



DX-120 ION CHROMATOGRAPH OPERATOR'S MANUAL

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1 • Introduction

The Dionex DX-120 Ion Chromatograph performs isocratic ion analysis applications using conductivity detection. The DX-120 is an integrated system, which includes a pump, detector, and injection valve. The chromatography components, including the column(s), Self-Regenerating Suppressor™ (SRS™), and conductivity cell are ordered separately. These components are mounted on the inside of the DX-120 door.

The DX-120 can be controlled locally, using the front panel keypad and display, or remotely, from a PeakNet workstation. The PeakNet workstation consists of a computer with a Dionex DX-LAN™ interface card and PeakNet software (Release 4.30 or higher) installed. Limited remote control is also available using TTL signals.

The DX-120 can be ordered as a single-column system or a dual-column system. The dual-column system allows switching between two sets of columns (column select mode) or between two eluents (eluent select mode). In the eluent select mode, the DX-120 can perform step gradients.

The following models are available:

Configuration	Voltage/Line Frequency*	Without DX-LAN	With DX-LAN
Single-column	115 VAC/60 Hz	P/N 050100	P/N 050200
	100 VAC/50 Hz	P/N 050103	P/N 050203
	230 VAC/50 Hz	P/N 050106	P/N 050206
Dual-column	115 VAC/60 Hz	P/N 050102	P/N 050202
	100 VAC/50 Hz	P/N 050105	P/N 050205
	230 VAC/50 Hz	P/N 050108	P/N 050208

*Must match the voltage and line frequency of the installation site's power source.

1.1 About This Manual

Chapter 1 Introduction	Introduces the DX-120 and explains the conventions used in the manual, including safety-related information.
Chapter 2 Description	Describes the DX-120 operating features, the chromatographic fluid path, and the control modes.
Chapter 3 Operation and Maintenance	Provides operating and routine preventive maintenance procedures.
Chapter 4 Troubleshooting	Lists problems, with step-by-step procedures to isolate and eliminate their sources.
Chapter 5 Service	Provides step-by-step instructions for routine service and parts replacement procedures.
Appendix A Specifications	Lists the DX-120 specifications and installation site requirements.
Appendix B Installation	Describes how to install the DX-120.
Appendix C Integrator Programming	Describes how to program a Dionex 4400 or 4600 integrator for automated control of the DX-120.
Appendix D Conductivity Detection	Describes conductivity detection and its applications.
Appendix E Glossary	Defines terms commonly used in ion chromatography.

1.1.1 Typefaces and Conventions

- Capitalized bold type indicates a front panel button. For example:

Press **Pump** to turn on the pump.

- Upper-case bold type indicates information displayed on the front panel screen. For example:

LEAK ALARM displays when a leak occurs.

- Upper-case italic type indicates a grouping of front panel buttons. For example:

Use the buttons in the *DISPLAY* group to select the type of information shown on-screen.

- When a function can be controlled by a DIP switch setting, the switch and position numbers are in parentheses. For example:

(SW1-3) indicates DIP switch 1, position 3.

(SW2-5, 6, 7, 8) indicates DIP switch 2, positions 5, 6, 7, and 8.

1.1.2 Safety Messages and Notes

The DX-120 meets European, EMC, and safety requirements per Council Directives 73/23/EEC and 89/336/EEC, EN 61010-1:1993 (safety), EN 50082-1:1992 (susceptibility), and EN 55011:1991 (emissions). The TUV/CE and GS safety label on the DX-120 attests to compliance with these standards.

The DX-120 is designed for ion chromatography applications and should not be used for any other purpose. If there is a question regarding appropriate usage, contact Dionex before proceeding.

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 follows:



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



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



Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury.



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

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.

1.1.3 Symbols

The symbols below appear on the DX-120, or on DX-120 labels.



Alternating current



Protective conductor terminal



Power supply is on



Power supply is off

1.2 Related Manuals

During installation and operation of the DX-120, you may need to refer to one or more of the following manuals for information about other components or instruments in the system.

- *4440 Integrator User's Guide* (Document No. 034200) or *4600 Integrator User's Guide* (Document No. 034408)
- *AS40 Automated Sampler Operator's Manual* (Document No. 034970)
- *Installation of Dionex Ferrule Fittings* (Document No. 034213)

DX-120 Operator's Manual

- *PeakNet Software User's Guide* (Document No. 034914)
- *Installing the PeakNet System* (Document No. 034941)

2 • Description

2.1 Operating Features

Figures 2-1 and 2-2 illustrate the main operating features of the DX-120.

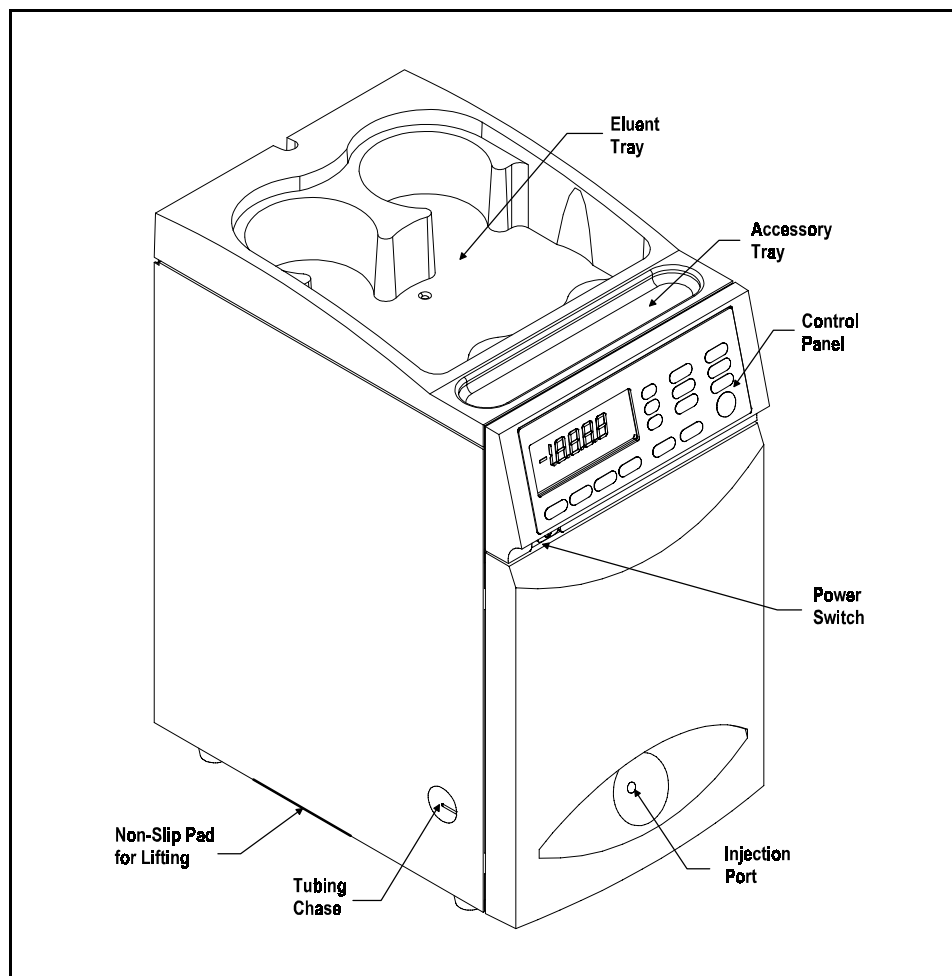


Figure 2-1. DX-120 Operating Features (Exterior)

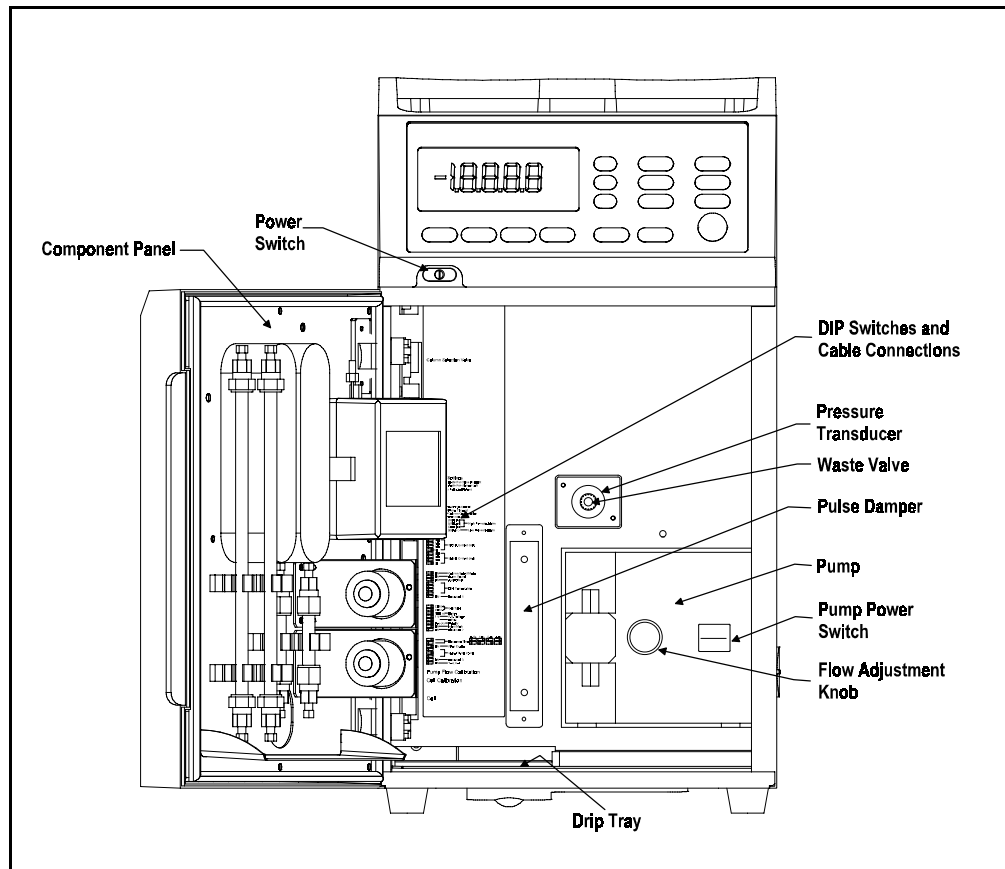


Figure 2-2. DX-120 Operating Features (Interior)

2.1.1 Front Control Panel

The control panel liquid crystal display (LCD) shows status information and alarm conditions. Press a button in the group labeled *DISPLAY* to determine the type of status information shown. The remaining buttons control DX-120 operation.

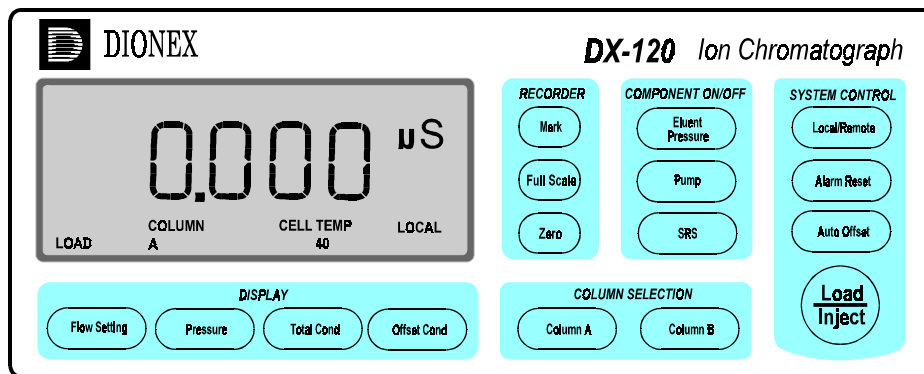


Figure 2-3. DX-120 Control Panel

Button	Function
DISPLAY	
Flow Setting	Displays the pump flow rate setting (0.5 to 4.5 mL/min).
Pressure	Displays the pump pressure transducer reading (0 to 27.6 MPa or 0 to 4000 psi)
NOTE MPa is the default pressure unit. For psi, set DIP SW1-4 on. See Section B.10 for details.	
Total Cond	Displays the total conductivity reading (0 to 999.9 μ S).
Offset Cond	Displays the offset conductivity reading (-999.9 to 999.9 μ S). Offset conductivity is the total conductivity minus the offset for the current run (see the description of the Auto Offset button).

Table 2-1. Control Panel Button Functions



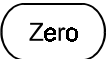

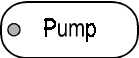





Button	Function
RECORDER	
	Delivers a chart mark signal to the analog output. The mark is 10% of the full-scale voltage.
	Delivers a 100% signal to the analog output. Pressing the button continuously keeps the output signal at full scale. The default full-scale voltage is 1 V, but can be changed to 10 V (SW4-4).
	Reduces the analog output signal to zero. Pressing the button continuously keeps the output signal at zero.
COMPONENT ON/OFF	
	Turns the gas pressure to the eluent reservoirs on and off. When on, the LED on the button is illuminated. This button is disabled when the DX-120 is in Remote mode (see Local/Remote below).
	Turns the pump flow on and off. When on, the LED on the button is illuminated. This button functions in Local or Remote mode.
	Turns the SRS power on and off, as well as the DS4 Detection Stabilizer, if installed. When on, the LED on the button is illuminated. In the dual-column configuration, this button controls the power to the SRS and DS4 of the selected column set (see Section 2.2.1). This button is disabled when the DX-120 is in Remote mode (see Local/Remote below).
SYSTEM CONTROL	
	Toggles between Local and Remote control modes. Local is control from the DX-120 control panel and Remote is control from a PeakNet workstation via the DX-LAN. The selected mode is shown in the lower right-hand corner of the display.
	A red LED on the left side of the button indicates an alarm condition. The top line of the display indicates the source of the alarm: cabinet or cell leak, high or low pressure, or SRS. Pressing the button clears the alarm. If the alarm condition is still present, the alarm reappears after 15 seconds.

Table 2-1. Control Panel Button Functions (continued)

Button	Function
	Offsets the background conductivity. After start-up, allow the system to equilibrate. The display shows the background conductivity (the eluent conductivity before sample injection). Press Auto Offset to offset this background reading and zero the chromatogram baseline.
	<p>Switches the injection valve between the Load and Inject positions. The current position is shown in the lower left-hand corner of the display.</p> <p>When switched from Load to Inject, the DX-120 also:</p> <ul style="list-style-type: none"> - sends a chart mark signal to the analog output (SW4-7) - performs an auto offset (SW3-3) - sends a TTL signal indicating that an injection has occurred <p>After 1 minute, the valve automatically returns to the Load position (SW1-1).</p> <p>This button is disabled when the DX-120 is in Remote mode.</p>

COLUMN SELECTION

The COLUMN SELECTION buttons are active in the dual-column configuration only.

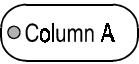
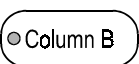
	In column select mode, this button initiates the column switching sequence from column set B to column set A (see Section 2.2.1). In eluent select mode, this button switches to eluent delivery from line A (see Section 2.2.2).
	In column select mode, this button initiates the column switching sequence from column set A to column set B (see Section 2.2.1). In eluent select mode, this button switches to eluent delivery from line B (see Section 2.2.2).
These buttons are disabled when the DX-120 is in Remote mode.	

Table 2-1. Control Panel Button Functions (continued)

2.1.2 Pump

The pump is located on the right side of the main compartment (see Figure 2-2). The knob on the front of the pump adjusts the flow rate from 0.5 to 4.5 mL/min.

Eluent Save Mode

If the DX-120 is idle (i.e., no control panel buttons have been pressed and no PeakNet commands have been received) for 90 minutes, the pump flow automatically decreases to 1/20th of its current flow rate and the SRS cycles on and off. When this occurs, the LEDs on the **Pump** and **SRS** buttons flash. Press any button to return to the last selected flow rate.

To turn off this feature, reset the Pump Time-out DIP switch (SW1-2).

2.1.3 Configuration DIP Switches

The DIP switches on the left side of the main compartment control system parameters. The factory-set defaults can be changed to meet specific system and application requirements (see Section B.10).

NOTE

In this manual, when a function is controlled by a DIP switch setting, the switch and position numbers are shown in parentheses. For example: (SW1-3, 4) indicates DIP switch 1, positions 3 and 4.

2.1.4 Eluent Reservoirs

Dionex strongly recommends degassing all eluents and storing them in reservoirs pressurized with helium. This helps prevent bubbles (resulting from eluent outgassing) from forming in the pump head and the detector cell. Degassed eluents and pressurized reservoirs are especially important when combining aqueous and non-aqueous components (e.g., water and methanol). With non-aqueous components, glass reservoirs are recommended.

The single-column DX-120 includes one 2-liter plastic reservoir (P/N 044129). The dual-column DX-120 includes two 2-liter plastic reservoirs.

The following additional reservoirs are available from Dionex:

- 1-liter plastic reservoir (P/N 044128)
- 1-liter glass reservoir with shatterproof plastic coating (P/N 044126)
- 2-liter glass reservoir with shatterproof plastic coating (P/N 044127)



The 2-liter plastic reservoir is not designed for vacuum degassing. Do not use it for this purpose.

2.1.5 Component Panel

Chromatography components are mounted on the inside front door. Figure 2-4 shows the single-column component panel layout. Figure 2-5 shows the dual-column layout.

The DX-120 is equipped with a Rheodyne injection valve (see Section 2.1.6). The following additional components must be ordered separately:

- *Self-Regenerating Suppressor(s) (SRS)*: The SRS neutralizes the eluent and enhances analyte conductivity. For a dual-column system, order two suppressors.
- *Column(s)*: One or two analytical columns and one or two guard columns can be installed on column clips. The clips have larger clasps on one side for supporting 4 mm columns and smaller clasps on the other side for 2 mm columns.
- *Column switching valve*: The column switching valve is installed only in a dual-column system. The valve controls liquid flow to the selected column (in column select mode) or from the selected eluent (in eluent select mode). See Section 2.2 for details about dual-column systems.
- *Detector cell*: Only one flow-through cell is required in either the single- or the dual-column system. Two cell models are available: a DX-120 standard cell (the CDM-3) and a DX-120 high-performance cell with heater (the DS4 Detection Stabilizer). The DS4 is recommended for applications requiring enhanced thermal stability. See sections 2.1.7 and 2.1.8 for details about the cells and the DS4.

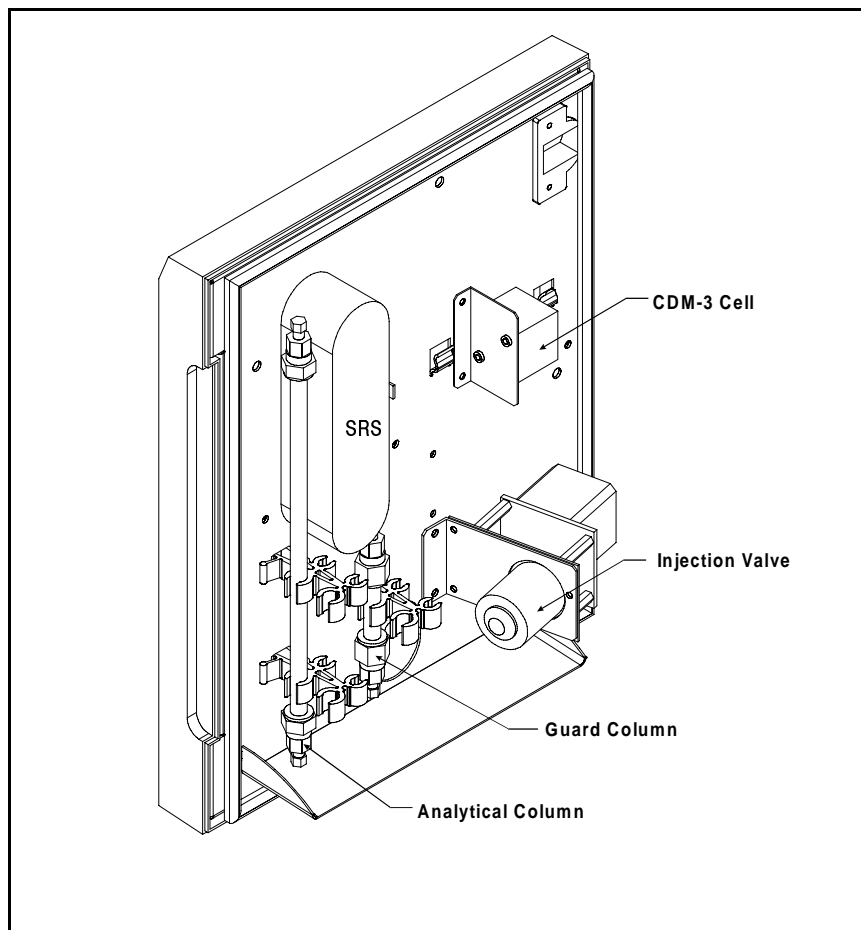


Figure 2-4. Single-Column Component Panel Layout

NOTE

A DS4 Detection Stabilizer can be installed instead of the CDM-3 cell.

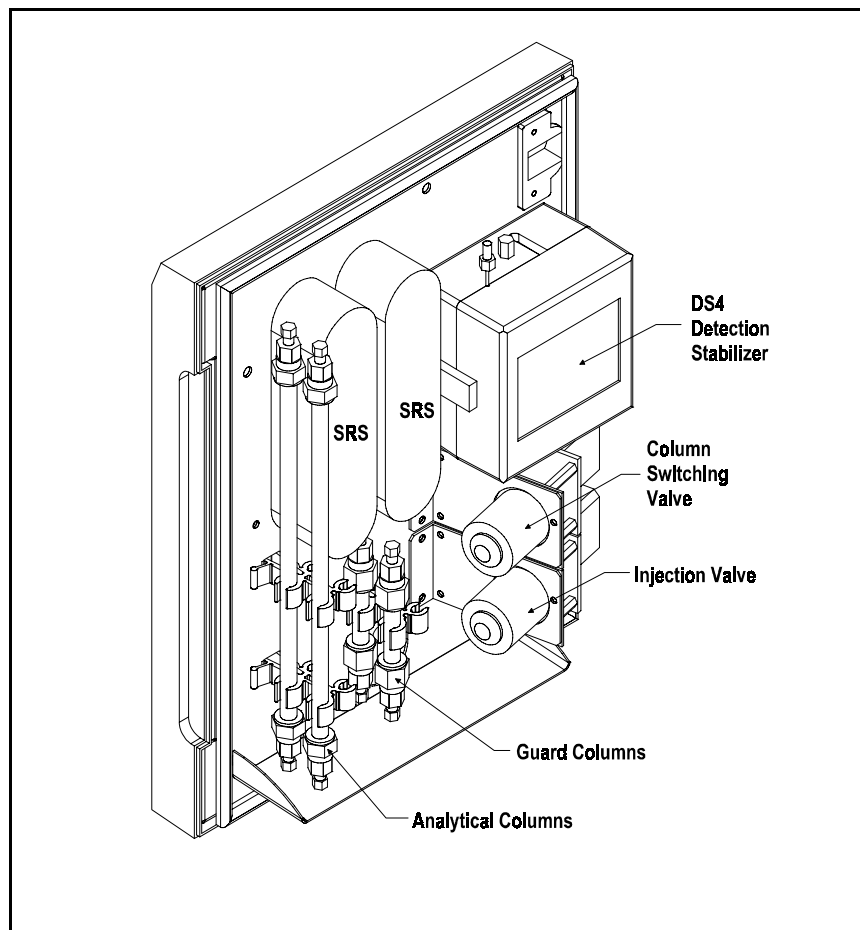


Figure 2-5. Dual-Column Component Panel Layout

NOTE

A CDM-3 cell can be installed instead of the DS4 Detection Stabilizer.

2.1.6 Rheodyne Injection Valve

The Rheodyne injection valve has two operating positions: Load and Inject. In the Load position, sample is loaded into the sample loop, where it is held until injection. In the Inject position, sample is swept to the column for analysis. Eluent flows through one of two paths, depending on the valve position:

- In the Load position, eluent flows from the pump, through the valve, and to the column, bypassing the sample loop. Sample flows from the syringe or autosampler line, through the valve, and into the sample loop; excess sample flows out to waste.
- In the Inject position, eluent flows from the pump, through the sample loop, and on to the column, carrying the contents of the sample loop with it.

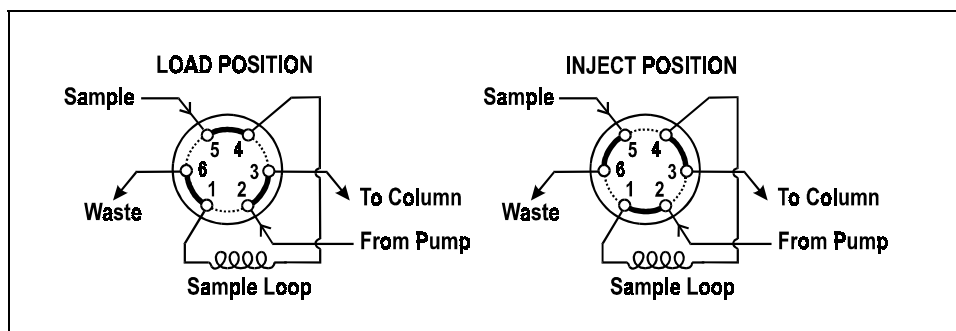


Figure 2-6. Rheodyne Injection Valve Flow Schematics

2.1.7 Detector Cells

The DX-120 accommodates two detector cell models. The DX-120 standard cell (CDM-3; P/N 050776) is used for applications that do not require the enhanced baseline stability gained through thermal stabilization. For increased thermal stabilization in high-sensitivity applications, use a DX-120 high-performance cell with heater (DS4 Detection Stabilizer; P/N 050218).

NOTE

If you change the cell model, the Cell Type DIP switches (SW4-1, 2) must be reset to select the new cell type.

