standard feature

Trail

Multiple Optional feature

Network Control and Network

Failure Protection

Settings

Storage of Factory, current, and previous calibration values are stored; the user

System can reset current values to factory or previous values **Calibration**

Dionex ICS-1100 Ion Chromatography System

Customized Standard feature; unlimited report workbooks **Reporting**

Automated Detailed, comprehensive qualification reports

System

Qualification

Oetailed, comprehensive qualification

B.1 TTL and Relay Connections

A 12-pin connector strip for TTL/relay control is located on the Dionex ICS-1100 rear panel. The connector provides two relay outputs, two TTL outputs, and four TTL inputs (see <u>Figure B-1</u>).

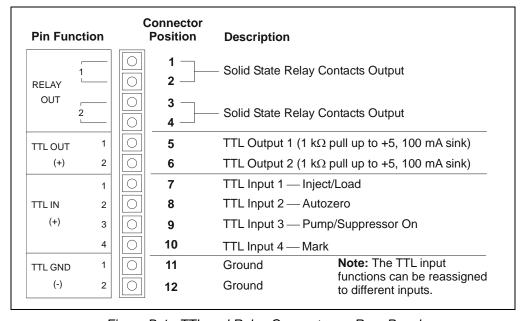


Figure B-1. TTL and Relay Connector on Rear Panel

IMPORTANT

Relay loads in excess of 200 mA or with included power supplies over 60 V may damage the relay drivers on the CPU.

NOTE TTL 1 Input - Inject/Load applies only to the injection valve (not to the auxiliary valve, if installed).

The outputs can be used to control functions in external devices such as an autosampler or another Dionex module. When connected to a controlling device, the inputs can be programmed to perform the following Dionex ICS-1100 functions:

- Switch the injection valve position (load/inject)
- Perform an autozero command (set the conductivity to zero)
- Turn the pump on and off (also turns the suppressor on and off)
- Send a chart mark signal to the analog output. The mark is 10% of the full-scale voltage, and the duration is 0.5 seconds. A mark can be used, for example, to indicate the injection.

Relay outputs 1 and 2 can be programmed to switch any low-voltage control. Switched current must be less than 200 mA and 42 V peak.

Connecting a TTL or Relay

- 1. Locate the twisted pair of wires (P/N 043598) and the 12-position connector plug (P/N 923687) (see Figure B-2) in the Dionex ICS-1100 Ship Kit (P/N 057905).
- 2. Follow these basic steps to connect the TTL or relays.

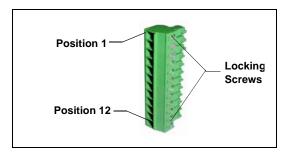


Figure B-2. 12-Position Connector Plug

a. For each relay or TTL to
 be used, connect an active wire (red) and a ground wire (black) to the 12-position connector plug at the appropriate pin locations. Refer to
 Figure B-1 or the label on the Dionex ICS-1100 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 TTL input/output ground pin.

IMPORTANT

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

- b. Plug the connector into the 12-pin connector on the Dionex ICS-1100 rear panel.
- c. Connect the wires from the Dionex ICS-1100 connector plug to the TTL or relay connector pins on the other module(s). 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.

3. 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 Chromeleon (see Section B.1.1).

B.1.1 Selecting TTL Input Functions and Control Types

To select TTL input functions and the control type, open the Chromeleon Server Configuration program and double-click the Dionex ICS-1100 icon under the timebase. Select the **TTL Inputs** tab (see Figure B-3).

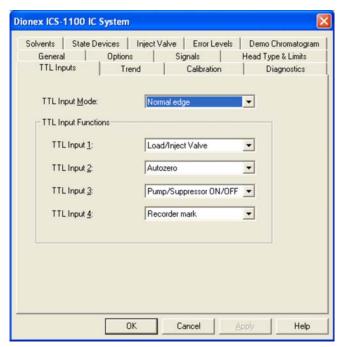


Figure B-3. Dionex ICS-1100 Server Configuration Properties: TTL Inputs

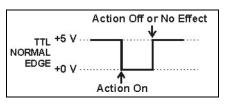
TTL Input Control Types



The Dionex ICS-1100 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.

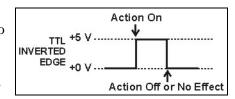
If the device connected to the Dionex ICS-1100 does not send a normal edge signal, select the appropriate control type. Refer to the documentation provided with the controlling device and the information below to select the correct type.

Normal Edge: In normal edge operation, the negative (falling) edge of a signal turns on the function. For example, for the Load/Inject function, the negative edge switches the injection valve position to Load.

