



Post Column Reactor

- Dual Channel -

Operator's Manual

- Preliminary -

WELCOME

The SSI/Lab Alliance's Sensivate brings you a new level of post-column performance and size. Sensivate post-column reactor features a compact footprint and independent reagent control. The reactor units are disposable, and heaters allow reactions at temperatures as high as 150°C. All connections are accessible from the front panel and the system offers the ability to set upper and lower pressure and temperature limits. Based on proven pump technology combined with a unique reactor design and disposable heating reactors, you can now achieve post-column derivatization with great performance.

USING THIS MANUAL

This manual provides information and instruction needed to install, operate, and perform maintenance on the Post Column Reactor.

GETTING TO KNOW YOUR POST-COLUMN REACTOR

This section provides basic information about your Post-Column Reactor. Read this section for information on:

- Typical Post Column Applications
- Location of Components and Connectors
- Sensivate Configurations
- Available Accessories

These diagrams are typical post-column configurations. Your system may or may not appear the same.

TYPICAL POST COLUMN APPLICATIONS

Applications	Reagent A	Heated Reactor	Temp, °C	Reagent B	Ambient Reactor	Detection	Part Number
Carbamates	NaOH	500uL	100	OPA	150uL	Fluorescence	PCR2-R050-R015
Glyphosates	NaOCl	500uL	40	OPA	150uL	Fluorescence	PCR2-R050-R015
Aflatoxins	Iodine	1.44mL	40			Fluorescence	PCR1-R144
Carbohydrates	NaOH	500uL	40			PAD	PCR1-R050
Lipids	phenacyl	700uL	100			UV/Visible	PCR1-R070
Amino Acids by Ninhydrin	Ninhydrin	500uL	120-140			UV/Visible	PCR1-R050
Amino Acids by OPA	NaOCl	500uL	40	OPA	150uL	Fluorescence	PCR2-R050-R015
Amino Acids, Primary only	OPA	150uL	30			Fluorescence	PCR1-R015
Chondroitin Sulfate	2-Cyanoacetamide/NaOH	1.44mL	120		500uL	Fluorescence	PCR2-R144-R050