Detector Cell Features

- Both cells are flow-through conductivity cells with polymeric bodies. Two 316 stainless steel electrodes are permanently sealed into the cell bodies.
- A sensor (thermistor) located slightly downstream from the electrodes senses the temperature of the liquid as it exits the cell. The measured value is used for temperature compensation.
- The active volume is nominally 1.25 μL for the CDM-3 cell and 1.0 μL for the DX-120 high-performance cell.
- The detector cell constant for both cells has a nominal value of 160 cm^{-1} .

The advanced geometry of the cells provide several benefits:

- Excellent accuracy and linearity over a broad working range
- Efficient sweepout and low volume for low dispersion
- Reduced sensitivity to electrode surface conditions
- Low electrode mass
- Effective temperature compensation

Temperature Control and Compensation

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 chromatography, suppressing eluent conductivity minimizes the effect of temperature variation. Temperature compensation further improves baseline stability. When the conductivity cell is housed in a DS4 Detection Stabilizer, the heater enhances the ability of these techniques to reduce temperature effects on conductivity.

Temperature compensation also ensures that there is no major change in the baseline or peak heights, should it be necessary to change the DS4 operating set point. Readings will be normalized to 25 °C.

2.1.8 DS4 Detection Stabilizer

The DS4 is a temperature-controlled chamber consisting of a cast aluminum base and cover enclosed in insulating foam. The chamber houses both the conductivity cell and the eluent heat exchanger. Figure 2-7 identifies the major components of the DS4.

The DS4 provides the following benefits:

- Conductivity measurements nearly impervious to laboratory temperature variation
- Very low dispersion in the eluent heat exchanger
- Excellent peak height reproducibility
- User-selected temperature set point

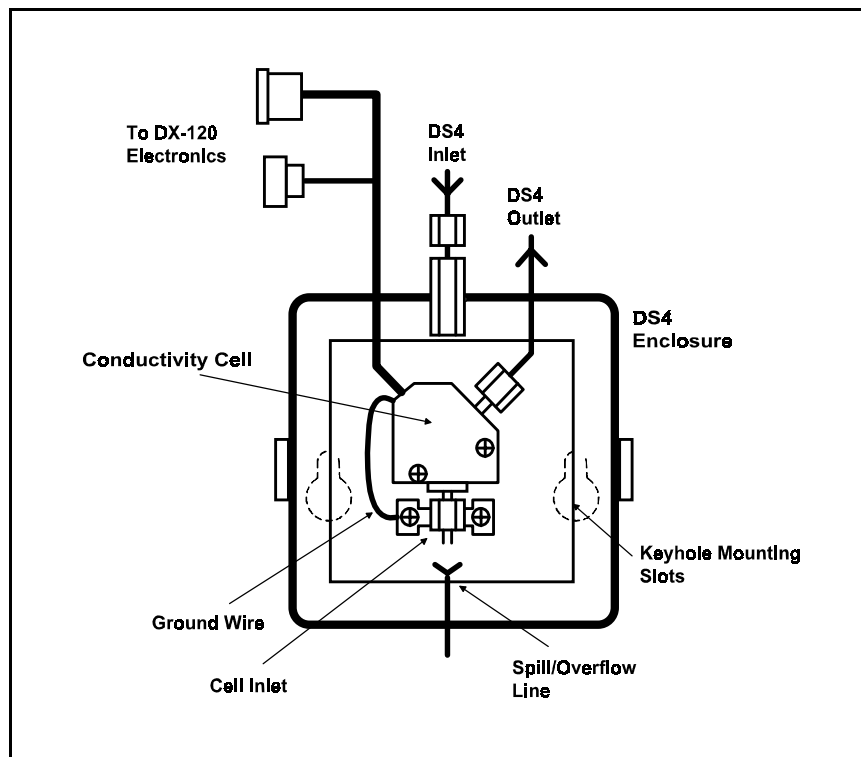


Figure 2-7. DS4 Detection Stabilizer Features

Power input to a pair of transistors on opposite sides of the DS4 heats it to a user-selected temperature from 30 °C to 45 °C (SW3-4, 5, 6, 7). A sensor near the heat exchanger outlet senses the eluent temperature. The DX-120 circuitry compares this temperature with the selected temperature and adjusts the heat input in real time to hold the temperature within a few millidegrees.

The DS4 is sealed with an O-ring to trap eluent that may leak from the cell. If 5 mL of liquid accumulates, a thermistor sensor signals a leak to the CPU. Any additional leakage will be discharged via the spill/overflow line. A second thermistor, above the discharge level, acts as a temperature reference for the leak sensor.

2.2 Dual-Column Configuration Features

The dual-column configuration has two operating modes:

- Column select mode allows switching of flow from one column set to the other.
- Eluent select mode allows switching of flow from one eluent to the other (the column set is not switched).

The Column Select DIP switch (SW3-1) selects the mode:
on=column; off=eluent.

The dual-column system option adds the following features:

- The **Column A** and **Column B** buttons on the front control panel are enabled.
- An eluent selection valve selects which eluent reservoir is used.
- A column switching valve directs flow to the selected column set (column select mode only).

2.2.1 Column Select Mode

In column select mode, you can switch the flow path from one column set to the other by pressing **Column A** or **Column B** or by sending a command from PeakNet.

Figure 2-8 illustrates the sequence of events when you switch from column set A to column set B.

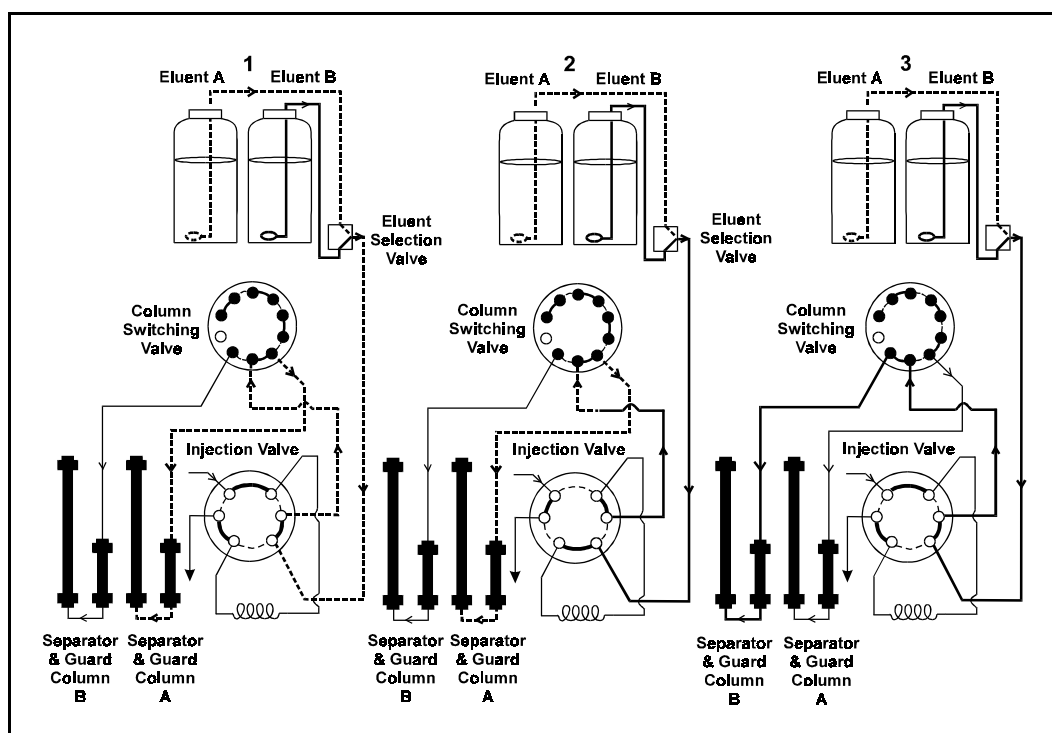


Figure 2-8. Column Selection Sequence

1. In Step 1, eluent A is flowing to column set A.
2. In Step 2, the following occurs:
 - A command is received to switch to column B.
 - The eluent selection valve switches and eluent B begins flowing to the injection valve.
 - The display flashes **RINSE**.
 - The injection valve switches to the Inject position and the previous eluent is cleared from the sample loop.
 - A short delay occurs before the column switching valve switches to the new position. This allows eluent A to continue through to column set A. The duration of the delay depends on the current flow rate. Lower flow rates require a longer rinse time.
3. In Step 3, the following occurs:
 - When eluent A has been cleared from the lines, the column switching valve switches and flow proceeds to the selected column set.
 - The injection valve returns to the Load position.
 - The **RINSE** indicator stops flashing and the display shows the new column selection. The system is now ready for use.

NOTE

There is a small amount of eluent carryover when switching column sets. For this reason, ignore the first injection run after switching columns.

2.2.2 Eluent Select Mode

In eluent select mode, pressing **Column A** or **Column B**, or sending an eluent switching command from PeakNet, switches the eluent selection valve to the new position.

In this mode, the column switching valve is disabled. The selected column set remains at what it was when the DX-120 was switched to eluent select mode. The LED on the button for the selected column set is always illuminated and the display indicates the selected eluent (see the example in Figure 2-9).

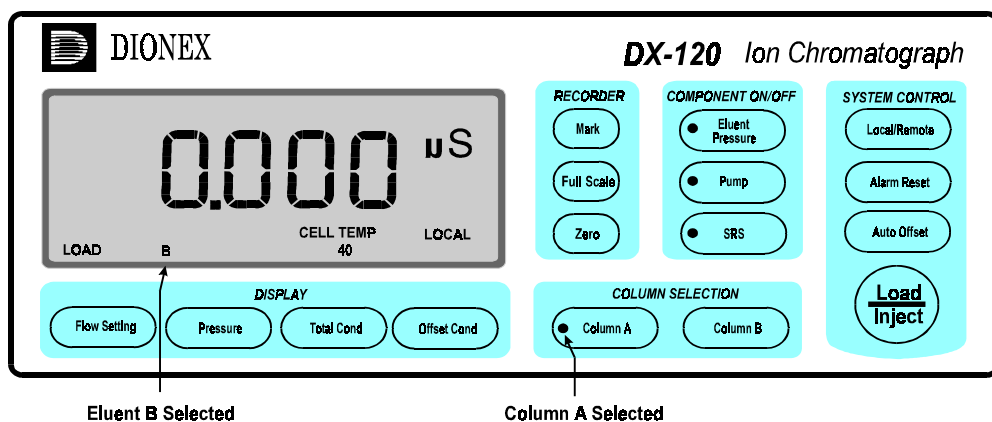


Figure 2-9. Display During Eluent Select Mode

2.3 Fluid Schematics

Figure 2-10 shows the flow path through a single-column DX-120 Ion Chromatograph.

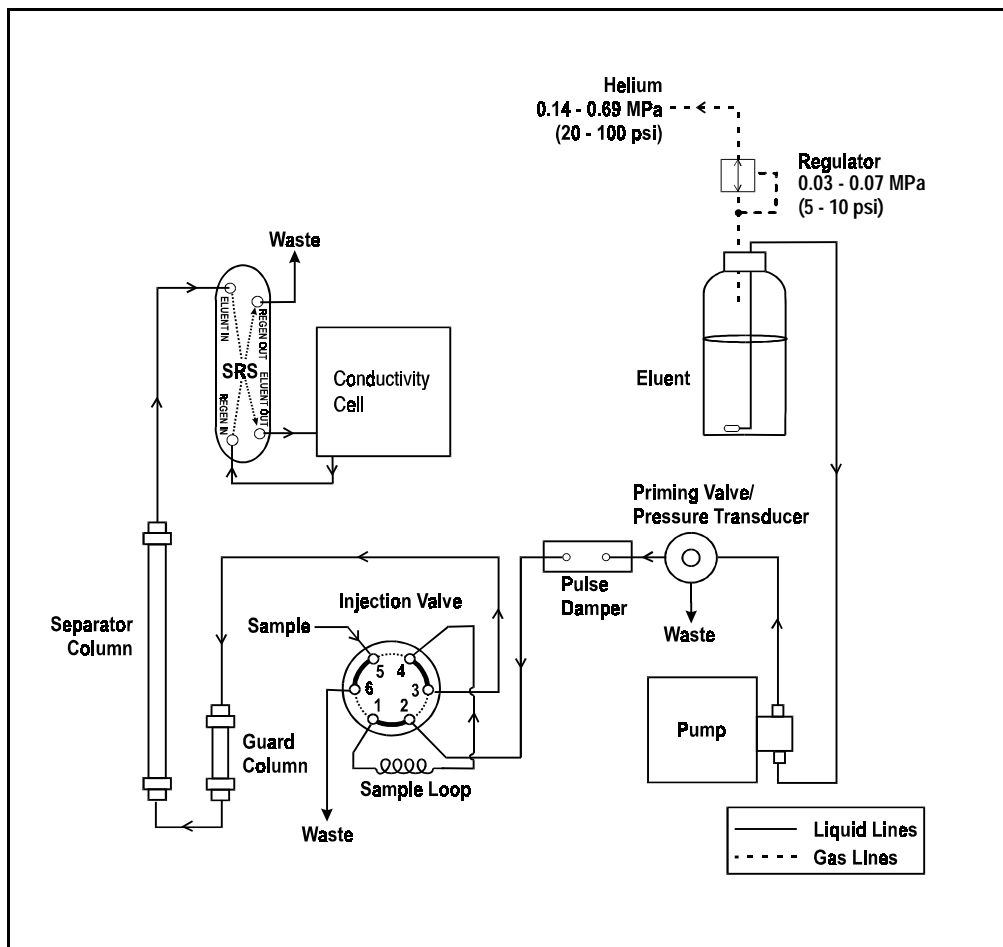
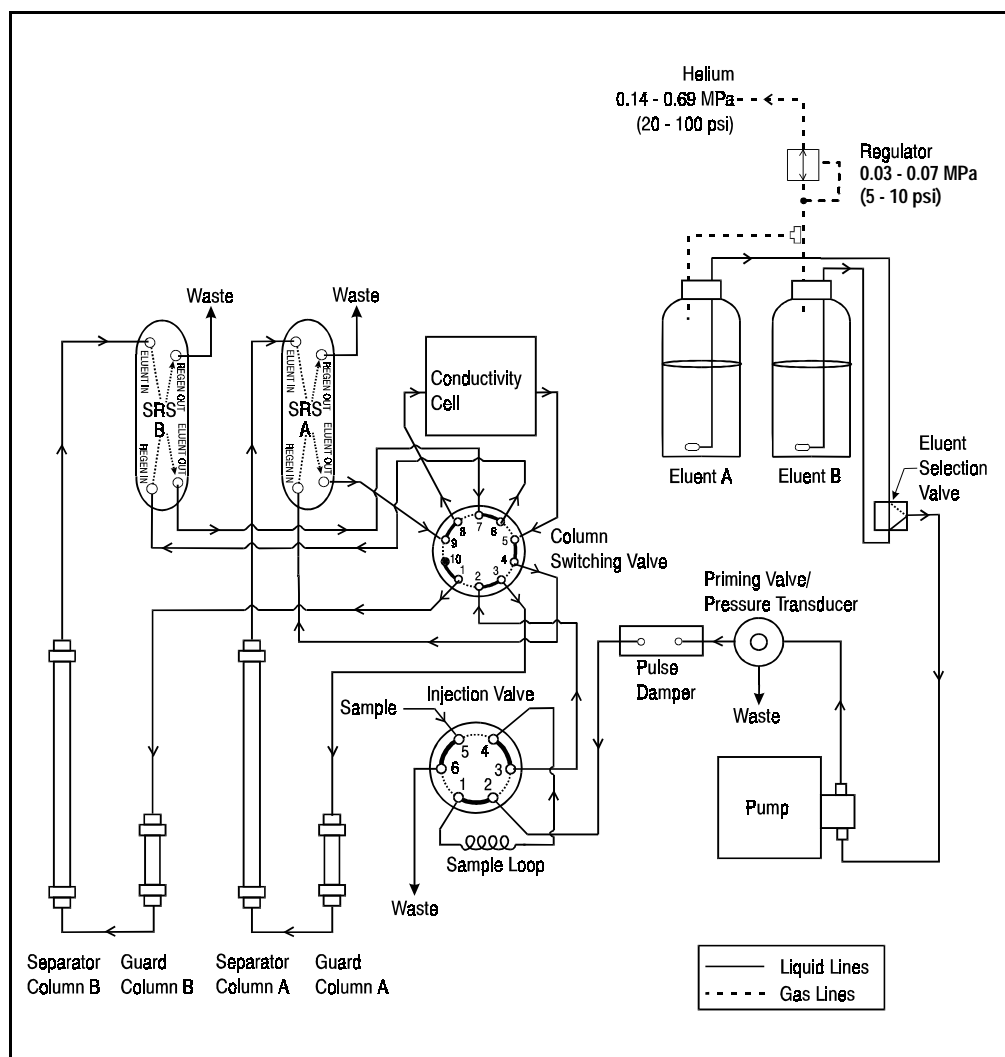


Figure 2-10. DX-120 Flow Schematic: Single-Column

Figure 2-11 shows the flow path through a dual-column DX-120 Ion Chromatograph in column select mode.



*Figure 2-11. DX-120 Flow Schematic: Dual-Column System
Column Select Mode*

Figure 2-11 shows the flow path through a dual-column DX-120 Ion Chromatograph in eluent select mode.

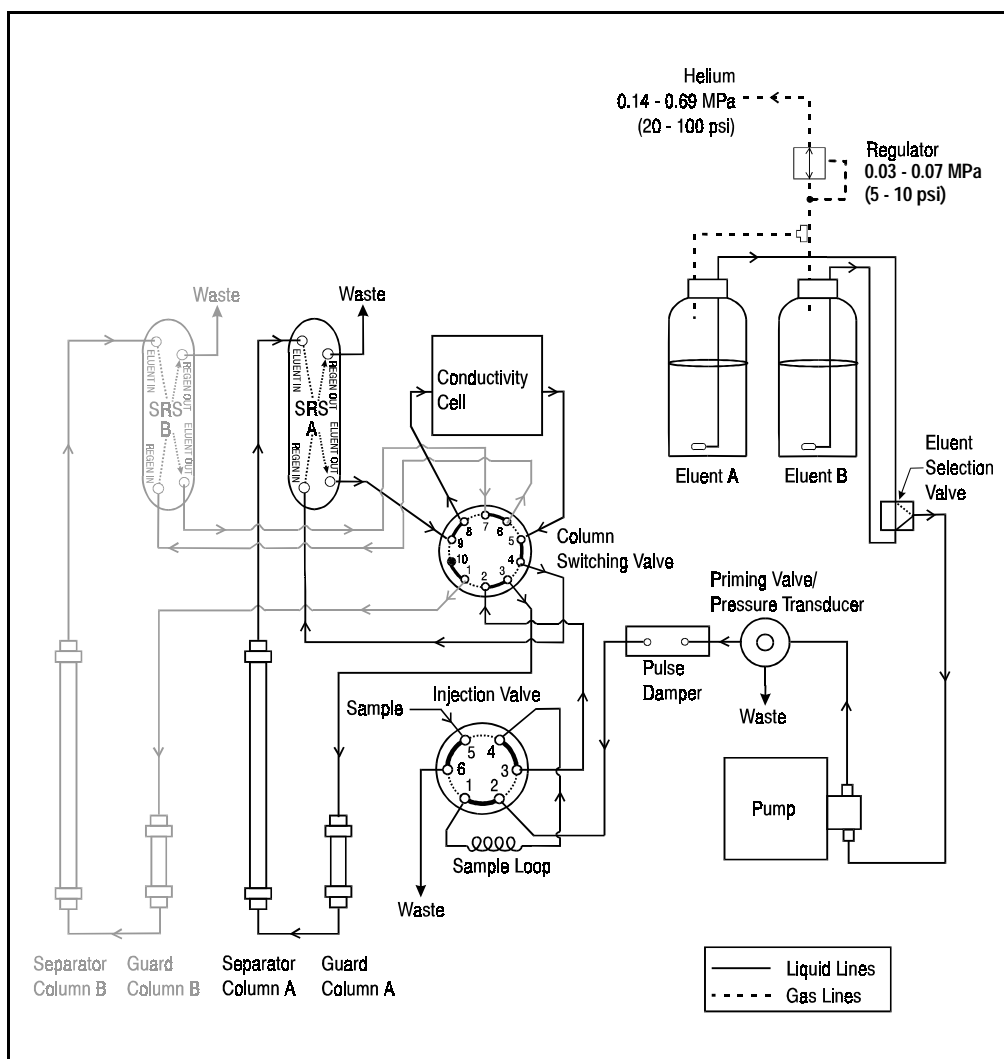


Figure 2-12. DX-120 Flow Schematic: Dual-Column System
Eluent Select Mode (Column Set A Active)

2.4 Control Modes

The DX-120 has two control modes: Local and Remote. Select Local for control from the DX-120 front control panel and select Remote for control from a PeakNet workstation.

To change the mode, press **Local/Remote**. The selected mode is shown in the lower right-hand corner of the display.

2.4.1 Local Mode

When the DX-120 is powered up, it defaults to Local mode. Local mode allows two types of operating commands:

- Direct input from the DX-120 front control panel buttons
- TTL inputs from a remote controller, such as an integrator or recorder

2.4.2 Remote Mode

In Remote mode, the DX-120 accepts operating commands from PeakNet software via the DX-LAN interface. Sending an operating command from PeakNet automatically selects Remote mode, if it was not already selected.

Several DX-120 control panel buttons are disabled in Remote mode, as indicated in the following table.

Disabled Buttons in Remote Mode	Active Buttons in Remote Mode
Load/Inject	<i>DISPLAY</i> group (all)
Eluent Pressure (on/off)	<i>RECORDER</i> group (all)
SRS (on/off)	Pump (on/off)
Column A and Column B	Local/Remote
	Alarm Reset
	Auto Offset

2.5 TTL Control

The TTL input connectors on the DX-120 rear panel allow limited remote control of the DX-120. With TTL input, a controlling device (such as an integrator or automated sampler) sends TTL signals to the DX-120. TTL input signals can be used to:

- Toggle the position of the injection valve and start a PeakNet run (these two functions are controlled simultaneously)
- Offset the background conductivity
- Change the recorder range by 10x
- Turn the pump flow on and off

TTL control is available when the DX-120 is in either Local or Remote mode.

Refer to Section B.7 for TTL connection instructions.

2.5.1 Injection Valve/Remote Start Control

A TTL output signal from another device, such as an automated sampler, can switch the injection valve to the Inject position. The same TTL input signal is also used to start a PeakNet run when the DX-120 is connected to a PeakNet workstation.

NOTE

If required for an application, disable the TTL injection valve control and use only the remote start function. See Section B.8.3 for instructions.

2.5.2 Recorder Range Control

The default full-scale recorder output signal range is 1000 μ S. This is set by the Range DIP switch (SW4-3). Sending a 10X Range TTL input signal when the recorder output is at 100 μ S changes the full-scale recorder range to 1000 μ S. The range will remain at 1000 μ S as long as the input signal is being sent from the controlling device.

If the Range DIP switch is set to 1000 μ S, sending the 10X Range TTL input signal will have no effect.

The table below summarizes the relationship between the 10X Range TTL input and the Range DIP switch.

Range DIP Switch (SW4-3) Setting	10X Range TTL Input Status	Signal Output
1000 μ S*	1X*	1000 μ S*
1000 μ S	10X	1000 μ S
100 μ S	1X	100 μ S
100 μ S	10X	1000 μ S

*Default settings

NOTE

The Range DIP switch setting and the 10X Range TTL input are only for recorder output control; they do not affect either the conductivity readings shown on the control panel display or the PeakNet data.

3 • Operation and Maintenance

3.1 Preparing Eluents

3.1.1 Degassing Eluents

Dionex strongly recommends degassing all eluents and storing them in reservoirs pressurized with filtered inert gas (see Section 3.1.3). This helps prevent bubbles (resulting from eluent outgassing) from forming in the pump and the detector cell. Degassed eluents and pressurized reservoirs are especially important when combining aqueous and nonaqueous components (for example, water and methanol).

Several degassing procedures can be used, including vacuum degassing, sparging with helium, or sonication without vacuum. Follow the steps below for vacuum degassing:

1. Prepare the eluent required for the application. Pour it into a clean vacuum flask and attach the flask to a vacuum pump or water aspirator.
2. Vacuum degas the eluent for 5 minutes while agitating the solution by shaking or sonication.

IMPORTANT

When using nonaqueous components, do not degas eluents for longer than 5 minutes; volatile compounds may be lost.

3. Remove the flask from the vacuum. **Do not allow water to flow from the aspirator back into the flask.**
4. Pour the degassed eluent into a pressurizable reservoir. Be careful not to shake the eluent.
5. Install end-line filters and pressurize the reservoirs (see Sections 3.1.2 and 3.1.3).

3.1.2 Filtering Eluents

Always filter eluents before operation to remove small particulates that may contaminate the pump check valves and cause erratic flow rates or loss of prime. End-line filters (P/N 045987) are supplied in the pressurizable reservoir ship kits for this purpose.

Install an end-line filter on the end of the eluent line inside the reservoir. To prevent air from being drawn through the lines, make sure that the end of the filter reaches the bottom of the eluent reservoir.

3.1.3 Pressurizing Eluent Reservoirs

Pressurize eluent reservoirs with filtered inert gas (preferably helium). Refer to the *Pressurizable Reservoir Installation Instructions* for details.

1. Verify that the gas supply is connected to the HELIUM INPUT connector on the rear panel and is regulated to between 0.14 and 0.69 MPa (20 and 100 psi).
2. Press **Eluent Pressure** to turn on the gas pressure to the eluent reservoir(s). A regulator inside the DX-120 regulates the pressure to between 0.03 and 0.07 MPa (5 to 10 psi).