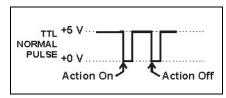


The action of the positive (rising) edge depends on the function: For the **Load/Inject** function, the rising edge switches the injection valve to the Inject position. For the **Pump On** function, the rising edge turns off the pump (and suppressor). For **Autozero** and **Mark**, the rising edge has 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.

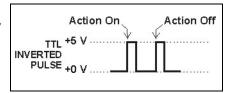


 Normal Pulse: In normal pulse operation, the negative (falling) edge of the TTL signal is the active edge and the positive (rising) edge is ignored.



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.



B.2 Controlling TTL and Relay Outputs

The Dionex ICS-1100 provides two TTL outputs and two relay contacts for control of functions in external devices, such as an integrator or autosampler. The relay outputs can be used to switch any low-voltage control. Switched current must be less than 200 mA and 60 V peak blocking. The relay-contact closures are normally open. When the relay is closed, current flows to the connected device.

IMPORTANT

Relay loads in excess of 200 mA or with included power supplies over 60 V may damage the relay drivers on the CPU.

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

The TTL and relay output states can be controlled, either by issuing direct control commands from the Chromeleon Control panel or by including the commands in a control program.

C • Reordering Information

Part Number	Item	
Reservoirs		
046548	2-L plastic reservoir assembly (includes stopper and cap)	
039164	4-L plastic reservoir assembly (includes stopper and cap)	
Pump		
057937	Primary pump head assembly	
057938	Secondary pump head assembly	
045721	Outlet check valve assembly, 10-32	
045722	Inlet check valve assembly, 1/4-28	
045994	Check valve cartridges	
052840	Piston	
055870	Piston seal	
048722	Piston rinse seal	
055752	O-ring for waste valve or priming valve	
057945	Eluent valve	
079803	10 mL syringe (for priming eluent lines)	
	Sample Loop and Injection Valve	
042857	25-μL sample loop	
057968	Injection valve	
024305	Luer adapter fitting, 1/4-28 (for manual injections)	
016388	1-mL syringe (for manual injections)	
057896	Injection Valve Rebuild Kit	
Auxiliary Valve		
069472	Auxiliary Valve Kit (6-port valve)	
069473	Auxiliary Valve Kit (10-port valve)	
057896	Injection Valve Rebuild Kit (for use with auxiliary valve, also)	

Part Number	Item	
061947	Auxiliary valve pod assembly (6-port valve)	
061948	Auxiliary valve pod assembly (10-port valve)	
07.44.5	Suppressors, Cell, and Column Heater	
056116	Dionex AAES Anion Atlas Electrolytic Suppressor	
056118	Dionex CAES Cation Atlas Electrolytic Suppressor	
064554	Dionex ASRS 300 4-mm Anion Self-Regenerating Suppressor	
064556	Dionex CSRS 300 4-mm Cation Self-Regenerating Suppressor	
064555	Dionex ASRS 300 2-mm Anion Self-Regenerating Suppressor	
064557	Dionex CSRS 300 2-mm Cation Self-Regenerating Suppressor	
057985	Dionex DS6 heated conductivity cell	
069564	Column heater assembly	
059979	Column heater heat exchanger, 0.25-mm (0.010-in) ID tubing	
060943	Column heater heat exchanger, 0.125-mm (0.005-in) ID tubing (for use with 2-mm columns)	
079910	Column heater cover assembly	
052324	Microbore tubing kit	
	Relay/TTL	
923686	12-position connector plug	
043598	Twisted pair of wires	
RFIC-ER Kits		
069570	RFIC-ER Anion Startup Kit, ICS-1100/ICS-1600	
067791	RFIC-ER Anion Consumables Kit, ICS-1100/ICS-1600	
069569	RFIC-ER Cation Startup Kit, ICS-1100/ICS-1600	
067792	RFIC-ER Cation Consumables Kit, ICS-1100/ICS-1600	
070061	ER Tubing Kit	
Miscellaneous		
954745	IEC 127 fast-blow fuses, rated 3.15 amps	
060494	USB cable, 5 m (16 ft)	
060392	External USB hub	

Part Number	Item
	Preventive Maintenance Kits
057954	Dionex ICS-1100 Preventive Maintenance Kit
060581	Dionex AS Preventive Maintenance Kit
055647	Dionex AS-DV Preventive Maintenance Kit

D.1 How do I hook up an autosampler?

For instructions on how to connect the Dionex ICS-1100 to an autosampler, refer to the Dionex ICS-1100 installation instructions. Also refer to the autosampler operator's manuals, which are included on the Thermo Scientific Reference Library DVD (P/N 053891).

D.2 How do I print?

Click the **Print** toolbar button in Chromeleon.

