DIONEX

ICS-90 Ion Chromatography System
Operator's Manual

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Document No. 031851
Revision 08
December 2006

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PRINTING HISTORY

Revision 01, April 2002
Revision 02, August 2003
Revision 03, April 2004
Revision 04, July 2004
Revision 05, December 2004
Revision 06, September 2005
Revision 07, December 2005
Revision 08, December 2006

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1 • Introduction

1.1 Introduction to Ion Chromatography (IC)

NOTE **Appendix E contains a glossary of chromatography terms.**

The Dionex ICS-90 Ion Chromatography System (ICS-90) performs isocratic ion analyses using suppressed conductivity detection. An ion chromatography system typically consists of a liquid eluent, a high-pressure pump, a sample injector, a separator column, a chemical suppressor, and a conductivity cell. Before running a sample, the ICS-90 is calibrated using a standard solution. By comparing the data obtained from a sample to that obtained from the standard, sample ions can be identified and quantitated. A computer running chromatography software automatically converts each peak in a chromatogram to a sample concentration and produces a tabulated printout of the results.

The IC analysis consists of four stages (see Figure 1-1):

1. **Eluent Delivery**
    - Eluent, a liquid that helps to separate the sample ions, carries the sample through the ion chromatography system. The ICS-90 is an isocratic delivery system. This means that the eluent composition and concentration remain constant throughout the run.
    - Liquid sample is injected into the eluent stream either manually or automatically (if an automated sampler is installed).
    - The pump forces the eluent and sample through a separator column (a chemically-inert tube packed with a polymeric resin).
2. **Separation**
    - As the eluent and sample are pumped through the separator column, the sample ions are separated. In the ICS-90, the mode of separation is called ion exchange and it 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.

3. Detection

- 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.
- A conductivity cell monitors and 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.

4. Data Analysis

- The conductivity cell transmits the signal to a computer running chromatography software.
- The chromatography software analyzes the data by comparing the sample peaks in a chromatogram to those produced from a standard solution. The software identifies the ions based on retention time, and quantifies each analyte by integrating the peak area or peak height. The results are displayed as a chromatogram, with the concentrations of ionic analytes automatically determined and tabulated.

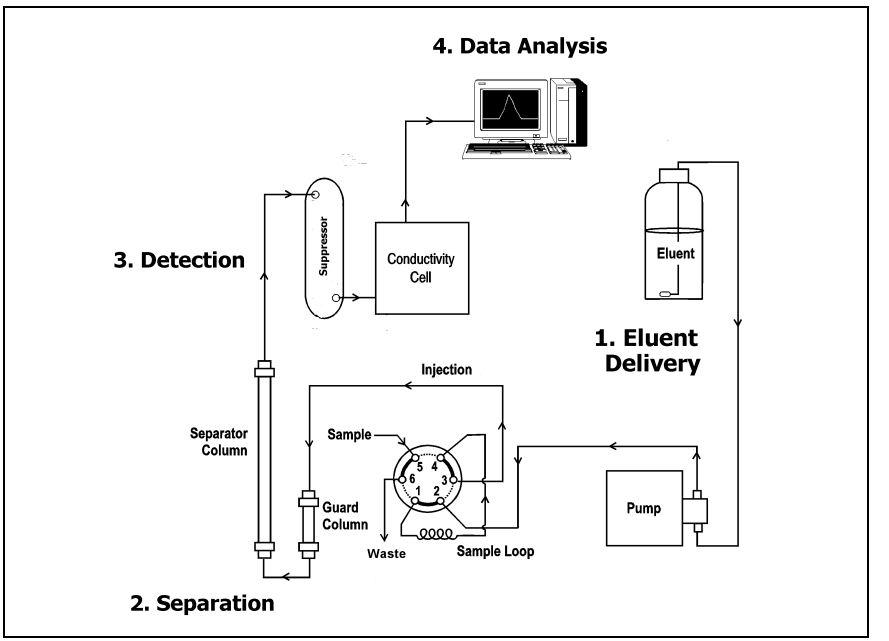


Figure 1-1. Ion Analysis Process

1.2 Overview of the ICS-90

In all major respects, the ICS-90 operating process is similar to the overview of ion analysis described in Section 1.1.

The ICS-90 is an integrated ion chromatography system containing a pump, an injection valve, and a conductivity cell. Other system components, including a guard column, separator column, and suppressor, must be ordered separately. These components vary, depending on the analyses to be performed. System components are described and illustrated in Section 2.1.3.

ICS-90 operation is controlled remotely by a PC (personal computer) running Microsoft® Windows® XP or Windows® 2000 operating system and either the Chromeleon® Chromatography Management System or Chromeleon Xpress chromatography software. Chromeleon provides all of the system control features found in Chromeleon Xpress, plus complete data acquisition and data processing functions.

For communication between the ICS-90 and the PC on which Chromeleon or Chromeleon Xpress is installed, the ICS-90 must be connected to a USB (Universal Serial Bus) port on the PC or a USB hub. For installation instructions, see Appendix B of this manual.

Product Versions

A newer version of the ICS-90, called the ICS-90A, has a feature that allows the approximate flow rate to be displayed in Chromeleon or Chromeleon Xpress (see Section B.15). Unless otherwise specified, all of the information in this manual applies to both the ICS-90 and the ICS-90A. If you are unsure which system you have, check the model data label on the rear panel.

1.3 About This Manual

Chapter 1 Introduction	An introduction to ion analysis and the ICS-90. Explanations of the conventions used in this manual, including safety-related information.
Chapter 2 Description	Descriptions of ICS-90 operating features and the chromatographic flow path. An introduction to the software required for ICS-90 control.
Chapter 3 Operation and Maintenance	Instructions for routine operation with Chromeleon or Chromeleon Xpress. Routine preventive maintenance procedures for the ICS-90.
Chapter 4 Troubleshooting	Minor problems that may occur during operation, with step-by-step procedures for how to isolate and eliminate the cause of each problem. A list of Chromeleon and Chromeleon Xpress Audit Trail error messages, with an explanation of the possible cause of each message and the corrective action to take.
Chapter 5 Service	Step-by-step instructions for routine service and parts replacement procedures for the ICS-90.
Appendix A Specifications	Specifications and installation site requirements for the ICS-90.
Appendix B Installation	Installation instructions for the ICS-90.
Appendix C Reordering Information	Spare parts for the ICS-90.
Appendix D FAQ	Answers to frequently asked questions about ICS-90 operation.
Appendix E Glossary	Definitions of terms commonly used in ion analysis.

1.3.1 Safety Messages and Notes

The ICS-90 is designed for ion analysis applications and should not be used for any other purpose. If there is a question regarding appropriate usage, contact Dionex at 1-800-346-6390 before proceeding. Outside the United States, call the nearest Dionex office.

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, may 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.

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.

1.3.2 Safety Labels

The TUV GS, C, US Mark safety label and the CE Mark label on the ICS-90 indicate that the ICS-90 is in compliance with the following standards: EN 61010-1:2001 (safety), CAN/CSA-C22.2 No. 1010.1-92+A2:97 (safety), UL 3101-1/10.93 (safety), and EN 61326:1997+A1:1998 (EMC susceptibility and immunity).

The symbols below appear on the ICS-90 or on labels affixed to the ICS-90.

Alternating current

Protective conductor terminal

Power supply is on

Power supply is off

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

2 • Description

The ICS-90 Ion Chromatography System (ICS-90) is designed for simplicity and ease of use. It is configured for optimal performance at the factory. ICS-90 operation is controlled by Chromeleon or Chromeleon Xpress software. This chapter presents a brief description of key ICS-90 features and components and the software user interface.

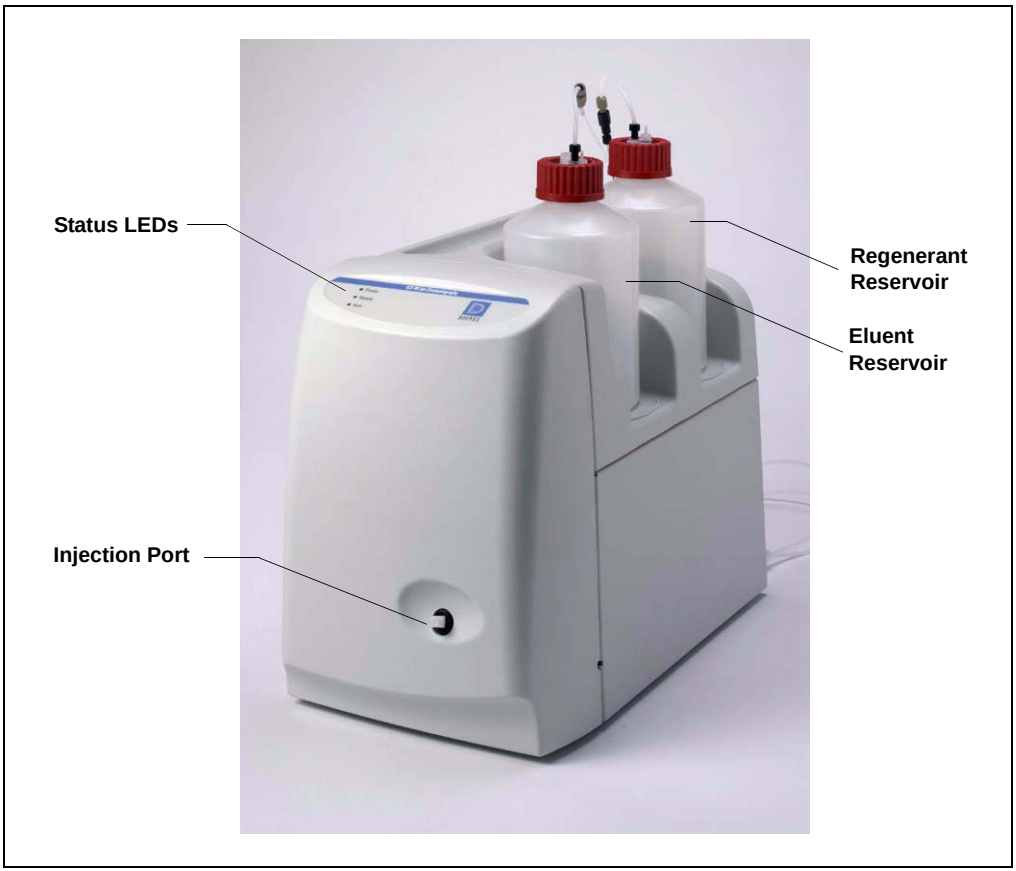


Figure 2-1. ICS-90 Ion Chromatography System

2.1 Operating Features

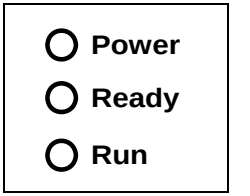
2.1.1 Front Door and Top Cover

Figure 2-1 illustrates the front door and top cover of the ICS-90.

The front door contains a sample injection port and LEDs that indicate the ICS-90 operating status. The top cover is molded to hold the eluent and regenerant reservoirs.

LEDs

There are three status LEDs on the ICS-90 front panel. Other status information and alarm messages are displayed in the Audit Trail in Chromeleon or Chromeleon Xpress. See Section 4.1 for a description of these messages.



LED Label	If On (Green)	If Flashing
Power	ICS-90 power is on	Does not flash
Ready	System check passed, but sequence not yet started (LED stays on until run starts or sequence is aborted)	System check failed (occurs if system check executes for 10 minutes without success)
Run	Running/acquiring data	Error/alarm/fault (including injection valve position)

Injection Port

The sample to be analyzed is injected into the injection port, using either a syringe or the AS40 Automated Sampler. For more information about sample injection, see Section 3.5.6.

Eluent and Regenerant Reservoirs

The ICS-90 top cover is molded to hold one eluent reservoir assembly (P/N 057711) and one regenerant reservoir assembly (anion, P/N 057712; cation, P/N 057713).

- Eluent carries the sample through the ICS-90 and facilitates the ion separation process. The type of eluent used depends on the analyses performed. For example, an ICS-90 configured for anion analyses uses carbonate eluent, while an ICS-90 configured for cation analyses uses methanesulfonic acid (MSA) eluent.
- Regenerant “regenerates” the suppressor’s ability to suppress eluent conductivity. An ICS-90 configured for anion analyses uses dilute sulfuric acid regenerant. An ICS-90 configured for cation analyses uses tetrabutylammonium hydroxide (TBAOH) regenerant.

2.1.2 Rear Panel

Figure 2-2 illustrates the ICS-90 rear panel.

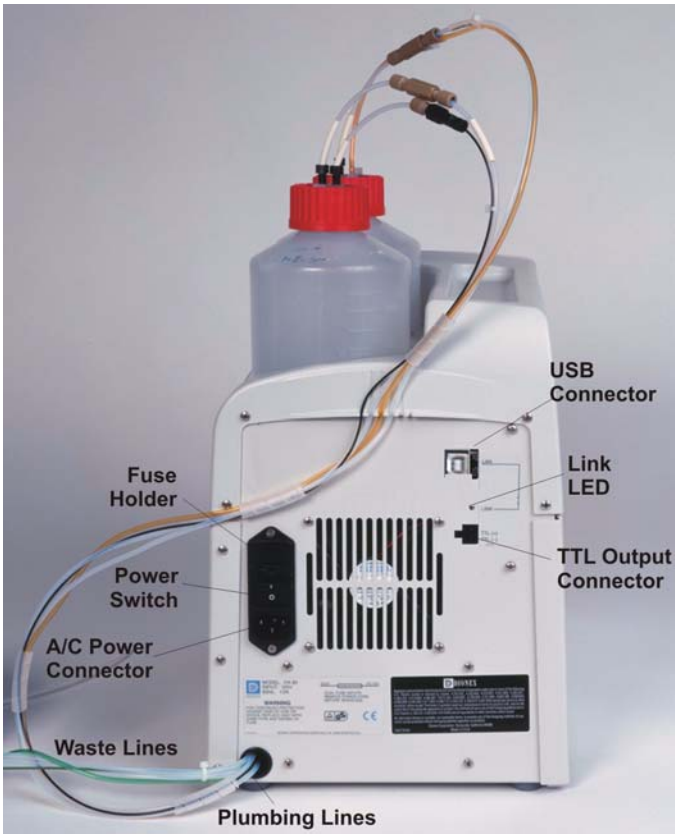


Figure 2-2. ICS-90 Rear Panel

Power Switch

The power switch provides on/off control of power to the ICS-90.

Main Power Receptacle

The AC power outlet is located directly below the power switch.

The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the ICS-90 and is easily accessible.

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

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

USB Port

The USB port provides a connection to the PC on which Chromeleon or Chromeleon Xpress is installed. For the standard system configuration of one ICS-90 connected to a PC, connect a USB cable between the USB connector on the ICS-90 and a USB port on the PC. See Section B.7.2 for detailed connection instructions.

Link LED

The Link LED indicates the communication status between the ICS-90 and the PC on which Chromeleon or Chromeleon Xpress is installed.

LED Name	Status	Description
Link	On	A link has been established between the ICS-90 and the PC, but no data is currently being transmitted or received
	Flashing	A link has been established between the ICS-90 and the PC and data is being transmitted
	Off	No link between the ICS-90 and the PC has been established

TTL Output Connector

The TTL output connector allows an AS40 Automated Sampler to receive TTL signals from the ICS-90. See Section B.18 for instructions on how to connect the AS40 to the ICS-90.

Tubing Connections

Eight lines exit the ICS-90 from the lower left corner of the rear panel. These lines include:

- 1 eluent line
- 1 regenerant line
- 1 cell outlet line
- 2 air lines
- 3 waste lines (sample, regenerant, and pump priming waste)

2.1.3 Component Mounting Panel

Figure 2-3 shows the user-accessible components installed inside the ICS-90. The component panel is accessed by opening the front panel door. All of the components that the user can service are located on this panel.

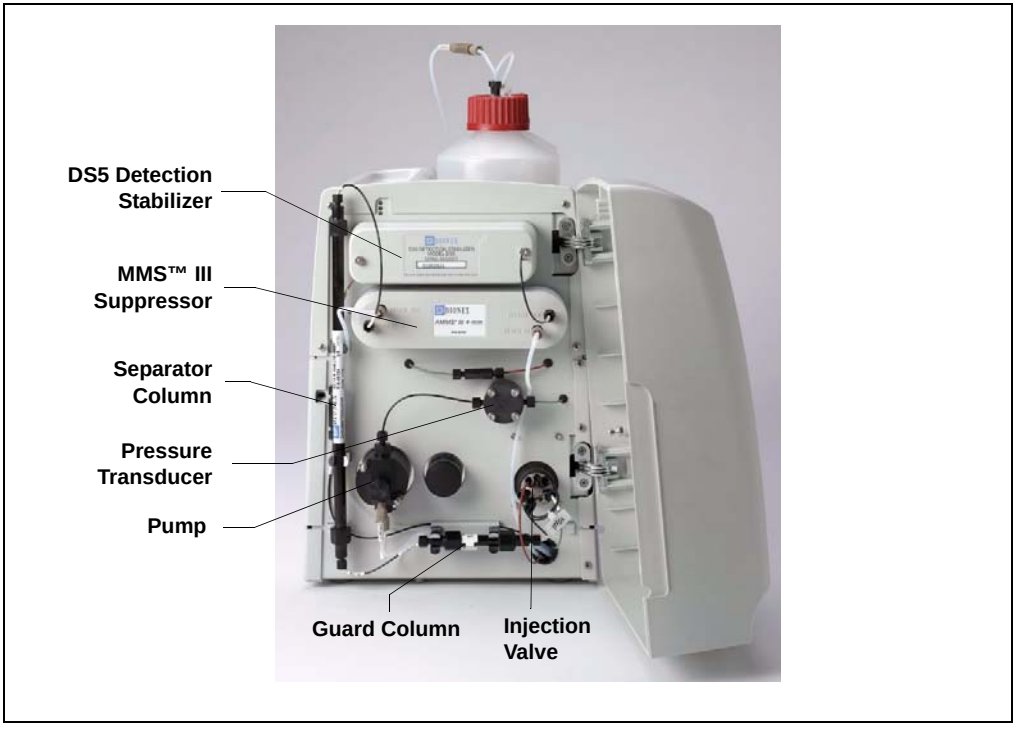


Figure 2-3. ICS-90 Component Mounting Panel

DS5 Detection Stabilizer

The DS5 Detection Stabilizer (P/N 057290) houses a flow-through conductivity cell. Two 316 stainless steel electrodes are permanently sealed into the PEEK cell body. The cell measures the electrical conductance of analyte ions as they pass through the cell.

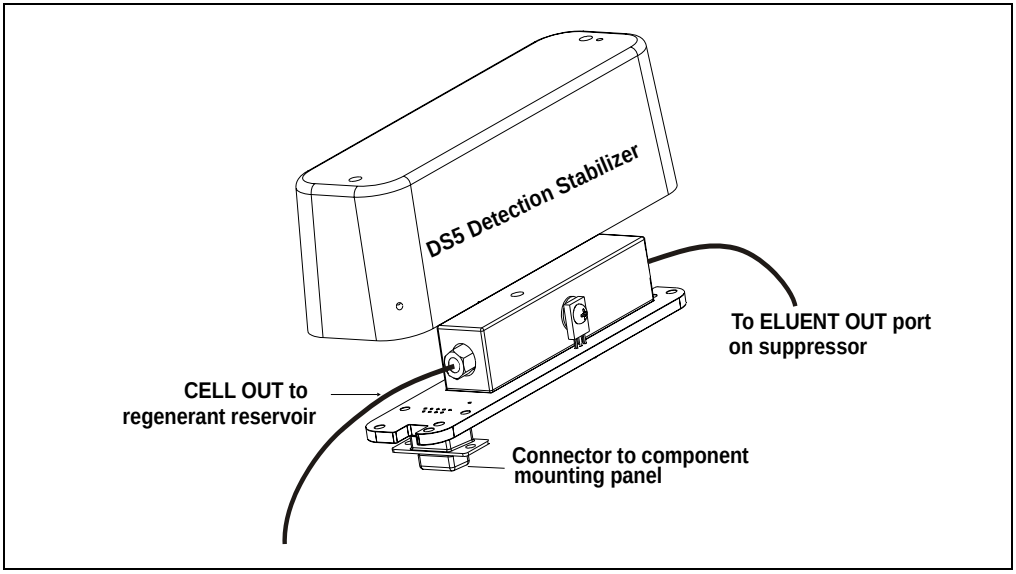


Figure 2-4. DS5 Detection Stabilizer

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. Built-in preset temperature compensation also ensures that there is no major change in the baseline or in peak heights. Temperature compensation further improves baseline stability.

Direct conductive heating is used in the ICS-90 to provide temperature control and compensation. A heat exchanger inside the ICS-90 cell regulates the temperature. All data is collected at 40 °C (104 °F).

MMS III Suppressor

The MMS III suppressor reduces the eluent conductivity and enhances the conductivity of the sample ions, thereby increasing detection sensitivity.

As illustrated in Figure 2-5, a constant flow of regenerant over the membrane continually restores the suppression ability of the MMS III.

A process called Displacement Chemical Regeneration (DCR) pushes regenerant from the regenerant reservoir and through the suppressor. See Section 2.2.1 for details.

For more information about the MMS III, see *Installation Instructions and Troubleshooting Guide for the Anion MicroMembrane Suppressor® III* (Document No. 031727) or *Installation Instructions and Troubleshooting Guide for the Cation MicroMembrane Suppressor® III* (Document No. 031728).