Never pressurize the reservoirs above 0.07 MPa (10 psi). If using glass reservoirs, inspect them periodically for scratches or cracks.

3.2 Preparing Samples

3.2.1 Collecting and Storing

Collect samples in high density polyethylene containers that have been thoroughly cleaned with deionized water. Do not clean containers with strong acids or detergents because they will leave traces of ions on the container walls. These ions may interfere with 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 will minimize, 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.

3.2.2 Pretreating

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

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

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

3.2.3 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 deionized water or eluent to dilute the sample. When using carbonate/bicarbonate 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 to prepare the calibration blank and 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.

3.3 Operating

3.3.1 Starting Up

1. Press the power switch below the DX-120 front control panel (see Figure 2-2) to turn on the system power. Microprocessor code revision levels are displayed briefly on the screen, and then the offset conductivity reading is displayed.

These are the conditions at power-up:

- The DX-120 is in Local mode.
 - The eluent pressure, pump, and SRS are off.
 - The DS4 Detection Stabilizer (if installed) is on.
 - The injection and column selection valves are in their last selected positions.
 - The offset value is reset to zero.
2. Press the power switch on the front of the pump (see Figure 2-2) to turn on the pump power.
 3. Press **Eluent Pressure** to pressurize the eluent reservoirs.
 4. Press **Pump** to turn on the pump flow.
 5. Press **SRS** to turn on the SRS power. The screen briefly displays the SRS current setting in mA.
 6. Press **Flow Rate** and verify that the pump flow rate is correct. If necessary, pull out the knob on the front of the pump and turn it right or left to increase or decrease the flow rate. When the correct rate is displayed, push in the knob.
 7. Press **Offset Cond** to display the offset conductivity reading.

8. Allow the system to equilibrate for 15 to 20 minutes. The screen displays the background conductivity (the conductivity of the eluent before injecting sample). Press **Auto Offset** to offset the background and zero the reading.

If a DS4 is installed, system equilibration must also include the time required for the DS4 to reach operating temperature. The DS4 warms up at about 1 °C/minute above ambient. Baseline conductivity should stabilize once the DS4 reaches the selected temperature. The DS4 temperature status appears at the bottom of the display:

- **LO CELL TEMP** appears and the temperature set point flashes when the DS4 is below temperature.
- **CELL TEMP** appears and the set point stops flashing when the DS4 has reached operating temperature.
- **CELL TEMP HI** appears and the temperature set point flashes when the DS4 is above operating temperature.

Figure 3-1 shows the display after all start-up steps are complete and the DX-120 is ready for a sample injection.

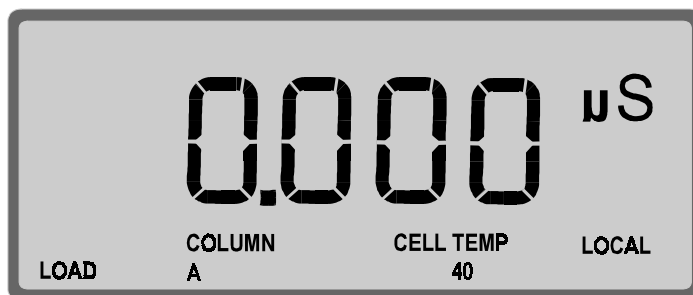


Figure 3-1. Display after Start-Up Completed

NOTE

If the DX-120 receives no input for 90 minutes, the pump flow is reduced to 1/20th of its current rate and the SRS cycles on and off (SW1-2). The LEDs on the Pump and SRS buttons flash when this occurs. Press any button to return to the previous flow rate.

3.3.2 Injecting the Sample

This section describes how to inject sample when the DX-120 is in Local control. PeakNet software can also be used to switch the injection valve position.

Using a Syringe

1. Verify that **LOCAL** is shown at the lower-right corner of the display. If necessary, press **Local/Remote** to toggle to Local mode.
2. Verify that **LOAD** is shown at the lower-left corner of the display. If necessary, press **Inject/Load** to switch the injection valve to the Load position.
3. Fill the syringe with a calibration standard or sample.
4. Insert the syringe into the port on the front of the DX-120 (see Figure 2-2).
5. Overfill the sample loop with several sample loop volumes. Excess sample will exit through the waste line.
6. Leave the syringe in the port.
7. Press **Inject/Load** to switch the injection valve to Inject.

Using an Autosampler

The autosampler output line connects to port 5 on the injection valve. See Section B.8 for installation instructions.

1. Verify that **LOCAL** is shown at the lower-right corner of the display. If necessary, press **Local/Remote** to toggle to Local mode.
2. Verify that **LOAD** is shown at the lower-left corner of the display. If necessary, press **Inject/Load** to switch the injection valve to the Load position.
3. Follow the instructions included with the autosampler to load the injection valve loop.

4. Press **Inject/Load** to switch the injection valve to Inject.

Injection Events

By default, the following events occur after injection:

- An auto offset occurs (SW3-3), which includes two steps:
 - The analog output signal is set to zero.
 - The background conductivity is offset from the total, thereby zeroing the baseline conductivity value. This is the same function as pressing **Auto Offset** on the front panel.
- An inject mark is sent out on the analog output (SW4-7).
- The Inject TTL output sends out a pulse indicating that injection occurred.
- After 1 minute, the injection valve returns to the Load position (SW1-1).

Injection Duration

To ensure complete injection of the sample, at least 10 sample volumes must be pumped through the loop before the valve is switched back to the Load position. For most applications, automatically returning to Load after 1 minute is sufficient. Here are the maximum loop sizes for a one-minute injection at 1 mL/min and 2 mL/min:

- At 1 mL/min, use a loop of 100 μ L or less.
 $(1000 \mu\text{L}/\text{min})(1 \text{ sample vol}/100 \mu\text{L})=10 \text{ sample vol}/\text{min}$
- At 2 mL/min, use a loop of 200 μ L or less.
 $(2000 \mu\text{L}/\text{min})(1 \text{ sample vol}/200 \mu\text{L})=10 \text{ sample vol}/\text{min}$

If your flow rate/loop combination requires more time, disable the automatic return (SW1-1) (see Section B.10).

3.4 Using an Integrator

You can connect an integrator, such as the Dionex 4400 or 4600 Integrator, to the DX-120 and use a DIALOG or BASIC program to automate analyses. If you also connect an autosampler, sample loading can also be controlled. See Appendix C for integrator programming examples.

3.5 Running under PeakNet Control

When the DX-120 is connected to a PeakNet workstation via the DX-LAN interface, PeakNet software (Release 4.30 or higher) can monitor DX-120 status and control the following functions:

- Select the position of the injection and column switching valves
- Turn the pump flow, SRS power, and eluent pressure on and off
- Perform an auto offset
- Select the pressure units displayed on the screen (MPa or psi)
- Control TTL1 and TTL2 output signals
- Control the auxiliary AC outlet (PeakNet control only)

For more information, refer to the *PeakNet Software User's Guide*.

3.6 Optimizing Temperature Compensation

The DX-120 built-in temperature compensation stabilizes conductivity readings by correcting for changes in ambient temperature that occur during a run. For more information about temperature control and compensation, see Section 2.1.7.

3.6.1 With a DS4

Housing the cell in a DS4 Detection Stabilizer ensures that there is no more than a minor temperature variation in liquid reaching the cell. Thus, the temperature compensation DIP switch setting can remain at the default of 1.7% per °C.

Many users are able to keep their systems at a single operating temperature. For optimal accuracy, calibrate the cell at this temperature. If you later reset the temperature, the DX-120 temperature compensation will normalize conductivity measurements to 25 °C (77 °F) to prevent a major upset in system calibration. If you change the DS4 set point, recalibrate the cell.

If temperature-induced baseline cycling occurs, it is probably caused by another component of the chromatography system. If the variation increases as the eluent reservoir empties, move the reservoir to a more temperature-stable environment and/or wrap the reservoir in thermal insulation.

3.6.2 With a CDM-3 Cell

When the CDM-3 cell is installed, conductivity drifts up and down with fluctuations in laboratory temperature. This is especially noticeable in laboratories with very high air turnover rates or no air conditioning. Selecting the proper temperature compensation factor will minimize the effect of temperature fluctuations.

The temperature compensation setting is selected with a DIP switch (SW5-3, 4, 5). Three settings are available: 1.5%, 1.7%, and 1.9%. Start with the default setting of 1.7%. If a

sinusoidal baseline variation of the same period as the laboratory cooling or heating occurs, increase or decrease the temperature compensation setting. If the baseline variation still occurs, try the other setting.

3.7 Maintenance

This section describes routine maintenance procedures that users can perform. All other maintenance procedures must be performed by Dionex personnel.

Daily

- Check the interior of the main compartment for leaks or spills. Wipe up spills. Isolate and repair leaks (see Section 4.3). Rinse off any dried eluent or reagent with deionized water.
- Check the waste container daily and empty when needed.

Weekly

- Once a week, check air lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
- Check the junction between the pump head and the metal 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 5.9).

4 • Troubleshooting

This chapter is a guide to troubleshooting problems that may occur while operating the DX-120. If an alarm sounds, check Section 4.1 for possible causes. If an error code is displayed, check Section 4.2 for possible causes. To resolve other problems, turn to the section that best describes the operating problem.

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

Three events signal an alarm condition: a tone sounds, the LED on the **Alarm Reset** button blinks, and the display indicates ALARM and the alarm's source. To clear the alarm, press **Alarm Reset**. If the alarm condition still exists, the alarm will reappear after 15 seconds. The alarm tone can be disabled with a DIP switch (SW3-2).

- **LEAK ALARM**

A horizontal rectangular display with a grey background. The word "ALARM" is centered in black capital letters.A horizontal rectangular display with a grey background. The word "LEAK" is centered in black capital letters.

There is liquid in the drip tray. Wipe up spills in the tray and check for leaks (see Section 4.3).

- **CELL LEAK ALARM**

A horizontal rectangular display with a grey background. The word "ALARM" is centered in black capital letters.A horizontal rectangular display with a grey background. The words "CELL LEAK" are centered in black capital letters.

There is a leak in the DS4 (see "Leaking DS4" in Section 4.3).

- **LOW PRESSURE ALARM**

A horizontal rectangular display with a grey background. The word "ALARM" is centered in black capital letters.A horizontal rectangular display with a grey background. The words "LO PRESSURE" are centered in black capital letters.

The system pressure is less than or equal to the low pressure limit of 1.7 MPa (250 psi). This condition automatically turns off the pump and SRS. This alarm can be turned off with a DIP switch (SW1-8).

1. Make sure the eluent reservoirs are full.
2. Check for liquid leaks (see Section 4.3).

3. Make sure the pressure transducer 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 pressure transducer housing.**
4. Restart the pump.
5. Prime the pump (see Section B.5.1).

- **HIGH PRESSURE ALARM**



The system pressure is greater than or equal to the high pressure limit selected (SW1-5, 6, 7). This condition automatically turns off the pump and SRS.

1. Make sure the selected flow rate is not too high.
2. Check for blockages in the liquid line from the pump pressure transducer to the waste container.
3. Make sure the columns are not the source of the high pressure.
4. Set the high pressure limit to 3.4 MPa (500 psi) above the normal system operating pressure.
5. Restart the pump.

- **SRS ALARM**



The SRS has exceeded the accepted current/voltage range.

1. The SRS may be dry. Make sure the eluent reservoir is filled and sufficiently pressurized. Check all liquid lines and valves for leaks, crimping, or blockage.
2. Make sure the cable connecting the SRS to the DX-120 electronics card is connected (see Figure B-10).
3. Refer to the SRS manual for more troubleshooting information.

4.2 Error Codes

When an error occurs, an error code number displays in the middle of the screen. The error code remains for several seconds, and then the display returns to normal.

- **E000**

Cause: No *Moduleware* is installed. Moduleware is the DX-120 instrument control microprocessor code.

Action: Download a new copy of the Moduleware, if the system includes PeakNet software, or contact Dionex for assistance.

- **E001**

Cause: The **Column B** button was pressed, although the system is configured for a single column.

Action: If it is a dual-column system, verify that the Column Configuration DIP switch (SW1-3) is set to dual-column.

- **E002**

Cause: Two or more conflicting DIP switch settings.

Action: Reset the DIP switches (see Section B.10).

- **E003**

Cause: More than one high-pressure alarm setting is selected. The pump and SRS will turn off when this error occurs.

Action: Check the high-pressure alarm setting (SW1-5, 6, 7). One switch position must be on and the other two off.

- **E004**

Cause: During the rinse portion of the column switching sequence, a command to switch columns was received from the **Column A** or **Column B** button, or from PeakNet.

Action: The DX-120 cannot begin another column switching sequence during the rinse cycle. Wait until the column switching sequence is completed.

- **E005**

Cause: A command to switch columns was received from either the **Column A** or **Column B** button, or from PeakNet, while the pump was off or the flow rate was very low. The rinse cycle step of the column switching sequence requires an adequate flow rate to flush the previous eluent from the system before switching.

Action: Turn on the pump or increase the flow rate.

- **E006**

Cause: The SRS will not turn on. Either the pump is not on or the suppressor is disconnected.

Action: Turn on the pump. Make sure the cable connecting the SRS to the DX-120 electronics card is connected (see Figure B-10).

4.3 Liquid Leaks

- **Leaking fitting**

Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection (see Section 5.1). Refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

- **Broken liquid line**

Replace the line and fittings (see Section 5.1).

- **Blocked or improperly installed waste line**

Make sure the waste lines are not crimped or otherwise blocked. Also make sure waste lines are not elevated at any point after they exit the DX-120.

- **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 5.9).
2. If the problem persists, replace the piston (see Section 5.10).

- **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 transducer are tight. Refer to *Installation of Dionex Ferrule Fittings* for tightening requirements. Replace any damaged fittings.

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 pressure transducer housing.**

Inspect the pressure transducer. If the waste valve is the source of the leak, replace the waste valve O-ring (see Section 5.11). If the leak is from the rear of the transducer, contact Dionex for assistance.

- **Leaking SRS**

See the SRS manual for troubleshooting procedures.

- **Leaking injection valve or column switching valve**

Liquid leaks from behind the valve stator may indicate a scratched rotor seal. Contact Dionex for assistance.

- **Leaking DS4**

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 DS4 is clear; a blockage may overpressurize the DS4, causing it to leak.

Make sure the downstream backpressure coils are appropriate for the operating flow rate (see Section B.6.1).

Follow the steps below to disassemble the DS4 and inspect it for the source of the leak.

1. Turn off the DX-120 power.
2. Disconnect the DS4 cables.
3. Disconnect the DS4 inlet and outlet lines. Do not misplace the ferrule fittings at the end of the tubing (see Figure 4-1).
4. Remove the DS4 by lifting it upward and then pulling it away from its mounting location. Place the DS4 on the workbench.
5. Open both latches on the DS4 and carefully lift off the top half of the cover, along with its insulating foam insert, exposing an aluminum box (see Figure 4-1).
6. Remove the box from the bottom half of the cover.
7. Remove the four Phillips screws securing the top of the box to its bottom plate. Carefully separate the two parts, exposing the cell (see Figure 4-2). Lay the top aside, being careful not to pull or stress the group of wires that connect the two parts.
8. Check the inlet and outlet cell fittings for leaks. Tighten or replace if necessary.
9. Dry the DS4, test for leaks, and reassemble. Make sure the foam insert is adjusted evenly around the various DS4 components, with no pinching or folding. Before latching the cover, make sure the top and bottom inserts meet on all sides.

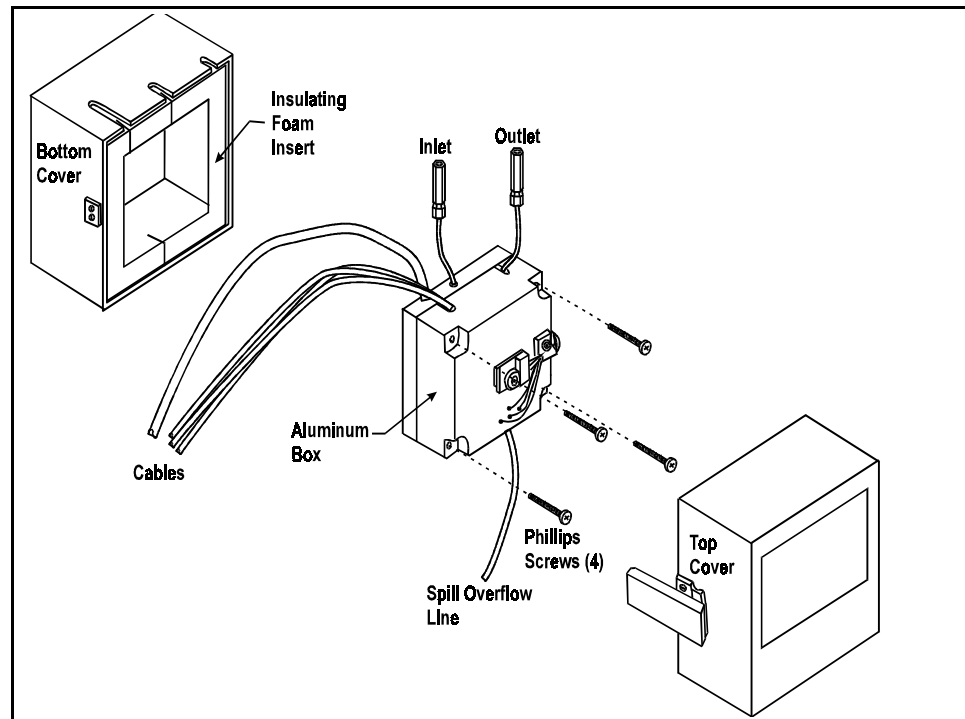


Figure 4-1. DS4 Exploded View

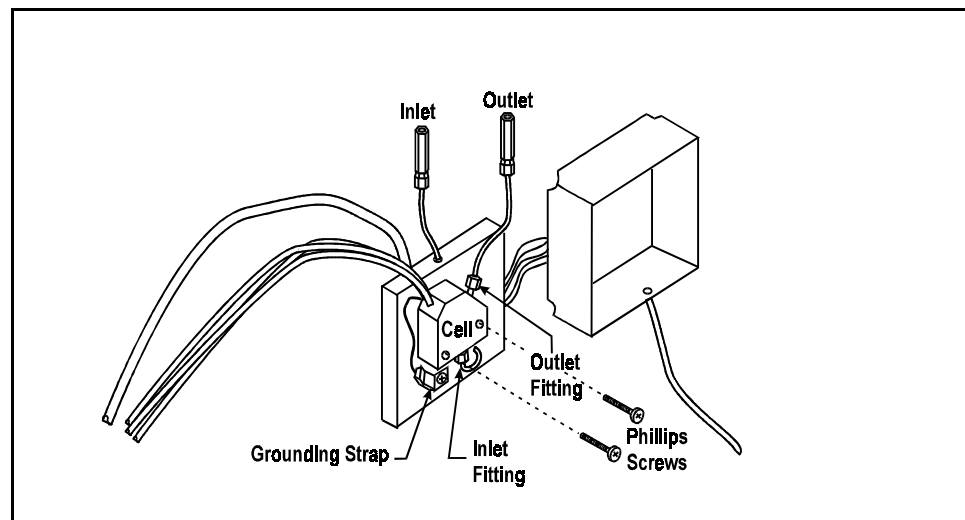


Figure 4-2. DS4 Interior Components

- **Liquid seeping from around cell cables**

The cell has an internal leak and must be replaced. Contact Dionex for assistance.

4.4 Pump Difficult to Prime

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

Connect the pump inlet line to the eluent reservoir. Fill the reservoir.

- **Eluent reservoir not pressurized**

Connect the air line to the reservoir and press **Eluent Pressure** to turn on the pressure to the reservoir.

- **Partially blocked end-line filter**

If the end-line filter (P/N 045987) is no longer pure white, replace it.

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

Replace the piston seal (see Section 5.9).

- **Dirty or defective check valves**

Clean the inlet and outlet check valves (see Section 5.8).

4.5 Pump Loses Prime

- **Eluent reservoir empty**

Refill the reservoir.

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

Replace the piston seal (see Section 5.9).

- **Dirty or defective check valves**

Clean the inlet and outlet check valves (see Section 5.8).

4.6 Pump Does Not Start

- **Power switch on pump or Pump button on front panel is off**

Turn on both switches.

- **No power (control panel LED indicators are not lighted)**

Check that the power cord is plugged in.

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

4.7 No Flow

- **Pump not primed**

Prime the pump (see Section B.5.1).

- **Flow rate set to zero**

Reset the flow rate.

- **Broken pump piston**

Replace the piston (P/N 036904) (see Section 5.10).

4.8 Excessive System Backpressure

- **Restriction in the hydraulic system**

Check all liquid lines for crimping or blockage. Make sure the ferrule fittings are not overtightened onto tubing. Refer to *Installation of Dionex Ferrule Fittings* for details.

- **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 5.1).

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

1. Verify that the column flow rate matches the flow rate set for the pump.

2. Measure the pump flow rate, using a 10 mL graduated cylinder and stopwatch. Calibrate the flow rate if needed (see Section 5.7).

- **Clogged column bed supports**

Replace the bed supports as instructed in the column manual.

- **Contaminated columns**

Clean the columns as instructed in the column manual.

- **Plugged Rheodyne valve passages**

Contact Dionex for assistance.

4.9 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 (see Section 3.3.2).

- **Malfunctioning injection valve**

Contact Dionex for assistance.

4.10 Nonreproducible Peak Height or Retention Time

- **Column overloading**
 1. Dilute the sample.
 2. Change to a sample loop with a smaller volume (see Section 5.2).
- **Liquid leaks**

Locate and eliminate the leaks (see Section 4.3).
- **Incomplete or imprecise filling of the sample loop**
 1. Fill the sample loop until excess sample exits the waste line.
 2. Inspect the syringe (P/N 016387, 10 cc; 016388, 1 cc) and replace if damaged.

4.11 Abnormal Retention Time or Selectivity

- **System not equilibrated following an eluent change**

Allow the system to equilibrate with at least 20 column volumes of eluent (for example, 30 minutes at 2.0 mL/min for 4 mm anion separator columns).
- **Incorrect flow rate through system**
 1. Check that the correct flow rate is selected.
 2. Calibrate the pump flow rate (see Section 5.7).
 3. Locate and eliminate any liquid leaks (see Section 4.3).
- **Contaminated or incorrect eluent**

Remake the eluent using reagent grade chemicals and ASTM filtered, Type I (18-megohm) deionized water.
- **Contaminated or degraded sample**

Take appropriate precautions when preparing and storing samples to prevent contamination and degradation (see Section 3.2).

- **Contaminated column**

1. Clean the column as instructed in the column manual.
2. If cleaning is unsuccessful, replace the column.

4.12 DS4 Temperature Inaccurate

- **CELL TEMP HI displays continuously**

Verify that the set temperature is at least 5 °C above ambient (SW4-4, 5, 6, 7). Allow 30 to 60 minutes for the initial warm-up period. If you later select a higher set point, allow an additional 3 to 7 minutes for each 5-degree increment in the set point.

The DS4 can take from 1 to 2 hours to completely cool down.

- **LO CELL TEMP displays continuously**

At high flow rates and temperature settings far above ambient, the DS4 requires more time to heat. In extreme cases, such as a very cold room and a high DS4 set point, the DS4 may not be able to reach the set point temperature.

- **DS4 does not heat**

Make sure the Cell Type DIP switches (SW4-1, 2) are set to the DS4 position (off).

Make sure that one of the DS4 temperature switch positions (SW4-4, 5, 6, 7) is on. If all switches are off, the DS4 will not heat.

Remove the cover and inspect the DS4 for broken or shorted wires or for moisture bridging the control thermistor. If a wire is broken or shorted, replace the wire or call Dionex for assistance. If a leak has caused a short, fix the leak (see Section 4.3, "Leaking DS4") and dry the control sensor.

4.13 No Detector Response

- **Cell not connected**

Check the cell cable connection.

- **Analog output range too high; although the display indicates a response, no recorder response observed**

Select the 100 μ S range setting (SW4-3, off).

- **Full-scale output too low**

Select the 10.0 V full-scale setting (SW4-4, off).

- **No flow from pump**

Check that the LED on the **Pump** button is lighted. Check the power switch on the front of the pump.

Make sure the flow rate is not set to zero.

- **Detector offset out of range**

Press **Auto Offset** on the front control panel.

- **Cell electronics malfunctioning**

Test the electronics as follows:

1. Disconnect the cell cable from the electronics card at the left side of the pump compartment (see Figure B-10).
2. Set SW4-1, 2 to the off position.
3. Set SW5-8 to the on position.
4. The conductivity reading on the display should be 25.0 μ S. If this is not the case, there may be a problem with the cell electronics. Contact Dionex for assistance.

4.14 Low Detector Output

- **Analog output range set too high; although the display indicates a response, no recorder response observed**

Select the 100 μ S range setting (SW4-3, off).

- **Insufficient sample injected**

Increase the injection size or concentration. See Section 5.2 for information on changing the sample loop size.

- **Cell out of calibration**

Recalibrate the cell (see Section 5.6).

4.15 High Detector Output

- **Auto offset not activated recently**

Press **Auto Offset** on the front panel before making an injection.

- **Background not suppressed by SRS**

Check that the SRS is on (the LED on the **SRS** button should be illuminated).

Check the SRS regenerant out line for bubbles; if there are no bubbles, the suppressor may be contaminated. Refer to the SRS manual for troubleshooting guidance.

- **Sample concentration too high**

Dilute the sample or install a smaller sample loop (see Section 5.2).

- **Wrong eluent**

Make sure you are using the correct eluent.

- **Cell out of calibration**

Recalibrate the cell (see Section 5.6).