D.3 Why are the retention times moving?

Retention times can shift if the pump flow is erratic or if the column or eluent is contaminated. See Section 4.7 for pump flow rate troubleshooting. If a contaminated column is suspected, clean the column as instructed in the column manual. The column manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

D.4 How do I adjust retention times?

Retention times are calculated during calibration. The **Use Recently Detected Retention Time** parameter in the Chromeleon QNT Editor (**General** tab) can be used to compensate for some types of retention time drifts; for example, evaporation of volatile components in pre-mixed solvents or an aging column. Refer to the Chromeleon Help or user's manual for details.

D.5 When should I remake standards?

Standards are used only for calibration and should always be made fresh (they have a lifetime of only one week).

D.6 When should I remake eluents?

Eluents should be remade every two or three weeks.

D.7 How do I start Chromeleon?

Click **Start** on the Windows taskbar, and then select **All Programs** > **Chromeleon** > **Chromeleon**.

D.8 How do I delete data?

In the Chromeleon Browser, highlight the sequence you want to delete and then select **File > Delete**.

D.9 How do I back up data?

In Chromeleon, select **File > Export/Backup**. Back up the data and indicate the backup source.

D.10 How do I shut off the system?

In Chromeleon, click the **System Shutdown** button on the Dionex ICS-1100 Control panel. On the instrument, turn off the power switch on the rear panel (see <u>Figure 2-6</u>).

D.11 How do I store columns?

Columns should be stored in eluent. See the column manual for complete instructions. The column manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).

D.12 How do I know when a column is dirty?

See the troubleshooting section of the column manual.

D.13 How do I clean a column?

See the troubleshooting section of the column manual.

D.14 Why is the conductivity high?

Possible reasons for high conductivity include:

- The suppressor is not on. Turn on the suppressor from the Chromeleon Control panel.
- The suppressor needs regeneration. See the suppressor manual for troubleshooting information. The suppressor manual is included on the Thermo Scientific Reference Library DVD (P/N 053891).
- The cell is out of calibration. See <u>Section 5.1.2</u> for calibration instructions.
- See <u>Section 4.13</u> for additional troubleshooting information.

D.15 How do I configure and operate the auxiliary valve?

See *Installing the ICS-1100/ICS-1600/ICS-2100 Auxiliary Valve* (Document No. 065288), provided in the Auxiliary Valve Kit.

Analytical Column

Synonymous with **Separator Column**.

Band Spreading

The broadening of the sample band as it travels through the column. Band spreading can also occur in the injection valve, detector cell, and interconnecting tubing.

Calibration Curve

A graph showing detector response in peak height or area versus analyte concentration.

Capacity Factor (k')

The number of column volumes of eluent, pumped through the column, required to elute an analyte. Capacity factor is a dimensionless measure of retention which is independent of column length or eluent flow rate. It is calculated as follows:

$$k' = \frac{t_r - t_o}{t_o}$$

Where: t_r = retention time

t_o = retention time of unretained solute (column void volume)

Cell Constant (k)

A factor determined experimentally by measuring the conductance (G) of a standard solution of known equivalent conductivity (k).

$$k = \kappa/G$$

The value of k depends upon the surface area of, and distance between, the electrode faces in the conductivity detector cell.

$$k = l/A$$

Where: l = length

A = area of one electrode (the other electrode is equal to the first)

Channeling

The preferential flow of liquid along more open, less resistant paths through the column packing. This causes **Band Spreading**.

Column Efficiency (N)

A measure of the *narrowness* of analyte bands as they elute from the column. High efficiency is desirable because resolution between closely spaced bands improves with greater efficiency. For a symmetrical (Gaussian) peak, column efficiency can be determined by the following:

$$N = 5.54(t_1/W_{1/2})^2$$

Where: t_1 = the peak retention time (in seconds)

 $W_{1/2}$ = the peak width at 1/2 height (in seconds)

Column efficiency is proportional to column length: for a given resin and column diameter, increasing the column length increases the column efficiency. Synonymous with **Theoretical Plates**.

Column Selectivity (a)

Describes the relative separation of the band maxima between two adjacent peaks. Selectivity can be determined by the following:

$$a = (t_2 - t_0)/(t_1 - t_0)$$

Where: t_1 and t_2 = retention time of components 1 and 2, respectively t_0 = retention time of unretained components (void volume)

Concentrator Column

A short column used to retain and concentrate analytes from a measured volume

of relatively clean sample. This allows large volumes of sample to be injected, lowering concentration detection limits.

Conductivity

A measure of the ease with which electrical current flows through a liquid contained between two opposite charged electrodes. Conductivity is a characteristic of ions in solution. Units are siemens.