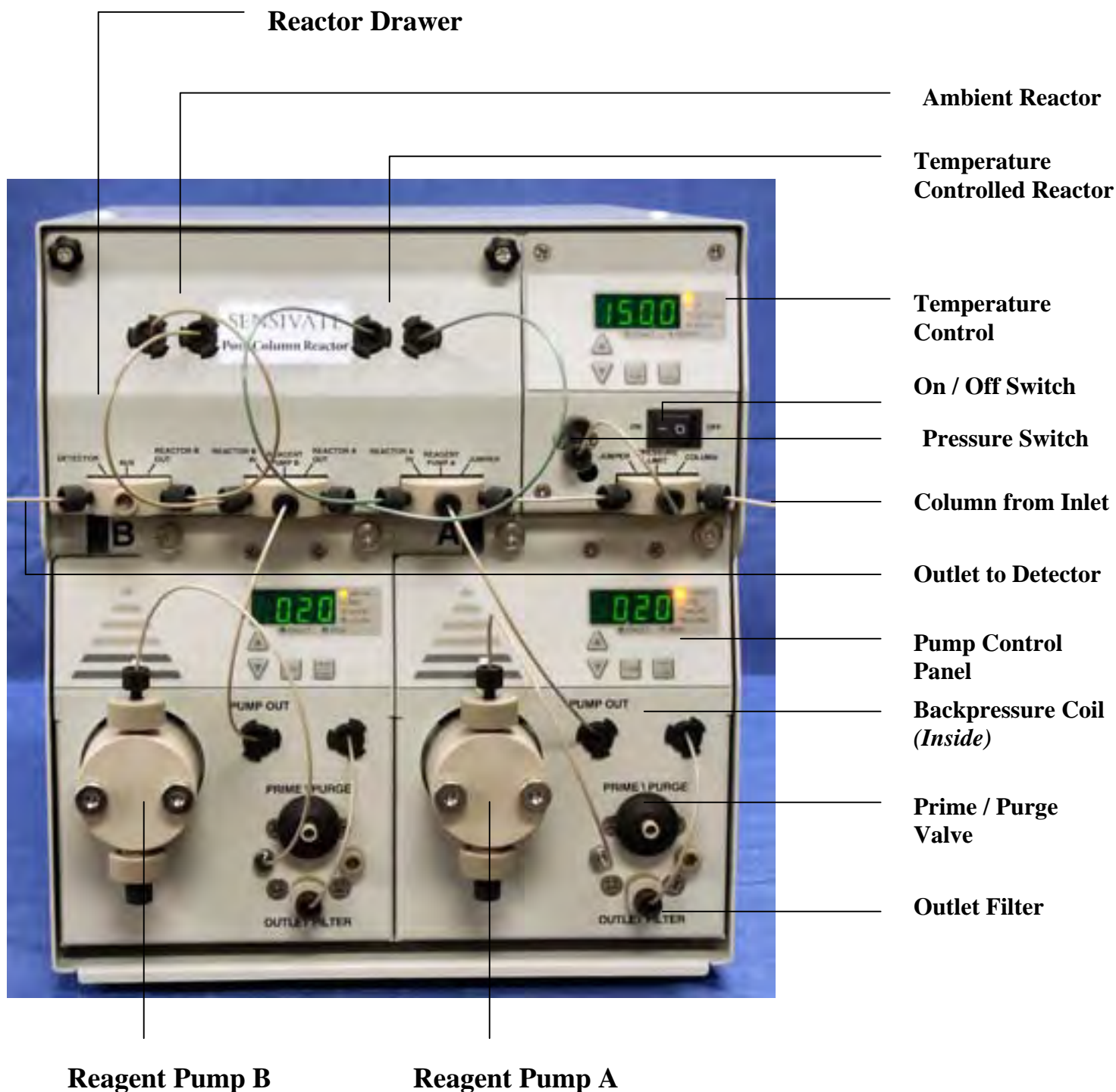
OPA=ortho-phthaldehyde

PAD=pulsed amperometric

This diagram shows the general recommendations for the Post Column Systems. Your system may be configured differently in order to meet specific application requirements.

SENSIVATE FRONT

CONFIGURATION
DUAL REAGENT
DUAL REACTOR
PCR2



Component**Description**

Reagent Pumps

Pump Control Panel	Set reagent flow rates, monitor pressure and set pressure limits.
Backpressure Coil (<i>Inside</i>)	The Backpressure Coil is a coil of .005 ID PEEK tubing to produce backpressure on pumps.
Prime / Purge Valve	Connect a Syringe here to Prime the pump.
Outlet Filter	The Outlet Filter port contains a high pressure 0.5 micron filter and is designed for a 1/16" OD tubing connection.