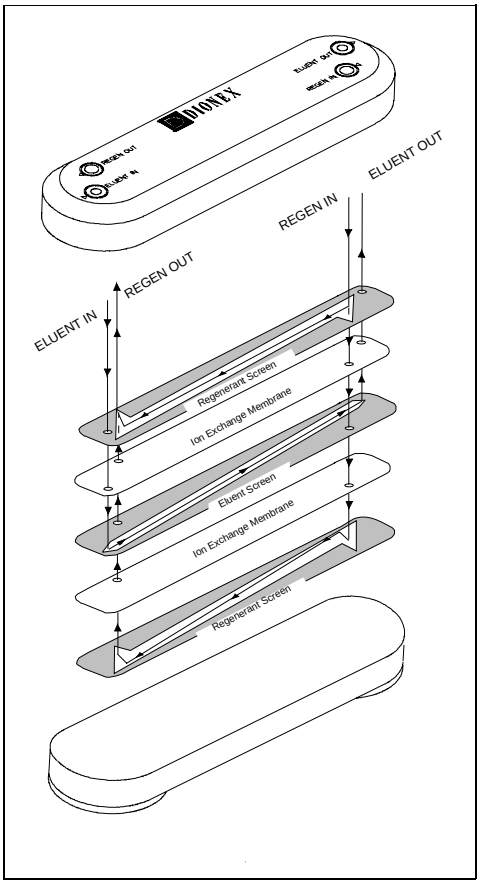


Figure 2-5. MMS III Suppressor Flow

Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

Pressure Transducer

The pressure transducer measures the system pressure at the point that the eluent flows from the pump head outlet check valve into the pressure transducer. Pressure measurements, which can be monitored from

Chromeleon or Chromeleon Xpress, indicate that the pumping system is delivering smooth, accurate flow. Pressure limits are used to detect high or low pressure conditions that might require the pump to be shut down.

The system backpressure should remain consistent. A significant increase in backpressure indicates a blockage in the liquid lines. A significant decrease in backpressure may indicate leaks or decreased pump flow due to an unprimed pump or insufficient eluent.

Injection Valve with Sample Loop

The injection valve is a six-port, electrically-activated Rheodyne valve. A 10 µL sample loop (P/N 042949) is installed on the valve at the factory.

The valve has two operating positions: Load and Inject (see Figure 2-6). 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.

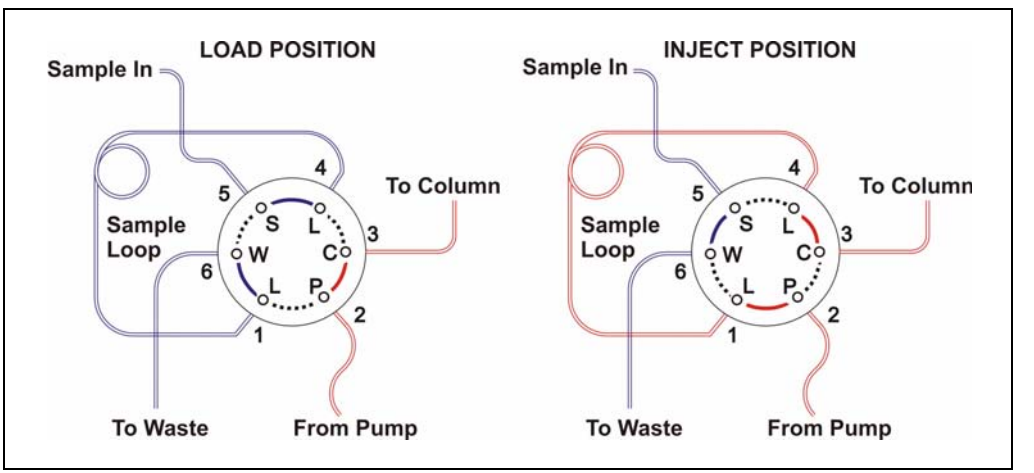


Figure 2-6. Rheodyne Injection Valve Flow Schematics

Pump

The ICS-90 uses a high-speed reciprocating pump with PEEK components. The flow rate can be set to between 0.0 and 5.0 mL/min. The recommended operating flow rate is between 0.5 and 2.0 mL/min.

Prime Valve

The prime valve is mounted on the pump head. The pump should be primed at installation and whenever the flow rate seems outside normal parameters. For priming instructions, see Section B.13.

Separator and Guard Columns

Both the separator and guard columns are packed with resin filling and perform the separation of the sample ions. However, the main function of the guard column is to absorb organic contaminants and remove particulates that might poison the separator column.

2.2 Fluid Schematic

Figure 2-7 shows the flow path through the ICS-90.

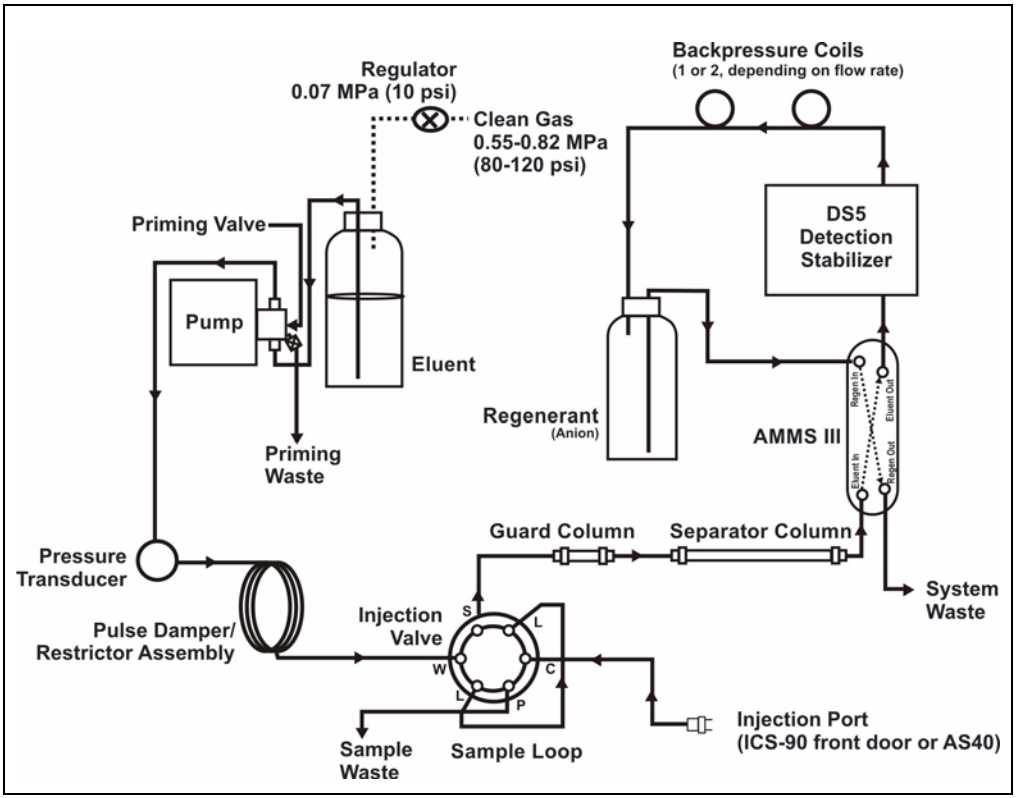


Figure 2-7. ICS-90 Flow Schematic

Liquid flows through the ICS-90 along the following flow path:

- Eluent from the eluent reservoir is pressurized by the gas, forced into the pump, and passes through the pressure transducer. From there, it is pushed through a pulse damper/restrictor assembly, which smooths minor pressure variations from the high-speed pump to minimize baseline noise. The eluent then flows into the injection valve.
- When sample is loaded into the sample loop, the injection valve toggles to the Inject position. This combines the injected sample with the eluent and pushes it through the sample loop.

- The eluent/sample mixture is pumped through the guard and separator columns, where the ions are separated by the ion exchange process (that is, different sample ions migrate through the columns at different rates, depending upon their interactions with the ion exchange sites).
- The eluent/sample mixture flows through the suppressor. There, detection sensitivity is enhanced by suppressing the conductivity of the eluent and enhancing the conductivity of the analyte. Regenerant flows continuously through the suppressor, restoring the ion exchange sites to their original state.
- The eluent/sample mixture then flows through the DS5, where the analytes are detected and a signal is produced and sent to Chromeleon or Chromeleon Xpress software.
- Finally, the eluent flows out of the cell and is directed into the regenerant reservoir, where it pressurizes the regenerant and forces it into the suppressor.

2.2.1 Displacement Chemical Regeneration (DCR)

Displacement Chemical Regeneration (DCR) is the process that regenerates the ability of the MMS III suppressor to suppress eluent. In DCR, the eluent/sample mixture that exits the cell is pumped into the regenerant reservoir. The eluent pressurizes the reservoir and pushes the regenerant into the suppressor. However, because the eluent is a different density than the regenerant, it remains separate.

In the anion DCR process (see Figure 2-8), the eluent/sample mixture is lighter than the regenerant and it remains on the top, forcing the regenerant to the bottom of the reservoir and out into the suppressor.

In the cation DCR process, the eluent/sample mixture is heavier than the regenerant and it flows to the bottom of the reservoir, displacing the regenerant and pushing regenerant out of the reservoir and into the suppressor.

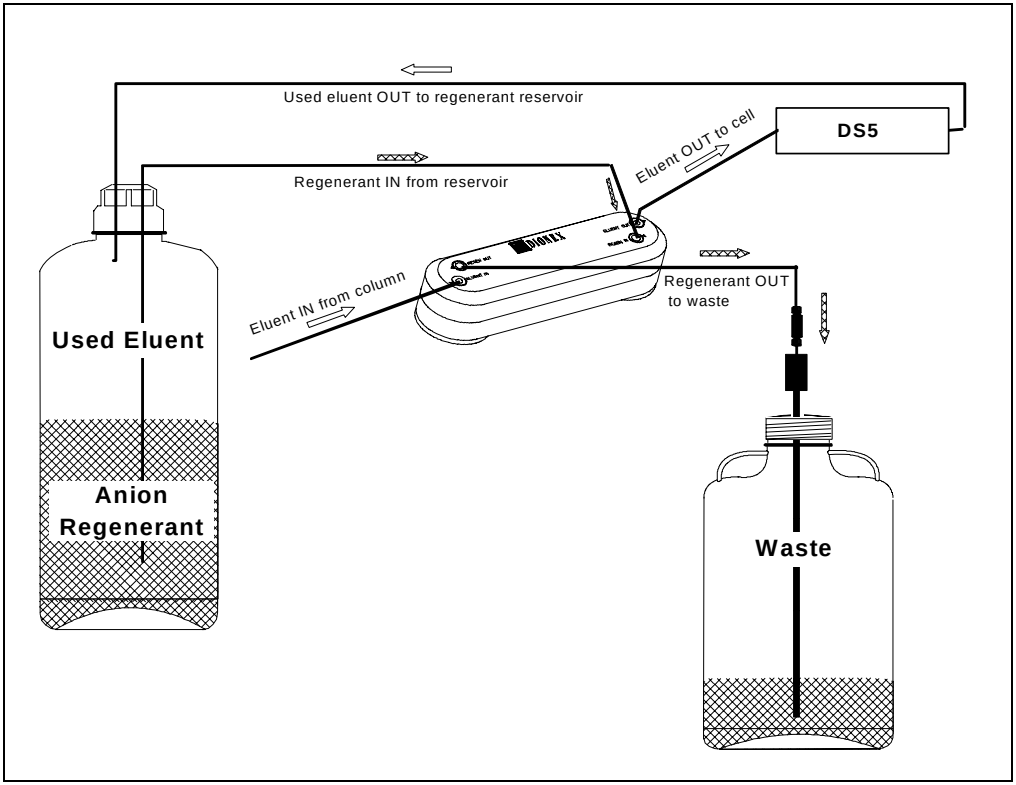


Figure 2-8. Anion ICS-90 Displacement Chemical Regeneration (DCR)

2.3 Chromeleon and Chromeleon Xpress Software

2.3.1 Overview

The ICS-90 can be controlled by either Chromeleon Xpress or the Chromeleon Chromatography Management System.

- Chromeleon Xpress is a system controller that replaces and combines the front panels of all system modules into one centralized system Control panel, called a *panel tabset*. The **ICS-90** tab provides access to ICS-90 functions, as well as to detailed status and diagnostics information for the system (see Figure 2-9).
- Chromeleon provides all of the system control features found in Chromeleon Xpress, plus complete data acquisition and data processing functions.

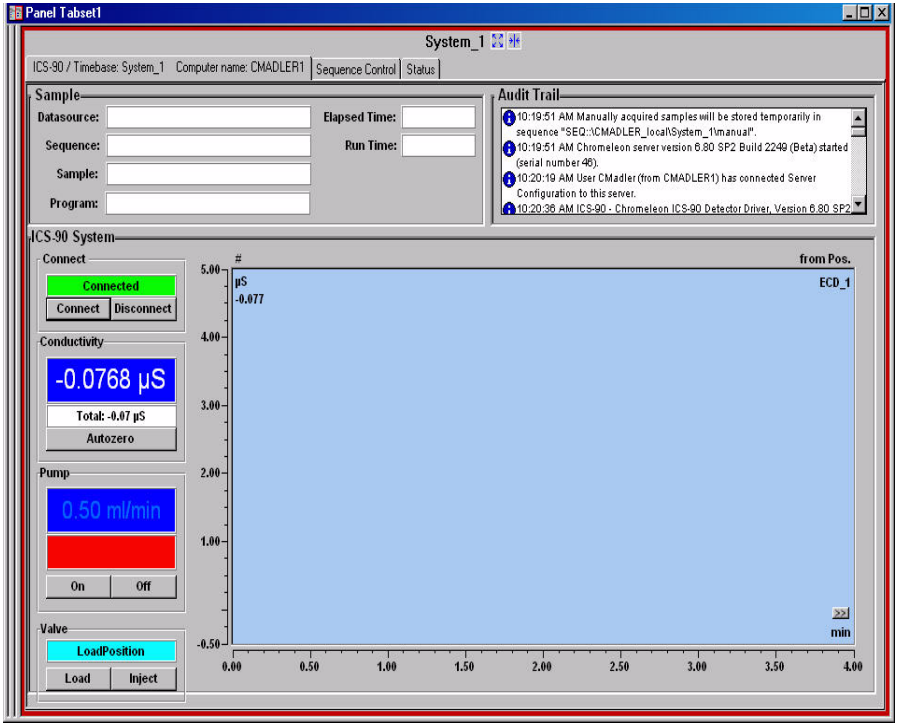


Figure 2-9. ICS-90 Control Panel on the Panel Tabset

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

- With *direct* control, you select operating parameters and commands from Control panels or the Commands dialog box. (To open the Commands dialog box, press F8.) Direct control commands are executed as soon as they are entered. See Section 3.5.4 for details about direct control.
- With *automated* control, you create a program that contains a list of control commands, to be executed in chronological order, for automated operation of the detector. Programs (sometimes called PGM files) can be created automatically (with the help of a software wizard) or manually (by editing an existing program). See Section 3.5.5 for details about automated control.

2.3.2 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.

You can perform diagnostic and calibration procedures and monitor ICS-90 functions from the Wellness panel (see Figure 5-1). In addition, all System Wellness parameters are available in the Commands dialog box.

For more information about System Wellness, refer to Section 5.1.

3 • Operation and Maintenance

Operation of the ICS-90 Ion Chromatography System (ICS-90) is controlled through Chromeleon or Chromeleon Xpress software. However, three functions take place at the instrument:

- Eluent and regenerant preparation and/or reservoir installation
- On/off power control
- Manual sample or standard injection

The ICS-90 is designed for IC (ion chromatography) applications and should not be used for any other purpose. Operation of the ICS-90 in a manner not specified by Dionex may result in personal injury.

3.1 Preparing the Eluent

For instructions on preparing the eluent, refer to the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

3.2 Preparing the Regenerant

The type of regenerant used with the ICS-90 depends on the type of analysis to be run. A dilute sulfuric acid regenerant is used for anion analyses; a tetrabutylammonium hydroxide (TBAOH) regenerant is used for cation analyses. Follow the instructions below to prepare either anion or cation regenerant.

1. Fill the regenerant reservoir (anion, P/N 057712; cation, P/N 057713) about halfway with ASTM filtered, Type I (18-megohm) deionized (DI) water.

For acid concentrates (such as the anion regenerant), always pour the concentrate into deionized water, not into the empty reservoir.

Pour les concentrés acides (comme le régénérant anionique), versez toujours le concentré dans de l'eau désionisée et non dans le réservoir vide.

VORSICHT

Gießen Sie bei säurehaltigen Konzentraten (beispielsweise dem Kationenelutionsmittel) das Konzentrat immer in entionisiertes Wasser und nicht in den leeren Behälter.

2. Empty the entire bottle of concentrate into the reservoir.
3. Fill the concentrate bottle with DI water and empty the contents into the reservoir.
4. Continue to pour DI water into the reservoir until it is filled all the way to the top. If a few drops spill over, then it is full enough.

IMPORTANT **The regenerant reservoir must be filled all the way to the top at all times.**

5. Insert the stopper assembly tubing into the reservoir and tighten the stopper.

IMPORTANT **Do not shake the regenerant reservoir to mix the contents. The ICS-90 will stratify the contents.**

For additional information, refer to the *Installation Instructions and Troubleshooting Guide for the Displacement Chemical Regeneration (DCR) Kit* (Document No. 031664). The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

3.3 Pressurizing the Eluent Reservoir

1. Before beginning operation, verify that the clean gas supply is regulated to between 0.55 and 0.83 MPa (80 and 120 psi) and is connected to the gas inlet of the ICS-90 air regulator (see Figure 3-1).
2. Verify that the gas line from the eluent reservoir is connected to the ICS-90 air regulator outlet line.
3. Make sure the eluent and regenerant reservoirs are filled, the caps are tightly screwed onto the reservoirs, and the lines connecting the eluent and regenerant reservoirs to the ICS-90 lines are securely connected.
4. Pull out the air regulator knob and turn it clockwise to pressurize the eluent reservoir.
5. Adjust the pressure to between 30 and 40 kPa (5 and 6 psi). Push the regulator knob back in.

CAUTION

Do not pressurize the eluent reservoir above 70 kPa (10 psi).

MISE EN GARDE

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

VORSICHT

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

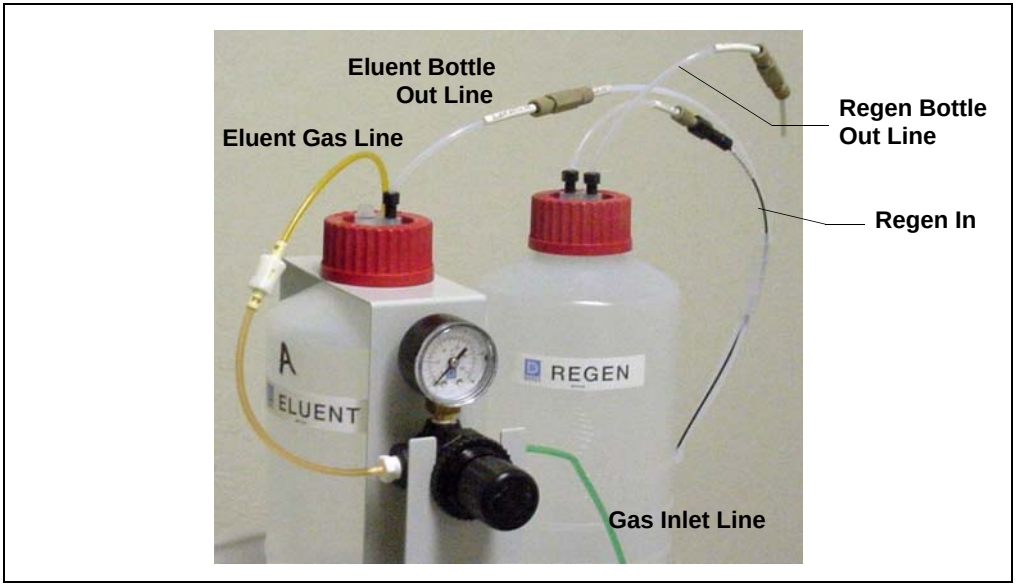


Figure 3-1. Reservoir Connections

3.4 Preparing Samples

3.4.1 Collecting and Storing

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 will 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 µm 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.

3.4.2 Pretreating

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. For instructions, refer to *Installation and Troubleshooting Guide for OnGuard Cartridges* (Document No. 032943). The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

3.4.3 Diluting

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 (DI) 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.

3.5 ICS-90 Operation

3.5.1 Starting Up the ICS-90

1. Make sure the eluent and regenerant reservoirs are filled, the caps are tightly screwed onto the reservoirs, and the lines connecting the reservoirs to the ICS-90 lines are securely connected (see Figure 3-1).
2. Make sure the clean gas inlet line is regulated to between 0.55 and 0.83 MPa (80 and 120 psi), and is connected to the gas inlet of the ICS-90 air regulator (see Figure 3-1).
3. Make sure the gas line from the eluent reservoir is connected to the ICS-90 air regulator outlet line.
4. Make sure the air regulator is adjusted to between 30 and 40 kPa (5 and 6 psi) (see Section 3.3).
5. Make sure the USB cable is connected to the USB port on the ICS-90 rear panel. See Section B.4 for installation instructions.
6. Press the power switch on the ICS-90 rear panel (see Figure 2-2) to turn on the system power. These are the conditions at power-up:
   - The pump is off.
   - The injection valve is in the Load position.
   - The DS5 is reading its current value.