4.16 Noisy or Drifting Baseline

- **Flow system leak ahead of cell; erratic baseline**

Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace all liquid line connections. Refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

- **Pump not properly primed**

Prime the pump (see Section B.5.1).

- **Rapid changes in ambient temperature**

Redirect heating and air conditioning vents away from the DX-120.

Replace the CDM-3 cell with a DS4 Detection Stabilizer (P/N 050218).

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

Allow longer system equilibration time before starting operation.

- **Air trapped in cell; excessive regular pulses in baseline**

Check that the correct backpressure coils are installed after the cell and before the SRS (see Section B.6.1).

- **Incorrect SRS operating conditions**

Refer to the SRS manual for troubleshooting information.

- **Temperature compensation setting not optimized**

Optimize the setting (see Section 3.6).

- **DS4 above or below set point**

Wait for the DS4 to reach the selected temperature before beginning operation. The display will indicate **CELL TEMP** and the selected temperature. If the temperature is above or below the set point, **CELL TEMP HI** or **LO CELL TEMP** is displayed.

5 • Service

This chapter describes routine service procedures that users may perform. Other service procedures must be performed by Dionex personnel.

NOTES

Electronics components are not customer-serviceable. Any repairs involving the DX-120 electronics must be performed by Dionex personnel.



The CPU card contains a lithium battery. If it is necessary to replace the CPU card, dispose of the used battery according to the manufacturer's instructions.

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 DX-120 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.

Substituting non-Dionex parts may impair DX-120 performance, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

5.1 Replacing Tubing and Fittings

The DX-120 is plumbed with the tubing and tubing assemblies listed in Table 5-1.

Tubing Size and Type	Used For
0.125-mm (0.005-in) ID (P/N 044221)	Connection from the pump pulse damper to the injection valve
0.25-mm (0.010-in) ID (P/N 042690)	Connections between other system components
0.5-mm (0.020-in) ID (P/N 042855)	Detector cell waste line
Waste Separator Tubing Assembly (P/N 045460)	SRS REGEN OUT connections
4-mm Backpressure Tubing Assembly (P/N 045877)	SRS REGEN IN connections

Table 5-1. Tubing Requirements

- 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 Ferrule Fittings*.
- 1/8-in flangeless fittings (P/N 048951) and ferrules (P/N 048949) are used for the SRS **REGEN OUT** port connections.
- 1/16-in flangeless fittings (P/N 048952) and ferrules (P/N 048950) are used for the SRS **REGEN IN** port connections.

5.2 Changing the Sample Loop

Peak response is directly related to sample concentration and injection volume. To change the sample volume, change the volume of the injection valve sample loop. Sample loops of several sizes are available from Dionex.

With most samples, use of a sample loop larger than 100 μ L results in column overloading and nonlinear response. This overloading volume can be even smaller with some sample types.

1. Turn off the pump.

2. Open the DX-120 door.
3. Disconnect the sample loop from ports 1 and 4 on the injection valve (see Figure B-20).
4. Install the new sample loop between ports 1 and 4 on the injection valve.

5.3 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) at the flow rate normally used.
2. Follow the appropriate hydraulic schematic (see Figure 2-10 or 2-11) 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 either by back flushing or by replacing the tubing or fitting.

5.4 Replacing the DS4 Cell

Follow the steps below to disassemble the DS4 and replace the cell. After replacing the cell, recalibrate it (see Section 5.6).

1. Turn off the DX-120 power.
2. Disconnect the DS4 cables.
3. Disconnect the DS4 inlet and outlet lines. Do not misplace the ferrule fittings at the end of the tubing (see Figure 5-1).

4. Remove the DS4 by lifting it upward and then pulling it away from its mounting location. Place the DS4 on the workbench.
5. Open both latches on the DS4 and carefully lift off the top half of the cover, along with its insulating foam insert, exposing an aluminum box (see Figure 4-1).
6. Remove the box from the bottom half of the cover.
7. Remove the four Phillips screws securing the top of the box to its bottom plate. Carefully separate the two parts, exposing the cell (see Figure 5-1). Lay the top aside, being careful not to pull or stress the group of wires that connect the two parts.

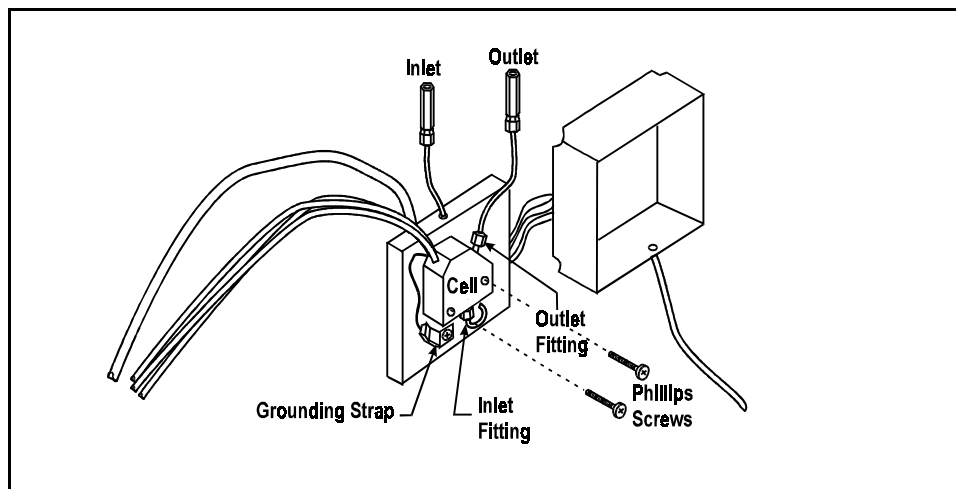


Figure 5-1. DS4 Interior Components

8. Disconnect the grounding strap. Remove the two Phillips cell mounting screws.
9. Rotate the cell inlet tube fitting counterclockwise. Let the cell body back away from the fitting until the fitting threads are fully disengaged. **Do not lose the ferrule.**

10. Check that the end of the inner Tefzel tubing is flush with the end of the plastic sleeve. If necessary, trim the sleeve slightly to prevent dispersion. Push the tubing into the new cell until it bottoms out in the hole; then, hold the tubing in place while tightening the nut.
11. After testing for liquid leaks, dry the DS4 and reassemble. Make sure the foam insert is adjusted evenly around the various components, with no pinching or folding. Before latching the cover, make sure the top and bottom inserts meet on all sides.
12. Reconnect the DS4 cables and turn on the DX-120 power. Now, calibrate the cell constant (see Section 5.6).

5.5 Cleaning Cell Electrodes

If you suspect fouling, clean and recalibrate the cell (see Section 5.6).

1. Prepare the solutions listed below, using filtered ASTM Type I (or better) deionized water. Pour the prepared solutions into labeled containers.



HNO₃ is corrosive and a strong irritant. Avoid breathing the vapors. Always prepare the cleaning solution in a fume hood. Wear gloves and goggles.

- a. 3 M HNO₃ cleaning solution: Dilute 200 mL concentrated HNO₃ (s.g. 1.42) to one liter with deionized water.
 - b. 0.01 M KCl stock solution: Dissolve 0.7456 g of reagent-grade KCl in one liter of deionized water.
 - c. 0.001 M KCl calibration solution: Dilute 100 mL of stock solution to 1 liter with deionized water.
2. Connect the container of 0.001 M KCl to the pump.
 3. Disconnect the line between the suppressor outlet and the cell (or DS4 inlet) at the suppressor. In a dual-column system, disconnect the cell inlet line from port 8 on the column switching valve.

4. Connect a female luer adapter (P/N 024305) to the tubing, using a union (P/N 042806).
5. Fill a 10 mL syringe (P/N 016387) with 3 M HNO₃ solution. Screw the syringe into the luer adapter.
6. Turn off the DX-120 main power switch.
7. Inject 5 mL of HNO₃ through the cell.
8. After two minutes, push the remaining 5 mL of solution through the cell. Wait 2 minutes.
9. Fill the syringe with 10 mL of deionized water. Inject the water through the cell.
10. Turn on the DX-120 main power switch. Continue to Step 3 of Section 5.6 to calibrate the cell constant.

5.6 Calibrating the Cell Constant

Calibrate the cell after installing a new cell or after cleaning the cell electrodes. The cell does not require routine calibration.

1. Disconnect the pump eluent line from port 2 on the injection valve.
2. Disconnect the line between the suppressor outlet and the cell or DS4 inlet at the suppressor. In a dual-column system, disconnect the cell inlet line from port 8 on the column switching valve.
3. Connect the eluent line from the pump directly to the inlet of the cell or DS4, using a union (P/N 042806).
4. Pump 0.001 M KCl calibration solution through the cell at 2.0 mL/min. After 5 minutes, reduce the flow rate to the value typically used during analysis and pump for an additional minute. Conductivity is slightly flow-rate sensitive, so select the flow rate used in the majority of your applications.

5. Set the temperature compensation to 1.7% (SW5-5, on; SW5-4 and 6, off). This temperature compensation value can be used for most applications. When measuring absolute conductivity, determine the optimal value for each batch of calibration solution (see Section 3.6).
6. Disconnect the cell cable from the cell connector on the edge of the electronics card (see Figure B-10).
7. Press the **Auto Offset** button. With the cell disconnected, this is the zero conductivity point. Press **Offset Cond** to display the offset conductivity.
8. Reconnect the cell cable.
9. Locate the Cell Calibration potentiometer on the electronics card (see Figure B-10). Continue pumping calibration solution through the cell. Use the tuning screwdriver (P/N 035617) from the Ship Kit to adjust the potentiometer until the display value equals $147 \pm 2 \mu\text{S}$.
10. Replace the container of 0.001 M KCl calibration solution with a container of deionized water. Pump deionized water through the liquid lines at 2.0 mL/min for at least 5 minutes to flush the calibration solution from the system.
11. Reconnect the pump to the injection valve.
12. In a single-column system, reconnect the liquid line from the cell or DS4 inlet to the suppressor outlet. In a dual-column system, reconnect the line from the cell inlet to the column switching valve.
13. Reset the temperature compensation to the optimal value for the eluent in use (SW5-4, 5, 6). The cell is now calibrated and ready for operation.

5.7 Calibrating the Pump Flow Rate

1. Disconnect the line from port 3 on the injection valve (see Figure B-20).
2. Connect an 11 MPa (1600 psi) backpressure device, or a piece of narrow-bore PEEK tubing that will provide 11 MPa (1600 psi) of backpressure, to port 3 on the injection valve. Route the other end of the backpressure tubing to waste.
3. Fill the eluent reservoir with deionized water.
4. Turn on the pump and set the flow rate to 1.2 mL/min.
5. Pump deionized water through the system for at least 15 minutes to allow the system to equilibrate.
6. Collect the water into a tared beaker for 5 minutes. Weigh the collected water. It should be 6.00 ± 0.05 g.
7. If the collected water is not within the above range, locate the Pump Flow Setting Adjust potentiometer on the electronics card (see Figure B-10). Continue pumping deionized water through the cell. Use the tuning screwdriver (P/N 035617) from the Ship Kit to adjust the potentiometer until the flow rate display corresponds to the weight of the collected water. For example, if you collected 5.5 g, adjust the flow rate to 1.1 mL/min.

5.8 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.5.1). If the pump does not stay primed, proceed to the next step.
2. Turn off the main power switch on the DX-120 and disconnect the main power cord.
3. Disconnect the tube fittings from both the inlet and outlet check valve housings (see Figure 2-2).

4. Using a 1/2-in box wrench or an adjustable wrench, carefully remove both check valve housings from the pump head.
5. Place the check valves in a beaker of methanol and sonicate or agitate for several minutes.
6. Rinse each check valve thoroughly with filtered, deionized water.
7. Reinstall the check valves in the pump head. Be sure to install the inlet check valve on the bottom of the head and the outlet check valve on the top. Tighten only enough to seat.



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

8. Reconnect the liquid lines. Turn on the power switch on the DX-120 front panel.
9. Prime the pump (see Section B.5.1). 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 038273; outlet check valve, P/N 042761).

5.9 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 main power switch on the DX-120 and disconnect the main power cord.
2. Disconnect the tube fittings from the inlet and outlet check valves.

3. Hold the head firmly against the pump housing, to compensate for the spring loading, and remove the two nuts.



Lateral motion when disengaging the head from the piston can break the piston.

4. 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. Be especially careful not to snap the piston if the internal spring is stuck to the piston guide.
5. Place the head, front end down, on a clean work area. Lift the piston guide and back-up washer off, to expose the seal (see Figures 5-2 and 5-3).
6. Remove the piston seal from the head. Discard the seal.
7. Remove the O-ring and back-up seal from the back-up washer.
8. Carefully push the new piston seal (P/N 035686) into the head. When properly installed, the piston seal is almost flush with the indented surface of the head.

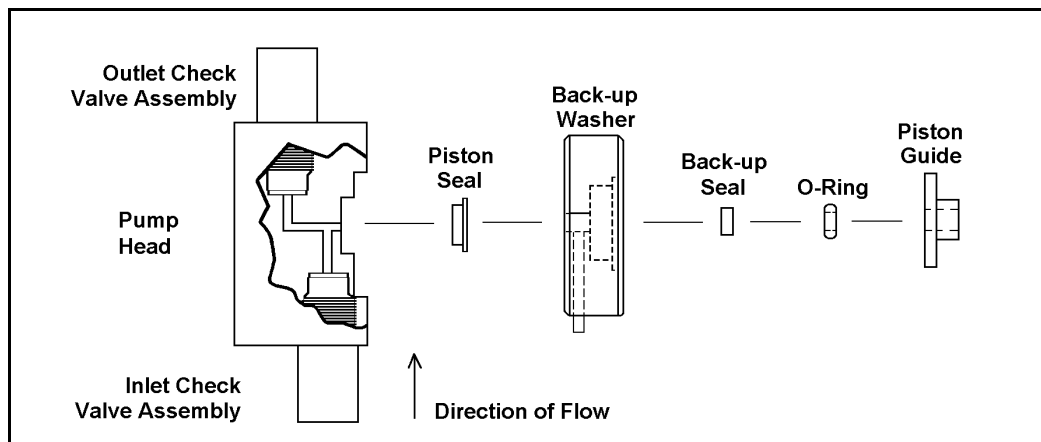


Figure 5-2. Pump Head Assembly

9. 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.
10. Press the back-up washer into the head, followed by the piston guide.
11. Remove the spring, spring guide, spring retainer, and piston from the pump housing (see Figure 5-3). 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.
12. Clean and inspect the piston. If it is scored or scratched, replace it (see Section 5.10).
13. Reinstall the piston, spring retainer, spring guide, and spring in the pump housing.
14. 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.



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.

15. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly.
16. Reconnect the liquid lines to the inlet and outlet check valves.
17. Reconnect the main power cord and turn on the main power switch.
18. Prime the pump (see Section B.5.1).

5.10 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 main power switch on the DX-120 and disconnect the main power cord.
2. Disconnect the tube fittings from the inlet and outlet check valves.
3. While holding the head firmly against the pump housing, to compensate for the spring loading, remove the two nuts.



Lateral motion when disengaging the head from the piston will break the piston.

4. 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. Be especially careful not to snap the piston if the spring is stuck to the piston guide.
5. Remove the piston guide, spring, spring guide, spring retainer, and piston by pulling them straight out, away from the pump housing (see Figure 5-3).
6. If the piston is broken, replace the piston seal and the back-up seal (see Section 5.9). This will prevent pieces of broken piston from scratching the new piston assembly.
7. Remove the retainer ring (P/N 035010) from the old piston assembly and install it on the new piston assembly (P/N 036904).
8. Carefully slide the spring retainer onto the piston assembly.
9. Slide the assembled piston back into the piston housing.

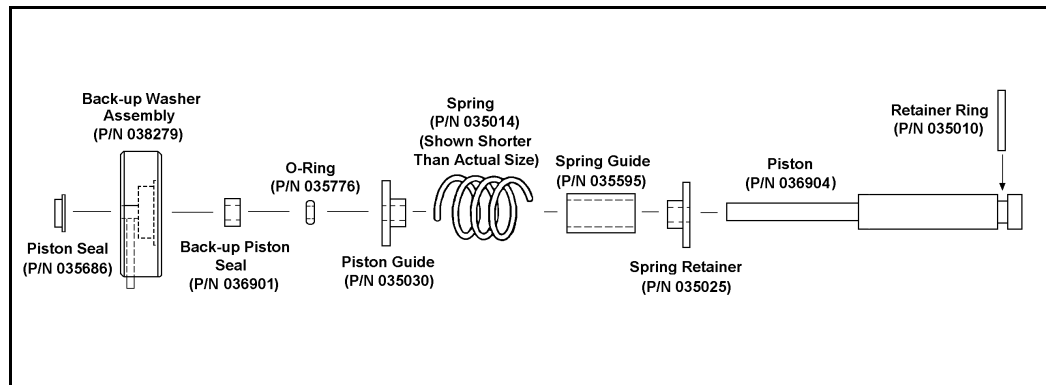


Figure 5-3. Piston Pump Assembly

10. Slide the spring over the piston, positioning it flush against the spring retainer.
11. 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 over the piston will break the piston, as well as damage the piston seal and back-up seal.

12. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly.
13. Reconnect the liquid lines to the check valve housings.
14. Reconnect the main power cord and turn on the main power switch.
15. Prime the pump (see Section B.5.1).

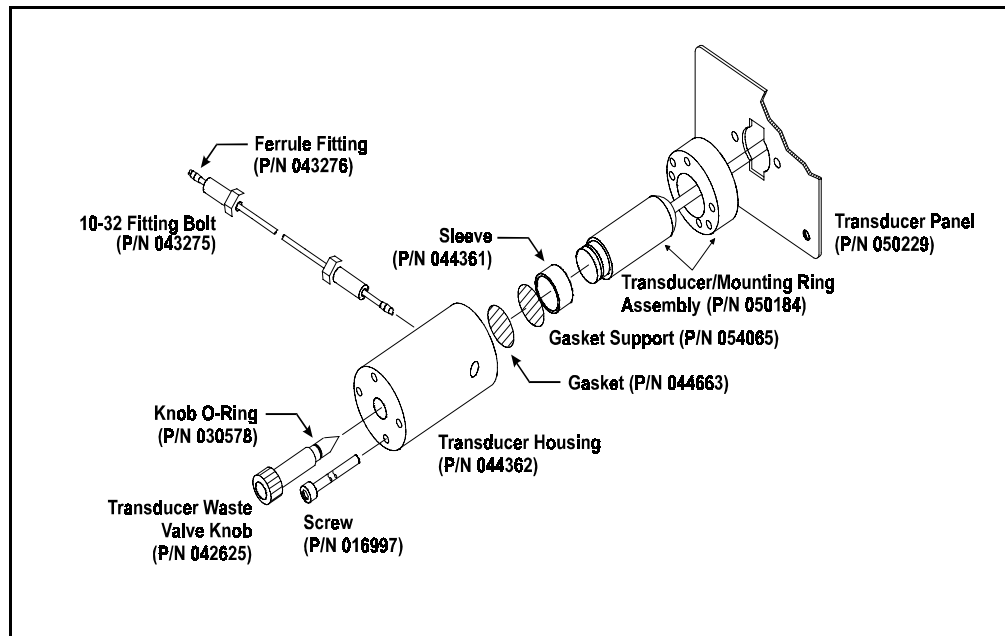


Figure 5-4. Pressure Transducer Assembly

5.11 Replacing the Pressure Transducer Waste Valve O-Ring

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

1. Press the **Pump** button to turn off the pump.
2. Remove the valve from the pressure transducer housing by turning the knob counterclockwise until it comes loose from the housing (see Figure 5-4).
3. Remove the O-ring.
4. Carefully slide a new O-ring (P/N 030578) over the end of the valve and push it into the groove.
5. Reinstall the valve in the housing, turning the knob clockwise until the valve is seated. **Do not overtighten the valve knob.**

5.12 Changing the Main Power Fuses

1. Turn off the main power.



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the DX-120 rear panel.

2. The fuse holder is part of the main power receptacle on the rear panel. A recessed lock is located on each side of the fuse holder (see Figure 5-5). Using a small screwdriver, push each lock toward the center to release it. The fuse holder pops out approximately 0.16 cm ($\frac{1}{16}$ in) when the locks release. When both locks are released, pull the fuse holder straight out of its compartment.
3. The holder contains two fuses. Replace both with new fast-blow fuses rated 5 amps 250 Vac (P/N 214339). Dionex recommends replacing both fuses even though only one is open.

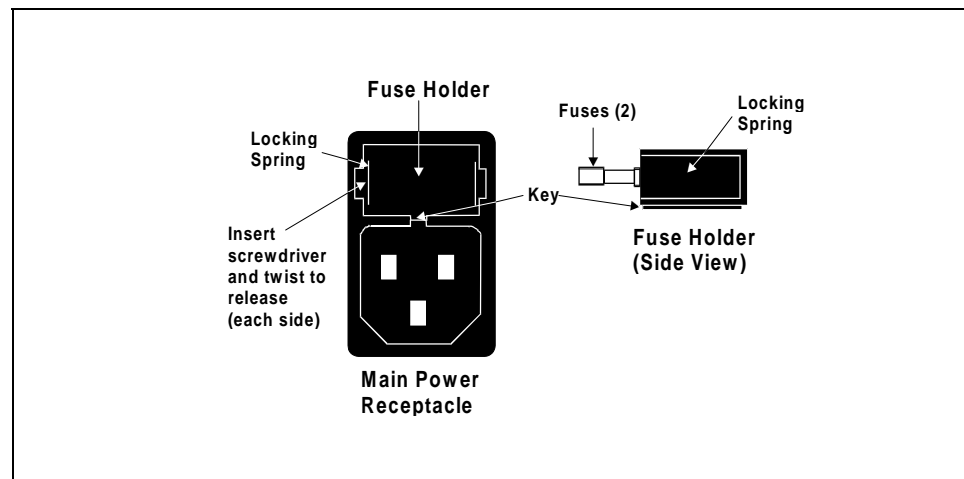


Figure 5-5. Main Power Fuse Holder

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	Three voltage/frequency configurations (not user-selectable): 100 Vac/50 Hz 115 Vac/60 Hz 230 Vac/50 Hz
Fuses	Two fast-blow fuses rated 5.0 A (P/N 214339)
Analog Output	1 V or 10 V, full-scale output

A.2 Environmental/Physical

Operating Temperature	10 °C to 40 °C (50 °F to 104 °F)
Operating Humidity	5% to 95% relative humidity, noncondensing
Decibel Level	53 db (at “A WEIGHING” setting)
Gas Pressure	Laboratory-quality helium, regulated to between 0.14 and 0.69 MPa (20 and 100 psi)
Operating Pressure	28 MPa (4000 psi) maximum liquid path (tubing, valves, columns, etc.)
Dimensions	60 cm high x 30 cm x 40 cm deep (23 in x 12 in x 16 in)
Weight	30 kg (65 lbs)

A.3 Control Panel

Display	Liquid crystal display
Buttons	16 front panel buttons for controlling the display and operating functions

A.4 Pump

Type	Single-reciprocating piston with metal-free flow path with PEEK components
Operation Mode	Constant volume
Flow Range	0.5 to 4.5 mL/min

A.5 Pulse Damper

Type	Coiled restricted tubing (PEEK), controlled compliance
Maximum Pressure	28 MPa (4000 psi)

A.6 Detector

Range	1000 μ S, full-scale
Temperature Compensation	1.5, 1.7, or 1.9% per $^{\circ}$ C
Cell Drive	Pulsed, bipolar fixed frequency
Auto Offset	-999 to 999 μ S
Local Operation	Front panel controls and display status of all functions
Remote Operation	Control of four functions via TTL inputs

DX-LAN Operation (Optional)	Control of all functions by PeakNet software via the DX-LAN interface
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A.7 Conductivity Cell

DX-120 Standard Cell (CDM-3)	Cell body: Chemically inert polymer Electrodes: 316 stainless steel Active Volume: 1.25 μ L Maximum Pressure: 2.0 MPa (300 psi)
DX-120 High- Performance Cell	Note: This cell is included with the DS4 Detection Stabilizer Cell body: PEEK Electrodes: 316 stainless steel Active Volume: 1.0 μ L Maximum Pressure: 2.0 MPa (300 psi)

A.8 DS4 Detection Stabilizer (Optional)

Warm-up Time	10 minutes (typical)
Temperature Alarms	High or low temperature message displayed when over or under the set temperature

A.9 Valves

Injection	Two-position, six-port, electrically-activated Rheodyne valve with PEEK wetted components
Column Switching (Optional)	Two-position, ten-port, electrically-activated Rheodyne valve with PEEK wetted components

A.10 Delay Volume

System Total <5 mL

B • Installation

This chapter provides instructions for the initial installation of the DX-120. Instructions for installing optional features, such as the DS4 Detection Stabilizer and the DX-LAN, are also provided.

B.1 Facility Requirements

- Make sure the DX-120 installation site meets the power and environmental specifications listed in Appendix A.
- Install the DX-120 on a sturdy workbench at a height that ensures convenient viewing of the front panel display and access to the interior.



Use caution when lifting the DX-120, which weighs 30 kg (65 lbs). Lift the DX-120 only from the non-slip pads on each side of the cabinet bottom (see Figure 2-1). Lifting from the front panel door will damage the door hinges.

B.2 Installation Overview

- Connect the power cord
- Place the waste lines in a waste container
- Connect the gas supply line
- (Optional) Connect the DX-LAN network cable
- Connect the eluent reservoir(s)
- Set up the pump
- Install the chromatography components (SRS and columns)
- (Optional) Connect an autosampler
- (Optional) Connect a chart recorder or integrator

- Set the configuration DIP switches

The DX-120 Ship Kit (P/N 050130) contains all items necessary for completing the installation.