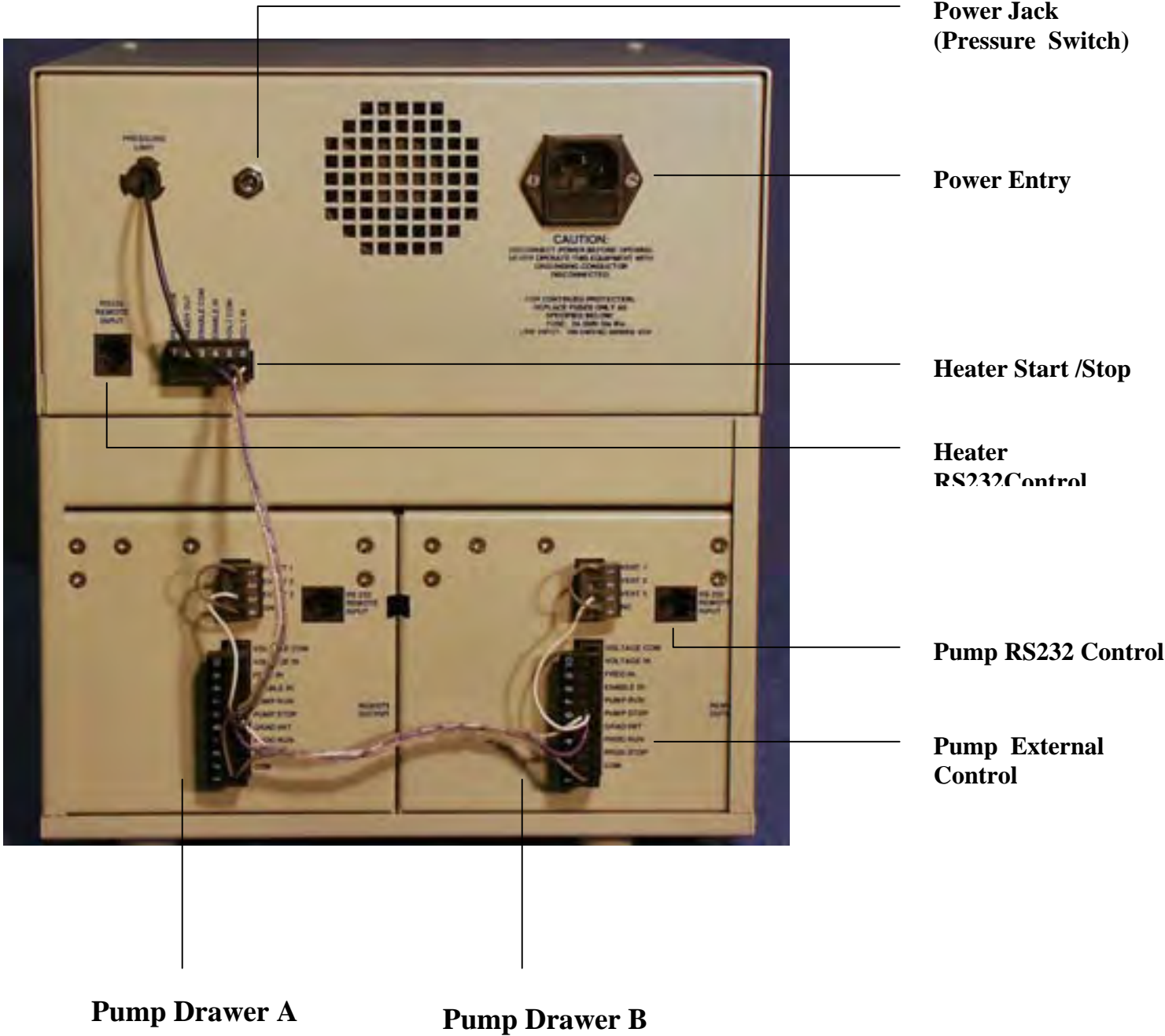
Reactor Drawer

Ambient Reactor	Ambient Disposable Reactor Unit
Temperature Controlled Reactor	Temperature Controlled Disposable Reactor Unit (30 to 150°C)

Temperature Control Panel	Set and monitor heated reactor temperature.
On / Off Switch	Press this button to turn the power to the PCR on or off.
Column from Inlet	Connect column outlet line into this port.
Outlet to Detector	Connect detector inlet line into this port.

SENSIVATE REAR PANEL

CONFIGURATION
DUAL REAGENT
DUAL REACTOR
PCR2



SENSIVATE REAR PANEL

CONFIGURATION

Component

Description

Power Jack	Plug the Pressure Control Switch into this jack.
Power Entry	Plug power cable into this connector.
Heater Start /Stop	6-PIN Connector: Connect the appropriate wiring for heater Start and Stop functions.
Heater RS232 Control	Connect an RS232 cable into this port for PC heater control functions.
Pump RS232 Control	Connect an RS232 cable into this port for PC pump control functions.
Pump External Control	A 10-PIN Connector: Connect the appropriate wiring for pump input functions.

SENSIVATE

OTHER CONFIGURATIONS



PCR 1 SINGLE REAGENT

Single Reactor



PCR 1 SINGLE REAGENT

Dual Reactor



PCR 0 SINGLE REACTOR



PCR 0 DUAL REACTOR



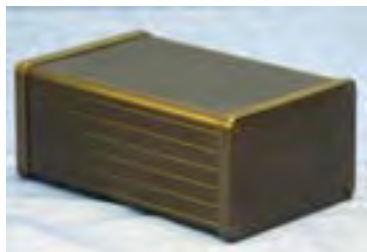
ACCESSORIES

SSI/Lab Alliance offers various accessories that can help you make the most of using your Post-Column System. To order accessories, visit www.laballiance.com.