3.5.2 Connecting to Chromeleon or Chromeleon Xpress

1. Turn on the computer.
2. Start the Chromeleon Server, if it is not already running:

   If the Chromeleon Server icon on the Windows taskbar is crossed out in red  , the Server is not running. Start the Server by right-clicking the icon and selecting **Start Server**. When the server is running, the icon is gray  .

   If the Server Monitor icon is not on the Windows taskbar, click **Start** on the taskbar and select **All Programs** (or **Programs**, depending on the operating system) **> Chromeleon > Server Monitor**. Click **Start** to start the server.
3. Start the Chromeleon client:

   a. Click **Start** on the Windows taskbar and select **All Programs** (or **Programs**) **> Chromeleon > Chromeleon** to start the Chromeleon client.

   b. If Chromeleon is installed, select **View > Default Panel Tabset** or click  on the toolbar to display the panel tabset.

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

   c. To display the ICS-90 (or ICS-90A) Control panel, select the **ICS-90** tab on the panel tabset (see Figure 3-2). (The ICS-90A Control panel includes a **Wellness Panel** button; otherwise, the panels are identical.)

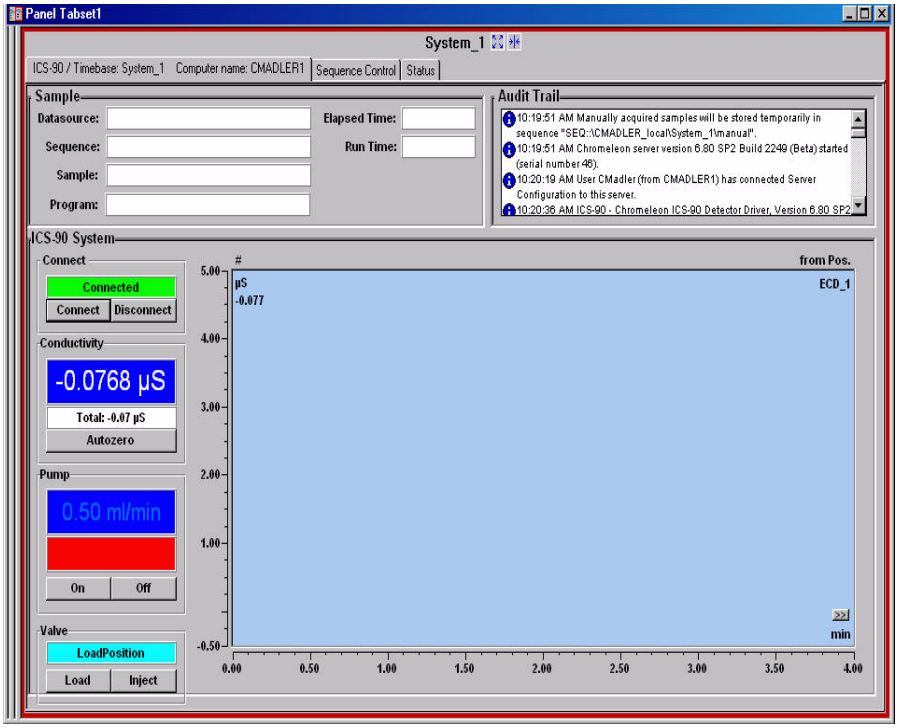


Figure 3-2. ICS-90 Control Panel

4. Verify that the **Connected** check box is selected. If it is not, click the box to connect the ICS-90 to Chromeleon or Chromeleon Xpress.
5. Check the Control panel for the following:
   - A steady operating pressure.
   - The operating total conductivity background depends on the type of analysis to be performed. In general, it should be <30 µS for a system set up for anion analyses, and <2 µS for a system set up for cation analyses.

3.5.3 Preparing the ICS-90 for Operation

1. Turn on the pump by clicking the **On** button on the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress (see Figure 3-2).
2. Prime the pump (for instructions, see Section B.13).
3. Allow the system to equilibrate for 30 minutes. During equilibration, the Control panel displays the background conductivity (the conductivity of the eluent before sample injection). Offset the background and zero the reading by clicking the **Autozero** button on the Control panel (see Figure 3-2).

NOTE **If the ICS-90 pump is running but no data is collected, the flow rate will remain constant indefinitely. However, if the ICS-90 receives no input from Chromeleon or Chromeleon Xpress (you do not turn the pump on or off, run a sequence, toggle the injection valve position, etc.) for 6 hours after data is collected, the module enters standby mode. In standby mode, the pump flow is reduced to 1/20th of its normal rate. To return to the normal flow rate, either begin a new sequence or turn on the pump from the Control panel.**

3.5.4 Manual Sample Processing

To manually process a sample, select operating parameters and commands from the Chromeleon or Chromeleon Xpress menu bar, toolbars, and Control panel. Commands are executed as soon as they are entered.

Summary of manual sample processing

1. Complete the steps in Sections 3.5.1, 3.5.2, and 3.5.3 to start the ICS-90, start Chromeleon or Chromeleon Xpress, and prepare the ICS-90 for operation.
2. Inject the sample via a syringe, vacuum syringe, or AS40 autosampler (see Section 3.5.6).
3. Click the **Autozero** button on the ICS-90 (or ICS-90A) Control panel.

4. Select **Control > Acquisition On**. Chromeleon records the signal supplied by the detector and displays the signal plot on the Control panel.
5. To stop acquisition, select **Control > Acquisition Off**.

NOTES

- **In Chromeleon, data from manual processing is saved in the MANUAL sequence folder under the local datasource. This data is overwritten each time a new manual sample is processed. To save the data from a manual run, select the MANUAL folder, select File > Save As, and enter a new name for the sequence.**
- **Chromeleon Xpress does not allow data to be saved.**

3.5.5 Automatic Sample Processing

For automated control, you create a list of samples (a sequence) to be processed automatically. For each sample, the sequence includes a program with commands and parameters for controlling the ICS-90, acquiring sample data, and generating reports. Additional sample processing parameters are included in the sequence (for example, the sample name, sample type, injection volume, etc.). After the sequence has been defined, automatic sample processing (also called batch processing) is started.

Summary of automatic sample processing

1. Complete the steps in Sections 3.5.1, 3.5.2, and 3.5.3 to start the ICS-90, start Chromeleon or Chromeleon Xpress, and prepare the ICS-90 for operation.
2. Create a new control program or modify an existing one.

   To create a new program:

   a. Select **File > New**. Select **Program File** and click **OK**.

   b. Complete the steps in the Program Wizard, selecting the desired processing options. For help with any of the steps, click the **Help** button on the Program Wizard page.

3. Create a new sequence or modify an existing one.

   To create a new sequence:

   a. Select **File > New**. Select **Sequence (using Wizard)** and click **OK**.

   b. Complete the steps in the Sequence Wizard, adding the desired number of samples and standards to the list and specifying the control program created in Step 2. For help with any of the steps, click the **Help** button on the Sequence Wizard page.

4. To start the sample processing, select **Batch > Edit**.

5. Click the **Add** button on the **Batch List** tab and add the sequence you created in Step 3.

6. To start the batch, click the **Start** button.

Sample Processing Events (Default)

The following events occur if samples are processed using the default control program created with the Program Wizard:

- The pump is turned on.
- If an AS40 Automated Sampler is being used, the AS40 load cycle is started. If no autosampler is being used, the program pauses for you to manually load the sample into the sample loop.
- After the AS40 load cycle is complete—or after you manually load the sample—the background conductivity is offset from the total, thereby zeroing the baseline conductivity value.
- The injection valve position is switched to the Inject position.
- Data acquisition is turned on.
- The injection valve returns to the Load position after 1 minute.
- Data acquisition is turned off after 30 minutes.

3.5.6 Sample Injection

There are three ways to inject sample into the ICS-90: via syringe, vacuum syringe, or autosampler.

Injection via a Syringe

1. Make sure the injection port on the ICS-90 front door (see Figure 2-1) is connected to the sample port S (5) on the injection valve (see Figure 3-3).
2. Fill the 1 cc syringe (P/N 016388) provided in the ICS-90 Ship Kit (P/N 059947) with a calibration standard or sample.
3. Insert the syringe into the injection port on the ICS-90 front door.
4. Overfill the sample loop with several sample loop volumes. Excess sample will exit through the injection valve waste line.
5. Leave the syringe in the port.

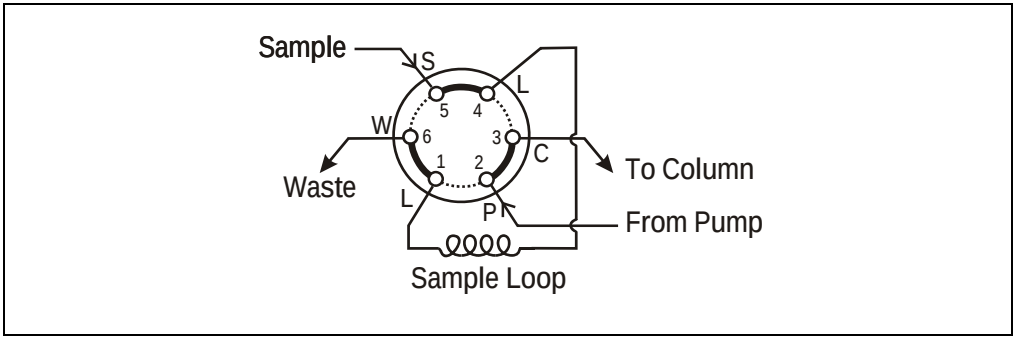


Figure 3-3. Injection Valve Connections

6. Switch the injection valve to start the injection. The method used depends on how you are processing samples:
   - For manual sample processing (see Section 3.5.4), click the **Inject** button on the ICS-90 (or ICS-90A) Control panel (see Figure 3-2).
   - For automatic sample processing (see Section 3.5.5), Chromeleon or Chromeleon Xpress pauses at the start of the program to allow you to load the sample loop (Step 1 through Step 4). After loading the loop, click **OK**. Chromeleon or Chromeleon Xpress

automatically switches the injection valve position and continues running the program.

Injection via a Vacuum Syringe

1. Disconnect the waste line from port W (6) of the injection valve (see Figure 3-3) and attach a shorter line (10 to 12 inches of PEEK or Teflon® tubing).
2. Place the free end of the line into the sample.
3. Insert the 1 cc syringe (P/N 016388) provided in the ICS-90 Ship Kit (P/N 059947) into the injection port on the ICS-90 front door (see Figure 2-1) and pull the plunger out to "suck" the sample into the injection valve.
4. Switch the injection valve to start the injection. The method used depends on how you are processing samples:
   - For manual sample processing (see Section 3.5.4), click the **Inject** button on the ICS-90 (or ICS-90A) Control panel (see Figure 3-2).
   - For automatic sample processing (see Section 3.5.5), Chromeleon or Chromeleon Xpress pauses at the start of the program to allow you to load the sample loop (Step 1 through Step 3). After loading the loop, click **OK**. Chromeleon or Chromeleon Xpress automatically switches the injection valve position and continues running the program.

Injection via the AS40 Automated Sampler

1. Verify that the AS40 output line is connected to port S (5) of the ICS-90 injection valve, and the **LOAD** connector from the AS40 Relay Control cable is connected to the ICS-90 **TTL Output** connector on the rear panel. See Section B.18 for detailed installation instructions.
2. The injection process depends on how you are processing samples:
   - For manual sample processing (see Section 3.5.4), load the injection valve sample loop as instructed in the *AS40 Automated Sampler Operator's Manual* (Document No. 034970) included on the Dionex Reference Library CD-ROM (P/N 055405). Then, click the **Inject** button on the ICS-90 (or ICS-90A) Control panel

(see Figure 3-2) in Chromeleon or Chromeleon Xpress to start the injection.

- For automatic sample processing (see Section 3.5.5), the Chromeleon or Chromeleon Xpress program contains commands that start the AS40 sample load cycle and switch the ICS-90 injection valve position.

3.6 Maintenance

This section describes routine maintenance procedures for the ICS-90 that users may perform. All other maintenance procedures must be performed by Dionex personnel.

As Needed

- Make fresh eluent as needed.
- Regularly check the eluent reservoir to see if it needs to be refilled.
- Whenever the eluent reservoir is refilled, the regenerant reservoir should also be emptied, rinsed, and refilled with regenerant.

Daily

- Check the ICS-90 component mounting panel (see Figure 2-3) for leaks or spills. Wipe up spills. Isolate and repair leaks (see Section 4.2). Rinse off any dried eluent or regenerant with deionized water.
- Check the waste container daily and empty when needed.

Weekly

- Once a week, check gas and fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
- Check the junction between the pump head and the pump casting for evidence of liquid leaks. 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 5.6).

Yearly

- Change the pump seals (see Section 5.6).
- Rebuild the injection valve (see Section 5.9).
- Replace the AS40 Automated Sampler tip and tubing. The ASM/AS40 Sample Tip Replacement Kit (P/N 040835) contains all of the components required to replace the sampling tip and the tubing between the tip and the injection valve. For instructions on how to replace the sampling tip, see the *AS40 Automated Sampler Operator's Manual* (Document No. 034970). The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

4 • Troubleshooting

This chapter is a guide to troubleshooting problems that may occur while operating the ICS-90 Ion Chromatography System (ICS-90).

- Section 4.1 describes the error messages displayed in the Chromeleon or Chromeleon Xpress Audit Trail.
- Section 4.2 through Section 4.13 describe other operating problems and how to resolve them.

If you are unable to eliminate a problem, contact Dionex for help. In the U.S., call Dionex Technical Support at 1-800-346-6390. Outside the U.S., call the nearest Dionex office.

4.1 Alarms and Error Conditions

If any of the following alarm conditions occurs, an error message is displayed in the Chromeleon or Chromeleon Xpress 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
	Warning	A message is displayed in the Audit Trail, but the current run is not interrupted.
	Error	A message is displayed in the Audit Trail and the system attempts to correct the problem (sometimes by using an alternative parameter). An Error never interrupts the current analysis; however, if it occurs during the Ready Check, the analysis will not be started.
	Abort	A message is displayed in the Audit Trail and the running batch is aborted.

- **PUMP OVER PRESSURE ALARM**

  The ICS-90 has a maximum system pressure limit of 28 MPa (4000 psi). In addition, the pressure limit can be lowered from Chromeleon or Chromeleon Xpress (in the Server Configuration or in the control program). If the system pressure exceeds the set limit for at least 0.5 second, Chromeleon or Chromeleon Xpress will stop the pump and the following error message will appear in the Audit Trail:

  The system pressure has exceeded the limit.

  To troubleshoot:

  1. Check for blockages in the liquid lines by working your way backward from the cell to the pump (see Figure 2-7 for the flow schematic).
  2. Restart the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.

- **PUMP UNDER PRESSURE ALARM**

  The system pressure has fallen below the low pressure limit, which is set from Chromeleon or Chromeleon Xpress (in the Server Configuration or in the control program). Chromeleon or Chromeleon Xpress will stop the pump and display the following error message in the Audit Trail:

  The pump has lost prime.

  To troubleshoot:

  1. Make sure the eluent reservoir is full.
  2. Check for liquid leaks (see Section 4.2).
  3. Prime the pump (see Section B.13).
  4. Restart the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.

- **LOAD/INJECT VALVE ERROR**

  If the injection valve fails to switch positions within 1 second of being toggled, the ICS-90 Moduleware will report an error to Chromeleon or Chromeleon Xpress and the following error message will be displayed in the Audit Trail:

   Load/inject valve error.

  To troubleshoot:

  1. If a sequence is being executed, terminate the sequence by selecting **Stop** from the **Batch** menu.
  2. Turn off the ICS-90 power briefly and then restart.
  3. Try to toggle the valve from Load to Inject by clicking the **Inject** button on the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.
  4. If the problem persists, call Dionex for assistance.

- **OVER CONDUCTIVITY**

  This message usually appears for the first few minutes after the ICS-90 is started. If the message appears at other times, it indicates a problem.

   Conductivity exceeds limit.

  To troubleshoot:

  1. Perform the suppressor QuickStart procedure:
     a. Disconnect the separator column outlet line and connect it to the DS5 cell inlet line.
     b. Turn on the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress and allow the suppressor to regenerate for 5 minutes.
     c. Turn off the pump and allow the suppressor to hydrate for 5 minutes.
     d. Reconnect the separator column outlet line and cell inlet line.

2. If the high background persists:
   - Prepare fresh eluent (see Section 3.1).
   - Prepare fresh regenerant (see Section 3.2).
   - Verify the cell calibration by pumping deionized water through the cell (bypassing the column(s) and suppressor). The total background should be less than 1.0 µS.
   - Perform the suppressor off-line regeneration procedure. For instructions, refer to the suppressor manual. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).
   - If the problem persists, replace the suppressor (see Section 5.11).

- **DATA BUFFER OVERFLOW ERROR**

  Various electronics-related problems can cause the following error message to be displayed in the Audit Trail:

   Module data buffer overflow. Data may have been lost.

  If this warning appears, call Dionex for assistance. The ICS-90 electronics components cannot be serviced by the user.

4.2 Liquid Leaks

- **Leaking fitting**

  Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection (see Section 5.2). Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

- **Broken liquid line**

  Replace the line and fittings (see Section 5.2).

- **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 ICS-90. If a line is blocked, replace it (see Section 5.2).

- **Loose pump check valve housing**

  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.

- **Damaged pump piston seal**

  1. Replace the piston seal (see Section 5.6).
  2. If the problem persists, replace the piston (see Section 5.7).

- **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), included on the Dionex Reference Library CD-ROM (P/N 055405), for tightening requirements. Replace any damaged fittings.

- **Leaking pump head waste valve**

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

  Inspect the pump head. If the waste valve is the source of the leak, replace the waste valve O-ring (see Section 5.8).

- **Leaking MMS III**

  Refer to the suppressor manual for troubleshooting procedures. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

- **Leaking injection valve**

  Make sure the liquid line connections to the transducer are tight. Replace any damaged fittings. Refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432) for tightening requirements. The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

  Liquid leaks from behind the valve stator may indicate a scratched rotor seal. Rebuild the injection valve (see Section 5.9).

- **Leaking DS5 Detection Stabilizer**

  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.

  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 Dionex for assistance.

4.3 Pump Difficult to Prime or Loses Prime

- **Empty eluent reservoir and/or no eluent connected**

  Fill the reservoir. Make sure all connections are secure.

- **Eluent reservoir not pressurized**

  Make sure the gas line is properly connected between the air regulator and the reservoir, and that the inlet gas supply is regulated to between 0.55 and 0.83 MPa (80 and 120 psi) and connected to the air regulator. Make sure the eluent reservoir cap is screwed on tightly. See Section B.9 and Section B.10 for detailed connection instructions.

- **Dirty check valve**

  Clean and/or replace the pump check valve (see Section 5.5).

- **Liquid leaks at junction between pump head and pump casting**

  Replace the piston seal (see Section 5.6).

4.4 Pump Does Not Start

- **No power (front door Power LED indicator fails to light)**

  Check that the power cord is plugged in.

  Check the main power fuses and replace if needed (see Section 5.12).

- **No communication between ICS-90 and Chromeleon or Chromeleon Xpress (Link LED on rear panel fails to light)**

  The USB cable is not connected correctly. See Section B.4 for installation instructions.

4.5 No Flow

- **Pump not primed**

  Prime the pump (see Section B.13).

- **Broken pump piston**

  Replace the piston (see Section 5.7).

4.6 Erratic Flow

- **Pump needs priming**

  Prime the pump (see Section B.13).

- **Damaged piston seal**

  Replace the piston seal (see Section 5.6).

- **Dirty pump check valve**

  Clean or replace the check valve (see Section 5.5).

4.7 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 Dionex Reference Library CD-ROM (P/N 055405).

- **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 5.2).

- **Flow rate through the columns too high**

  Measure the pump flow rate and set it to the correct rate (see Section B.14).

- **Clogged column bed supports**

  Refer to the instructions in the column manual for troubleshooting guidance. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

- **Contaminated columns**

  Clean the columns as instructed in the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

- **Plugged Rheodyne valve passages**

  Rebuild the injection valve (see Section 5.9).

4.8 Peak "Ghosting"

Ghosting is the appearance of extraneous peaks in a chromatogram. These may be late-eluting peaks from a previous injection or they may result from a contaminated, malfunctioning, or incorrectly installed 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 (see Section 3.1).

- **Malfunctioning injection valve**

  Contact Dionex for assistance.

4.9 Nonreproducible Peak Height or Retention Time

- **Column overloading**

  Dilute the sample (see Section 3.4.3).

- **Liquid leaks**

  Locate and eliminate the leaks (see Section 4.2).

- **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 B.13).

4.10 Abnormal Retention Time or Selectivity

- **Contaminated or incorrect eluent**

  Remake the eluent, using concentrated eluent and ASTM filtered, Type I (18-megohm) deionized water (see Section 3.1).

- **Contaminated or degraded sample**

  Take appropriate precautions when preparing and storing samples to prevent contamination and degradation (see Section 3.4).

- **Contaminated column**

  1. Clean the column as instructed in the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405), located in the ICS-90 Ship Kit (P/N 059947).
  2. If cleaning is unsuccessful, replace the column.