Counterion

Ions carrying a charge opposite that of the sample ions (e.g., Na⁺) may be the counterion of a Cl⁻ analyte. These ions preserve electrical neutrality in solution.

% Crosslink

Divinylbenzene content in a polystyrene/divinylbenzene (PS-DVB) resin; this contributes to the mechanical strength of the resin and determines chromatographic properties.

Equivalent Conductivity (λ)

The contribution of an ionic species to the total conductivity of a solution as measured in a standard cell having electrodes 1 cm² in area and exactly 1 cm apart.

Guard Column

A small column that prevents poisoning of the separator column by sorbing organic contaminants and removing particulates. It is filled with the same packing as the separator column. Synonymous with **Pre-Column**.

HETP (H)

<u>H</u>eight <u>E</u>quivalent to a <u>T</u>heoretical <u>P</u>late. A measure of column efficiency which allows comparison between columns of different lengths.

$$HETP = H = L/N$$

Where: L =the column length (in mm)

N = the number of theoretical plates

Ion-Exchange Capacity

The number of active ion exchange sites in a given weight or volume of resin; this is usually expressed in meq/g or meq/mL.

Ion-Exchange Resin

An insoluble polymer matrix containing fixed-charge exchange sites (anionic or cationic). IC resins are formed into small spherical particles (beads).

Packing

The material that fills a chromatographic column; usually a resin or silica-based material.

Pellicular Resin

A resin with a solid, nonporous core coated with a thin layer of more porous material. The exchange sites of pellicular ion exchange resins are located only on the surface layer of the bead. These resins have a low ion-exchange capacity.

Pre-Column

Synonymous with **Guard Column**.

Regenerant

A dilute acid or base that converts ion exchange sites in a MicroMembrane Suppressor back to the form that suppresses the eluent conductivity.

Resin

See Ion-Exchange Resin.

Resolution (R)

A measure of the separation between two sample components. This is expressed as the ratio of the distance between the two peak maxima to the mean value of the peak width at the baseline.

$$R = 2(t_2 - t_1)/(W_2 + W_1)$$

Where: t_1 and t_2 =the retention times of components 1 and 2, respectively W_1 and W_1 = the baseline width of peaks 1 and 2, respectively (measured in the same units as the retention time)

R is proportional to the square root of efficiency (N). A value of R = 1.5 represents "baseline separation" of the two peaks.

Retention Time

The time from injection to peak maximum; the basis for identification of a species in chromatographic analysis.

Separator Column

The column used to perform a chromatographic separation; also called an analytical column.

Siemens (S)

Unit measure of conductance; the reciprocal of the electrical resistance of a solution.

Suppressor

A device used to minimize eluent conductivity and convert sample species to a common form, thus increasing detection sensitivity.

Temperature Coefficient

The percent of change in the conductivity of a solution with a 1 °C change in temperature. Every solution has a characteristic temperature coefficient which is determined experimentally.

Theoretical Plates (N)

See Column Efficiency.

Void Volume (V_0)

The volume occupied by the eluent in a packed column. This volume includes the volume between the injection valve and the column, as well as between the column and the detector cell. Unretained components are eluted in the void volume.

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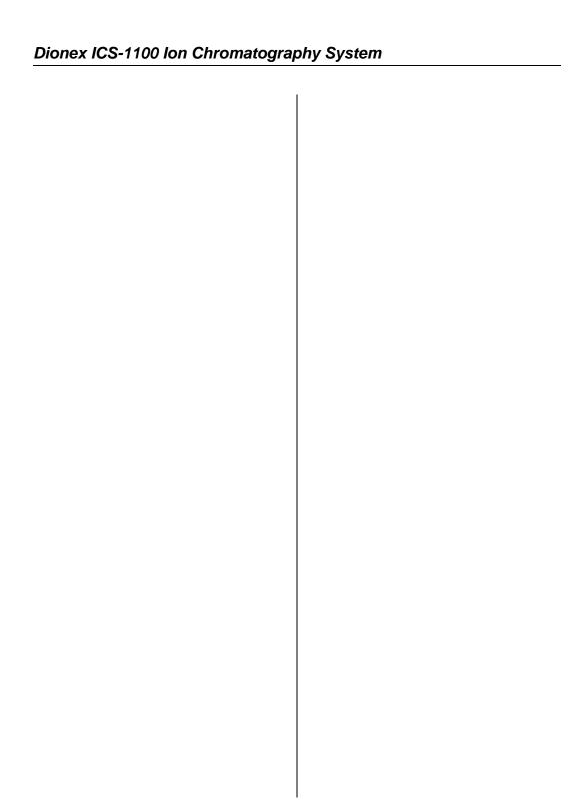
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