Sparging System



The Sparging System can be mounted on either side of the PCR. Nitrogen or Helium gas is typically used with the Sparging system. The Sparging System contains a 0-5 PSI range Pressure Regulator, Pressure Gauge, Mobile Phase Caps with three-hole bottle cap with valves, NO-OX Tubing (for pump inlet), Teflon Tubing (between regulator and bottle caps), and the Reagent Tray. Bottles are not included. We recommend the use of Safety-Coated Glass Bottles that can be pressurized to 8PSI, and which are designed to accommodate caps containing GL-45 threads. To block out UV light, Kontes Safety-Coated Glass Bottles can be used. The customer connects to a 1/8 NPT port in the regulator. Recommended supply pressure is 30 - 200 PSI.



Pressure Control Switch

A Pressure Control Switch is installed to prevent back flushing of harmful reagent into the analytical column. The Pressure Control Switch allows the system to come up to pressure before the PCR's reagent pumps and heating units are made operable. The pumps and heater remain on standby until the system reaches operating pressure, then the pumps and heater are automatically turned on. The switch also shuts down the reagent pumps and heating unit if LC pressure drops while the system is running; the user manually resumes operation when system comes back to pressure. The Pressure Switch protects the system and column if the LC pumps shut down or if there is a leak. The Pressure Control Switch is placed between the LC Pump and the Injector (or Auto Sampler) to minimize delay volume.

Reagent Tray



The Reagent Tray can be mounted on either side, one of two height levels, or on top of the PCR for system flexibility. The tray will accommodate two 1L reagent bottles and a 250ml Boston Round bottle for self flush solvent.

1. INTRODUCTION

This manual provides information and instruction needed to install, operate, and perform maintenance on the Post Column Reactor.

1.1 Description of the Post Column Reactor

The system consists of two Series 1+ high performance metering pumps, a temperature controlled reaction coil and an ambient coil. Applications include general laboratory or industrial use. System features include:

- ▶ Fast & easy setup:
 - One inlet connection (from column)
 - One outlet connection (to detector)All connections accessible from front panel
- ▶ Modular:
 - 1 or 2 Reaction Coils (1 heated) – Disposable
 - 1 or 2 pumps
 - Single Reagent Systems field upgradable to Dual Reagent
- ▶ All-PEEK Fluid path, including pumps, valves and fittings
- ▶ Modular pump bays for easy replacement and maintenance
- ▶ Modular reaction coil bays for easy method change-over and replacement
- ▶ Self flushing pump heads for extended seal life and reduced maintenance
- ▶ Pulse Damper and back pressure device for reduced pulsation.
- ▶ Very high performance / price ratio
- ▶ Over-pressure system shut-down to protect upstream LC column
- ▶ Easily user adjustable process set points (flow rate, temp., etc.) via front key pads
- ▶ Digital readout of process parameters
- ▶ User settable upper/lower pressure and temperature limits
- ▶ RS-232 interface standard on pumps and reactor temperature controller
- ▶ “No-Ox” tubing used for Reagent Inlet lines (for amino acid, carbamate & glyphosate analysis)
- ▶ Compact size—Requires only 11 inches of bench space

The low pulsation flow produced by the reciprocating, single-piston pump is achieved by using an advanced rapid-refill cam design, programmed stepper motor acceleration, and an internal pulse damper with back pressure coil.

1.1.1 Pump Features

The Post Column Reactor Pumps include:

- Rapid refill mechanism to reduce pulsation
- PEEK™ pump head
- LED front panel readout of flowrate, pressure and upper/lower pressure limits
- PRIME mode to flush out entrapped air bubbles upon start-up
- Flow adjustment in 0.01 ml increments, from 0.01 to 0.30 ml/min with a precision of 0.5%
- Microprocessor advanced control
- Digital stepper motor design to prevent flow rate drift over time and temperature
- Back panel RS232 serial communications port for complete control and status
- Remote analog inputs to control flow rate (optional)

1.1.2 Wetted Pump Materials

Pump heads, check valve bodies, and tubing are made out of PEEK™. Other materials are synthetic ruby and sapphire (check valve internals and piston).

1.1.3 Self-Flushing Pump Head

Self-flushing pump heads provide continuous washing of the piston surface without the inconvenience of a manual flush or gravity feed arrangement. The self-flushing pump head uses a diaphragm and secondary set of check valves to create a continuous and positive flow in the area behind the high pressure pump seal. The flushing solution washes away any buffer salts that have precipitated onto the piston. If not removed, these precipitates can abrade the high pressure seal and cause premature seal failure, leakage, and can possibly damage the pump.