4.11 No DS5 Response

- **DS5 not properly installed**

  Check that the DS5 is plugged into the component mounting panel and screwed down until the bottom of the DS5 is flush against the sheet metal panel (see Figure 2-3 and Figure 2-4).

- **No flow from pump**

  Several conditions may cause this condition; see Section 4.4 and Section 4.5 for details.

- **Cell electronics malfunctioning**

  Use the Chromeleon or Chromeleon Xpress Wellness panel diagnostics to test the electronics with a dummy cell (see Section 5.1 and the online Help for instructions). If the variance reading is outside the tolerance range (less than 1 µS), the electronics are malfunctioning. Call Dionex for assistance.

4.12 High DS5 Output

- **Background not suppressed by suppressor**

  If **Conductivity exceeds limit** is displayed in the Chromeleon or Chromeleon Xpress Audit Trail, follow the instructions in Section 4.1. For additional troubleshooting guidance, refer to the suppressor manual. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

- **Sample concentration too high**

  Dilute the sample (see Section 3.4.3).

- **Wrong eluent or regenerant**

  Check that you are using the correct eluent and regenerant for your system (see Section 3.1).

- **Cell out of calibration**

  Recalibrate the cell from the Wellness panel in Chromeleon or Chromeleon Xpress (see Section 5.1).

4.13 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 Dionex Reference Library CD-ROM (P/N 055405).

- **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 MMS eluent ports.

- **Pump not properly primed**

  Prime the pump (see Section B.13).

- **Contaminated or incorrect eluent and/or regenerant**

  Remake the eluent and regenerant (see Section 3.1).

- **Rapid changes in ambient temperature**

  If the ambient temperature does not meet the specification of 10 to 35 °C (50 to 95 °F), verify that air conditioning and heating vents are directed away from the ICS-90 and the ICS-90 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 Dionex Reference Library CD-ROM (P/N 055405).

- **Cell above or below temperature**

  Call Dionex for assistance.

- **Damaged piston seal**

  Replace the piston seal (see Section 5.6).

- **DCR backpressure tubing not installed**

  Install the tubing on the end of the suppressor waste line (see Section B.8).

5 • Service

This chapter describes routine service procedures for the ICS-90 Ion Chromatography System (ICS-90) that users may perform. Other service procedures must be performed by Dionex personnel.

NOTE The ICS-90 electronics components cannot be serviced by the user. Repair of electronics components must be performed by Dionex personnel.

Before replacing any part, refer to the troubleshooting information in Chapter 4 to isolate the cause of the problem. When ordering replacement parts, please include the ICS-90 model number and serial number. To contact Dionex in the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Dionex office.

IMPORTANT Substituting non-Dionex parts may impair the performance of the ICS-90, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

5.1 Diagnostics and Calibrations

Chromeleon and Chromeleon Xpress allow you to monitor ICS-90 functions and perform diagnostic and calibration procedures from the Wellness panel (see Figure 5-1). This section provides an overview of Wellness panel features. For instructions on performing calibrations, see the following sections:

- Calibrating the Pressure Transducer (Section 5.1.1)
- Calibrating the Cell (Section 5.1.2)
- Calibrating the Flow Rate (ICS-90A only) (see Section 5.1.3)

NOTE If you are unsure whether your system is an ICS-90 or ICS-90A, check the model data label on the rear panel.

To open the Wellness panel:

1. In the Chromeleon Browser, expand the **Dionex Templates** folder, and then the **Panels** and the **Wellness** folders.

2. In the right pane of the Browser window, double-click either **Dionex_ICS-90_wellness.pan** or **Dionex_ICS-90A_wellness.pan**.

   Note: You can also open the ICS-90A Wellness panel by double-clicking the **Wellness Panel** button on the ICS-90A Control panel in Chromeleon or Chromeleon Xpress.

   The selected Wellness panel appears. The ICS-90A version of the panel (see Figure 5-1) includes controls for flow rate calibration that are not available on the ICS-90 Wellness panel; otherwise, the panels are identical.

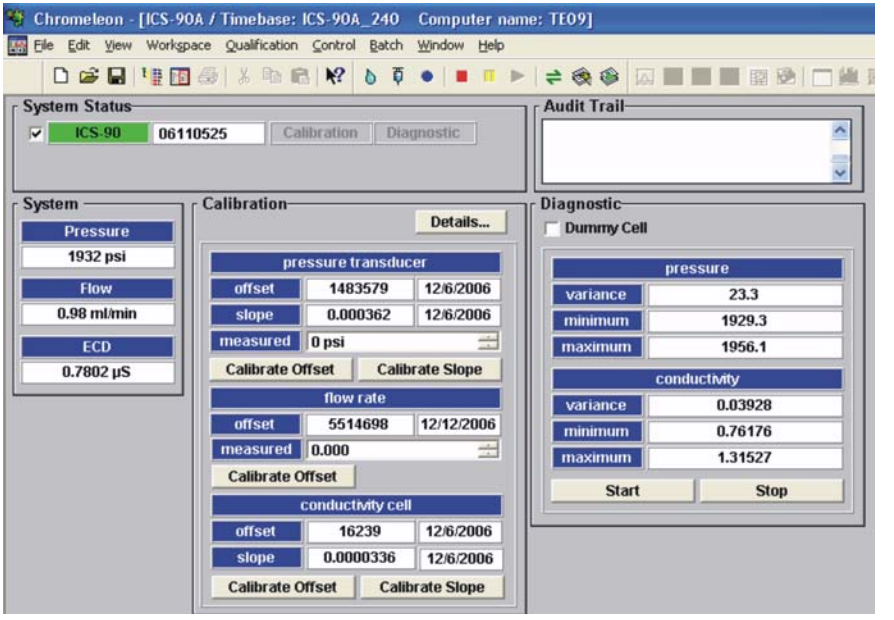


Figure 5-1. ICS-90 Wellness Panel (ICS-90A version shown)

Use the Wellness panel to monitor and/or perform the following functions:

System Status

- The check box to the left of the **ICS-90** LED indicates the connectivity status. When the check box is gray, there is no connectivity between the ICS-90 and the Chromeleon or Chromeleon Xpress computer.
- The ICS-90 serial number is displayed to the right of the **ICS-90** LED.
- While a calibration or diagnostic procedure is running, the **Calibration** or **Diagnostic** box is green.

Audit Trail

- The **Audit Trail** displays an account of every event that occurs during ICS-90 operation. This includes errors, status messages, operational events, etc.

System

- The current total system **Pressure** is displayed.
- The current electrochemical detector (**ECD**) conductivity is displayed.

Calibration

- **Pressure Transducer**
  - The **offset**, **slope**, and date of the last calibration are displayed.
  - The **Measured** field is in the slope calibration. See Section 5.1.1 for instructions.
  - The **Calibrate Offset** and **Calibrate Slope** buttons are used to calibrate the pressure transducer. See Section 5.1.1 for instructions.
- **Conductivity Cell**
  - The **offset**, **slope**, and date of the last DS5 calibration are displayed.
  - The **Calibrate Offset** or **Calibrate Slope** buttons are used to calibrate the cell. See Section 5.1.2 for calibration instructions.
- **Flow Rate**
  - The **offset** and date of the last calibration are displayed.
  - The **Calibrate Offset** button is used to calibrate the flow rate. See Section 5.1.3 for calibration instructions.
- Click the **Details** button to open a window that shows the factory-set calibration values, the current values, and the previous set of calibration values. The factory and last set of calibration values can be downloaded from this window.

Diagnostic

- The last calibrated **pressure** and **conductivity variance**, **minimum**, and **maximum** readings for the DS5 are displayed.
- The **Start** and **Stop** buttons start and stop the readings.
- To determine the conductivity variance of the electronics alone, select the **Dummy Cell** box and then click **Start**. The variance reading should be less than 1 µS.

5.1.1 Calibrating the Pressure Transducer

NOTE About 10 minutes before starting this calibration procedure, toggle the injection valve position a few times by clicking the valve Load and Inject buttons on the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress. This removes any air or contaminant buildup in the injection valve loop.

1. Turn off the pump flow from the ICS-90 (or ICS-90A) Control panel.
2. Open the waste valve on the front of the pump head (see Figure 5-2) by turning the knob counterclockwise two turns.

Figure 5-2. ICS-90 Interior Components

3. Open the ICS-90 (or ICS-90A) Wellness panel (see Section 5.1).
4. Click the **Calibrate Offset** command button under **pressure transducer** (see Figure 5-1). The pump calibrates the transducer and uploads the new value to Chromeleon or Chromeleon Xpress. The new offset is stored as the current value.
5. Close the waste valve.

6. Connect a pressure gauge between the pump outlet and the pressure transducer (see Figure 5-2). Turn on the pump and let the system stabilize. Note the average pressure reading on the gauge; enter this reading in the **measured** field.
7. Click the **Calibrate Slope** command button under **pressure transducer**. The pump calibrates the slope and uploads the new value to Chromeleon or Chromeleon Xpress. The new slope is stored as the current value.
8. Turn off the pump.
9. Disconnect the pressure gauge. Reconnect the pressure transducer to the pump.

5.1.2 Calibrating the Cell

Calibrate the cell every 6 months or after installing a new DS5 Detection Stabilizer.

Items Needed	Description
1.0 mM KCl solution	Prepare by dissolving 0.07456 g of reagent-grade KCl in one 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 ICS-90 (or ICS-90A) Wellness panel (see Section 5.1).
2. Use an Allen wrench to remove the two screws securing the DS5 to the ICS-90 component mounting panel (see Figure 5-2). Save the screws.
3. Pull the DS5 straight out from the component mounting panel to unplug the cell from its electronics. Let the DS5 hang by the tubing.
4. Allow the conductivity to stabilize for 5 to 10 minutes.
5. Click the **Calibrate Offset** command button under **conductivity cell** (see Figure 5-1).
6. Line up the 9-pin connectors on the DS5 and the component mounting panel and plug the DS5 back into the electronics. Replace the mounting screws (removed in Step 2) and tighten.

7. Disconnect the pump outlet line from port P (2) on the injection valve; connect the line directly to the cell inlet, using the yellow PEEK backpressure tubing (P/N 049715).
8. Fill an eluent bottle with 1.0 mM KCl solution and connect it to the eluent out line. Prime the pump (see Section B.13) and then turn on the pump.
9. Verify that there is a minimum of 7 MPa (1000 psi) of backpressure.
10. Allow the conductivity to stabilize for 5 to 10 minutes.
11. Click the **Calibrate Slope** command button under **conductivity cell**. The cell is calibrated and a new cell calibration constant is uploaded to Chromeleon or Chromeleon Xpress. The new value is stored as the current cell calibration constant.

    After calibration, the conductivity reading should be 147.00 ± 2 µS/cm. If this is not the case, call Dionex for assistance.
12. Flush the KCl solution from the system by pumping DI water through the cell. When the conductivity drops to near zero, stop the pump flow.
13. Reconnect the pump to the injection valve and reconnect the line from the suppressor to the cell inlet.

5.1.3 Calibrating the Flow Rate (ICS-90A only)

NOTE **If you typically operate the ICS-90A at a particular flow rate, you may wish to calibrate the system at that flow rate to improve flow rate accuracy.**

1. Measure the flow rate, using one of the methods described below:
   - Volumetrically (recommended for most applications): Measure the volume of liquid collected over a measured time period (see page 57 or page 58).
   - Gravimetrically: Weigh the mass of liquid collected over a measured time period (see page 58). The gravimetric method is slightly more accurate than the volumetric method.
2. After measuring the flow rate, go on to page 59 to calibrate the flow rate.

Volumetric Method 1 (Recommended): Using a Syringe and Adapter

1. Locate the following items in the ICS-90 Ship Kit (P/N 059947): luer fitting (P/N 024305), luer coupler (P/N 042806), 1 mL syringe (P/N 016388), and 10 mL syringe (P/N 016387). You will also need a stopwatch or a clock or watch with a second hand (the computer's clock can be used), and a small beaker or other container for collecting waste.
2. Connect the luer coupler to the luer fitting.
3. Select the syringe to be used:
   - For a flow rate of 0.5 mL/min, use the 1 mL syringe.
   - For a flow rate above 0.5 mL/min, use the 10 mL syringe.
4. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor. Connect the luer coupler to the **ELUENT IN** line, and connect the syringe to the luer fitting (see Figure 5-3).

Figure 5-3. Assembled Syringe and Luer Adapter

5. Using a stopwatch or clock's second hand, time the flow of eluent into the syringe for 60 seconds. Determine the volume collected by noting the beginning and ending positions of the syringe plunger.
6. The volume of eluent collected in 60 seconds should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 1 mL/min, the volume should be 1.0 ± 0.1 mL.

7. If necessary, adjust the flow rate knob, and then repeat the measurement. Continue adjusting until the desired milliliters per minute is collected.
8. Reconnect the **ELUENT IN** line to the suppressor.

Volumetric Method 2: Using a Graduated Cylinder

1. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor.
2. Using a clock or watch with a second hand (the computer's clock can be used), collect eluent from the **ELUENT IN** line into a small graduated cylinder for 2 minutes.
3. The collected eluent should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 1 mL/min, 2.0 ± 0.2 mL should be collected in 2 minutes.
4. If necessary, adjust the flow rate knob, and then repeat the measurement. Continue adjustments until the desired eluent per minute is collected.
5. Reconnect the **ELUENT IN** line to the suppressor.

Gravimetric Method

1. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor.
2. Using a tared beaker and a calibrated stopwatch, collect the eluent from the **ELUENT IN** line for 60 seconds.
3. Weigh the mass of the eluent. The mass (in grams) should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 0.5 mL/min, the mass of the collected eluent should be 0.5 ± 0.02 g.
4. If necessary, adjust the flow rate knob, and then repeat Step 2 and Step 3. Continue adjusting until the desired mass per minute is collected.
5. Reconnect the **ELUENT IN** line to the suppressor.

Calibrating the Flow Rate

1. Open the ICS-90A Wellness panel (see Section 5.1).
2. Enter the flow rate obtained by the volumetric or gravimetric method in the **measured** field.
3. Click the **Calibrate Offset** command button under **flow rate** (see Figure 5-1). The pump calibrates the flow rate and uploads the new value to Chromeleon or Chromeleon Xpress. The new offset is stored as the current value.

5.2 Replacing Tubing and Fittings

The ICS-90 is plumbed with the tubing and tubing assemblies listed below.

Tubing Size and Type	Used For
0.125-mm (0.005-in) ID PEEK (P/N 044221)	Connection from pump pulse damper to injection valve
0.25-mm (0.010-in) ID PEEK (P/N 042690)	Connections between other system components
0.30-mm (0.012-in) ID Tefzel® (P/N 048904)	Connection from injection port to injection valve
0.75-mm (0.030-in) ID PEEK (P/N 044777)	Connection from injection valve to waste and between the DS5 and regenerant reservoir

- 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 Dionex Reference Library CD-ROM (P/N 055405).
- 1/8-in fittings (P/N 052267) and ferrules (P/N 048949) are used for connections to the MMS III **REGEN OUT** port and the eluent and regenerant reservoirs.
- 1/16-in fittings (P/N 052230) and ferrules (P/N 052231) are used for connections from the injection port to the injection valve.

5.3 Changing the Sample Loop

Figure 5-4 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, and column. A 10 µL PEEK sample loop (P/N 042949) is installed at the factory between ports L and L. Always replace the pre-installed 10 µL loop with another of the same size.

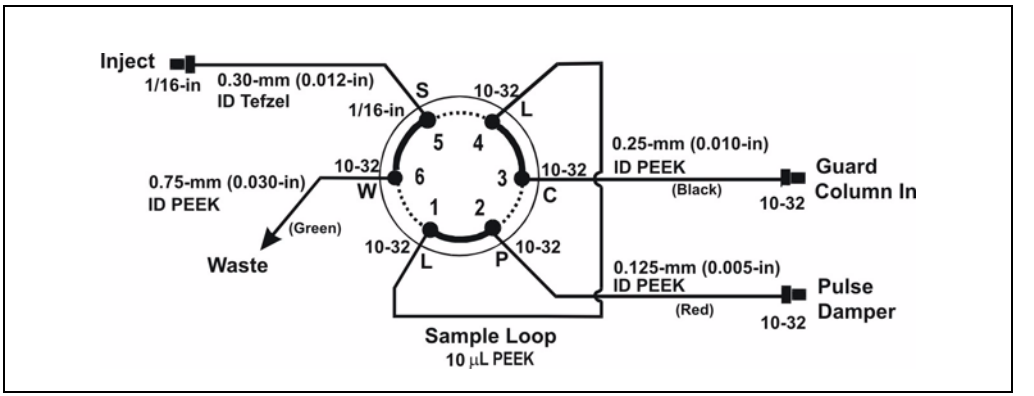


Figure 5-4. Injection Valve Plumbing

1. Turn off the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.
2. Open the ICS-90 front door.
3. Disconnect the sample loop from ports L and L on the injection valve (see Figure 5-4).
4. Install the new sample loop between ports L and L on the injection valve.
5. Make sure the loop is tightly coiled so the door can close securely.
6. Close the door.
7. Turn on the pump from the Control panel.

5.4 Isolating a Restriction in the Liquid Plumbing

A restriction in the liquid plumbing will cause excessive system backpressure.

1. Begin pumping eluent through the system (including the columns).
2. Follow the ICS-90 flow schematic (see Figure 2-7) and work backward through the system, beginning at the cell exit. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops abnormally indicates the point of restriction.

   If the 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.

5.5 Cleaning and Replacing Pump Check Valves

A dirty check valve causes an erratic flow rate. It may also cause the pump to lose prime and/or be difficult to reprime.

1. Prime the pump (see Section B.13). If the pump does not stay primed, go to the next step.
2. Turn off the power switch on the ICS-90 rear panel and disconnect the power cord.
3. Open the front door of the ICS-90.
4. Disconnect the tube fittings from both the inlet and outlet pump check valve housings (see Figure 5-5).
5. Using a 1/2-in box wrench or an adjustable wrench, carefully remove both check valve housings from the pump head.
6. Place the check valves in a beaker of methanol and sonicate or agitate for several minutes.
7. Rinse each check valve thoroughly with filtered, deionized water.

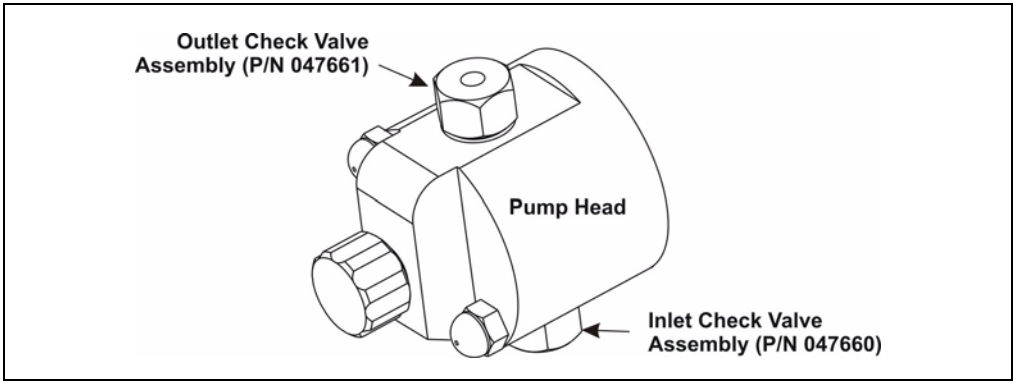


Figure 5-5. Pump Head Assembly

8. Reinstall the check valves in the pump head. Be sure to install the *inlet* check valve (which a 1/4-28 port) on the bottom of the head and the *outlet* check valve (which has a 10-32 port) on the top. Tighten only enough to seat.

CAUTION

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

9. Reconnect the liquid lines. Close the front door.
10. Reconnect the power cord, and turn on the power switch on the ICS-90.
11. Prime the pump (see Section B.13). If the pump will not prime, and you have eliminated all other possible causes of the problem, replace both check valves (inlet check valve, P/N 047660; outlet check valve, P/N 047661).

5.6 Replacing a Pump Piston Seal

A damaged piston seal allows leakage around the piston at the head mounting plate or around the base of the pump head. Flow rates will be unstable and there may be baseline noise.

1. Turn off the power switch on the ICS-90 rear panel and disconnect the power cord.
2. Open the ICS-90 front door.
3. Disconnect the tube fittings from the inlet and outlet check valves (see Figure 5-5).
4. Hold the head firmly against the pump housing, to compensate for the spring loading, and remove the two nuts (see Figure 5-6).

Lateral motion when disengaging the head from the piston can 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.