B.3 Rear Panel Connections

B.3.1 Power Connection

1. A label on the DX-120 rear panel indicates the line frequency (50 or 60 Hz) and voltage (100, 115, 230 VAC) for which the system is designed (see Figure B-1). 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-1) to a grounded power source.



SHOCK HAZARD—To avoid electrical shock, a grounded receptacle must be used. Do not operate or connect to AC power mains without an earthed ground connection.



The power supply cord is used as the main disconnect device. Make sure the outlet is located near the DX-120 and is easily accessible.

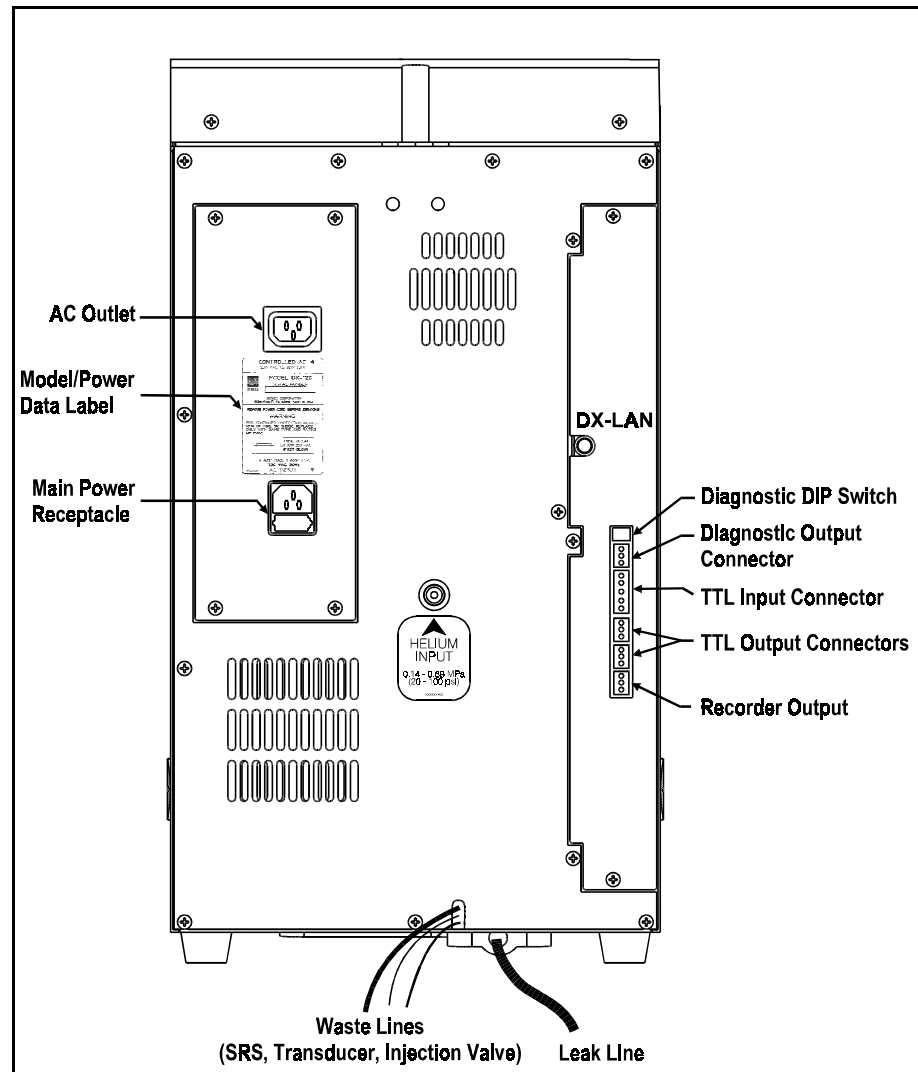


Figure B-1. DX-120 Rear Panel

B.3.2 Switched AC Outlet Connection (Optional)

The AC power outlet provides on-off control of an external accessory, such as a pre-concentration pump. PeakNet software controls the outlet; there is no control from the DX-120.

Use one of the following cables (cables must be ordered separately from the DX-120):

- The accessory jumper cable (P/N 960748) is for modules with IEC 320 connectors.
- The accessory power adapter cable (P/N 960749) is for modules with NEMA 5-15 connectors (the 3-prong American line cord plug).

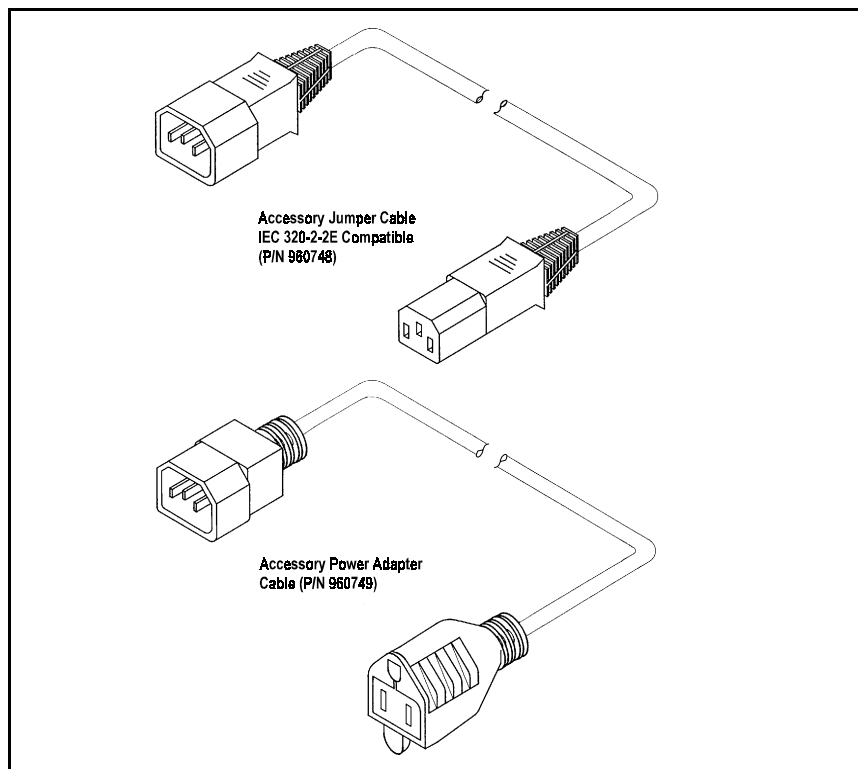


Figure B-2. Accessory Cables

B.3.3 Waste Lines

Untape the coiled waste lines from the rear panel and place the ends into a waste container. To prevent waste siphoning back into the system, make sure the tubing is not bent, pinched, or elevated at any point.

B.3.4 Gas Connection

Locate the 3-mm (0.062-in) ID tubing (P/N 030089) provided in the DX-120 Ship Kit. Push one end into the gas connector on the rear panel (see Figure B-1). Connect the other end to a source of laboratory-quality helium regulated to between 0.14 and 0.69 MPa (20 and 100 psi).

B.3.5 DX-LAN Cable Connection (Optional)

NOTE

For the DX-120 to communicate with PeakNet software, a DX-LAN interface card (P/N 050225) must be installed in the DX-120. If you order a DX-120 with the DX-LAN option, the card is installed at the factory. If you order the DX-LAN at a later time, see Section B.15 for installation instructions.

Figure B-3 illustrates the cable connections for a single DX-120 connected to a PeakNet workstation. Refer to *Installing the PeakNet System* for details about installing more than one instrument on the network.

1. Connect a BNC tee connector (P/N 921914) to the DX-LAN BNC connector on the rear panel (see Figures B-1 and B-3).
 - a. Note the two small locking pins on each side of the DX-LAN BNC connector.
 - b. Push the tee connector onto the DX-LAN BNC connector and twist until the locking pins are fully engaged in the slots on the tee connector.

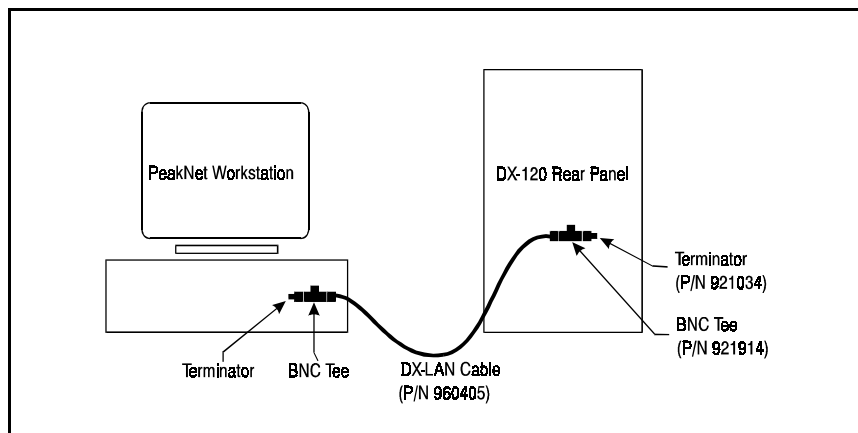


Figure B-3. DX-LAN Cable Connection

- c. Pull firmly on the tee connector to verify that it cannot move.
2. Locate the cylindrical ferrite core (P/N 918014) in the Ship Kit. The core is required for EMI suppression. Wind the DX-LAN cable (P/N 960405) three turns around the core (see Figure B-4). The core should be approximately 7.5 cm (3 in) from the end of the cable. Plug the end of the DX-LAN cable that is nearest to the ferrite cylinder into the BNC tee on the DX-120 rear panel.
3. If the DX-120 is the last (or only) instrument on the network, cap the unconnected end of the tee with a terminator plug (P/N 921034).
4. Connect the other end of the cable to the BNC tee connector on the PeakNet workstation. If this is the last device to be connected, cap the unconnected end of the tee with a terminator plug.

IMPORTANT

Do not connect the DX-LAN cable directly to the BNC connectors on the PeakNet workstation or DX-120. BNC tee connectors and terminator plugs are required for correct data link/transfer.

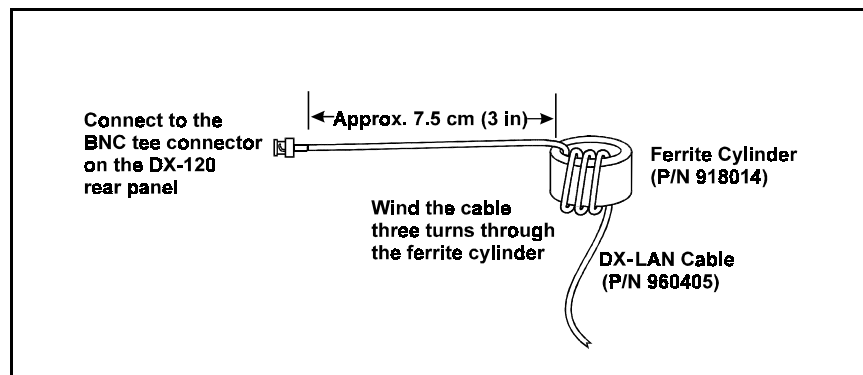


Figure B-4. Ferrite Core Installation on DX-LAN Cable

B.4 Eluent Reservoir Connections

1. Untape the coil of red air tubing from the reservoir tray on the top of the DX-120. Push the end of the tube firmly onto the barbed connector on the eluent reservoir cap assembly.
2. Untape the coil of white eluent tubing from the reservoir tray and screw the tube end-fitting securely into the eluent fitting on the reservoir cap.

B.5 Pump Setup

NOTE

Always filter eluents through a 0.45 μ m filter to remove small particulates that may contaminate the pump. Install an end-line filter (P/N 045987) on the end of each eluent reservoir line. Filters are supplied in the pressurizable reservoir ship kits. See the *Pressurizable Reservoir Installation Instructions* for details.

B.5.1 Priming the Pump

1. Fill the eluent reservoir with the eluent required for the application or, if you will be calibrating the pump flow rate, with deionized water.
2. Lower the outlet line of the reservoir cap assembly into the reservoir. Handtighten the cap.
3. Press **Eluent Pressure** on the front control panel to pressurize the reservoir.
4. Open the pressure transducer waste valve (see Figure 2-2) by turning the knob counterclockwise two turns. When the valve is open, eluent flows directly from the pump to waste.
5. Press **Pump** on the front control panel to turn on the pump. Press **Flow Setting** to display the pump flow rate.
6. Pull out the pump flow adjustment knob and adjust the flow rate to 2.0 mL/min. Push the knob back in.

7. After about 10 seconds, adjust the flow rate to the setting required for the application.
8. Close the pressure transducer waste valve and let the system flush and equilibrate for 10 minutes.

B.5.2 Checking the Pump Flow Rate Calibration

Check the pump flow rate calibration at installation and whenever you change applications.

1. Disconnect the line from port 3 on the injection valve if it is currently connected to either the guard column or the column switching valve (see Figure B-20).
2. Connect one of the following to port 3 on the injection valve: either an 11 MPa (1600 psi) backpressure device or a piece of narrow-bore PEEK tubing that will provide 11 MPa (1600 psi) of backpressure. Route the other end of the backpressure tubing to waste.
3. Fill the eluent reservoir with deionized water.
4. Turn on the pump and set the flow rate to 1.2 mL/min.
5. Pump deionized water through the system for at least 15 minutes to allow the system to equilibrate.
6. Collect the water into a tared beaker for 5 minutes.
7. Weigh the collected water. If it is not 6.00 ± 0.05 g, see Section 5.7 for flow rate calibration instructions.

B.6 Connections to Chromatography Components

The DX-120 injection valve, column switching valve (if installed), pump, and cell or DS4 connections are plumbed before shipping. The DX-120 Ship Kit contains tubing assemblies for completing the SRS and column connections.

NOTE

For shipping purposes, unions are installed between unconnected lines from the cell and valves. Remove these unions before beginning the SRS and column installation procedure.

The DX-120 can be plumbed for three operating modes:

- AutoSuppression™ Recycle mode (SRS required), shown in Figure B-5.
- Nonsuppressed mode, shown in Figure B-6. Conductivity measurements made when no suppressor is in-line may show noticeable baseline drift.
- AutoSuppression with External Regenerant mode. This mode is required if the eluent contains organic solvents.

This section contains installation instructions for the AutoSuppression and Nonsuppressed modes. Refer to the SRS manual for information about using the AutoSuppression with External Regenerant mode.

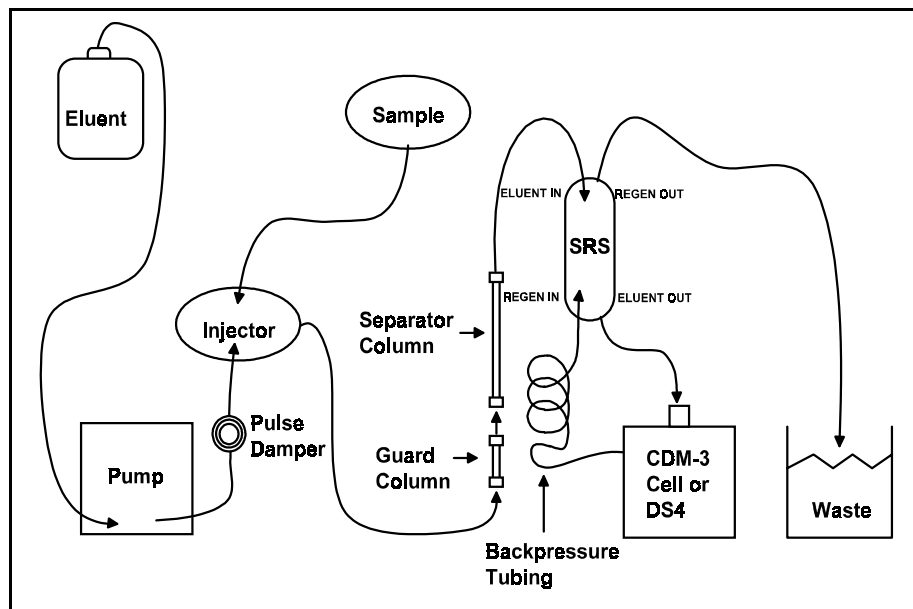


Figure B-5. AutoSuppression Recycle Mode

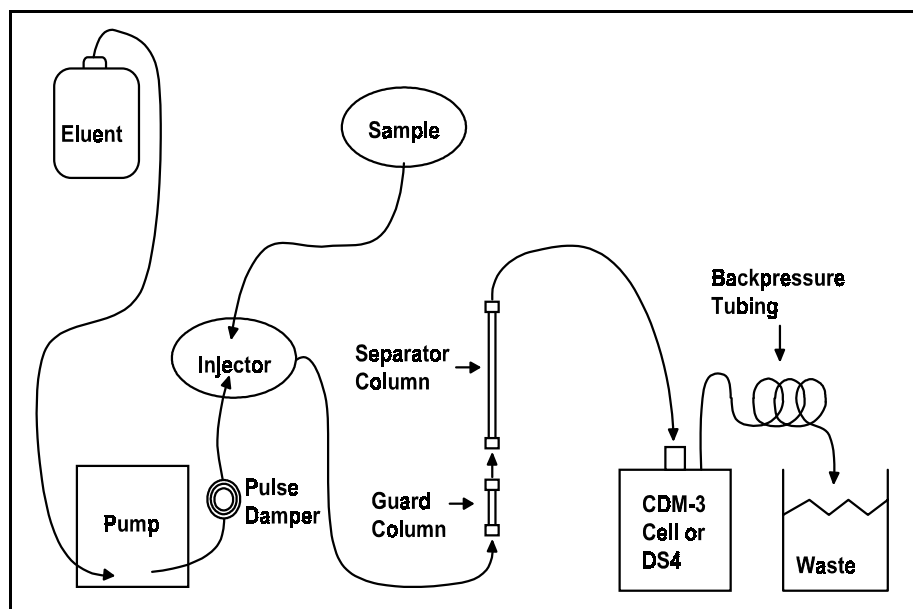


Figure B-6. Nonsuppressed Mode

B.6.1 Backpressure Requirements

All detector cells require enough backpressure to prevent eluent in the cell from degassing due to abrupt volume changes between the small inner diameter of the connecting tubing and the relatively larger volume of the cell. Degassing creates bubbles in the cell and disrupts detector responsiveness.

The DX-120 Ship Kit includes backpressure coils (P/N 045877) for installation after the cell outlet (see Figures B-5 and B-6). The number of coils required depends on the application flow rate and the type of system (see Table B-1).

Flow Rate (mL/min)	Tubing ID	Coil Length	Number of Coils Single-Column Systems	Number of Coils Dual-Column Systems
0.5 to 1.5	0.25-mm (0.010-in)	0.76 M (2.5 feet)	2	1
1.5 to 3.0	0.25-mm (0.010-in)	0.76 M (2.5 feet)	1	0

Table B-1. Coils for Backpressure Requirements

In the AutoSuppression mode, the backpressure generated by the DS4 (if installed), cell, and backpressure tubing is applied to the SRS. For example, with 1 meter of 0.25-mm (0.01-in) ID tubing at a flow rate of 1.0 mL/min, the backpressure is about 0.21 MPa (30 psi). The optimal backpressure is 0.28 MPa (40 psi). Refer to the SRS manual for details about SRS backpressure requirements.

In the Nonsuppressed mode, the backpressure generated by the backpressure tubing is applied to the cell. The optimal backpressure is 0.21 to 0.34 MPa (30 to 50 psi).

B.6.2 Self-Regenerating Suppressor (SRS) Installation

NOTE

The instructions here do not replace the instructions in the SRS manual. For complete SRS installation instructions, as well as the initial SRS start-up procedure, refer to the SRS manual.

The SRS mounts on tabs on the component panel (see Figure B-7). Orient the SRS with the **REGEN OUT** port and the cable at the top; align the slots on the back of the SRS with the tabs on the panel. Press in, and then down, to lock the SRS in place. Lift up and pull out to remove the SRS. Install a single SRS on the left; when installing two suppressors, install SRS A on the left and SRS B on the right.

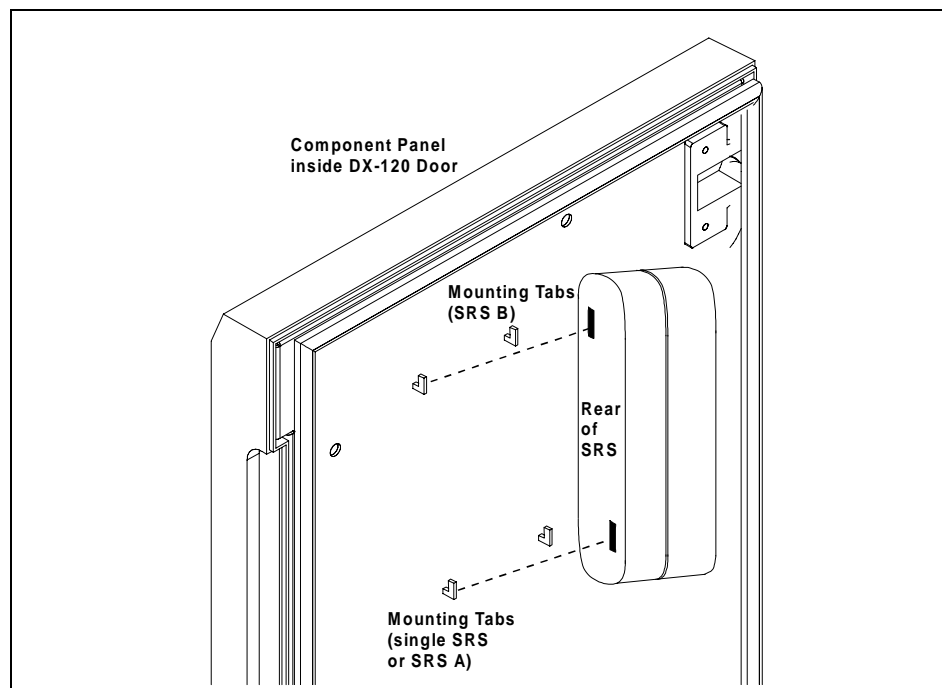


Figure B-7. Installing the SRS

The DX-120 Ship Kit includes certain items required for SRS installation:

- SRS gas separator waste tube (P/N 045460)
- Backpressure coils for 4 mm SRS (P/N 045877)

The SRS plumbing connections depend on whether you are installing one or two column sets. Refer to the appropriate following section.

Single-Column SRS Installation

Figure B-8 shows the connections for a single-column system.

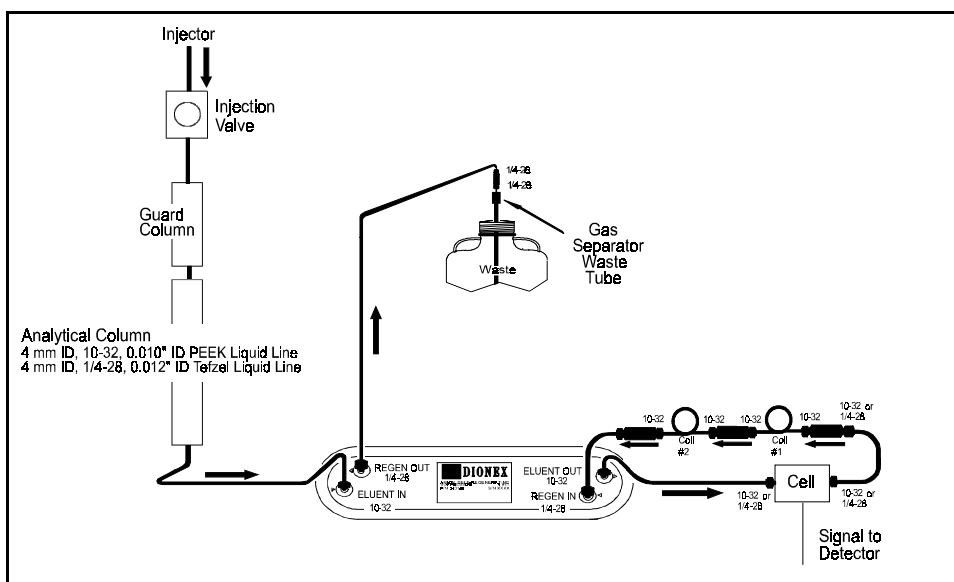


Figure B-8. SRS Connections: Single-Column DX-120

1. Connect the line from the cell or DS4 outlet to the SRS **REGEN INLET**.
2. Connect the line from the cell or DS4 inlet to the SRS **ELUENT OUTLET**.
3. Connect backpressure tubing and a waste line to the SRS **REGEN OUTLET**. Refer to the SRS manual for instructions.

Dual-Column SRS Installation

Figure B-9 shows the connections for a dual-column system.