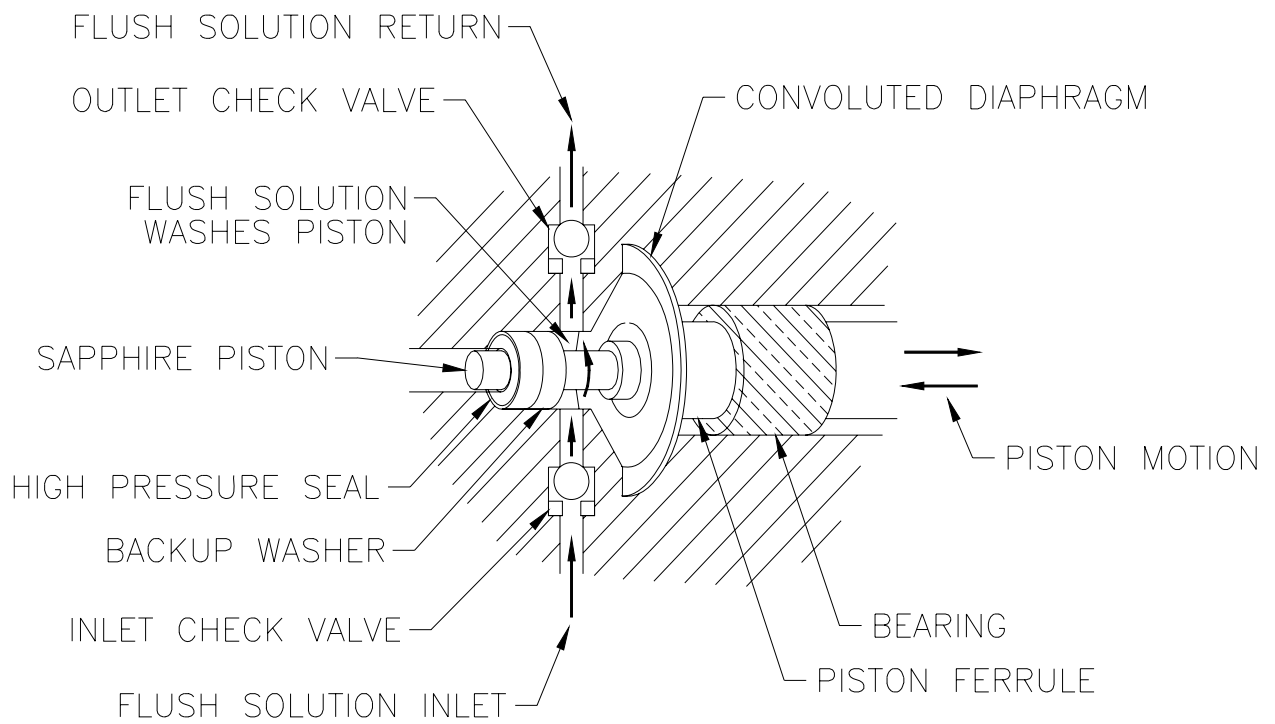


Figure 1-1. Self-Flushing Pump Head

1.1.4 Temperature Controlled Reaction Coil Features

- LED readout on the front panel—displays the setpoint and current temperature in °C or °F, has a HEATING light to indicate when the heating element is receiving power, and a FAULT light to indicate a fault condition has occurred.
- Tactile response, chemically resistant front panel keypad
- Microprocessor advanced control
- RTD temperature sensor for accuracy and stability
- PID control algorithm
- The Heated Reaction Coil can be controlled remotely with the RS-232 serial communications interface. The set point and current temperatures can be read and the set point temperature can be written. See chapter 3 for a complete list of commands.
- A thermal safety switch removes power from the heating element if the temperature reaches 160°C. This protects against damage to the heated coil.

1.1.5 Wetted Reaction Coil Materials

Wetted surfaces in the temperature controlled reaction coil are PEEK™.