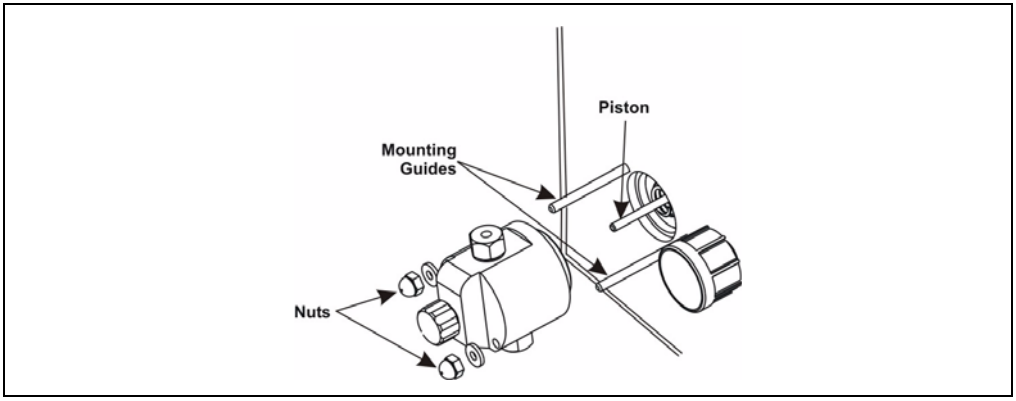


Figure 5-6. Removing the Pump Head

5. Slowly release the head, allowing it to separate from the housing. **CAREFULLY** disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides (see Figure 5-6). Be especially careful not to snap the piston if the internal spring sticks to the piston guide.
6. Place the head, front end down, on a clean work area. Lift off the piston guide and back-up washer to expose the seal (see Figure 5-7).

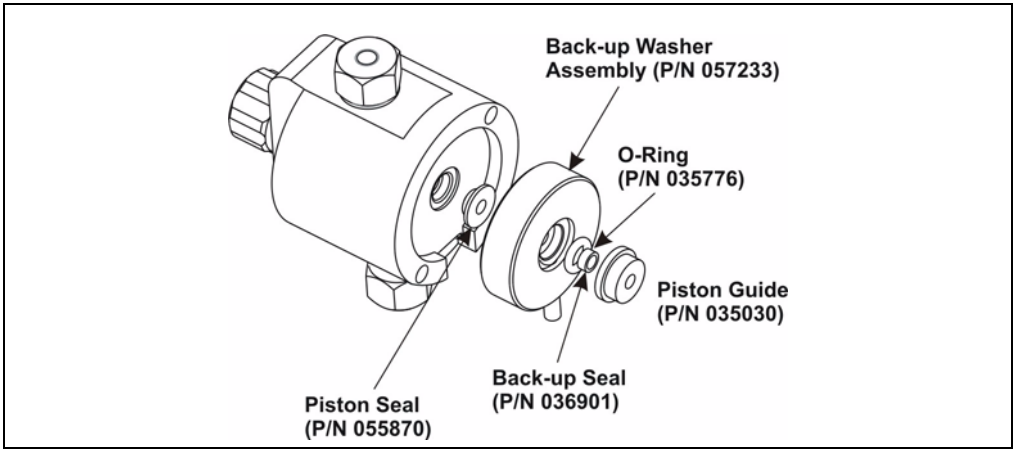


Figure 5-7. Pump Head Assembly

7. Remove the piston seal from the head. Discard the seal.
8. Remove the O-ring and back-up seal from the back-up washer.
9. Carefully push the new piston seal (P/N 055870) into the head. When properly installed, the piston seal is almost flush with the indented surface of the head.
10. Press a new back-up seal (P/N 036901) into the O-ring (P/N 035776). If necessary, also replace the O-ring. Then, press the O-ring and back-up seal into the back-up washer assembly (P/N 057233).
11. Press the back-up washer assembly into the head, followed by the piston guide (P/N 035030).
12. Remove the spring, spring guide, piston, spring retainer, and retainer ring from the pump housing (see Figure 5-8). Inspect the interior of the housing for liquid and corrosion. Clean up any spills and carefully clean any signs of corrosion from the interior of the housing.

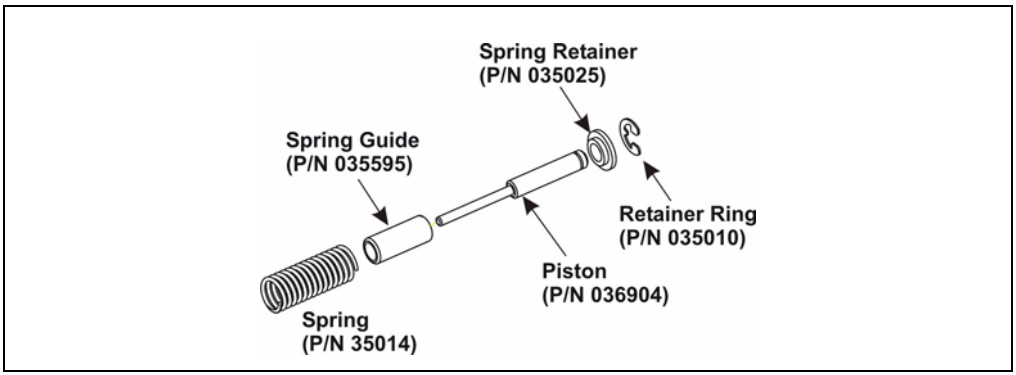


Figure 5-8. Piston Assembly

13. Clean and inspect the piston. If it is scored or scratched, replace it (see Section 5.7).
14. Reinstall the retainer ring, spring retainer, piston, spring guide, and spring in the pump housing.
15. Carefully slide the pump head straight onto the mounting guides. Guide the spring onto the piston guide. Apply gentle pressure to push the piston through the seal.

CAUTION

Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight on will break the piston, as well as damage the piston seal and back-up seal.

MISE EN GARDE

Évitez tout mouvement latéral lorsque vous faites coulisser la tête sur le piston. Un coulissement non rectiligne de la tête cassera le piston et en endommagera le joint étanche et le joint étanche de sécurité.

VORSICHT

Vermeiden Sie Seitwärtsbewegungen, wenn Sie den Kopf auf den Kolben schieben. Andernfalls kann der Kolben abbrechen und die Kolbendichtung sowie die Hinterspülungsdichtung zerstört werden.

16. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly.
17. Reconnect the liquid lines to the inlet and outlet check valves on the pump head assembly (see Figure 5-5).
18. Reconnect the power cord and turn on the power switch.
19. Prime the pump (see Section B.13).

5.7 Replacing a Pump Piston

Continued leaking from around the pump head after replacing the piston seal indicates a scratched or broken piston.

1. Turn off the power switch on the ICS-90 rear panel and disconnect the power cord.
2. Open the ICS-90 front door.
3. Disconnect the tube fittings from the inlet and outlet check valves (see Figure 5-5).
4. While holding the head firmly against the pump housing to compensate for the spring loading, remove the two nuts (see Figure 5-7).

Lateral motion when disengaging the head from the piston can 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. Slowly release the head and allow it to separate from the housing. **CAREFULLY** disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides (see Figure 5-6). Be especially careful not to snap the piston if the spring is stuck to the piston guide.
6. Remove the piston guide, spring, spring guide, spring retainer, and piston by pulling them straight out, away from the pump housing (see Figure 5-8).
7. If the piston is broken, replace the piston seal and the back-up seal (see Section 5.6). This will prevent pieces of broken piston from scratching the new piston assembly.
8. Remove the retainer ring (P/N 035010) from the old piston assembly and install it on the new piston assembly (P/N 036904).

9. Carefully slide the spring retainer onto the piston assembly.
10. Slide the assembled piston back into the piston housing.
11. Slide the spring over the piston, positioning it flush against the spring retainer.
12. Carefully slide the pump head straight onto the alignment rods. Guide the spring over the piston guide. Gently push the piston through the seal.

Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight on will break the piston, as well as damage the piston seal and back-up seal.

Évitez tout mouvement latéral lorsque vous faites coulisser la tête sur le piston. Un coulissement non rectiligne de la tête cassera le piston et en endommagera le joint étanche et le joint étanche de sécurité.

Vermeiden Sie Seitwärtsbewegungen, wenn Sie den Kopf auf den Kolben schieben. Andernfalls kann der Kolben abbrechen und die Kolbendichtung sowie die Hinterspülungsdichtung zerstört werden.

13. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly.
14. Reconnect the liquid lines to the check valve housings.
15. Reconnect the power cord and turn on the power switch.
16. Prime the pump (see Section B.13).

5.8 Replacing the Waste Valve O-Ring

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

1. Turn off the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.
2. Open the front door of the ICS-90.
3. Turn off the gas pressure to the eluent reservoir.
4. Remove the valve from the pump head housing by turning the knob counterclockwise until it comes loose from the housing (see Figure 5-9).
5. Remove the O-ring.
6. Carefully slide a new O-ring (P/N 035776) over the end of the valve and push it into the groove.
7. Reinstall the valve in the pump head housing, turning the knob clockwise until the valve is firmly seated. **Do not overtighten the valve knob.**
8. Turn on the gas pressure to the eluent reservoir.
9. Close the ICS-90 front door.
10. Prime the pump (see Section B.13).

NOTE **If the pump head leaks after replacing the waste valve O-ring, the valve knob may not have been seated firmly enough. Tighten the knob further, but only by hand. Never use a tool.**

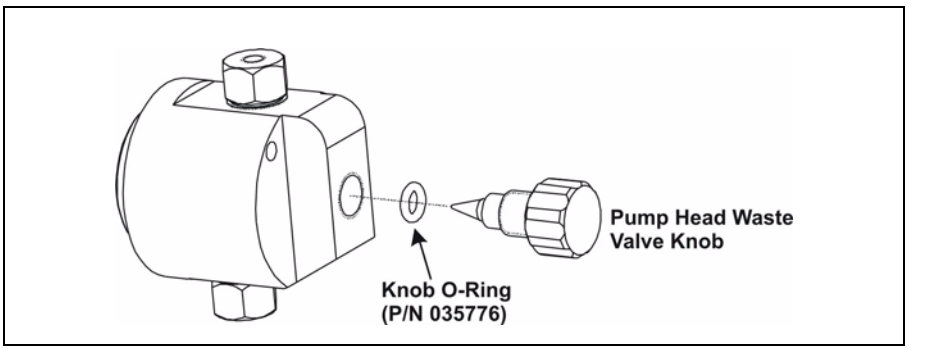


Figure 5-9. Pump Head Assembly

5.9 Rebuilding the Injection Valve

Dionex recommends rebuilding the injection valve annually. The Injection Valve Rebuild Kit (P/N 057896) contains all required replacement parts.

NOTE Substitution of non-Dionex parts may impair valve performance and void the product warranty.

1. Turn off the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.
2. Open the front door of the ICS-90.
3. Disconnect each liquid line connected to the injection valve.
4. Follow the instructions provided in the LC25 Injection Valve Maintenance Kit (P/N 055676) to replace the rotor seal, isolation seal, and stator face.
5. Reconnect all liquid lines to the injection valve.
6. Close the ICS-90 front door.
7. Turn on the pump from the Control panel.

5.10 Installing a New DS5 Detection Stabilizer

1. Turn off the power on the ICS-90 rear panel and disconnect the power cord.
2. Open the ICS-90 front door.
3. Disconnect the black **ELUENT OUT** line from the **ELUENT OUT** port on the suppressor (see Figure 5-10).
4. Disconnect the orange **CELL OUT** line from the union that connects it to the black **CELL OUT** line.
5. Use an Allen wrench to remove the two screws on the top of the DS5.
6. Remove the DS5 by unplugging it from the component mounting panel.
7. Plug the new DS5 (P/N 057290) into its mounting location.
8. Replace the screws on the top of the DS5 and tighten.
9. Attach the orange **CELL OUT** line from the DS5 to the union attached to the black **CELL OUT** line. Attach the **ELUENT OUT** line to the **ELUENT OUT** port on the suppressor.

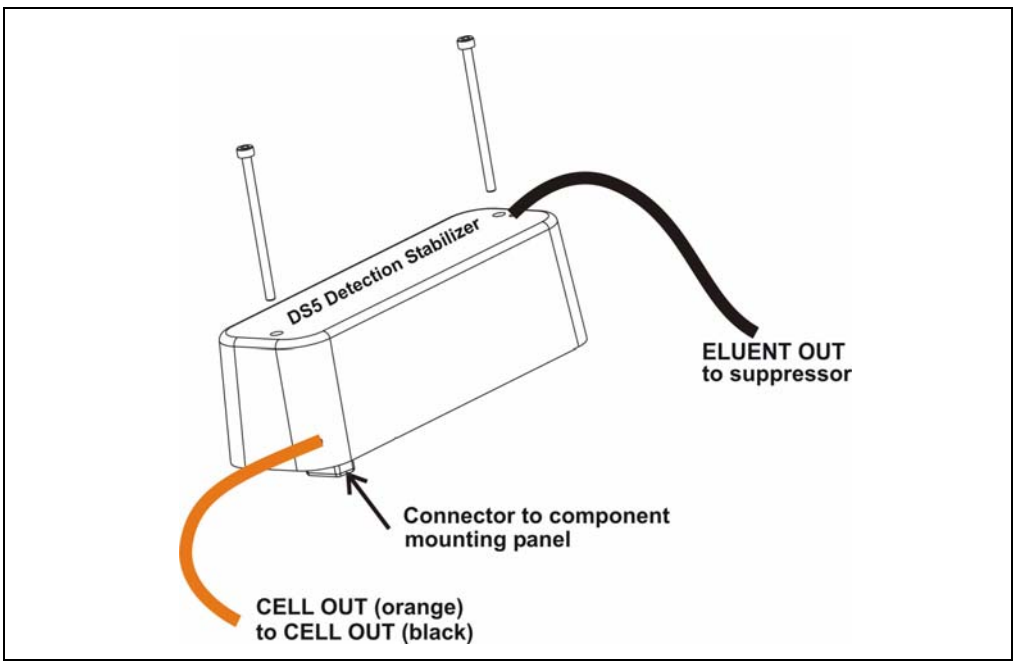


Figure 5-10. DS5 Detection Stabilizer Assembly

10. Close the front door.
11. Reconnect the power cord and turn on the ICS-90 power.
12. Calibrate the cell (see Section 5.1.2).

5.11 Installing a New Suppressor

Refer to the suppressor manual for guidance about when to replace a suppressor. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

1. Turn off the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress.
2. Open the front door of the ICS-90.
3. Disconnect the four eluent and regenerant lines from the suppressor.
4. Slide the suppressor to the left to detach it from the component mounting panel.
5. Slide the new suppressor (AMMS III suppressor, P/N 056751; CMMS III suppressor, P/N 056753) to the right until it locks into place on the mounting panel.
6. Connect the four eluent and regenerant lines to the new suppressor.
7. Close the ICS-90 front door.
8. Prime the pump (see Section B.13).

5.12 Changing the Main Power Fuses

DANGER

HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the ICS-90.

DANGER

HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du ICS-90.

ACHTUNG

HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des ICS-90.

1. Turn off the main power and disconnect the power cord.
2. The fuse holder is part of the main power receptacle on the ICS-90 rear panel. Note the recessed lock located on each side of the fuse holder (see Figure 5-11).

   Using a small screwdriver, push each lock toward the center to release it. When both locks are released and the fuse holder pops out slightly, pull the fuse holder straight out of its compartment.

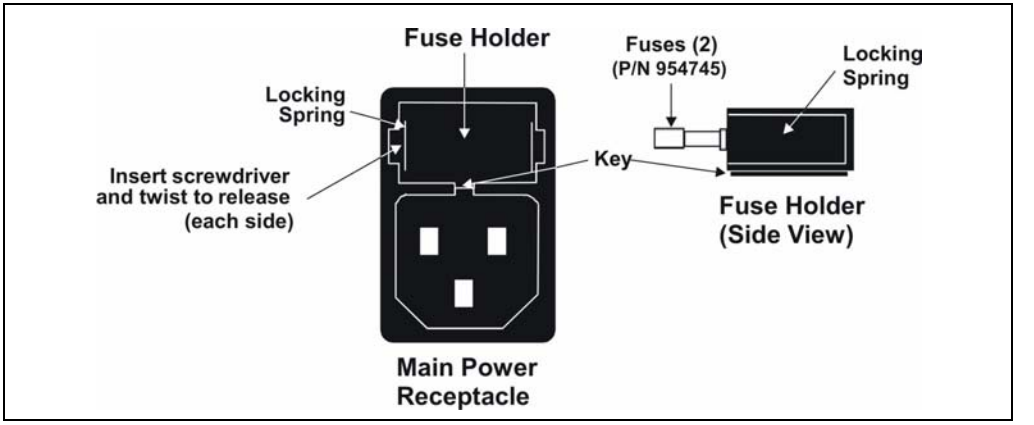


Figure 5-11. Main Power Fuse Holder

3. Replace the two fuses in the holder with new IEC 127 fast-blow fuses rated 3.15 amps (P/N 054745). Dionex recommends always replacing both fuses.
4. Reinsert the fuse holder into its compartment. The fuse holder is keyed to fit only in its proper orientation. Apply sufficient pressure evenly against the holder to engage the two locks. The holder is flush against the panel when both locks are engaged.
5. Reconnect the main power cord and turn on the power.

A • Specifications

A.1 Electrical

Main Power	Two voltage/frequency configurations (not user-selectable): 110–120 VAC/60 Hz 220–240 VAC/50 Hz 100 VAC/50 Hz
Fuses	Two fast-blow IEC 127 fuses rated 3.15 A (P/N 954745)

A.2 Physical

Dimensions (with reservoirs and tubing)	Height without reservoirs: 32.125 cm (12.85 in) Height with reservoirs and cap tubing: 59.63 cm (23.85 in) Width: 23.23 cm (9.29 in) Depth: 39.73 cm (15.89 in)
Weight	14 kg (30 lb)
Decibel Level	< 53 dBA

A.3 Environmental

Operating Temperature 10 to 35 °C (50 to 95 °F)

Humidity 5% to 95% relative humidity, noncondensing

Gas Pressure Laboratory-quality clean gas regulated to between 0.55 and 0.83 MPa (80 and 120 psi)

Operating Pressure 28 MPa (4000 psi) maximum liquid path (tubing, valve, columns, etc.)

A.4 Front Panel

Operational LED Indicators LEDs display the ICS-90 operational status:

- **Power**

  Off: No power

  On: Power present

- **Ready**

  Off: Not connected to Chromeleon or Chromeleon Xpress

  On: System check passed and the ICS-90 is connected to Chromeleon or Chromeleon Xpress

  Flashing: System check failed (occurs if system check executes for 10 minutes without success)

- **Run**

  Off: Not running

  On: Running/acquiring data

  Flashing: Error/alarm/fault (including injection valve position fault)

A.5 Rear Panel

Link LED The **Link** LED displays the communication status between the ICS-90 and Chromeleon or Chromeleon Xpress:

Off: There is no communication link between the ICS-90 and Chromeleon or Chromeleon Xpress.

On: The communication link between the ICS-90 and Chromeleon or Chromeleon Xpress is activated, but data is not being transmitted or received.

Flashing: The communication link between the ICS-90 and Chromeleon or Chromeleon Xpress is activated and data is being transmitted or received.

A.6 Pump

Type	High performance pump with metal-free flow path and PEEK components
Operation Mode	Constant volume
Flow Rate	0.5 to 4.5 mL/min
Operating Pressure	28 MPa (4000 psi) maximum

A.7 Pulse Damper

Type	0.75-mm (0.030-in) ID tubing
Maximum Pressure	28 MPa (4000 psi)

A.8 Detector

Range	Digital signal range of 0 to 500 µS or 0 to 10,000 µS (user-selectable)
Temperature Compensation	Preset for accurate reading at 40 ºC
Cell Drive	8 kH square wave
Auto Offset	-999 to 999 µS
Control and Data Evaluation	Provided by Chromeleon or Chromeleon Xpress software; communication with the ICS-90 is via USB (Universal Serial Bus)
Linearity	1%

A.9 DS5 Detection Stabilizer

Cell Body	PEEK
Electrodes	Passivated 316 stainless steel
Active Volume	1 µL
Maximum Pressure	2.0 MPa (300 psi)

A.10 Injection Valve

Injection Valve	Two-position, six-port, electrically-activated Rheodyne valve with PEEK wetted components

A.11 Delay Volume

System Total	<5 mL

B • Installation

This chapter provides instructions for the initial installation of the ICS-90 Ion Chromatography System (ICS-90).

NOTE A printed installation manual (Document No. 031856) is shipped with the ICS-90.

B.1 Facility Requirements

- Make sure the ICS-90 installation site meets the power and environmental specifications listed in Appendix A.
- The ICS-90 requires a sturdy workbench of a height that ensures convenient access to the interior.
- Allow at least 15 cm (6 in) behind the ICS-90 for power connections and ventilation. For optimal performance, install the ICS-90 in a draft-free location, out of the path of air conditioning and heating vents.
- A clean gas source regulated to between 0.55 and 0.83 MPa (80 and 120 psi) is required for pressurization of the eluent reservoir.
- Use ASTM filtered, Type I (18-megohm) deionized water when preparing eluent and regenerant.

B.2 Unpacking the ICS-90 System

B.2.1 Unpacking the ICS-90

1. Open the shipping box.
2. Remove the eluent reservoir assembly and the ICS-90 Ship Kit (P/N 059947).
3. Remove the foam cover.
4. Using the cardboard side handles, carefully remove the ICS-90 from the box. You may need to remove the foam at the sides of the ICS-90 before you can lift the instrument from the box.

Lift the ICS-90 only from each side of the cabinet bottom. Lifting from the front door will damage the door hinges. Use caution when lifting the module: it weighs 19 kg (42 lb).

Ne soulevez le ICS-90 que par le fond ou les côtés. Son soulèvement par la porte du panneau avant endommagera les charnières de la porte. Soyez prudent lorsque vous soulevez le ICS-90: il pèse 19 kg (42 lb).

Heben Sie das ICS-90 nur an, indem Sie von beiden Seiten unter den Boden greifen. Wenn Sie das Gerät an der Vordertür anheben, beschädigen Sie die Türangeln. Vorsicht beim Heben des Moduls: Es wiegt 19 kg.