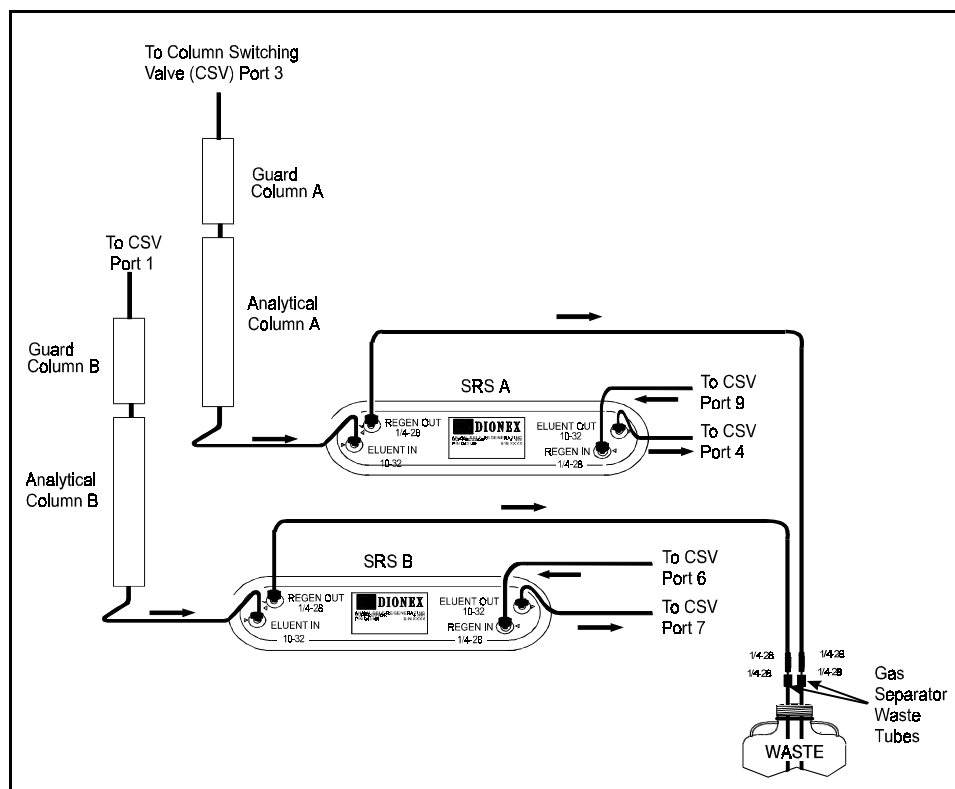


Figure B-9. SRS Connections: Dual-Column DX-120

1. Connect the line from port 4 on the column switching valve to the **REGEN INLET** of SRS A.
2. Connect the line from port 6 to the **REGEN INLET** of SRS B.
3. Connect port 9 to the **ELUENT OUTLET** of SRS A.
4. Connect port 7 to the **ELUENT OUTLET** of SRS B.
5. Connect waste lines to the **REGEN OUTLET** ports on both suppressors. Refer to the SRS manual for instructions.

Completing the SRS Installation

1. For a single-column system, plug the cable from the SRS into the matching cable extending from the SRS A connector on the electronics card (see Figure B-10).

For a dual-column system, also plug the cable from SRS B into the matching cable extending from the SRS B connector.

2. Select the SRS current setting(s) with SW2. Figure B-10 shows the default settings. Refer to the SRS manual for the recommended setting for your application.

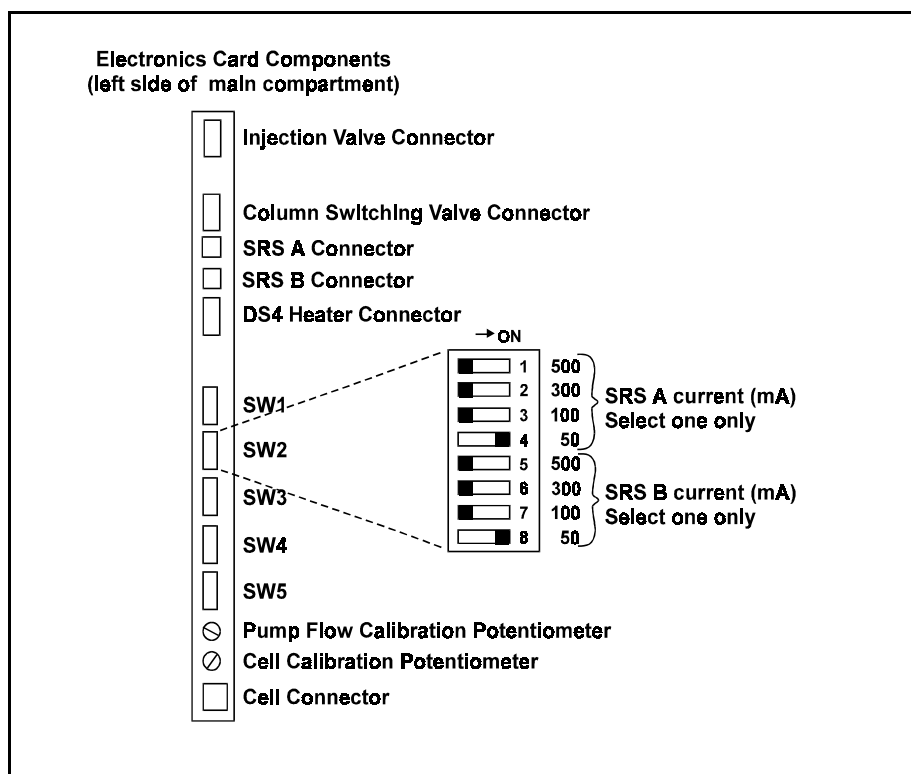


Figure B-10. Electronics Card Connections

B.6.3 Column Installation

Before installing the column(s) in the DX-120, refer to the column manual for specialized installation and start-up instructions.

Column Setup

1. Before installing the separator column, pump deionized water through the injection valve at 1.0 to 3.0 mL/min for 2 to 5 minutes to clear any air from the liquid lines. Trapped air reduces the column efficiency.
2. Switch the injection valve between the **INJECT** and **LOAD** positions several times. This allows water to flow through the sample loop and each valve port.
3. Set the Low Pressure Alarm DIP switch (SW1-8) to off. Set the flow rate to 2.0 mL/min and verify that the pressure through the system, with no columns installed, is less than 690 KPa (100 psi). Reset the Low Pressure Alarm switch to on.
4. Stop the pump.
5. Remove the end plugs from the separator column(s) and store them in a safe place. You must reinstall the end plugs in the column(s) before placing them in storage.
6. If they are not already installed, install the column mounting clips (P/N 046699) on the component panel. Orient each clip with the larger clasps toward the front of the panel; then, squeeze the open ends of the clip together and insert them into the square opening. Install two clips for the separator column(s) and one for the guard column(s).

NOTE

An arrow on each column indicates the liquid flow direction. The arrow points toward the column outlet.

7. Orient the guard column with the outlet facing down and snap the column into its clip. For dual-column systems, snap column A in the back and column B in the front.
8. Orient the separator column with the outlet facing up and snap the column into its clip. For dual-column systems, snap column A in the back and column B in the front.

The remaining steps depend upon whether you are installing one or two column sets.

Single-Column Set Connections

1. Connect the line from port 3 on the injection valve to the guard column inlet.
2. Connect the guard column outlet to the separator column inlet. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).
3. For AutoSuppression mode, connect the separator column outlet to the SRS **ELUENT INLET**. For Nonsuppressed mode, connect the separator column outlet to the cell or DS4 inlet. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

Dual-Column Set Connections

1. Connect the line from port 1 on the column switching valve to the inlet of guard column B.
2. Connect the line from port 3 on the column switching valve to the inlet of guard column A.
3. Connect the outlet of guard column A to the inlet of separator column A. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276). Repeat for guard and separator columns B.

4. Connect the outlet of separator column A to the **ELUENT INLET** of SRS A and connect the outlet of separator column B to the **ELUENT INLET** of SRS B. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

B.7 TTL Control Connections (Overview)

This section summarizes the functions of the TTL input and output connectors on the DX-120 rear panel (see Figure B-1). For example connections, see Sections B.8.2 and B.9.2.

B.7.1 TTL Inputs

The TTL input connectors can be connected to the TTL outputs of a controlling device, such as an integrator, to control the following DX-120 functions: pump on/off, 10X signal range, auto offset, and injection/remote start.

Figure B-11 summarizes the TTL inputs.

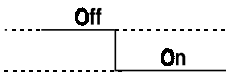
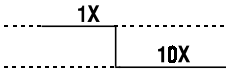

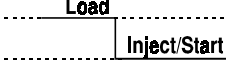
TTL Input Function	Logic Type	Waveform	Description
Pump Enable	Hold		Normally pulled high for pump off. Pull low for pump on.
10X Range	Hold		Normally pulled high for 1X signal range. Pull low for 10X range.
Auto Offset	Pulse Trigger		Temporarily pull to ground to cause an auto offset.
Inject/Remote Start	Hold		Normally pulled high for Load. Pull low for Inject and start PeakNet run.

Figure B-11. TTL Inputs

B.7.2 TTL Outputs

There are two types of TTL outputs:

- The Inject and Pressure Alarm outputs signal when an injection or a pressure alarm occurs. When connected to the TTL inputs of another device, such as an autosampler, these signals can trigger a response in the connected device. For example, the Inject output can start the autosampler load cycle.
- The TTL Out 1 and TTL Out 2 are controlled by PeakNet software only. When connected to the TTL inputs of other devices, these outputs can control functions in the connected devices.

Figure B-12 summarizes the TTL outputs.

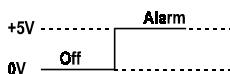
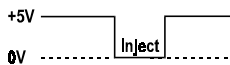
TTL Output Function	Logic Type	Waveform	Power-up Status	Description
Pressure Alarm	Hold		0V	Normally low. Goes to high when an alarm occurs.
Inject	Pulse Trigger		+5V	Normally pulled high. Pulses down at injection. 500 ms pulse duration.
TTL Out 1		- PeakNet Software Control Only -		
TTL Out 2		- PeakNet Software Control Only -		

Figure B-12. TTL Outputs

B.7.3 Ferrite Core Installation on TTL Output Cables

An EMI suppression core (P/N 918013) must be installed on any cable or wires used to connect the DX-120 TTL outputs to another instrument.

1. Gather multiple pairs of wires together.
2. Loop the wires or cable three turns through the core and snap it closed (see Figure B-13).
3. The core should be approximately 7.5 cm (3 in) from the **TTL Outputs** connectors on the DX-120 rear panel.

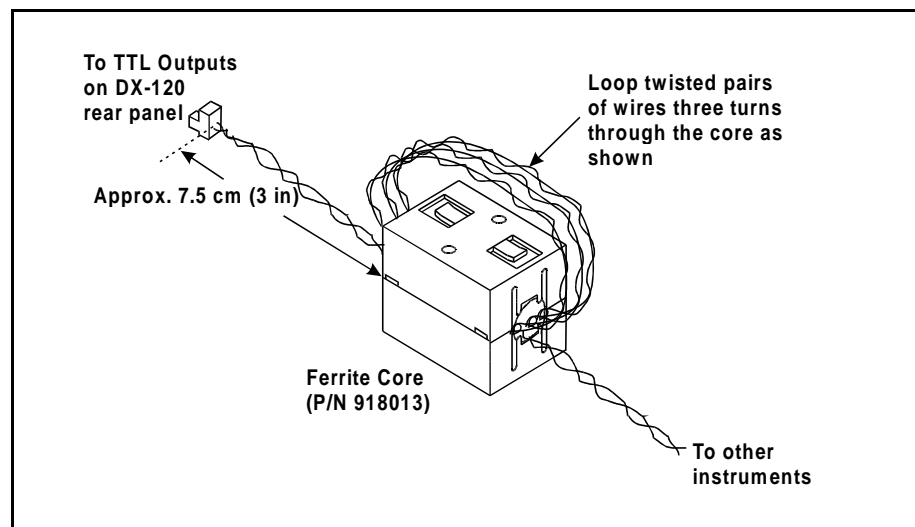


Figure B-13. Ferrite Core Installation on TTL Outputs Cable

B.8 Autosampler Connections (Optional)

B.8.1 Autosampler Outlet Line Connection

1. Disconnect the sample inject line from port 5 on the DX-120 injection valve (see Figure B-20).
2. Route the outlet line from the autosampler through the opening below the injection port on the DX-120 front door (see Figure 2-1). Connect the line to port 5.

B.8.2 AS40 Automated Sampler Connections

This section describes how to connect an AS40 Automated Sampler to a DX-120 that is connected to a PeakNet workstation. Figure B-14 illustrates the TTL connections. If the DX-120 is connected to an integrator, see Section B.9.2 for the AS40 connections.

1. Locate the Relay Control cable (P/N 047946) supplied with the AS40 Automated Sampler. Connect this cable's 10-pin connector to the **RELAY CONTROL** connector on the AS40 rear panel.
2. Remove the 2-pin connector from the pair of wires labeled **LOAD** on the Relay Control cable.



When connecting wires to the green connectors, be careful not to allow stray strands of wire to short to the adjoining terminal on the connector.

3. Locate a green 3-pin connector (P/N 921186) in the DX-120 Ship Kit. Insert the red wire from the **LOAD** pair into the last position on the connector and tighten the locking screw. Insert the ground wire into the first position on the connector and tighten.
4. Install an EMI suppression ferrite core on the wires (see Section B.7.3).

5. Plug the connector into the lower **TTL Outputs** connector on the DX-120 rear panel. Make sure the red wire connects to the **TTL1** position and the ground wire connects to the **Ground**.

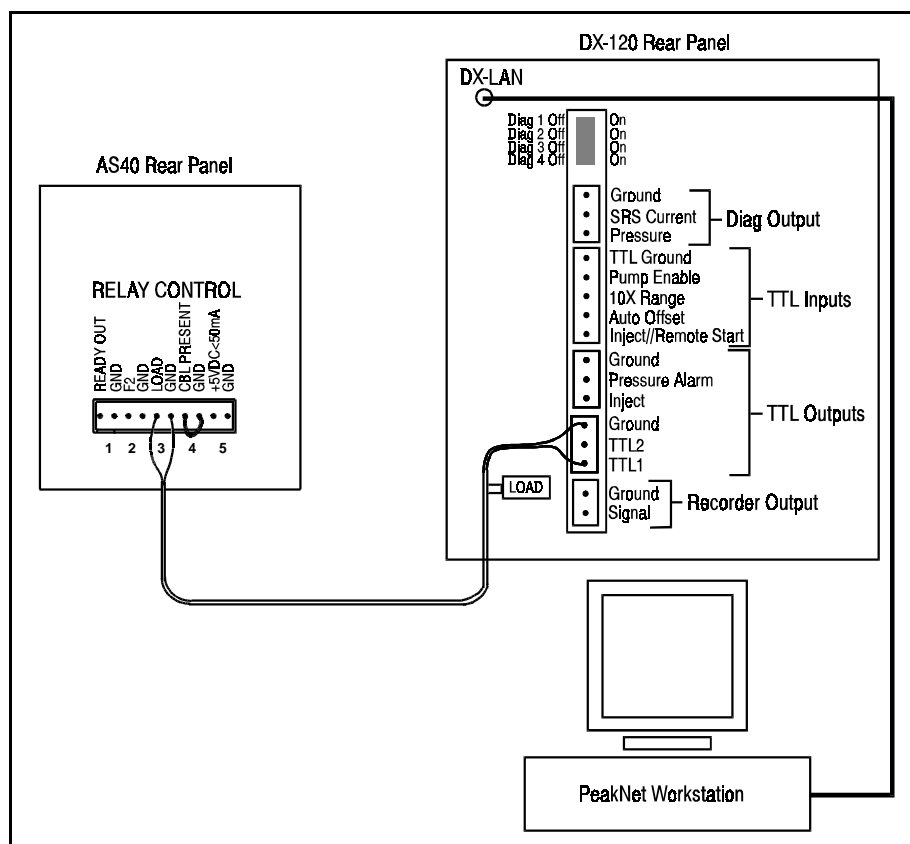


Figure B-14. AS40 Automated Sampler with PeakNet Connections

B.8.3 AS3500 Automated Sampler Connections

This section describes how to connect an AS3500 Automated Sampler to the DX-120.

Figure B-15 illustrates the TTL connections.

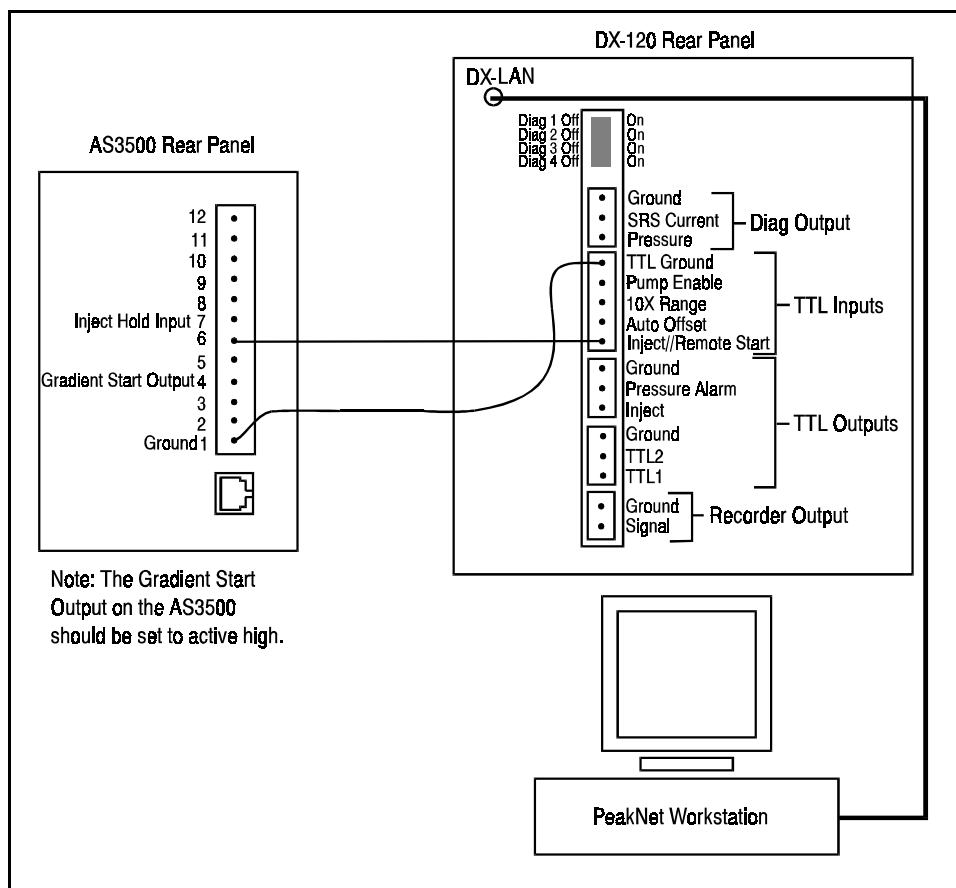


Figure B-15. AS3500 Automated Sampler TTL Connections

1. Locate the green 5-pin connector (P/N 921273) and the twisted pair of wires (P/N 043598) in the DX-120 Ship Kit. Insert the signal wire from the pair into the last position on the connector. Use a screwdriver to tighten the locking screw. Insert the ground wire into the first position on the connector and tighten.



When connecting wires to the green connectors, be careful not to allow stray strands of wire to short to the adjoining terminal on the connector.

2. Plug the 5-pin connector into the **TTL INPUTS** connector on the DX-120 rear panel. Make sure the signal wire connects to the **Inject** position and the ground wire to the **TTL Ground**.
3. Connect the other end of the signal wire to **Pin 6** on the AS3500 rear panel.

Disabling the DX-120 Injection Valve

With the above TTL connections, each time the AS3500 sends a remote start output signal to the DX-120 to start a PeakNet run, the DX-120 injection valve automatically switches to the Inject position. If desired, you can disable the DX-120 injection valve.

1. Locate the set of DIP switches above the TTL connectors on the DX-120 rear panel (see Figure B-16).
2. The default position for all these switches is Off.
3. To disable the DX-120 injection valve, use a small screwdriver to press switch 2 down (the On position).

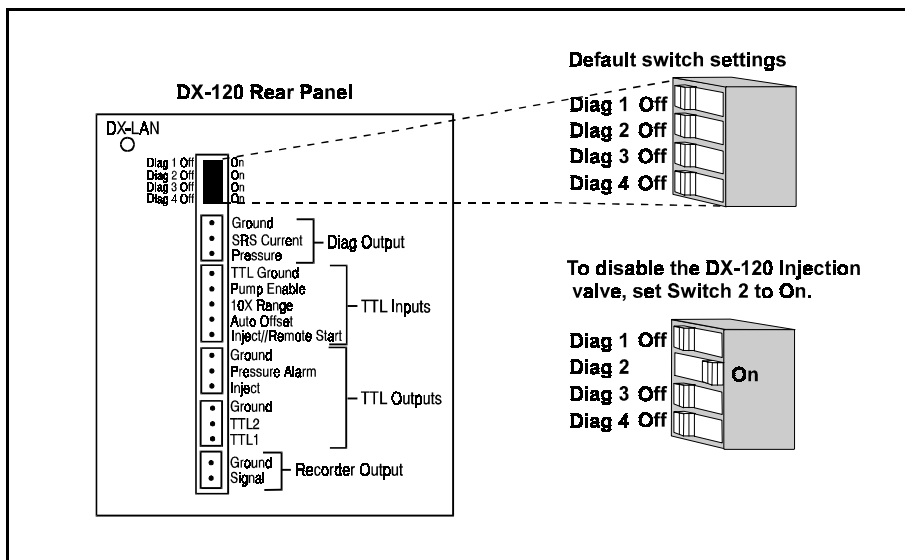


Figure B-16. Disabling the DX-120 Injection Valve

B.9 Recorder and Integrator Connections (Optional)

B.9.1 Chart Recorder Connections

1. Connect one end of the Recorder Signal Wire assembly (P/N 043598) to the Recorder Output connectors on the DX-120 rear panel (see Figure B-1).
2. Connect the other end to the appropriate input connectors on the chart recorder.

B.9.2 4400 or 4600 Integrator Connections

Recorder Data Transmission

To use the integrator as a recorder with integration functions:

1. Connect one end of the Recorder Signal Wire assembly (P/N 043598) to the Recorder Output connectors on the DX-120 rear panel (see Figure B-1).
2. Connect the other end to the 4400 or 4600 Integrator input connectors.

Time Functions Automation

To use the integrator for timed control of DX-120 and AS40 Automated Sampler functions, install an Automation Cable (P/N 043940), in addition to the Recorder Signal Wire assembly. Figure B-17 illustrates the wire connections.

1. Set the DX-120 to Local control.
2. Connect the Automation Cable's 36-pin connector to the **TIME FUNCTION** connector on the rear of the integrator.
3. Connect the Automation Cable's 10-pin green connector, labeled **A1**, to the AS40 **RELAY CONTROL** connector on the rear of the autosampler.
4. Connect the Automation Cable's 5-pin green connector, labeled **A3**, to the DX-120 **TTL Input** connector.

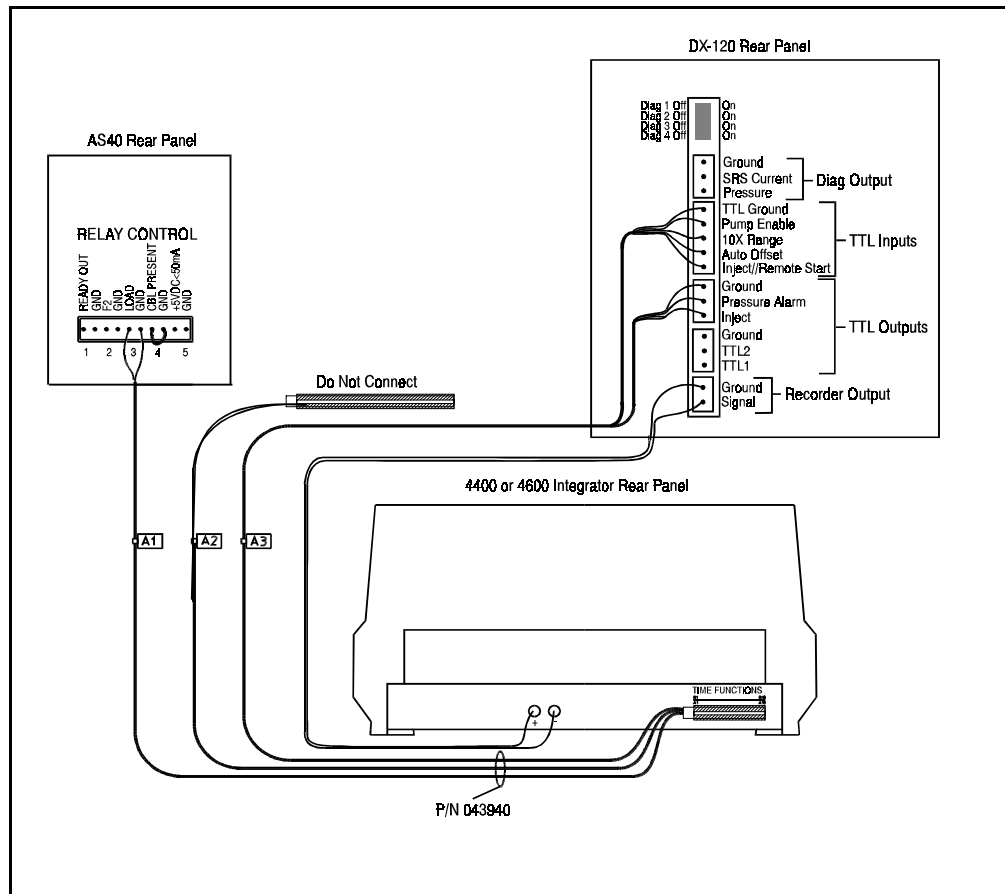


Figure B-17. Single-Channel Integrator Connections

5. Connect the Automation Cable's 3-pin green connector, labeled **A3**, to the DX-120 upper **TTL Output** connector.

Note: The 50-pin parallel connector, labeled **A2**, is not used.

B.10 Configuration DIP Switch Settings

The DIP switches on the left side of the main compartment set various DX-120 operating parameters. The factory default settings are appropriate for most applications. After reviewing the settings listed in Figure B-18, make any changes required for your system.

NOTE

The DIP switch labeled DIAGNOSTIC TESTS on the rear panel is for service procedures only. Do not change any of its settings unless directed to do so by a Dionex Service representative. For routine operation, all settings are off.