1.2 Specifications for the Pump

- ▶ 0.01 – 0.30 ml/minute flow range (limited by firmware except when priming)
- ▶ 2,500 p.s.i. upper pressure limit (limited by firmware)
- ▶ All-PEEK fluid path, including pump heads
- ▶ Automatic Piston Wash (significantly improves seal life)
- ▶ Stepper motor drive, with electronic fast refill via flag and sensor
- ▶ Dual Check Valves (inlet & outlet) – Ruby Ball, Sapphire Seat
- ▶ Prime-Purge Valve - PEEK
- ▶ Pulse Damper (PEEK)
- ▶ Outlet Filter (0.5 μ UHMW)
- ▶ Back Pressure Coil for proper Pulse Damper operation (approx. 1,000 p.s.i. @ 1.0 ml/min)
- ▶ Pressure Transducer (isolated in Pulse Damper)
- ▶ Interactive front keypad with digital read-out:
 - Flow rate set points
 - Pressure read-out
 - Set upper/lower pressure limit
- ▶ RS-232 Interface for remote control / monitoring
- ▶ Pulsation: < 0.5%
- ▶ Flow Accuracy: $\pm 2\%$ from set point

1.3 Specifications for the Temperature Controlled Reaction Coil

- ▶ Continuous loop, fully sealed
- ▶ Multi-directional path for effective mixing
- ▶ Operating Range: 10° C above ambient to 150° C
- ▶ Temp. Accuracy: $\pm 2^{\circ}$ C over entire range (outlet fluid temperature vs. set point)
- ▶ Temp. Repeatability: $\pm 1^{\circ}$ C.
- ▶ Safety Cutoff Temperature: 160° C
- ▶ Stabilizing Time: 45 minutes (maximum) to 150° C for "Ready" indication
- ▶ Interactive front key pad control with digital read-out
 - Temperature set point
 - Temperature read-out (°C or °F)
 - Ready light
- ▶ RS-232 Interface for remote control and status monitoring

2. INSTALLATION

2.1 Unpacking and Inspection

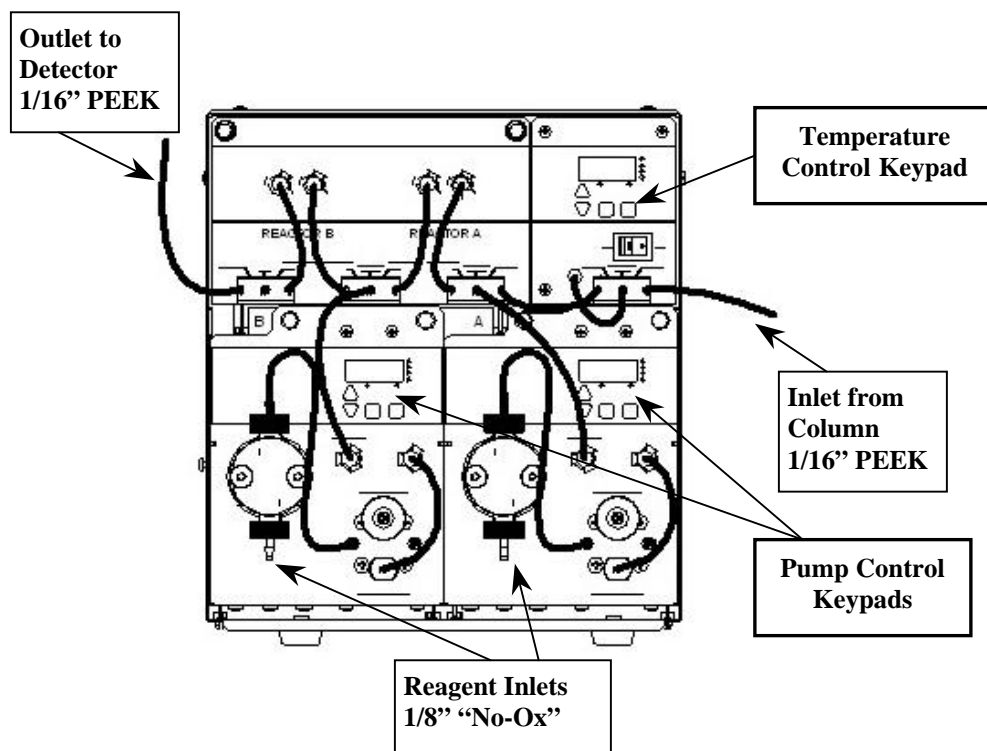
Prior to opening the shipping container, inspect it for damage or evidence of mishandling. If it has been damaged or mishandled, notify the carrier before opening the container. Once the container is opened, inspect the contents for damage. Any damage should be reported to the carrier immediately. Save the shipping container. Check the contents against the packing list.