5. Set the ICS-90 on a workbench and cut the tape to remove the plastic surrounding the instrument.
6. Inspect the ICS-90 for any shipping damage.

B.2.2 Removing the Pump Shipping Screw

1. Carefully lay the ICS-90 on its back or side.
2. Locate the middle screw on the bottom of the ICS-90 (see Figure B-1). This screw secures the pump in place during shipment.
3. Use a Phillips screwdriver to remove the shipping screw.
4. A screw holder is located to the right of the screw (see Figure B-1). Place the screw in the holder for safekeeping. You will need to reinstall the screw if the ICS-90 is shipped to another location.
5. Return the ICS-90 to an upright position.

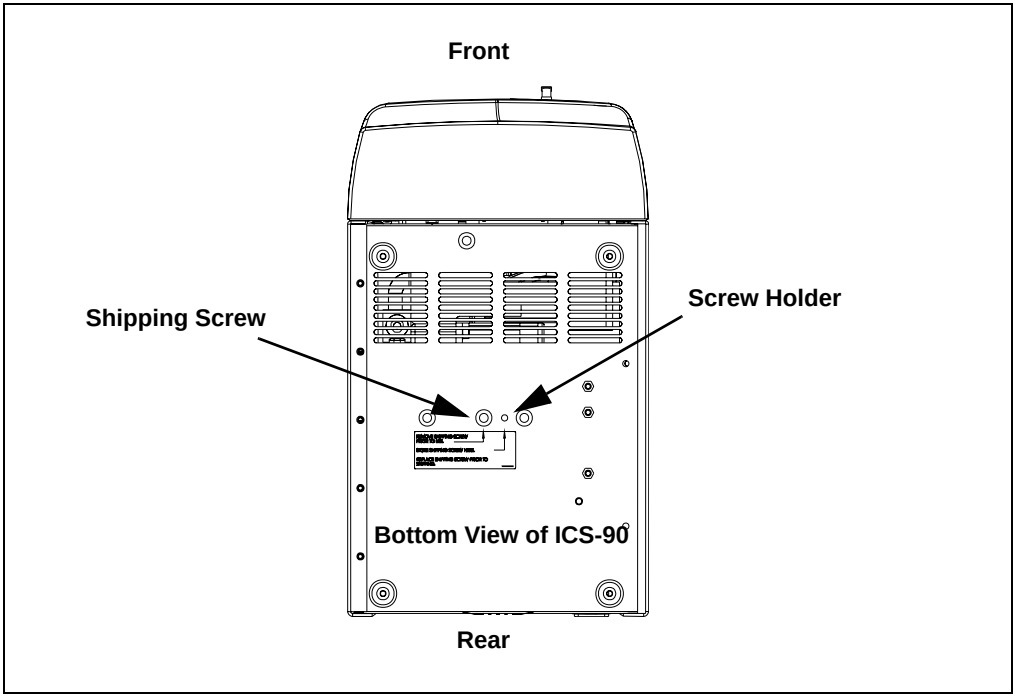


Figure B-1. Pump Shipping Screw Removal

B.2.3 Unpacking the Computer

Outside North America

1. Remove the computer and all documentation from the computer box and place them on a workbench.
2. Chromeleon and Chromeleon Xpress run under the Microsoft Windows XP and Windows 2000 operating systems. Refer to http://www.dionex.com to verify that the PC meets current system specifications.
3. Follow the instructions in the computer installation guide to hook up the PC components.
4. When you finish, go to Section B.3 to install the chromatography software.

North America Only

NOTE **These instructions assume that Chromeleon or Chromeleon Xpress and the software license were installed on the PC before shipment from Dionex.**

1. Remove the computer and all documentation from the computer box and place them on a workbench.
2. Follow the instructions in the computer installation guide to hook up the PC components.
3. When you finish, go to Section B.4 to connect the PC to the ICS-90.

B.3 Installing the Chromatography Software

B.3.1 Installing the Software

NOTE Dionex strongly recommends installing Chromeleon or Chromeleon Xpress *before* connecting the ICS-90 to the computer. When the chromatography software is installed first, USB driver information is loaded automatically and the Windows operating system will detect the ICS-90 when the power is turned on.

The SETUP.EXE file on the Chromeleon CD-ROM installs the software. After presenting a series of options for your consideration, the Setup program then copies the software onto the hard disk.

1. If this is a local computer, log onto Windows XP or Windows 2000 as an **administrator**. If this is a network computer, log on as a user with local computer administrator privileges.

   If necessary, ask the computer systems administrator for temporary administrator privileges. (Administrator privileges are required to install Chromeleon or Chromeleon Xpress, but not to operate it.)

2. Verify that the drive on which you plan to install the Chromeleon or Chromeleon Xpress application has at least 250 MB of free disk space. This is the amount of free disk space required for operation.

   Chromeleon only: 250 MB may not be enough disk space to store the large amounts of data that Chromeleon can generate. If necessary, you can store data at a different location (for example, on a network).

3. Insert the Chromeleon CD-ROM into the PC drive. The Chromeleon Setup menu should appear.

   If the Setup menu does not appear automatically, go to the Autorun folder on the Chromeleon CD-ROM and double-click **autorun.exe**. (The .exe extension is not always visible; it depends on the computer settings.)

4. Select **Launch Chromeleon Setup**.

5. Setup now guides you through the installation procedure; follow the instructions on the screen.

6. When prompted whether to add the Server Monitor program to the Startup group, select **Yes** if the computer will be physically connected to devices or **No** if the computer will never be connected to devices.

   When **Yes** is selected, the Server Monitor automatically starts when the computer is started and the program icon is displayed on the taskbar.

7. After installing the software, restart the computer.

8. Microsoft Windows should automatically detect the ICS-90 and launch the Found New Hardware Wizard. (If this does not happen, refer to the **Troubleshooting Tip** below.) Complete the wizard by selecting the following options:

   a. If asked whether Windows can connect to Windows Update to search for software, select **No, not this time**.

   b. Accept the default option (**Install the software automatically**) and click **Next >**.

   c. When the wizard reports that the software for the module has been installed, click **Finish**.

Troubleshooting Tip

If a Microsoft Windows message box asks for the USB configuration file (cmwdmusb.inf), it indicates that you connected the ICS-90 to the PC and turned on the ICS-90 power before installing Chromeleon or Chromeleon Xpress.

Follow these steps to resolve the problem:

1. Click **Cancel** in the Windows message box.
2. Turn off the power to the ICS-90 and unplug the USB cable from the computer.
3. Install Chromeleon or Chromeleon Xpress.
4. Reconnect the USB cable to the computer and turn on the power to the ICS-90. Windows will now automatically recognize the ICS-90.

B.3.2 Installing the Software License

In order for the Chromeleon or Chromeleon Xpress computer to control the ICS-90, a valid software license must be installed. To install the license, you must first install a dongle on the computer and then enter a license Key Code in Chromeleon. (A dongle is an adapter that is connected to the parallel PC interface or USB port. The dongle stores the serial number of a Chromeleon computer.)

NOTE If the software license is managed via a License Server, a dongle is not required.

1. Plug the dongle into the appropriate port on the computer (either a parallel or USB port, depending on the type of dongle). For a parallel port dongle, be sure to tighten the connector screws.
2. If it is not already running, start the Chromeleon Server Monitor (select **Start** > **Chromeleon** > **Server Monitor**).

   The Server Monitor window opens.

   - If Chromeleon or Chromeleon Xpress was installed on the computer before shipping, the Server Monitor status will display "Chromeleon Server is running idle." (This is because the license Key Code was entered before shipping.) If this is the case, click **Close** and go on to "Creating a Timebase" (see Section B.7.1).
   - If Chromeleon or Chromeleon Xpress was not pre-installed, the Server Monitor status displays "Chromeleon Server is running idle (Evaluation Mode)." (see Figure B-2). If this is the case, go on to Step 3.

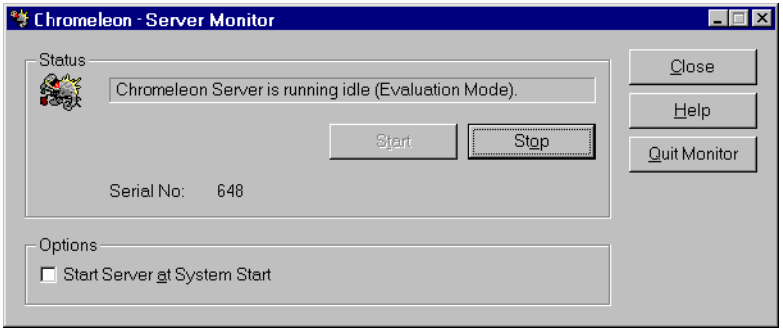


Figure B-2. Server Evaluation Mode

3. Open the Chromeleon Server Configuration program (select **Start** > **Chromeleon** > **Server Configuration**).

4. Select **Edit** > **Properties** to open the Server Configuration properties dialog box (see Figure B-3). Click **Dongle** and then enter the **Key Code** provided with the Chromeleon license. Click **OK**.

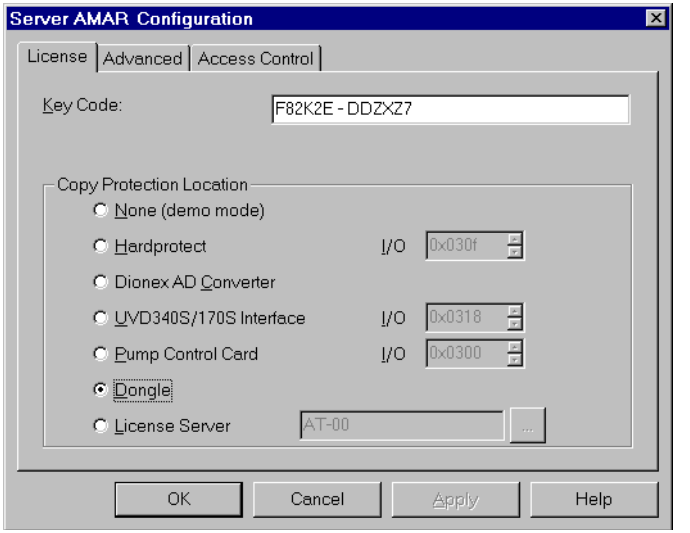


Figure B-3. Server Configuration Properties

5. Check the Server Monitor status. It should now display "Chromeleon Server is running idle." (see Figure B-4).

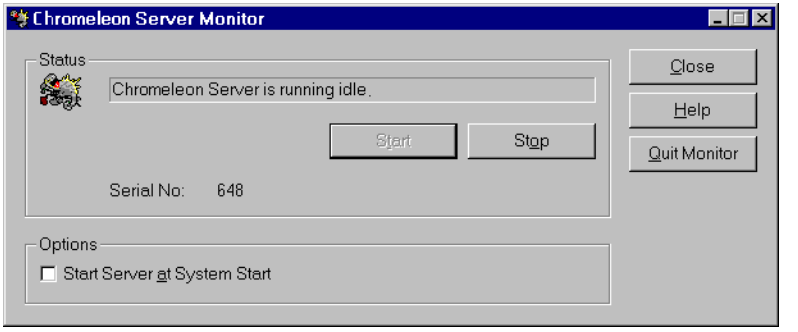


Figure B-4. Server Running

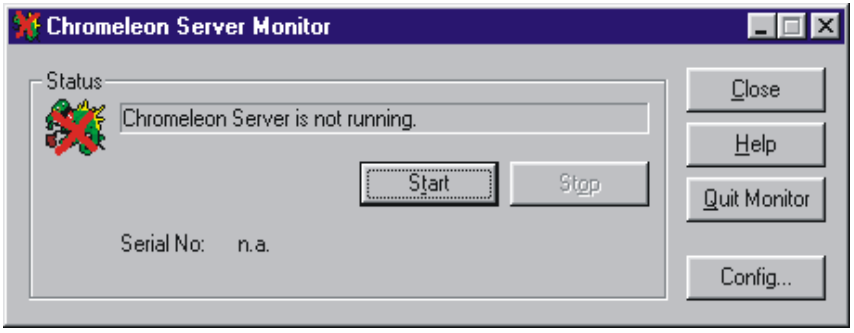
6. Click **Close** to close the Server Monitor window.

B.3.3 Starting the Chromeleon Server Monitor

1. If the Chromeleon Server Monitor program did not start automatically, start it now by double-clicking the Server Monitor icon on the Windows taskbar.

   If the Server Monitor icon is not on the taskbar, click **Start** on the taskbar and select **All Programs** (or **Programs**, depending on the operating system) > **Chromeleon** > **Server Monitor**.

2. If the server is not already running, start it by clicking **Start**.



3. Click **Close** to close the Server Monitor program window.

   The Server Monitor icon appears on the taskbar.

NOTE **Clicking the Quit Monitor button quits (or exits) the Server Monitor program, but does not stop the server. To stop the server, click the Stop button.**

B.4 Connecting the ICS-90 to the Computer

The ICS-90 rear panel (see Figure B-5) provides one USB receptacle. Select one of the following methods for connecting the ICS-90 to the PC on which Chromeleon or Chromeleon Xpress is installed:

- Connect the ICS-90 directly to a USB port on the PC.
- If there are no unused USB ports on the PC, connect the ICS-90 to an external USB hub (P/N 060392) and connect the PC to the hub.

If the dongle provided with Chromeleon or Chromeleon Xpress is for a USB port, remember to reserve a USB port for the dongle on either the PC or the hub.

IMPORTANT **Before connecting the USB cable and turning on the ICS-90 power, verify that Chromeleon or Chromeleon Xpress is installed on the PC (see Section B.3.1).**

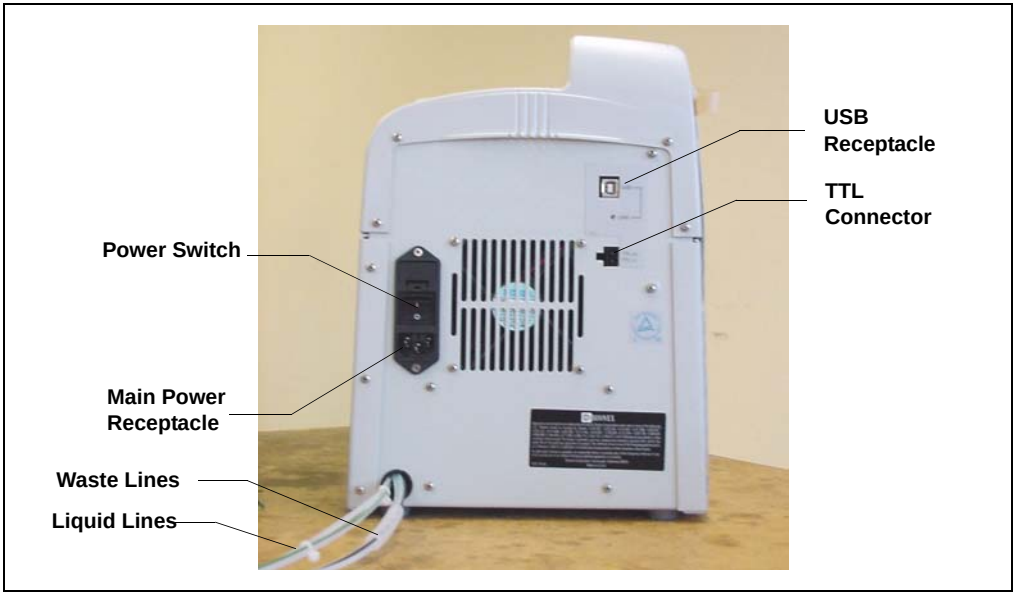


Figure B-5. ICS-90 Rear Panel

To connect the ICS-90 directly to the PC:

1. Locate the USB cable (P/N 960777) provided in the ICS-90 Ship Kit (P/N 059947).
2. Plug the "A" connector of the USB cable into the USB port on the PC (see Figure B-6).
3. Plug the "B" connector of the USB cable into the USB receptacle on the ICS-90 rear panel (see Figure B-6).

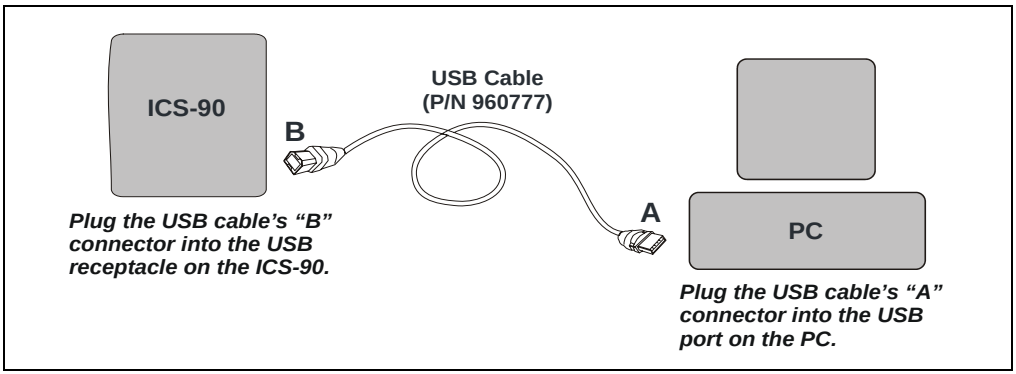


Figure B-6. Example Connections: One ICS-90 Connected to the Computer

To connect the ICS-90 to an external hub (P/N 060392):

The ICS-90 Ship Kit (P/N 059947) includes one USB cable (P/N 960777). You must order another USB cable for this configuration.

IMPORTANT **The USB standard limits the USB cable length to 5 m (5.5 yds). Each USB device can be separated from the PC by no more than five hubs. Thus, each USB device can be located no more than 30 m (32 yds) from the PC.**

IMPORTANT **Carefully secure all USB cables, the external hub, and the hub power cable so that they cannot be accidentally disconnected.**

1. Plug the "A" connector of a USB cable into a port on the external USB hub (see Figure B-7).
2. Plug the "B" connector of the cable into the USB receptacle on the ICS-90 rear panel (see Figure B-7).

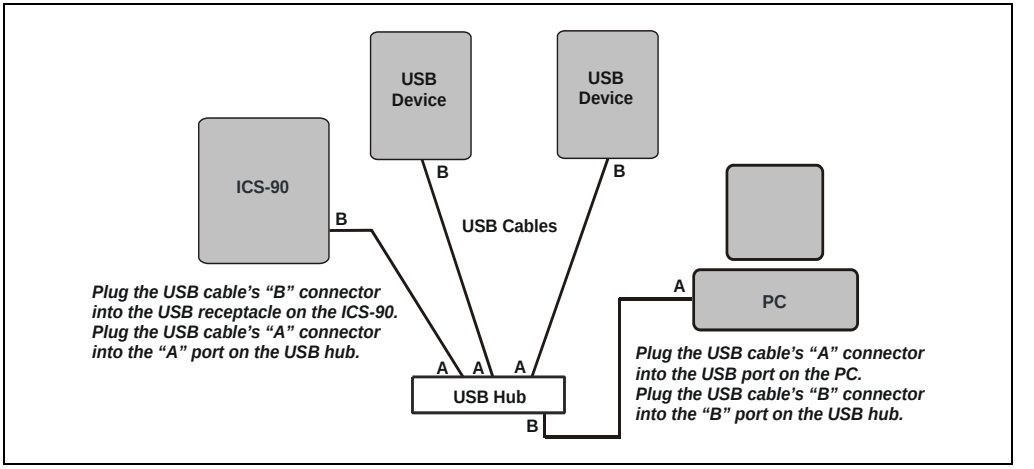


Figure B-7. Example Connections: Multiple Modules Connected to an External Hub

3. Plug the "A" connector of a USB cable into a USB port on the computer (see Figure B-7).
4. Plug the "B" connector of the cable into a port on the USB hub.

B.5 Connecting the Power Cord

1. A label on the ICS-90 rear panel indicates the line frequency (60 Hz or 50 Hz) and voltage (110 to 120 VAC or 220 to 240 VAC) for which the system is designed. Make sure the frequency and voltage are appropriate for your location. If you are unsure, consult an electrician.
2. Connect the power cord (IEC 320 C13) from the main power receptacle on the rear panel (see Figure B-5) to a grounded power source.

SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the ICS-90 or connect it to AC power mains without an earthed ground connection.

The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the ICS-90 and is easily accessible.

Operation at AC input levels outside of the specified operating voltage range may damage the ICS-90.

DANGER D'ÉLECTROCUTION—Pour éviter toute électrocution, il faut utiliser une prise de courant avec prise de terre. Ne l'utilisez pas et ne le branchez pas au secteur C.A. sans utiliser de branchement mis à la terre.

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.

STROMSCHLAGGEFAHR—Zur Vermeidung von elektrischen Schlägen ist eine geerdete Steckdose zu verwenden. Das Gerät darf nicht ohne Erdung betrieben bzw. an Wechselstrom angeschlossen werden.

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

B.6 Turning On the ICS-90 Power

IMPORTANT

Before turning on the ICS-90 power, verify that Chromeleon or Chromeleon Xpress is installed. If the chromatography software is not installed first, the Windows operating system will be unable to identify the new USB device.

1. Turn on the computer power. If this is a local computer, log onto Windows XP or Windows 2000 as an **administrator**. If this is a network computer, log on as a user with local computer administrator privileges.
2. Turn on the power to the ICS-90. The operating system automatically detects the new USB device. A message flashes on the screen to inform you that the device was detected.