Switch Set	Default Settings
SW1	<p>→ ON</p> <div> <div> <div>1</div> <div>2</div> <div>3</div> <div>4</div> <div>5</div> <div>6</div> <div>7</div> <div>8</div> </div> <div> <div>Auto Inject Reset</div> <div>Pump Time-out</div> <div>Dual — Column Configuration</div> <div>psi — Pressure Units</div> <div>24.1 MPa (3500 psi)</div> <div>17.3 MPa (2500 psi)</div> <div>13.8 MPa (2000 psi)</div> <div>2.0 MPa (250 psi)</div> </div> <div> <div>High Pressure Alarm</div> <div>Select one only</div> <div>Low Pressure Alarm</div> </div> </div>
SW2	<p>→ ON</p> <div> <div>1</div> <div>2</div> <div>3</div> <div>4</div> <div>5</div> <div>6</div> <div>7</div> <div>8</div> </div> <div> <div>500</div> <div>300</div> <div>100</div> <div>50</div> <div>500</div> <div>300</div> <div>100</div> <div>50</div> </div> <div> <div>SRS A Current (mA)</div> <div>Select one only</div> <div>SRS B Current (mA)</div> <div>Select one only</div> </div>
SW3	<p>→ ON</p> <div> <div>1</div> <div>2</div> <div>3</div> <div>4</div> <div>5</div> <div>6</div> <div>7</div> <div>8</div> </div> <div> <div>Column Select Mode</div> <div>Alarm Sound</div> <div>Auto Offset</div> <div>45 °C</div> <div>40 °C</div> <div>35 °C</div> <div>30 °C</div> <div>Reserved 1</div> </div> <div> <div>DS4 Temperature</div> <div>Select one only</div> </div>
SW4	<p>→ ON</p> <div> <div>DS4</div> <div>DS4</div> <div>100 µS</div> <div>10 V</div> <div>0%</div> <div>Neg (-)</div> <div>1</div> <div>2</div> <div>3</div> <div>4</div> <div>5</div> <div>6</div> <div>7</div> <div>8</div> </div> <div> <div>CDM-3</div> <div>CDM-3</div> <div>1000 µS Range</div> <div>1 V — Full Scale Voltage</div> <div>Offset 10%</div> <div>Pos (+) — Polarity</div> <div>Inject Mark</div> <div>Reserved 2</div> </div> <div> <div>Cell Type</div> <div>Select both</div> <div>Recorder Settings</div> </div>
SW5	<p>→ ON</p> <div> <div>1</div> <div>2</div> <div>3</div> <div>4</div> <div>5</div> <div>6</div> <div>7</div> <div>8</div> </div> <div> <div>0.5 sec</div> <div>1.0 sec</div> <div>2.0 sec</div> <div>4.0 sec</div> <div>1</div> <div>2</div> <div>Filter Enable</div> <div>1.9%</div> <div>1.7%</div> <div>1.5%</div> <div>Reserved 3</div> <div>Test Cell</div> </div> <div> <div>Response Time</div> <div>Signal Temperature Compensation</div> <div>Select one only</div> </div> <div> <div>KEY</div> <div> <div>ON</div> <div>= ON—Selects function on right</div> </div> <div> <div>OFF</div> <div>= OFF—Turns off function on right or selects function on left (if any)</div> </div> </div>

Figure B-18. DIP Switch Settings

DIP Switch Function	Description
Auto Inject Reset (SW1-1)	When on, the injection valve automatically returns to the Load position 60 seconds after it is switched to the Inject position.
Pump Time-out (SW1-2)	When on, if the DX-120 is idle for 90 minutes, the pump flow is reduced to 1/20th of its current rate. The LED on the Pump button flashes when the pump flow is reduced. Press any button to resume the last selected flow rate.
Column Configuration (SW1-3)	Selects the column configuration: dual (on) or single (off).
Pressure Units (SW1-4)	Selects the pressure units (psi or MPa) shown on the screen.
High Pressure Alarm (SW1-5, 6, 7)	Selects at what pressure the high pressure alarm occurs.
Low Pressure Alarm (SW1-8)	Enables an alarm when a low pressure condition (2.0 MPa or less) occurs.
SRS A Current (SW2-1, 2, 3, 4)	Selects the operating current for the SRS (or SRS A in a dual-column system).
SRS B Current (SW2-5, 6, 7, 8)	Selects the operating current for SRS B in a dual-column system.
Column Select Mode (SW3-1)	For dual-column configurations (SW1-3 on). When on, selects the column select mode (see Section 2.2.1). When off, selects the eluent select mode (see Section 2.2.2).
Alarm Sound (SW3-2)	Enables or disables the alarm tone.
Auto Offset (SW3-3)	When on, an auto offset is performed when injection occurs.
DS4 Temperature (SW3-4, 5, 6, 7)	Selects the DS4 operating temperature. Select a temperature at least 5 °C above the highest expected ambient temperature.

DIP Switch Function	Description
Cell Type (SW4-1, 2)	Select the type of cell (CDM-3 or DS4) installed in the DX-120. Both switches must be set.
Range (SW4-3)	Selects the microSiemens output (100 μ S or 1000 μ S) to the recorder of a full-scale detector response.
Full-Scale Voltage (SW4-4)	Selects the voltage output (1 V or 10 V) to the recorder of a full-scale detector response.
Offset (SW4-5)	When on, the analog output is offset 10% of full-scale. The 10% offset allows the recording device to plot the signal if it becomes negative.
Polarity (SW4-6)	Selects whether the output signal to the recording device is positive or negative. In nonsuppressed applications in which the analyte output is lower than the background conductance, the polarity must be inverted to have peaks instead of dips.
Inject Mark (SW4-7)	When on, a mark 10% of full-scale is sent to the recording device when the injection valve switches to the Inject position.
Response Time (SW5-1, 2)	When analog filtering is enabled, on/off combinations of these switch positions select the filter response time.
Filter Enable (SW5-3)	Enables or disables filtering of the analog output. Filtering is typically needed only for nonsuppressed applications, which have higher background and noise.
Signal Temperature Compensation (SW5-4, 5, 6)	Selects by how much to adjust the value of the temperature coefficient used by the detector. See Section 3.6 for details.
Test Cell (SW5-8)	Allows testing of the cell electronics for troubleshooting purposes. See Section 4.13 for the cell testing procedure.

If the DS4 is ordered at the same time as the DX-120, it is installed in the DX-120 at the factory. Follow the installation instructions below when you order a DS4 separately.

1. Position the keyhole slots on the rear of the DS4 over the shoulder washers on the DX-120 component panel, then pull the DS4 down into position.
2. Route both DS4 cables to the electronics card at the left rear of the main compartment.
3. Plug the connectors on the cables into the appropriate jacks on the electronics card (see Figure B-19).



Figure B-19. DS4 Cable Connections and DIP Switches

4. For single-column systems, connect the SRS **ELUENT OUT** port to the DS4 inlet (AutoSuppression Recycle mode) or to the column outlet (Nonsuppressed mode), using 0.25-mm (0.010-in) ID tubing (P/N042690), 10-32 fittings (P/N043275), and ferrules (P/N 043276).

For dual-column systems, connect the line from port 8 on the column switching valve to the DS4 inlet.

5. Connect the outlet of the DS4 (the 10-32 coupler) to either the SRS **REGEN IN** port (for AutoSuppression Recycle mode) or to the backpressure line leading to waste (for Nonsuppressed mode).
6. Direct the DS4 spill/overflow tubing to the spill tray at the bottom front of the DX-120.
7. Select a DS4 temperature set point (SW3-4, 5, 6, or 7) (see Figure B-19). Select a temperature at least 5 °C above the highest expected ambient temperature.
8. Set the Cell Type DIP switches (SW4-1, 2) to the off (DS4) position (see Figure B-19).

B.12 CDM-3 Cell Installation

If the CDM-3 cell is ordered at the same time as the DX-120, it is installed in the DX-120 at the factory. Follow the installation instructions below when you order a CDM-3 separately.

1. Orient the CDM-3 with its mounting bracket to the left and align the screw holes on the bracket with the mounting holes on the component panel (see Figure 2-4).
2. Secure the bracket to the component panel with the two Phillips screws supplied with the cell and bracket.
3. Route the cell cable to the electronics card at the left rear of the main compartment and plug the cable connector into the Cell Connector jack on the card (see Figure B-19).
4. For single-column systems, connect the SRS **ELUENT OUT** port to the cell inlet (AutoSuppression Recycle mode) or to the column outlet (Nonsuppressed mode), using 0.25-mm (0.010-in) ID tubing (P/N042690), 10-32 fittings (P/N043275), and ferrules (P/N 043276).

For dual-column systems, connect the line from port 8 on the column switching valve to the cell inlet.

5. Connect the cell outlet (the 10-32 coupler) to either the SRS **REGEN IN** port (for AutoSuppression Recycle mode) or to the backpressure line leading to waste (for Nonsuppressed mode).
6. Set the Cell Type DIP switches (SW4-1, 2) to the on (CDM-3) position (see Figure B-19).

B.13 Injection Valve Connections

NOTE

Refer to the following information if you need to replace any tubing or fittings.

The injection valve is plumbed at the factory with all tubing and fittings for connections to the pump, injection port, and column (or to the column switching valve in dual-column systems). A 25 μ L PEEK sample loop (P/N 042857) is installed between ports 1 and 4. Other sample loop sizes are available. Contact Dionex for information. Figure B-20 shows the connections.

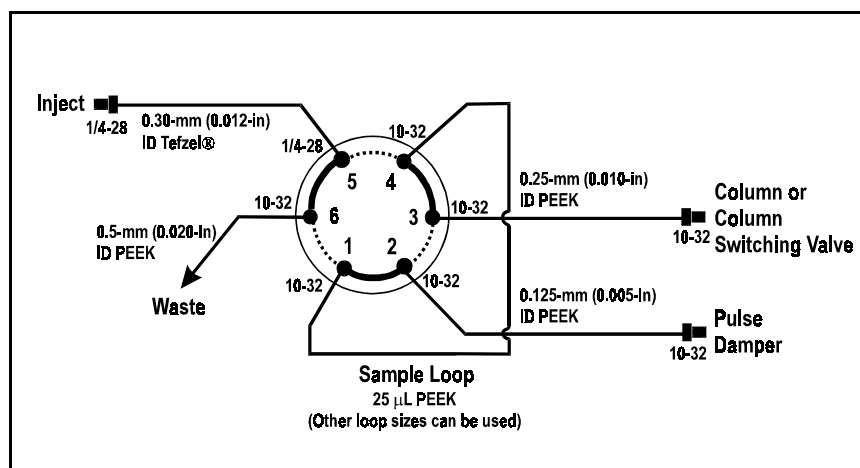


Figure B-20. Injection Valve Plumbing

B.14 Column Switching Valve Connections

NOTE

Refer to the following information if you need to replace any tubing or fittings.

The column switching valve for dual-column configurations is plumbed at the factory with the necessary tubing and fittings to connect the valve to the columns, suppressors, injection valve, and cell. Figure B-21 shows the connections.

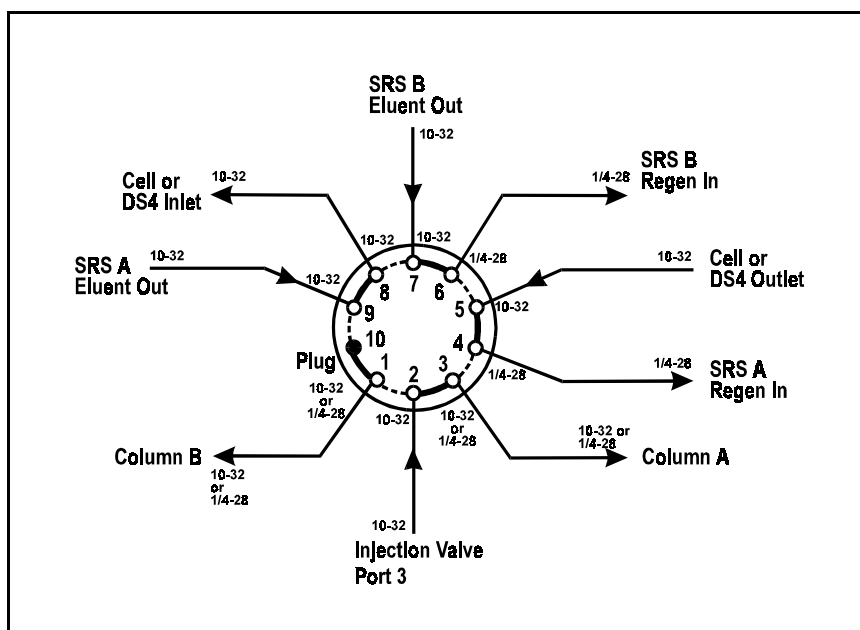


Figure B-21. Column Switching Valve Plumbing

B.15 DX-LAN Card Installation (Optional)

For the DX-120 to communicate with PeakNet software, a DX-LAN interface card (P/N 050225) must be installed in the DX-120. If you order a DX-120 with the DX-LAN option, the card is installed at the factory. Follow the installation procedure below if you order a DX-LAN at a later time.

IMPORTANT

STATIC—The DX-120 electronics are not user-serviceable and the DX-LAN interface card should be installed by qualified personnel only. Observe standard anti-static procedures when installing the interface card or handling the electronics card.



To prevent damage to the DX-120, turn off the main power and unplug the power cord before installing the interface card. Turning off only the front panel power switch is not sufficient.

1. Disconnect all cables from the electronics card at the left of the main compartment (see Figure 2-2).
2. Remove the card's holding screw located above the DIP switches.
3. Disconnect the gas supply line from the rear panel and unplug any TTL connectors.
4. Remove the screws from the rear panel and remove the rear panel from the DX-120. Remove the screws from the electronics card front plate (see Figure B-22).
5. Inside the DX-120 rear compartment, disconnect all cables from the electronics card. Note the connector location for each cable.
6. Pull out the electronics card far enough to allow access to the DX-LAN connector on the top edge of the card (see Figure B-23).

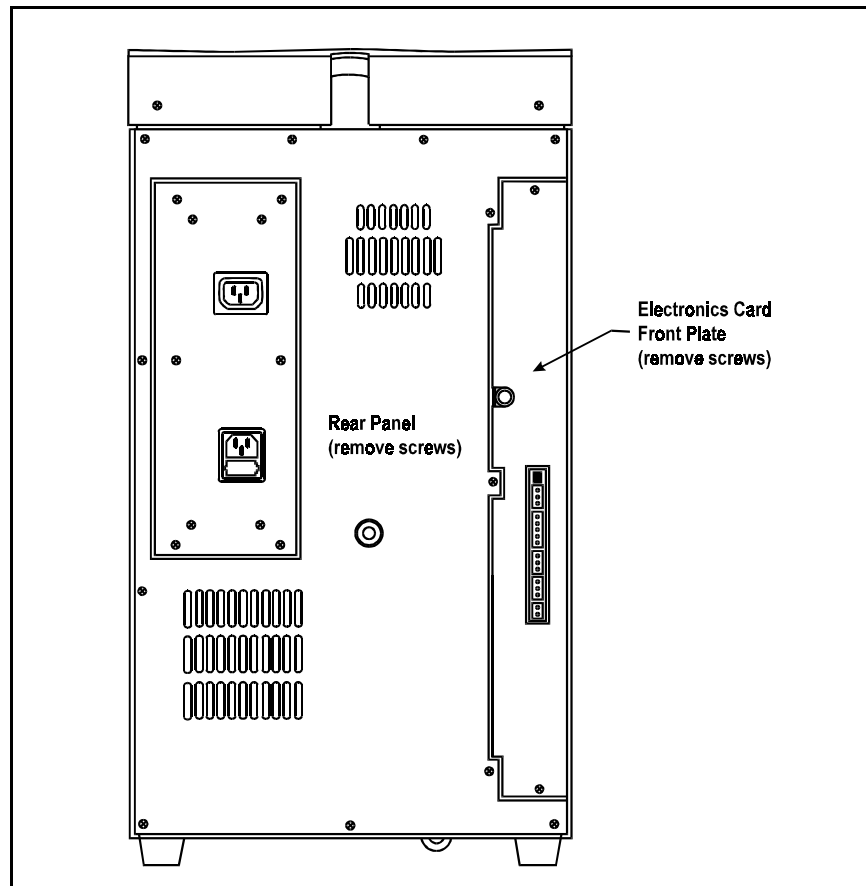


Figure B-22. Removing the Rear Panel

7. Align the BNC connector on the interface card with the opening on the electronics card front plate and insert the interface card connector into the connector on the electronics card (see Figure B-23). The small stainless steel grounding wipers on the BNC connector slide behind the front plate. Press firmly on the card until it mates fully with the electronics card connector.
8. Fasten the interface card to the standoffs on the electronics card with the two Phillips screws (P/N 045791) supplied in the Ship Kit.

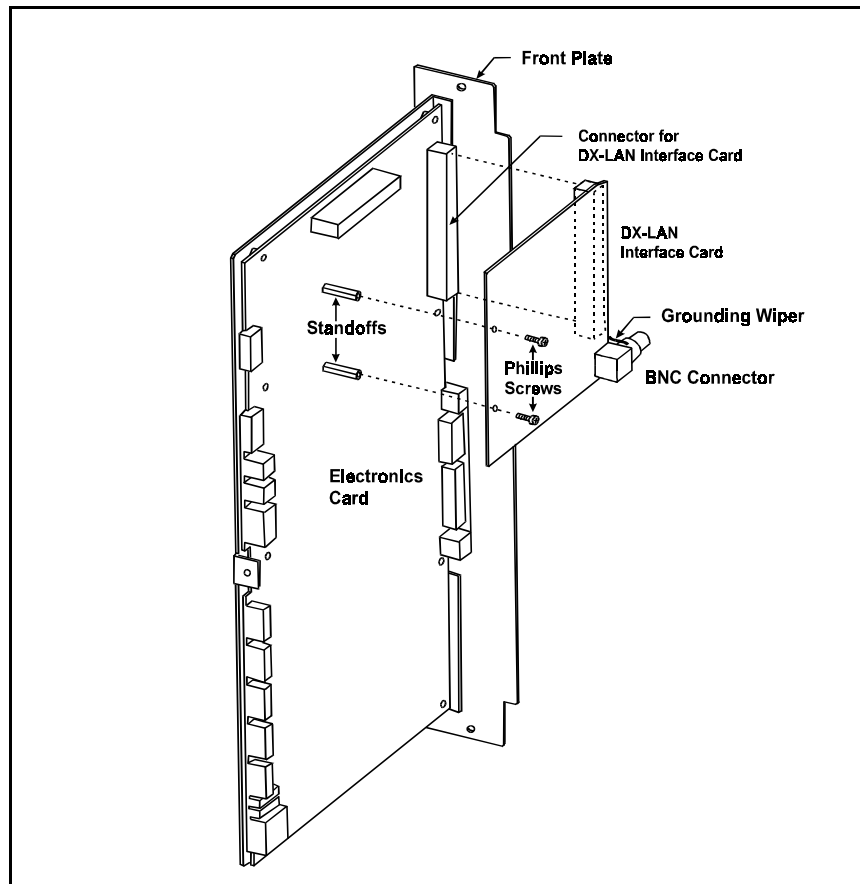


Figure B-23. Installing the DX-LAN Interface Card

9. Push the electronics card back in and replace the mounting screws. Reconnect the cables that were removed in Step 4.
10. Replace the rear panel and mounting screws.
11. Replace the gas supply line and the TTL connectors.
12. Reconnect the cables and the screw that were removed in Steps 1 and 2.
13. Connect the DX-LAN cable. See Section B.3.5 for instructions.

C • Integrator Programming

A Dionex 4400 or 4600 Integrator can collect and integrate data from the DX-120. If you install an Automation Cable (P/N 043940), the integrator can also control the following functions:

- Pump on/off
- Injection valve position
- Auto offset
- 10X range
- Start the AS40 Automated Sampler load cycle (if installed)

See Section B.9.2 for instructions on connecting the data signal wires and Automation Cable from the DX-120 to the integrator and (optionally) an autosampler.

Refer to the *4440 or 4600 Integrator User's Guide* for initial installation and setup of the integrator. After setup, use the instructions in this appendix to configure the integrator to operate the DX-120 and the optional autosampler.

C.1 Integrator Power-up Configuration

At power-up, the integrator performs various self-diagnostic tests, according to the options purchased with the integrator. Refer to the integrator user's guide for details.

After the diagnostic tests are completed, define the integrator parameters required for correct DX-120 data collection. **After setting up the integrator, leave the power on. If you turn off the power, the entered parameters are lost.**

1. Press the **LCD STATUS** key. The LCD displays several parameters:
 - FI=1 indicates that you are using File 1.
 - CS=1 indicates that the chart speed is 1 cm/min.
 - ATTEN=1 indicates that the recorder response is 1 mV full-scale.

2. Press the **PRINT FILE** key. Several file parameters will be printed. PW stands for peak width; PT stands for peak threshold.
3. The following parameters enable the integrator to operate with the DX-120.
 - a. Press the **CHART SPEED** key and enter 0.5 (=0.5 cm/min).
 - b. Press the **ATTEN** key and enter 1024 (=1024 mV full-scale).
 - c. Type in and enter **PT=1000** (a representative peak threshold for 1 V signal).

C.2 Setting Offsets

The default signal offset for the DX-120 is 10%. This is set with a DIP switch (SW4-4).

The recorder offset for the integrator is determined by the OF value. Dionex recommends that you use the DX-120 factory default setting of 10%, and enter **OF=0** for the integrator offset.

C.3 Area Percent Mode

After the offsets have been entered, the integrator is ready to operate in the area percent mode. This is a simple report format that records and integrates peaks from the DX-120 and then reports retention time and peak area.

1. To begin integration, press **INJ/END A** as the sample is being injected.
2. When all of the sample peaks have eluted, press **INJ/END A** again to stop integration and print the report. Use the retention times from this analysis when setting up the component table for automated operation (see Section C.4).

C.4 Using DIALOG to Create a Method

Once the application is running on the DX-120, you can create an integrator Method for peak identification, calibration, and reporting. Time Functions, defined in the Method, provide control of DX-120 and AS40 Automated Sampler functions.

Refer to Section 3, “Operation Overview,” and Section 8, “Time Functions,” in the 4400/4600 manual for details about using DIALOG and Time Functions.

C.4.1 Time Functions

When a DX-120 Automation Cable is installed, integrator Time Function (TFN) commands are assigned to the following DX-120/AS40 functions:

Time Function (TFN) Command	Value	DX-120/AS40 Function
T3	0	AS40 Sampler Ready
T3	1	AS40 Sampler Start Load
T4	0	Detector Range Normal
T4	1	Detector Range 10X
T5	0	Auto Offset Off
T5	1	Auto Offset On
T6	0	Injection Valve Load
T6	1	Injection Valve Inject
T7	0	Pump Off
T7	1	Pump On

At power-up, the Time Function values are 0. This is appropriate for functions T3 through T6. Before running a Method, the T7 Time Function, which controls the pump, must be set to 1 to turn on the pump. Press the **TFN** key on the integrator and enter **T7=1**.

C.4.2 Creating a Method

Use **DIALOG** to create a Method file that includes Time Functions for controlling the operation of the DX-120, the AS40, and the integrator. The Method must also include the Method number, a component table, and a sample table.

The Method number determines which calculation method the integrator uses to convert peak areas to concentrations. For most IC applications, a multilevel calibration for each analyte is appropriate. Calibration should also be performed with multiple injections at each calibration level.

1. Press **DIALOG** to begin entering function parameters into the data table. After keying in the response to each **DIALOG** query, press **ENTER**.

Figure C-1 illustrates the setup of a Method for the analysis of seven anions. The run is 10 minutes long. The analytes are calibrated at three different concentrations, with two replicate injections at each level. Figure C-2 illustrates the Method timed events.

The Time Functions for the example are shown below:

Time	Function	Value	Comment
TT= 0.01	TF=" T3	TV= 1	Start AS40 Load cycle
TT= 0.02	TF=" T3	TV= 0	Toggle the Load relay off
TT= 1.40	TF=" T5	TV= 0	Turn off Auto Offset
TT= 1.42	TF=" T5	TV= 1	Reset Auto Offset
TT= 1.50	TF=" T6	TV= 1	Injection valve set to Inject position
TT= 11.50	TF=" T6	TV= 0	Injection valve set to Load position
TT= 11.50	TF=" ER	TV= 1	End run
TT= 12.00	TF=" SR	TV= 1	Start next run

To delete a Time Function while in **DIALOG**, enter the TFN time preceded by a negative sign, and then re-enter the time function and value.

To list the completed Method file, press **PR FILE**.