2.2 Location/Environment

The preferred environment for the Post Column Reactor is normal laboratory conditions. The area should be clean and have a stable temperature and humidity. The specific temperature and humidity conditions are 10 to 30 °C and 20% to 90% relative humidity. The instrument should be located on a stable flat surface with surrounding space for ventilation and the necessary electrical and fluid connections.

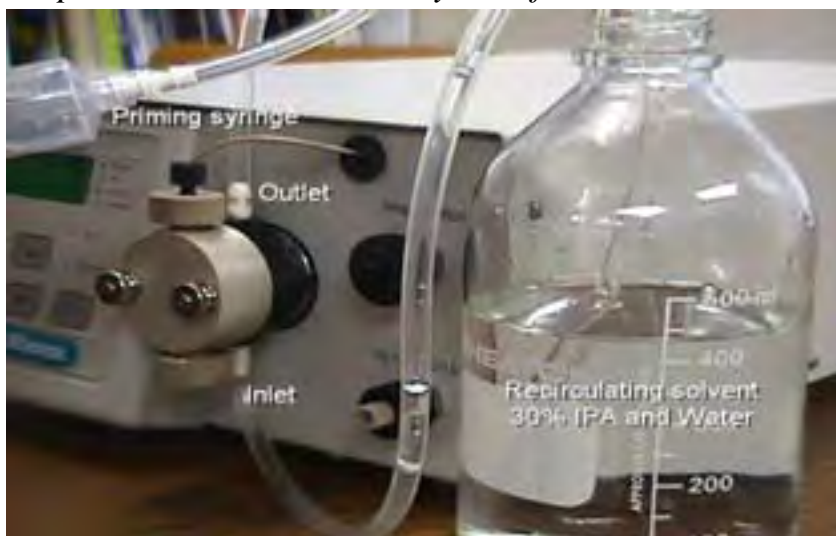
2.3 Fluid Connections

There are only four connections to be made. At the front mounted “T” connection blocks, connect the **Inlet from the Column** and the **Outlet to the Detector**, as shown below. Use standard PEEK tubing and 1/16” CPI fittings. Connect the two 1/8” “No-Ox” **Reagent Inlet Lines** (provided) to the pump inlets, as shown below.



Next, install the self flush. Connect solvent inlet and outlet (clear urethane) tubing as shown on the [similar pump](#) below (the PCR pump has a slightly different configuration). Prime self-flushing mechanism by drawing on syringe attached to outlet (pump does not need to run).

Replace wash solvent weekly with fresh solution.



Next, Prime the Pump, as shown on the [similar pump](#) below (the PCR pump has a slightly different configuration).

- Turn power on.
- Open prime purge valve by turning counterclockwise 2 complete turns
- Draw on syringe until approximately 15 mL of fluid comes through.
- Continue to draw on syringe until no bubbles are seen.
- Close prime purge valve.
- To aid in priming, the pump may be run by pressing the Run/Stop button. However, this is generally not required.



2.4 Electrical Connection

The system utilized Universal Switching Power Supplies, and will accept voltages from 90 – 240 VAC, 50-60 Hz.

WARNING: Do not bypass the safety ground connection as a serious shock hazard could result.

2.5 Solvent Preparation

Proper solvent preparation will prevent a great number of pumping problems. The most common problem is bubble formation, which may affect the flow rate consistency. Aside from leaky fittings, the problem of bubble formation arises from two sources: solvent out-gassing and cavitation. Filtration of HPLC solvents is also required.

2.5.1 Solvent Out-gassing and Sparging

Solvent out-gassing occurs because the mobile phase contains dissolved atmospheric gases, primarily N₂ and O₂. These dissolved gases may lead to bubble formation and should be removed by degassing the mobile phase before or during use. The best practical technique for degassing is to sparge the solvent with standard laboratory grade (99.9+%) helium. Helium is only sparingly soluble in HPLC solvents, so other gases dissolved in the solvent diffuse into the helium bubbles and are swept from the system. Solvent filtration is not an effective alternative to helium degassing.