B.7 Setting Up the Chromatography Software

B.7.1 Creating a Timebase

1. In the Server Configuration program, select **Edit > Add Timebase**.
2. A clock icon appears under the server name with a default name highlighted. To use the default name, press **Enter**. To use a different name, type the name and press **Enter**. Figure B-8 shows an example timebase named ICS-90.

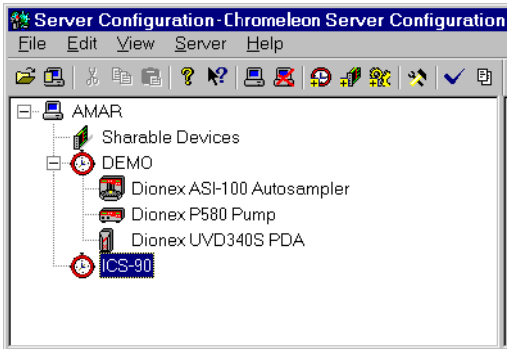


Figure B-8. Example: Creating a Timebase

3. Select **Edit > Add Device**. The Add device to timebase dialog box appears.

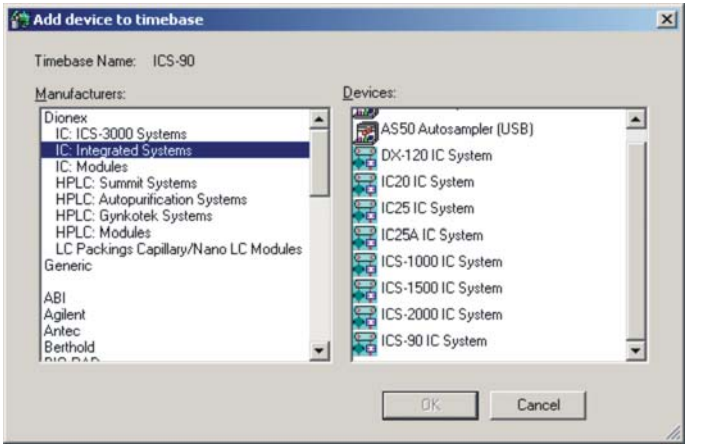


Figure B-9. Add Device to Timebase Dialog Box

4. Select **ICS-90 IC System** from the list and click **OK**. A dialog box appears.

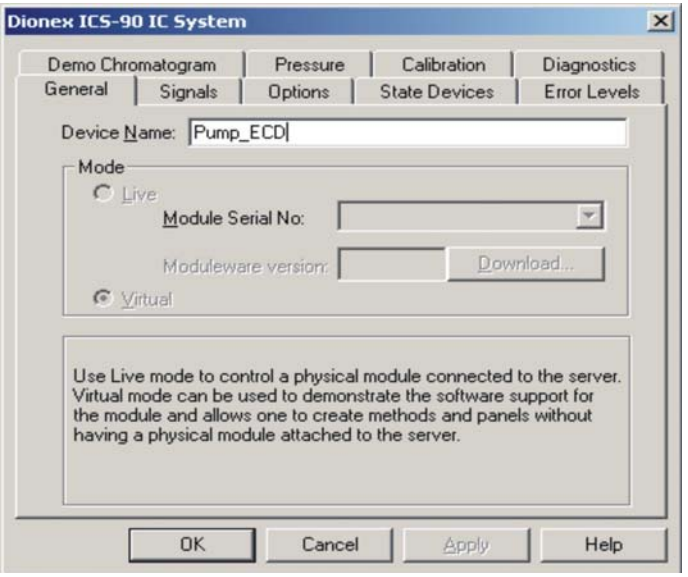
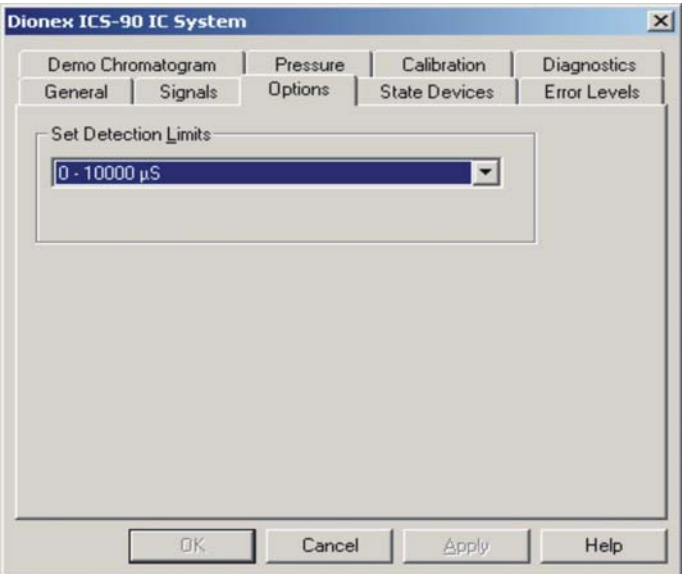


Figure B-10. ICS-90 Configuration Properties: Live Mode

5. On the **General** tab page, **Live Mode** is selected by default. Select the ICS-90 serial number from the drop-down list. If you are installing more than one ICS-90, you can verify the serial number for each system by checking the label at the top of the component panel (see Figure B-17).

6. Click **Options** to display the **Options** tab page.



7. Select the conductivity range (0 to 500 µS or 0 to 10,000 µS) from the drop-down list under **Set Detection Limits**. Selecting the larger (coarse) range reduces the detector's resolution by a factor of 20.
8. Click **OK** to close the properties dialog box.
9. Save the configuration (select **File > Save Installation**).
10. The Server Configuration Check message window appears. You can ignore the "no inject device installed" warning, if it appears. This simply means that the timebase does not include an autosampler. Click **Close**.
11. Exit the Server Configuration program.

B.7.2 Connecting to the Control Panel

1. Click **Start** on the Windows taskbar and select **All Programs** (or **Programs**) > **Chromeleon** > **Chromeleon** to start the Chromeleon client.
2. If Chromeleon is installed, select **View** > **Default Panel Tabset** or click  on the toolbar to display the panel tabset.

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

3. To display the ICS-90 (or ICS-90A) Control panel (see Figure B-11), select the **ICS-90** tab on the panel tabset. (The ICS-90A Control panel includes a **Wellness Panel** button; otherwise, the panels are identical.)

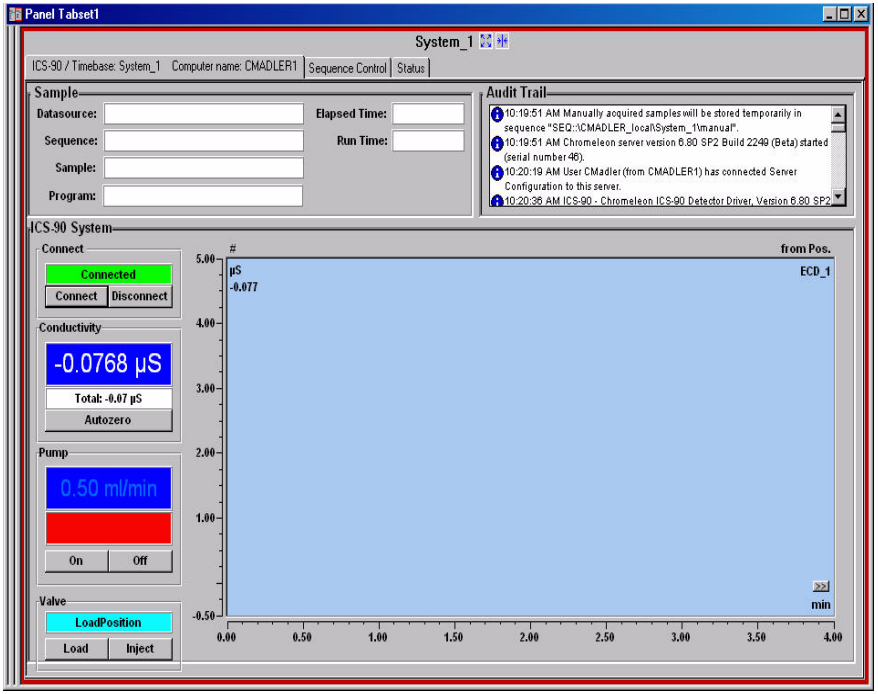


Figure B-11. ICS-90 Control Panel on the Panel Tabset

4. If a "Cannot connect to timebase…" message appears, click **OK** to close it.

   NOTE If the timebase was named "ICS-90" when it was created, the Control panel opens and connects automatically to the ICS-90 timebase.

5. If the Control panel opens with the controls not yet active, connect the system to the Control panel by clicking the **Connect** button.

   A dialog box appears (see Figure B-12).

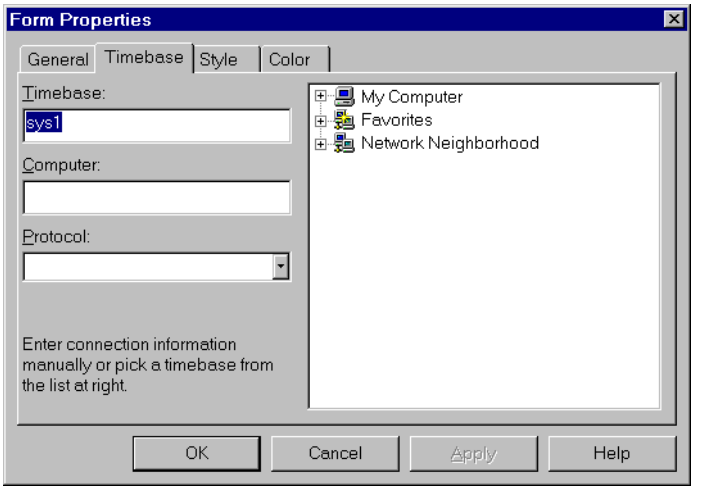


Figure B-12. Connect to Timebase

6. Click the plus sign beside **My Computer** on the right side of the dialog box and select the timebase you created in Section B.7.1. Click **OK** to close the dialog box.

   The controls on the panel are now connected.

B.7.3 Verifying Communication with the ICS-90

To verify that Chromeleon or Chromeleon Xpress is communicating with the ICS-90, click the **Disconnect** and then the **Connect** button on the Control panel while observing the ICS-90 front panel LEDs.

If the ICS-90 and the chromatography software are communicating correctly, the **Power** and **Ready** LEDs are on when the ICS-90 is connected to Chromeleon or Chromeleon Xpress, and only the **Power** LED is on when it is disconnected.

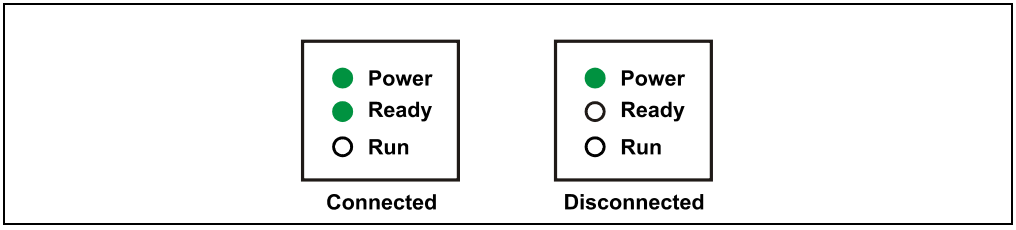


Figure B-13. ICS-90 LEDs Connected and Disconnected States

B.8 Connecting the Waste Lines

Untape the coiled waste and liquid lines from the rear panel. Place the ends of the three waste lines into a waste container. The waste lines siphon off prime waste from the pump head, sample overflow from the injection valve, and system waste from the suppressor.

NOTE To prevent waste from siphoning back into the system, check the lines periodically to be sure they are not bent, pinched, or elevated at any point.

IMPORTANT

A DCR waste backpressure tubing assembly (P/N 060214) is installed on the free end of the MMS III waste line (see Figure B-14). The assembly consists of a coupler and a 6-inch length of 0.25-mm (0.010-in) ID black tubing.

The tubing assembly provides a small amount of backpressure to compress any air bubbles formed in the suppressor's regenerant chamber. If you shorten or replace the MMS III waste line, remember to reinstall the backpressure tubing assembly. Failure to do so will result in high baseline drift.

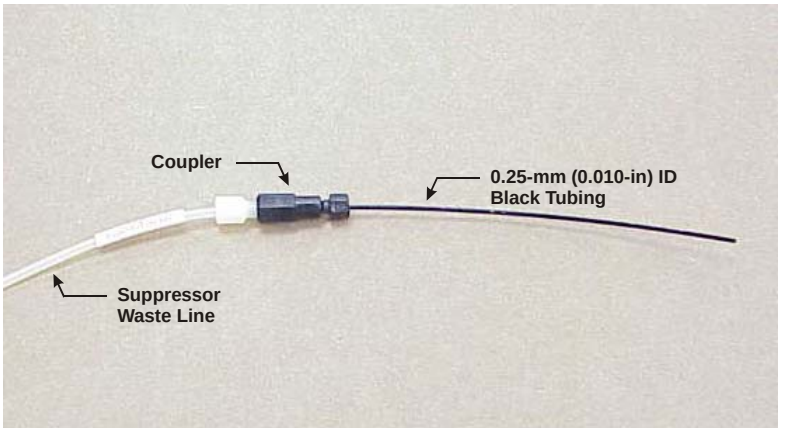


Figure B-14. DCR Waste Backpressure Tubing Assembly

B.9 Connecting the Gas Source

1. Locate the barbed fitting (P/N 030071), pipe thread reducer (P/N 030087), 1/8-in ID tubing (P/N 040793), and ICS-90 air regulator accessory (P/N 060054) in the ICS-90 Ship Kit (P/N 059947).
2. Use the barbed fitting and pipe thread reducer to connect the 1/8-in ID tubing to a clean gas source regulated to between 0.55 and 0.83 MPa (80 and 120 psi).
3. Push the other end of the tubing onto the inlet of the air regulator (see Figure B-15).

NOTE Do not turn on the gas source. Do not pressurize until the eluent is prepared and the ICS-90 is plumbed.

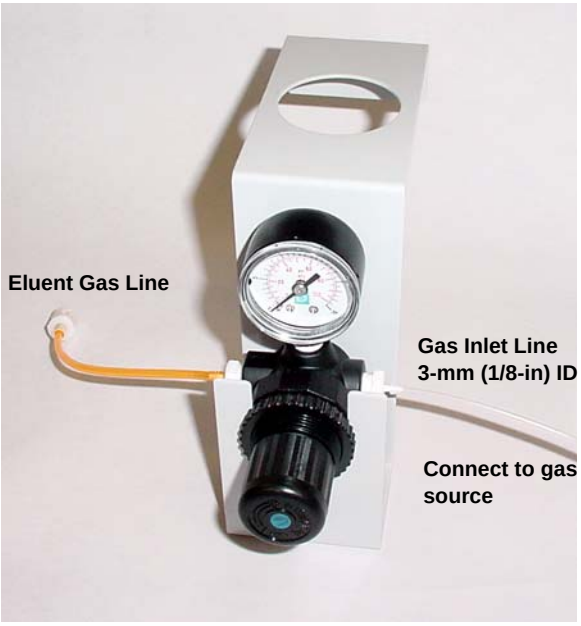


Figure B-15. ICS-90 Air Regulator Accessory

B.10 Setting Up the Eluent and Regenerant Reservoirs

1. Rinse the eluent and regenerant reservoirs.
2. Prepare 2 L of eluent. For instructions, see the manual for the column being installed. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).
3. Follow the instructions below to prepare either anion (dilute sulfuric acid) or cation (tetrabutylammonium hydroxide (TBAOH)) regenerant.
   a. Fill the regenerant reservoir (anion, P/N 057712; cation, P/N 057713) about halfway with ASTM filtered, Type I (18-megohm) deionized (DI) water.

For acid concentrates (such as the anion regenerant), always pour the concentrate into deionized water, not into the empty reservoir.

Pour les concentrés acides (comme le régénérant anionique), versez toujours le concentré dans de l'eau désionisée et non dans le réservoir vide.

Gießen Sie bei säurehaltigen Konzentraten (beispielsweise dem Kationenelutionsmittel) das Konzentrat immer in entionisiertes Wasser und nicht in den leeren Behälter.

   b. Empty the entire bottle of concentrate into the reservoir.
   c. Fill the concentrate bottle with DI water and empty the contents into the reservoir.
   d. Continue to pour DI water into the reservoir until it is filled all the way to the top. If a few drops spill over, then it is full enough.

IMPORTANT **The regenerant reservoir must be filled all the way to the top at all times.**

   e. Insert the stopper assembly tubing into the reservoir and tighten the stopper.

IMPORTANT **Do not shake the regenerant reservoir to mix the contents. The ICS-90 will stratify the contents.**

For additional information, refer to the *Installation Instructions and Troubleshooting Guide for the Displacement Chemical Regeneration (DCR) Kit* (Document No. 031664). The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

4. Make sure the regenerant reservoir is filled almost to overflowing.
5. Slip the air regulator accessory bracket over the neck of the eluent reservoir.
6. Install the caps on the eluent and regenerant reservoirs and hand tighten.
7. Place the reservoirs in the holders on the top cover of the ICS-90.
8. Connect the liquid lines from the ICS-90 to the lines exiting the eluent and regenerant reservoir caps (see Figure B-16) as follows:
   - Connect the two lines labeled **Eluent Bottle Out**.
   - Connect the two lines labeled **Regen Bottle Out**.
   - Connect the line labeled **Regen In** to the green **Regen Bottle In** line from the ICS-90.

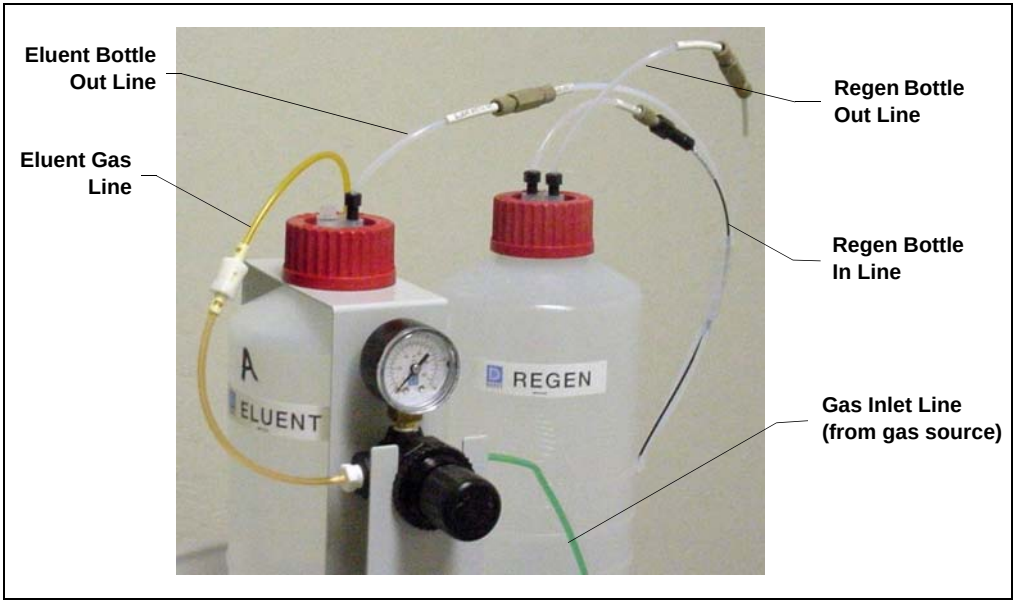


Figure B-16. Reservoir Connections

9. To ensure the correct operating pressure for the suppressor, at least one backpressure coil (P/N 045877) must be installed between the cell outlet and the regenerant reservoir inlet. The application flow rate determines the number of coils required:

Application Flow Rate	Number of Required Backpressure Coils
Less than 2 mL/min	2
2 mL/min or more	1

   a. For flow rates less than 2 mL/min, verify that two coils are installed.

   b. For flow rates of 2 mL/min or more, remove one of the backpressure coils.

NOTE **Refer to the flier entitled *Backpressure Coil Pressure Test for Dionex Suppressors* (Document No. 031759) (shipped with the suppressor) for details about suppressor operating pressure requirements.**

10. Connect the orange gas line from the eluent reservoir to the gas line on the air regulator outlet.

B.11 Installing and Plumbing the Columns and Suppressor

NOTE **This section provides brief installation and plumbing instructions for the columns and suppressor. For detailed installation information, refer to the column and suppressor manuals provided on the Dionex Reference Library CD-ROM (P/N 055405).**

1. Remove the guard column, separator column, and MMS III suppressor from their boxes. Remove the fitting plugs from the ends of each column and from all ports on the suppressor. Discard the tubing that temporarily connected the suppressor ports.
2. Plumb the guard and separator columns. Refer to the labels on the tubing, to Figure B-17, and to the following instructions.

a. Remove the union connecting **GUARD IN** and **GUARD OUT** and connect **GUARD IN** to the guard column inlet. Check the arrow on the column label; it should point away from the injection valve.

b. Connect **GUARD OUT** to the guard column outlet.

c. Push the guard column onto the clips.

d. Remove the union connecting **COL IN** and **COL OUT** and connect **COL IN** to the separator column inlet. Connect **COL OUT** to the separator column outlet. Check the arrow on the column label. It should point toward the cell (DS5).

e. Push the column onto the clips.