<pre> FI = 1. FE = 1. MN = 0. PRESS 'ENTER' TO SKIP ENTRY ENABLE BASELINE DRAWING? [Y/N] (N) STORAGE MENU? [Y/N] (N) FUNCTION NUMBER [0-10] (0) FILE NAME () = " ANIONS TIME FUNCTION VALUE TT= 0.01 TF=" T3 TV=1 TT= 0.02 TF=" T3 TV=0 TT= 1.40 TF=" T5 TV=0 TT= 1.41 TF=" T5 TV=1 TT= 1.50 TF=" T6 TV=1 TT= 11.50 TF=" T6 TV=0 TT= 11.50 TE=" ER TV=1 TT= 12.00 TE=" SR TV=1 TT= METHOD NUMBER [0, 1, 2, 5] (0) MN=5 IF NV=0 THEN NO CALIB IF NV=1 THEN NORMAL CALIB IF NV>1 THEN MULTI-LEVEL CALIB NUMBER OF LEVELS [0-26] (0.) NV= 3 INJECTIONS/LEVEL (1) = 2 TOTAL CALIB INJECTIONS, RC= 6 LINEAR[1] OR NONLINEAR[2] () LS= 1 COMPONENT TABLE... RET TIME NAME RT= 1.30 CN=" F RT= 1.70 CN=" Cl RT= 2.00 CN=" NO2 RT= 2.50 CN=" Br RT= 3.40 CN=" NO3 RT= 7.90 CN=" SO4 RT= 8.80 CN=" HPO4 RT= EXPECTED CONC [Y/N] (N) RRT REF PEAK: RP(1) = 1.7 RRT REF PEAK: RP(1) = MULTI LEVEL CALIB VALUES: RETENTION TIMES: RT(1) = 1.3 (2) = 1.7 (3) = 2 CONCENTRATIONS: VA(1) = 1 VA(2) = 2 VA(3) = 2 VB(1) = 2.5 VA(2) = 5 VA(3) = 5 VC(1) = 5 VA(2) = 10 VA(3) = 10 RETENTION TIMES: RT(4) = 2.5 (5) = 3.4 (6) = 3.8 CONCENTRATIONS: VA(4) = 2 VA(5) = 2 VA(6) = 2 VB(4) = 5 VA(5) = 5 VA(6) = 5 VC(4) = 10 VA(5) = 10 VA(6) = 10 RETENTION TIMES: RT(7) = 7.9 (7) = 8.8 CONCENTRATIONS: VA(7) = 2 VA(8) = 2 VB(7) = 5 VA(8) = 5 VC(7) = 10 VA(8) = 10 SAMPLE TABLE... ANALYST () AN=" DI ONEX INJECTIONS/ SAMPLE [0-254] (0.) RA= SAMPLES BETWEEN CALIB [0-254] (0.) C1= CONC UNITS () CU=" PPM SAMPLE NAME SAM AMT SCALE SI = END OF DIALOG </pre>	<p>① Enter Time Functions</p> <p>② Enter the Method number</p> <p>③ Enter the retention time and name of each component</p> <p>④ Enter the retention time of a reference peak</p> <p>⑤ Enter the concentrations for the calibration levels for each component. For F, enter 1, 2.5, and 5. For all others, enter 2, 5, and 10.</p>
--	--

Figure C-1. DIALOG Entry Example A

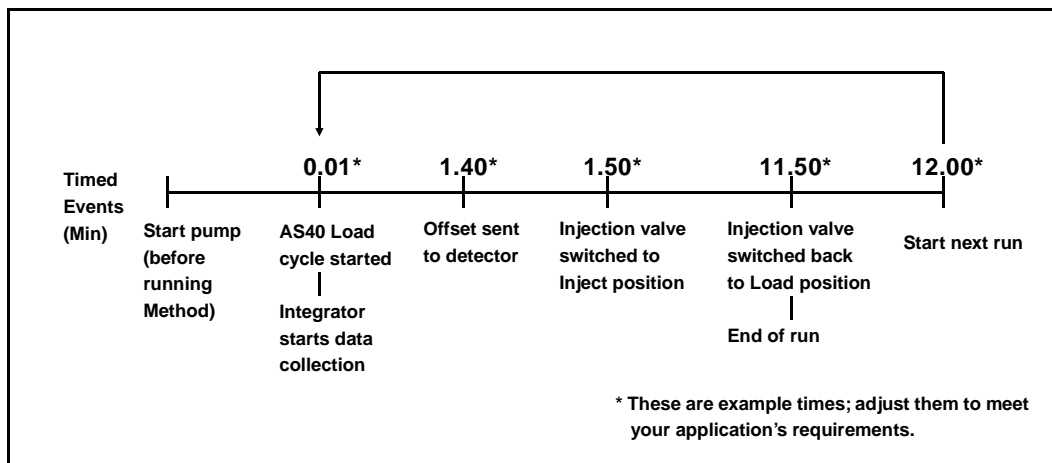


Figure C-2. DX-120 and Integrator Timed Events Example

2. Before running the Method, the T7 Time Function, which controls the pump, must be set to 1 to turn on the pump. If you have not already done so, press the **TFN** key on the integrator and enter **T7=1**.
3. Set the AS40 to **Lcl** control.
4. Set the DX-120 to **REMOTE** control.
5. When you are ready to start the calibration run, press **CALIB** and enter **1**. Start the first run by pressing the **INJ/END A** key on the integrator. Thereafter, the Method will continue automatically until you stop the integrator by again pressing the **INJ/END A** key.

This example provides limited automation with no status monitoring of the DX-120 or autosampler. For fully-automated operation, write a BASIC program (see Section C.5).

C.5 BASIC Programming

The BASIC program shown in Figures C-3 and C-4 can be used with a Method program in DIALOG. This program:

- Monitors the DX-120 and autosampler to see if they are ready to operate
- Monitors the DX-120 for the occurrence of a pressure alarm
- Prompts you in setting up the system to run a set of samples

Refer to the 4400/4600 user's guide for detailed information on BASIC programming.

To set up and run the program:

1. Type the program from the keyboard. To edit a line, retype the line and press **ENTER**. To delete a line, type the line number and press **ENTER**.
2. To print the program, type **LIST** and press **ENTER**.
3. To start the program, type **RUN** and press **ENTER**.

IMPORTANT

The program is erased from memory when the integrator power is turned off.

```
10 REM DX-120 AUTOMATION PROGRAM #1
0 REM VERSION 1.00 13 MAR 96
30 REM PROMPTING FOR SYSTEM SETUP
32 !!"CONFIRM THE AUTOMATION CABLE IS CONNECTED TO THE INTEGRATOR,"
34 !!"THE DX-120, AND THE AUTOSAMPLER."
36 REM "Aa" IS A NONSENSE VARIABLE TO ALLOW THE PROGRAM TO PAUSE
38 INPUT "PRESS 'ENTER' WHEN READY ", A
40 !!"PLACE THE DX-120 INTO 'LOCAL CONTROL',"
42 !!"START THE PUMP AND ALLOW THE SYSTEM TO EQUILIBRATE."
44 INPUT "PRESS 'ENTER' WHEN EQUILIBRATED." ,Aa
50 REM INITIALIZING VARIABLES
52 St=0 REM St = TOTAL # OF SAMPLES TO BE RUN
54 Sr=0 REM Sr = # OF SAMPLES WHICH HAVE RUN
60 TFN"T3",0 REM AUTOSAMPLER PROCEED/START LOAD
62 TFN"T4",0 REM RANGE NORMAL/*10
64 TFN"T5",0 REM AUTO OFFSET OFF/ON
66 TFN"T6",0 REM INJECTION VALVE LOAD/INJECT
68 TFN"T7",1 REM PUMP OFF/ON
70 TFN"T8",0 REM UNUSED TFN
80 !!"LOAD SAMPLES AND STANDARDS INTO THE AUTOSAMPLER AND SET TO 'RUN'."
82 INPUT "PRESS 'ENTER' WHEN READY.", Aa
90 !INPUT "WHAT IS THE TOTAL NUMBER OF INJECTIONS TO BE MADE ?",St
92 IF St>0 THEN 100
94 !"NO TOTAL WAS ENTERED. TO TERMINATE PROGRAM, PRESS 'ESC',"
96 GOTO 90
100 !!"PLACE THE DX-120 INTO 'RELAY CONTROL'."
102 INPUT "PRESS 'ENTER' WHEN READY.",Aa
110 !!"THE SYSTEM IS READY TO BEGIN."
112 INPUT "TO START THE FIRST INJECTION PRESS 'ENTER'." ,Aa
120 REM # OF SAMPLES RUN COMPARED TO TOTAL # OF SAMPLES
122 IF Sr=St THEN 212 REM DIALOG TFN"GO" COMMAND MUST COME TO THIS LINE
130 REM PRESSURE ALARM IS CHECKED
132 TFN"XD": XD(1)=PEEK#4C1F8
134 IF XD(1)=4 THEN 202
140 REM AUTOSAMPLER STATUS IS CHECKED
142 TFN"XD": XD(1)=PEEK#1C1F8
144 IF XD(1)=0 THEN 142
150 REM AUTOSAMPLER LOAD COMMAND GIVEN
152 TFN"T3",1
154 TFN"T3",0
160 REM INTEGRATOR WAITS FOR "SAMPLE READY" FROM AUTOSAMPLER
162 TFN"XD": XD(1)=PEEK#3C1F8
164 IF XD(1)<3 THEN 162
166 IF XD(1)=3 THEN 172
170 REM SAMPLE # INCREMENTED AND INJECTION COMMAND GIVEN
172 Sr=Sr+1
174 !!"INJECTION SAMPLE";Sr
```

Figure C-3. Integrator BASIC Program for the DX-120

```

176 INJECT A
200 REM PRESSURE ALARM STATEMENT
202 !"PRESSURE ALARM DETECTED AFTER INJECTION OF SAMPLE";Sr
204 !"LOCATE THE CAUSE, CORRECT, AND TYPE ''RUN'' TO RESTART THE PROGRAM."
206 GOTO 220
210 REM NOTICE OF RUN OR SCHEDULE FINISH
212 !"SCHEDULE FINISHED. TYPE ''RUN'' TO RESTART."
214 TFN"T7",0 REM PUMP OFF
220 END

```

Figure C-4. Integrator BASIC Program for the DX-120(Continued)

Figure C-5 shows the Time Functions for an example DIALOG Method, which runs with the BASIC program. The following main modifications were made to Example A:

- Example Method B does not include any commands to the autosampler; these are included in the BASIC program.
- A GO command occurs after the ER command. This replaces the SR command in Example A, and tells the integrator to go to line 122 (you must use this line number) to start the next run.
- A PT (peak threshold) Time Function is included to set peak threshold to a certain value.

```

FI= 1. FE= 1. MN= 0.
PRESS 'ENTER' TO SKIP ENTRY
ENABLE BASELINE DRAWING? [Y/N] (N)
STORAGE MENU? [Y/N] (N)

FUNCTION NUMBER [0-10] (0)

FILE NAME ( ) = " ANIONS WITH BASIC
TIME          FUNCTION      VALUE
TT= 0.01      TF=" PT       TV= 10000
TT= 0.01      TF=" T5       TV= 0
TT= 0.02      TF=" T5       TV= 1
TT= 0.10      TF=" T6       TV= 1
TT= 10.10     TF=" T6       TV= 0
TT= 10.10     TE=" ER       TV= 1
TT= 10.60     TE=" GO       TV= 122
TT=

```

Figure C-5. DIALOG Entry Example B

D • Conductivity Detection

Ions in solution conduct electrical current when voltage is applied between electrodes contacting the solution. Since the magnitude of this current is nearly proportional to the concentration of dissolved ions, conductivity detection is useful for quantifying ionic analytes.

D.1 How Conductivity Is Measured

The conductivity of a solution is measured by applying an alternating voltage between two electrodes in a conductivity cell. At any instant in time, negatively charged anions migrate toward the positive electrode and positively charged cations migrate toward the negative electrode. Since the detector applies a known voltage to the cell electrodes and the current is measured, the solution resistance, R , is calculated from *Ohm's law*:

$$R = \frac{E}{i}$$

The inverse of the measured solution resistance is the *conductance*, G , measured in siemens (S).

$$G = \frac{1}{R} = \frac{i}{E}$$

The measured conductance is corrected by the conductivity cell constant, K . The conductivity of the solution is the conductance which would be measured in a standard cell containing electrodes of 1 cm² surface area held 1 cm apart. This quantity is the conductivity, k . The units of conductivity are siemens per cm (S/cm).

$$\kappa = K \bullet G$$

D.2 Conductivity of Solutions

According to Kohlraush's law of independent migration, conductivity is directly proportional to concentration (that is, the conductivity of a dilute solution is the sum of the individual contributions to conductivity of all the ions in the solution multiplied by their concentration). Kohlraush's law further states that each ion carries its portion of the total conductivity without being affected by any of the other ions in solution.

Stated as an equation:

$$\kappa = \frac{\sum_i \lambda_i^o c_i}{1000}$$

Where:

κ is the measured conductivity in S/cm

c_i is the concentration of the ions in equivalents/L

(Equivalents/L equals moles/L times the charge on the ion.)

The *ionic limiting equivalent conductivity*, λ_i^o , is specific for each ion. It is the conductivity of the ion divided by the concentration and extrapolated to infinite dilution. Table D-1 lists limiting equivalent conductivities for a number of organic and inorganic ions. The unit for λ_i^o is S·cm²/equivalent.

Anions	λ_i^o	Cations	λ_i^o
OH ⁻	198	H ⁺	350
F ⁻	54	Li ⁺	39
Cl ⁻	76	Na ⁺	50
Br ⁻	78	K ⁺	74
I ⁻	77	NH ₄ ⁺	73
NO ₃ ⁻	71	Mg ²⁺	53
HCO ₃ ⁻	45	Ca ²⁺	60
SO ₄ ²⁻	80	Sr ²⁺	59
Acetate	41	CH ₃ NH ₃ ⁺	58
Benzoate	32	N(CH ₃ CH ₂) ₄ ⁺	33

Table D-1. Limiting Equivalent Conductivities at 25 °C

Values of λ_i^o from Table D-1 can be used to calculate conductivities of solutions containing ions. For example, the limiting equivalent conductivity for NaCl at 25 °C is 126.5. This is the sum of the ionic limiting equivalent conductivity for Na⁺, which is 50.1, plus that of Cl⁻, which is 76.4. A 0.1 mM solution of NaCl at 25 °C has a conductivity of 0.1×126.5 , or 12.65 μ S/cm.

So far, only dilute solutions have been discussed. As concentration increases, the direct proportionality between conductivity and concentration is lost. However, at the analyte concentrations normally encountered in ion chromatography (below 1 mM), conductivity is generally proportional to concentration. For example, the equivalent conductivity at 25 °C of KCl at infinite dilution is 149.9; at 1 mM, it is 146.9, a decrease of only 2%.

However, the conductivity of an eluting analyte is not necessarily directly proportional to concentration, because ionic components of the eluent may be contained in the eluting volume.

If the electrolyte is a weak electrolyte such as an acid or base with only partial dissociation, then c_i must be replaced by the concentration of the dissociated ions only, since only they contribute

to conductivity. For acids and bases, the pK values and the solution pH can be used to calculate the extent of dissociation.

D.2.1 Effect of Hydration Sphere and Solvent on Conductivity

The limiting equivalent conductivity of an ion, λ_i^o , is a measure of ionic mobility. Ionic mobility is greatly affected by the properties of the ion in the solvent. Ions with large hydration spheres are less mobile, and therefore less conductive, than ions with small hydration spheres. This explains why λ_i^o for extensively hydrated fluoride (55.4) is lower than λ_i^o for chloride (76.4), which is less hydrated. Solvent viscosity also affects ionic mobility; ions are more mobile in solvents of lower viscosity.

It is not necessary to know values such as hydration sphere and viscosity, since quantitative analysis is performed by comparing the conductivity of the analyte in the sample to the conductivity of the same analyte in a standard (or standards). Even if a solvent gradient is used, the composition of the solvent during the elution of the analyte will be the same in both the sample and the standard.

D.2.2 Effect of Temperature on Conductivity

Ionic mobility, and therefore conductivity, is greatly affected by temperature. The conductivity of an aqueous solution has been found experimentally to rise a little less than 2% per °C. Thus, it is necessary to hold the eluent temperature as constant as possible to maintain a stable baseline.

When the cell is housed in a DS4 Detection Stabilizer, the DS4 maintains the cell at a set temperature. In addition, the detector corrects the measured conductivity to that which would be measured at 25 °C. It does this by measuring the cell temperature with a thermistor and multiplying the conductivity by a temperature-dependent constant. This

constant, the *temperature compensation factor*, is expressed in units of $\%/^{\circ}\text{C}$.

When operating with a standard cell (without a DS4), setting an accurate temperature compensation factor helps minimize the baseline drift caused by fluctuations in ambient temperature. See Section 3.6 for instructions on optimizing the temperature compensation.

D.2.3 Species Detected by Conductivity

Conductivity detection is typically selected for species that are ionic when they enter the detector cell, especially those with weak UV absorbance. This includes both organic and inorganic ions. Conductivity detection is best suited to anions and cations of strong acids and bases, such as chloride, sulfate, trifluoroacetate, sodium, and potassium. Ions of weaker acids are detected, provided that the eluent pH is chosen to maximize analyte dissociation. (When a suppressor is used, the eluent pH which determines whether an ion is detected is the pH after suppression.)

Anions

Sensitivity is best for anions with pKa values below 6. As analyte ionization (dissociation) decreases, so does sensitivity. Anions with a pKa above 7 can be detected under certain conditions, but signal-to-noise ratios are generally poorer. All organic acids with either carboxylate, sulfonate, or phosphonate functional groups have pKa's below 4.75, so conductivity is the preferred detection method for these species. Common inorganic strong acid anions include chloride, nitrate, phosphate, and sulfate.

Cations

Inorganic cations detected include the alkali metals and alkaline earths. Most transition metal cations are not detected, since they either hydrolyze water to form anions or

precipitate in the suppressor. These metals are usually detected by visible wavelength absorbance following postcolumn reaction with a chelating indicator.

Nearly all organic cations are amines. Aliphatic amines have pKa's around 10 and are easily detected. Aromatic and heterocyclic amines have pKa's between 2 and 7, too low to be detected by suppressed conductivity following cation exchange separation. Although nonsuppressed (single-column) detection can be used for these species, sensitivity is generally poor. These amines are best detected by UV absorbance or by amperometric techniques.

D.2.4 Chemical Suppression

Species detected by conductivity are by their nature ionic, so ion-exchange and ion-pair chromatography are by far the most commonly used separation methods. These methods require eluents containing strong electrolytes, which presents a problem: how to detect the ionic analytes without the detector being overwhelmed by the ions in the eluent. The best solution is to neutralize the eluent in a suppressor.

Figure D-1 illustrates the suppression mechanism that occurs inside a Dionex Anion Self-Regenerating Suppressor (ASRS) when NaOH is the eluent for anion-exchange separation.

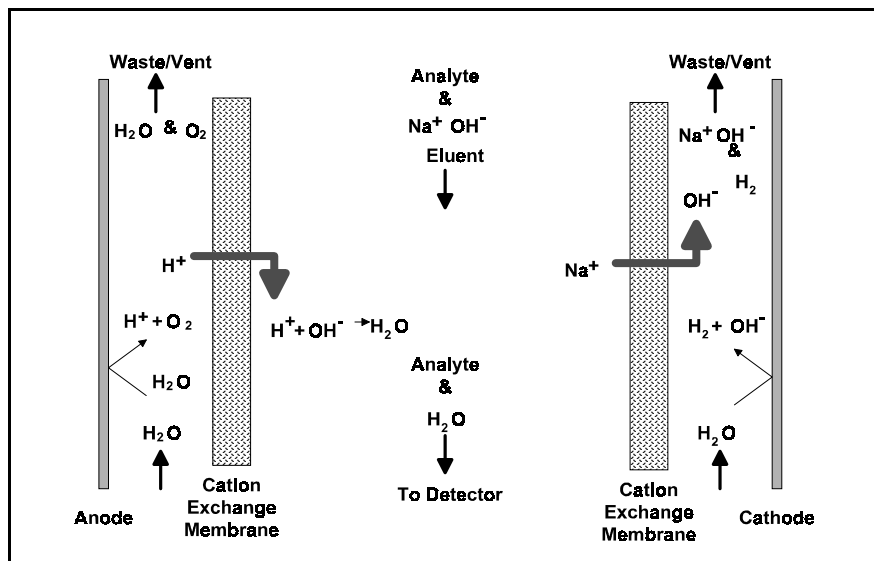


Figure D-1. ASRS Suppression Mechanism

Analyte anions elute from the column with sodium counterions. Two electrodes, one beside each membrane (on the side opposite the eluent) hydrolyze water to hydrogen and hydroxide ions. Hydrogen ions diffuse across the membrane next to the anode, neutralizing the eluent hydroxide to water, while sodium ions from the eluent diffuse across the other membrane, providing counterions to the hydroxide being generated at the cathode.

In effect, sodium hydroxide from the eluent is transferred across the membrane and does not reach the detector. The resulting eluent background conductivity is near zero, considerably lower than before suppression. Also, the counterion to the anion analytes is now a hydrogen ion with a conductivity seven times higher than the original sodium counterion. Since both the anion analyte and the cation counterion produce the detector response, response is increased. The suppressor lowers the background conductivity (and thus the baseline noise and drift) and increases the signal. Suppression can also be accomplished without water

electrolysis by pumping a dilute sulfuric acid solution (the regenerant) through the suppressor on the side of the membranes opposite the eluent.

For ion chromatography of cations, the suppressor membranes are anion exchange polymers that allow anions to pass freely but exclude cations. The eluent uses dilute acids such as methanesulfonic acid. In the Dionex Cation Self-Regenerating Suppressor (CSRS), methanesulfonate counterions are replaced by hydroxide generated by the electrolysis of water. This neutralizes the acidic eluent and provides the highly conductive hydroxide counterion to the analyte cations.

Use of a suppressor typically increases signal-to-noise ratios about one order of magnitude for strong acid or base ions. The improvement is somewhat less for ions of weak acids or bases, due to decreased ionization at the neutral pH of the eluent after suppression. Nonetheless, the benefits from reducing the background, and therefore the noise, almost always yield a net improvement in signal-to-noise ratio compared to nonsuppressed conductivity detection.

Suppressors provide several important advantages, listed below. The first three are a direct result of the increased signal-to-noise ratio.

- Lower detection limits
- Longer column life, due to a possible increase in the dilution of dirty samples
- Wider dynamic range
- Greater range of elution control and larger sample concentrations or volumes, due to more highly concentrated eluents
- Gradient elution capability
- Faster equilibration time

- Elimination of interference from counterions
- Elimination of system peaks

D.2.5 Eluents for Conductivity Detection

When choosing an eluent, the constraints imposed by the separation and detection methods must be considered. The most important criteria for obtaining optimal separation are the elution strength of the eluent, the separation efficiency, and the resolution of the analytes of interest. For conductivity detection, the criteria for a good eluent are the relative conductivity response of the analytes and the magnitude of the background.

Eluents can be wholly aqueous, containing only water and a strong electrolyte. Or, if an organic solvent-compatible column such as a Dionex OmniPac® or MPIC® column is used, typical reversed-phase solvents such as methanol or acetonitrile can be used. Organic solvents are essential components of ion-pair eluents and provide important selectivity control during ion-exchange separations. Since these solvents are nonconducting, they do not interfere with conductivity detection.

Anion-Exchange

When a suppressor is used, the ionic components of the eluent must be such that they are removed, or else converted to weakly conducting compounds by the suppressor. Sodium salts of weak acids are used because they are converted to the neutral free-acid form in the suppressor. The higher the pKa of the acid, the lower the background conductivity following suppression. Weak acids with a pKa above 6 can be used for isocratic separations. For gradient elution, pKa's should be above 8 to minimize baseline shift during the gradient.

Sodium hydroxide solutions make excellent eluents for anion-exchange chromatography because hydroxide is neutralized in the suppressor to water (the free acid form of

hydroxide). Another common eluent is a carbonate/bicarbonate buffer suppressed to carbonic acid ($pK_a = 6.2$). Carbonate/bicarbonate buffers are easily prepared and are routinely used for isocratic separations of inorganic anions.

Other eluents for suppressed anion chromatography include the sodium salts of boric acid (borax, tetraborate, $pK_a = 9.2$) and p-cyanophenol ($pK_a = 8.0$). Borate forms weak bonds with hydroxy-organic acids, producing changes in selectivity compared to hydroxide. p-Cyanophenol is a powerful monovalent displacer, useful for eluting strongly retained hydrophobic monovalent anions such as fumaric acid, iodide, and thiocyanate.

Cation-Exchange

Millimolar concentrations of dilute strong acids, often mixed with organic solvents, are good choices for cation exchange chromatography of both monovalent and divalent cations. Methanesulfonic acid (MSA) is commonly used, since it is compatible with the CSRS.

Some columns require a stronger displacing ion than hydrogen ion to elute more strongly retained divalent cations. The zwitterion 2,3-diaminopropionic acid (DAP), mixed with a strong acid to protonate the DAP, is a good choice.

The best eluents for nonsuppressed cation IC are dilute strong acids such as 1 mM nitric acid. Backgrounds are very high (around 1 μS), so signal-to-noise ratios are not good enough for low-level work. Since the conductivity of the hydrogen ion is greater than that of all other cations, elution of analytes causes decreases in conductivity. Instead of peaks, eluting analytes produce dips.

Ion-Pair

Eluents for ion-pair chromatography contain mixes of aqueous and organic solvent solutions with hydrophobic ion-pair reagents as additives. Commonly used reagents are

quaternary ammonium salts for anion separations and long-chain sulfonates for cation separations. For suppressed conductivity detection, these reagents are easily used provided the counterions are either hydroxide (anion-ion pair) or hydrogen ion (cation-ion pair). These are marketed by Dionex as IonSep® reagents. The reagents are purified solutions of quaternary ammonium hydroxide solutions and sulfonic acid solutions.

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 (κ).

$$k = \kappa / G$$

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 (α)

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

$$\alpha = (t_2 - t_0)/(t_1 - t_0)$$

Where: t_1 and t_2 = retention time of components 1 and 2, respectively

t_0 = retention time of unretained components (void volume)

Concentrator Column

A short column used to retain and concentrate analytes from a measured volume of relatively clean sample. This allows large volumes of sample to be injected, lowering concentration detection limits.

Conductivity

A measure of the ease with which electrical current flows through a liquid contained between two opposite charged electrodes.

Conductivity is a characteristic of ions in solution. Units are siemens.

Counterion

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

% Crosslink

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

Equivalent Conductivity (λ)

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

Guard Column

A small column which 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 Equivalent to a Theoretical Plate. A measure of column efficiency which allows comparison between columns of different lengths.

$$\text{HETP} = H = L/N$$

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, most often 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 which 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_2 = 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 °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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