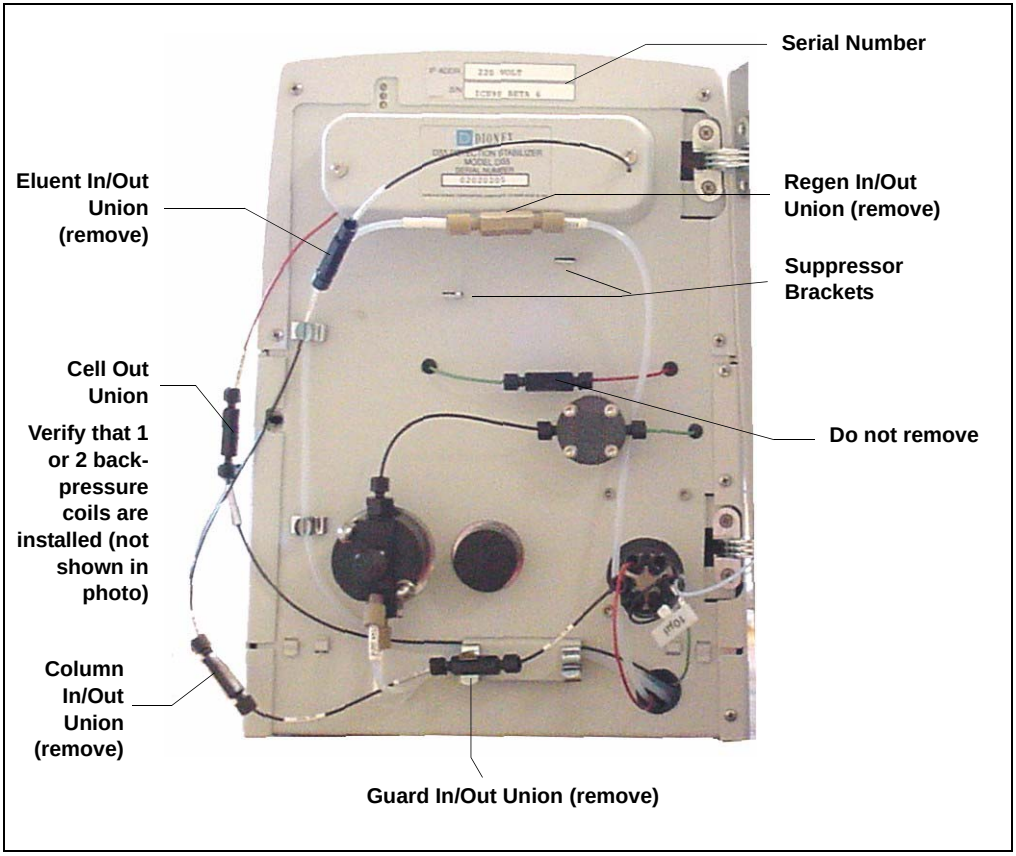


Figure B-17. ICS-90 Component Panel (Door not shown)

3. Plumb the MMS III suppressor as follows:
   a. Install the suppressor onto the brackets located below the DS5 (see Figures B-17 and B-18).
   b. Remove the union connecting **ELUENT IN** and **ELUENT OUT** and connect **ELUENT IN** and **ELUENT OUT** to the corresponding ports on the suppressor.
   c. Remove the union connecting **REGEN IN** and **REGEN OUT** and connect **REGEN IN** and **REGEN OUT** to the corresponding ports on the suppressor.

Figure B-18. ICS-90 with Columns and Suppressor Installed

4. To ensure the correct operating pressure for the suppressor, at least one backpressure coil (P/N 045877) must be installed between the cell outlet and the regenerant reservoir inlet. The application flow rate determines the number of coils required:

Application Flow Rate	Number of Required Backpressure Coils
Less than 2 mL/min	2
2 mL/min or more	1

a. For flow rates less than 2 mL/min, verify that two coils are installed.

b. For flow rates of 2 mL/min or more, remove one of the backpressure coils.

NOTE Refer to the flier entitled *Backpressure Coil Pressure Test for Dionex Suppressors* (Document No. 031759) (shipped with the suppressor) for details about suppressor operating pressure requirements.

B.12 Pressurizing the Eluent Reservoir

NOTE Do not pressurize the regenerant reservoir.

1. Pull out the air regulator knob and turn it fully counterclockwise to ensure there is no pressure to the reservoir when the gas source is turned on.
2. Turn on the gas source.
3. Pull out the air regulator knob and turn it clockwise to pressurize the eluent reservoir.
4. Adjust the pressure to between 30 and 40 kPa (5 and 6 psi). Push the regulator knob back in.

Do not pressurize the eluent reservoir above 70 kPa (10 psi).

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

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

B.13 Priming the Pump

1. Verify that the eluent and regenerant reservoirs are filled, the reservoir caps are installed and hand-tightened, and the gas and liquid lines are connected to the reservoirs (see Section B.10).
2. Verify that the gas supply is turned on, the eluent reservoir is pressurized (see Section B.12), and the regenerant reservoir is unpressurized. **Note:** The regenerant reservoir is unpressurized if the reservoirs are connected as described in Section B.10.
3. Verify that the waste lines are directed to a waste container.
4. To start the liquid flow, open the pump head waste valve (see Figure B-19) by turning the knob counterclockwise two turns. When the valve is open, eluent flows directly from the pump to waste.

Figure B-19. ICS-90 Interior Components

5. Let the liquid flow for at least 2 to 3 minutes to ensure that there are no trapped gas bubbles in the line from the eluent reservoir to the pump head.

6. Turn on the pump from the ICS-90 (or ICS-90A) Control panel in Chromeleon or Chromeleon Xpress. Allow the pump to run with the waste valve open for 30 seconds.
7. Close the waste valve knob.

B.14 Setting the Pump Flow Rate (ICS-90 only)

The instructions in this section apply to the ICS-90 only. If you are unsure whether your system is an ICS-90 or ICS-90A, check the model data label on the rear panel.

B.14.1 Setting the Flow Rate

1. Locate the production test chromatogram on the Dionex Quality Assurance Report (supplied with the separator column). Note the backpressure and flow rate listed for the column.
2. Pull out the flow rate knob on the component mounting panel (see Figure B-19) and turn the knob to the right to begin adjusting the pressure.

   NOTES

   - **There is about a 10-second delay between adjusting the knob and the pressure reading response on the ICS-90 Control panel in Chromeleon or Chromeleon Xpress.**
   - **The flow rate on the ICS-90 Control panel is a recorded value only and does not change when the flow rate knob is adjusted.**
3. Adjust the knob until the pump pressure reading on the ICS-90 Control panel reaches the figure listed on the production test chromatogram.

   NOTE The production test chromatogram is produced without a guard column. If a guard column is installed, adjust the knob until the pressure is about 1.4 to 2.1 MPa (200 to 300 psi) above the backpressure listed for the separator column alone.

4. Measure the flow rate, using one of the following methods:
   - Volumetrically (recommended for most applications): Measure the volume of liquid collected over a measured time period (see page 108 or page 109).
   - Gravimetrically: Weigh the mass of liquid collected over a measured time period (see page 109). The gravimetric method is slightly more accurate than the volumetric method.

Volumetric Method 1 (Recommended): Using a Syringe and Adapter

a. Locate the following items in the ICS-90 Ship Kit (P/N 059947): luer fitting (P/N 024305), luer coupler (P/N 042806), 1 mL syringe (P/N 016388), and 10 mL syringe (P/N 016387). You will also need a stopwatch or a clock or watch with a second hand (the computer's clock can be used), and a small beaker or other container for collecting waste.

b. Connect the luer coupler to the luer fitting.

c. Select the syringe to be used:
   - For a flow rate of 0.5 mL/min, use the 1 mL syringe.
   - For a flow rate above 0.5 mL/min, use the 10 mL syringe.

d. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor. Connect the luer coupler to the **ELUENT IN** line, and connect the syringe to the luer fitting (see Figure B-20).

Figure B-20. Assembled Syringe and Luer Adapter

e. Using a stopwatch or clock's second hand, time the flow of eluent into the syringe for 60 seconds. Determine the volume collected by noting the beginning and ending positions of the syringe plunger.

f. The volume of eluent collected in 60 seconds should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 1 mL/min, the volume should be 1.0 ± 0.1 mL.

g. If necessary, adjust the flow rate knob, and then repeat the measurement. Continue adjusting until the desired milliliters per minute is collected.

h. Reconnect the **ELUENT IN** line to the suppressor.

Volumetric Method 2: Using a Graduated Cylinder

a. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor.

b. Using a clock or watch with a second hand (the computer's clock can be used), collect eluent from the **ELUENT IN** line into a small graduated cylinder for 2 minutes.

c. The collected eluent should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 1 mL/min, 2.0 ± 0.2 mL should be collected in 2 minutes.

d. If necessary, adjust the flow rate knob, and then repeat the measurement. Continue adjustments until the desired eluent per minute is collected.

e. Reconnect the **ELUENT IN** line to the suppressor.

Gravimetric Method

a. Disconnect the **ELUENT IN** line from the **ELUENT IN** port on the suppressor.

b. Using a tared beaker and a calibrated stopwatch, collect the eluent from the **ELUENT IN** line for 60 seconds.

c. Weigh the mass of the eluent. The mass (in grams) should be approximately equal to the desired flow rate in milliliters per minute. For example, for a flow rate of 0.5 mL/min, the mass of the collected eluent should be 0.5 ± 0.02 g.

d. If necessary, adjust the flow rate knob, and then repeat Step b and Step c. Continue adjusting until the desired mass per minute is collected.

e. Reconnect the **ELUENT IN** line to the suppressor.

B.14.2 Recording the Measured Flow Rate

1. From the ICS-90 Control panel, press the **F8** key or select **Control** > **Command**.
2. Click the plus sign next to **Pump_ECD** to display the items underneath. Select **MeasuredFlowRate** (see Figure B-21).

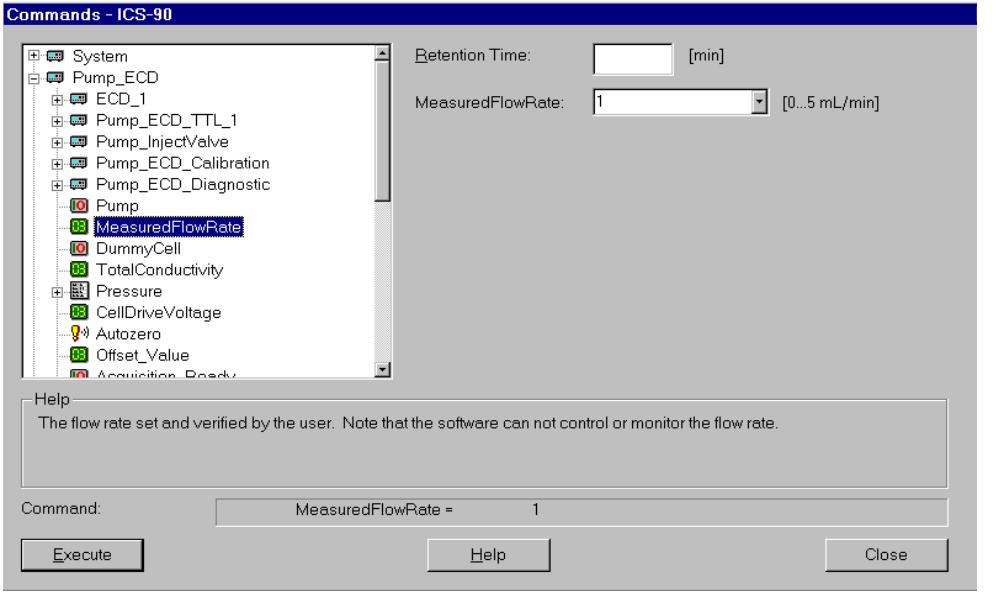


Figure B-21. ICS-90 Chromeleon Commands: MeasuredFlowRate

3. Enter the flow rate in the space provided and click **Execute**.
4. Click **Close**. The measured flow rate is now shown on the Control panel.

B.15 Setting the Pump Flow Rate (ICS-90A only)

NOTE **The instructions in this section apply to the ICS-90A only. If you are unsure whether your system is an ICS-90 or ICS-90A, check the model data label on the rear panel.**

The ICS-90A has a feature that allows the approximate flow rate to be displayed on the Control panel in Chromeleon or Chromeleon Xpress. The accuracy of the displayed flow rate depends on the flow rate at which the system was calibrated and the current pump pressure.

The ICS-90A flow rate is calibrated at the factory before shipment and does not need to be calibrated now. However, if you will typically operate the ICS-90A at a particular flow rate, you may wish to calibrate the system at that flow rate to improve flow rate accuracy. For calibration instructions, refer to Section 5.1.3.

To set the pump flow rate:

Pull out the flow rate knob on the component mounting panel (see Figure B-19) and turn the knob until the desired flow rate is displayed on the ICS-90A Control panel.

B.16 Equilibrating the System

1. After priming the pump and adjusting the flow rate, leave the pump on and flush the system for about 5 minutes to equilibrate.
2. Monitor the system pressure from the ICS-90 Control panel to make sure the pressure is between 107 and 153 MPa (1600 and 2300 psi). If the pressure is less than 107 MPa (1600 psi), gas may be trapped in the system. Release the gas by opening one fitting at a time from the pump head and along the plumbing to the column; pay close attention to the line from the pulse damper (see Figure B-19).
3. Check that there are no leaks in the regenerant reservoir.
4. Check that liquid is flowing out of the suppressor **REGEN OUT** waste line and that the pressure is stable.
5. Monitor the baseline conductivity. In general, it should be <30 µS for a system set up for anion analyses, and <2 µS for a system set up for cation analyses.

B.17 Verifying Operational Status

After the system has equilibrated, verify the actual pump pressure and stability by monitoring the pump pressure. Record the short-term pressure fluctuations; they should be less than 0.13 MPa (20 psi).

B.18 Connecting the AS40 Automated Sampler (Optional)

1. Open the front door of the ICS-90 and thread the outlet line from the AS40 through the side slot near the lower ICS-90 door hinge (see Figure B-18).
2. Connect the outlet line from the AS40 to port S (5) of the injection valve (see Figure B-22).

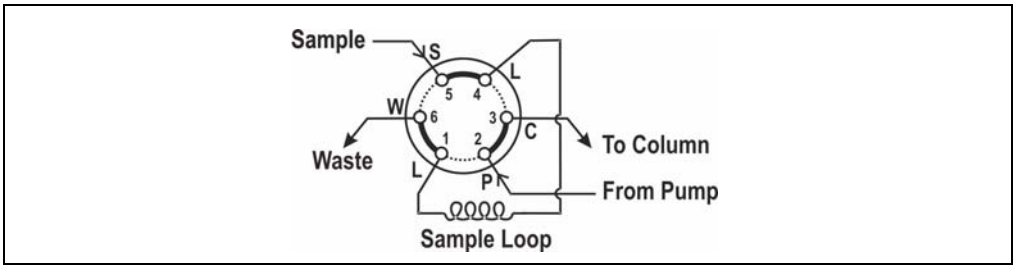


Figure B-22. Injection Valve Connections

3. Follow the steps below to connect the **TTL Output** connector on the ICS-90 rear panel to the **RELAY CONTROL** connector on the AS40 rear panel.
   a. Locate the Relay Control cable (P/N 047946) supplied with the AS40.
   b. Connect the Relay Control cable's 10-pin connector to the **RELAY CONTROL** connector on the AS40 rear panel (see Figure B-23).

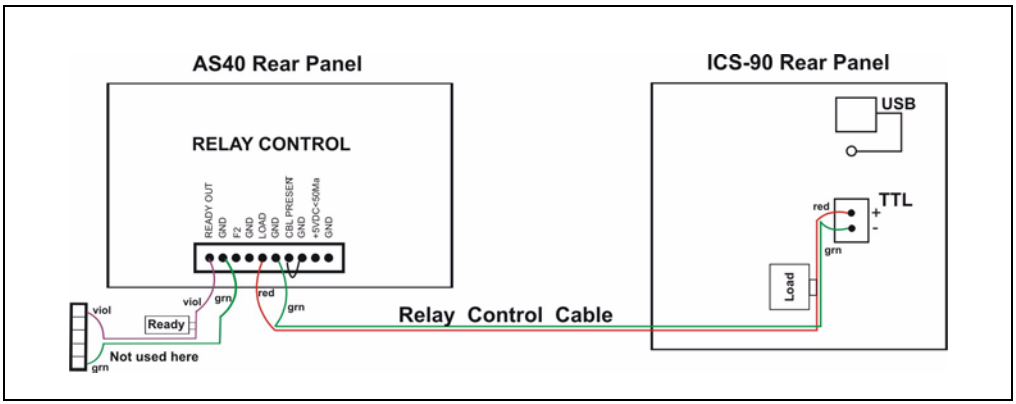


Figure B-23. AS40 Automated Sampler Connection

c. Plug the 2-pin connector from the pair of wires labeled **LOAD** on the Relay Control cable into the **TTL Output** connector on the ICS-90.

B.19 Operating the ICS-90

ICS-90 installation is complete. For routine operation, refer to Section 3.5.

B.20 Installation Troubleshooting

Problem: When the ICS-90 power is turned on for the first time, a Windows message box appears asking for a USB configuration file (cmwdmusb.inf).

Possible Cause: The USB cable was connected and the power turned on before Chromeleon or Chromeleon Xpress was installed.

Solution:

1. Click the **Cancel** button in the Windows message box.
2. Turn off the ICS-90 power and unplug the USB cable from the computer.
3. Install Chromeleon or Chromeleon Xpress (see Section B.3.2).
4. Reconnect the USB cable to the computer and turn on the ICS-90 power. Windows will now automatically recognize the new ICS-90.

C • Reordering Information

Part Number	Item
	Pump
057853	Pump head assembly
055870	Main piston seal
036901	Backup piston seal
035776	O-ring
047661	Outlet check valve assembly, 10-32
047660	Inlet check valve assembly, 1/4-28
057233	Backup washer assembly
035030	Piston guide
036904	Piston
035014	Piston return spring
035595	Piston spring guide
035885	Washer (installed under acorn nut on pump head assembly)
036884	Acorn nut (secures the pump head to the pump housing)
	Sample Loop and Injection Valve
042949	10 µL sample loop assembly
057896	Injection valve rebuild kit
024305	Luer adapter fitting, 1/4-28 (for manual injections)
	Reagent Reservoir Assemblies
057711	Eluent reservoir assembly (includes stopper and cap)
057712	Anion regenerant reservoir assembly (includes stopper and cap)
057713	Cation regenerant reservoir assembly (includes stopper and cap)
059068	O-ring for eluent or regenerant reservoir stopper

Part Number	Item
	Reagents
057559	Anion Regenerant Concentrate (75 mL of 2.0 N sulfuric acid)
057555	4-Pack of Anion Regenerant Concentrate
057561	Cation Regenerant Concentrate (100 mL of 2.06 M TBAOH)
057556	4-Pack of Cation Regenerant Concentrate
	Suppressors and Cell
056751	AMMS III suppressor
056753	CMMS III suppressor
057290T	DS5 Detection Stabilizer

D.1 How do I hook up an AS40 Automated Sampler?

For instructions on how to connect the ICS-90 to an AS40, see Section B.18 of this manual. Also see the *AS40 Automated Sampler Operator's Manual* (Document No. 034970). The manual is included on the Dionex Reference Library CD-ROM (P/N 055405).

D.2 How do I print?

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

D.3 How often should I perform calibrations?

Recalibrate every six months or after changing a major component (separator column, DS5, suppressor, etc.).

D.4 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 4.6 for pump flow rate troubleshooting. If a contaminated column is suspected, clean the column as instructed in the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

D.5 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 guide for details.

D.6 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.7 When should I remake eluents?

Eluents should be remade every two or three weeks.

D.8 How do I start Chromeleon or Chromeleon Xpress?

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

D.9 How do I back up data?

In Chromeleon, select **File** > **Export/Backup**. Back up the data and indicate the backup source. Chromeleon Xpress does not allow data to be saved.

D.10 How do I delete data?

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

D.11 How do I shut off the system?

In Chromeleon or Chromeleon Xpress, turn off the pump from the ICS-90 (or ICS-90A) Control panel (see Figure 2-9). On the ICS-90, turn off the power switch on the rear panel (see Figure 2-2).

D.12 How do I store columns?

Columns should be stored in eluent. See the column manual for complete instructions. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

D.13 How do I know when a column is dirty?

See the troubleshooting section of the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

D.14 How do I clean a column?

See the troubleshooting section of the column manual. Column manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).

D.15 Why is the conductivity high?

Possible reasons for high conductivity include:

- The suppressor needs regeneration. See the suppressor manual for troubleshooting information. Suppressor manuals are included on the Dionex Reference Library CD-ROM (P/N 055405).
- The regenerant is exhausted and should be remade. See Section 3.2 for instructions on preparing regenerant.
- The DS5 is out of calibration. See Section 5.1.2 for calibration instructions.

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.

Electrical Conductance

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 cm^2 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)
Height **E**quivalent to a **T**heoretical **P**late. A measure of column efficiency which allows comparison between columns of different lengths.

$$HETP = H = L/N$$

Where: L = the column length (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 which converts ion exchange sites in the suppressor back to the form which will suppress the eluent conductivity.

Resin
See **Ion-Exchange Resin**.

Resolution (R)
A measure of the separation between two sample components. It 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 separations; also called 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 ^{o}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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