It is recommended that you sparge the solvent vigorously for 10 to 15 minutes before using it. Then maintain a trickle sparge during use to keep atmospheric gases from dissolving back into the mobile phase. The sparged solvent must be continually blanketed with helium at 2 to 3 psi. Non- blanketed, sparged solvents will allow atmospheric gases to dissolve back into the mobile phase within four hours.

Solvent mixtures using water and organic solvents (like methanol or acetonitrile) hold less dissolved gas than pure solvents. Sparging to reduce the amount of dissolved gas is therefore particularly important when utilizing solvent mixture.

Even with sparging some out-gassing may be occur. A back pressure regulator installed after the detector flow cell will help prevent bubbles from forming and thus limit baseline noise.

WARNING: Always release pressure from the pump slowly. A rapid pressure release could cause the pulse damper diaphragm to rupture.

2.5.2 Cavitation

Cavitation occurs when inlet conditions restrict the flow of solvent and vapor bubbles are formed during the inlet stroke. The key to preventing cavitation is to reduce inlet restrictions. The most common causes of inlet restrictions are crimped inlet lines and plugged inlet filters. Inlet lines with tubing longer than 48" (120 cm) or with tubing of less than 0.085" (2 mm) ID may also cause cavitation.

Placing the solvent reservoirs below the pump level also promotes cavitation. The optimal location of the reservoirs is slightly above the pump level, but it is adequate to have them on the same level as the pump.

2.5.3 Filtration

Solvent filtration is good practice for the reliability of the Post Column Reactor and other components in a HPLC system. Solvents should always be filtered with a 0.5 micron filter prior to use. This ensures that no particles will interfere with the reliable operation of the piston seals and check valves. Solvents in which buffers or other salts readily precipitate out will need to be filtered more often. After filtration, the solvents should be stored in a closed, particulate-free bottle.

2.6 Instrument Installation

2.6.1 Mobile Phase Reservoirs

The mobile phase reservoir should be placed at the same level or slightly higher than the pump, never below the pump, and the inlet tubing should be as short as practical. These steps minimize pressure losses on the inlet side of the pump during refill and help to avoid bubble formation. These steps are particularly important when using high vapor pressure solvents (hexane, methylene chloride, etc.). Mobile phases should be degassed, filtered and covered. (See Section 2.4.)

2.6.2 Self-Flush Solution

Self-flush heads require 250-500 mL of 20% methanol in water as a flushing solution. A pH indicator that will indicate the concentration of salts in the solution is recommended as a reminder to change the solution. This flush solution should be replaced with a fresh solution weekly to avoid frequent pump maintenance.

2.6.3 Inlet Tubing and Filters

The table below shows the inlet tubing and filter used in the Post Column Reactors. All inlet lines are supplied in a 30" (76 cm) length and are made of a fluoropolymer material.

Pump Head Type	Inlet Tubing	Inlet Filter
10 ml PEEK™	0.085" ID X 1/8" OD	88-0721

2.6.4 Priming the Pump and the Flushing Lines

Connect a syringe to the outlet tubing. Run the pump at a flowrate of 3 to 5 ml/min. Prime the pump by pulling mobile phase and any air bubbles through the system and into the syringe (a minimum of 20 ml).

To prime the flush lines for a self-flush head, simply place the inlet line in the flush solution and connect a syringe to the outlet line and apply suction until the line is filled with flush solution. Place the outlet line in the flush solution. Secure both flush lines in the flush solution container so they stay immersed during pump operation.

2.6.6 Long Term Pressure Calibration Accuracy

This note applies if your pump is equipped with an electronic pressure transducer. The transducer has been zeroed and calibrated at the factory. Over the life of the pump, some drift may occur. For example, it is typical for the zero to drift < 10 p.s.i. after about 1 year of operation (i.e., with no back pressure on the pump a reading of 1-9 p.s.i. may be displayed). A similar drift may also occur at higher pressures, and are typically less than 1% (e.g. <50 p.s.i. at 6,000 p.s.i. back pressure).

If pressure calibration and/or drift is a concern, consult the factory. The pump can be shipped back to SSI for recalibration. Alternatively, written calibration and zero-reset procedures are available. Consult the factory to receive these instructions.

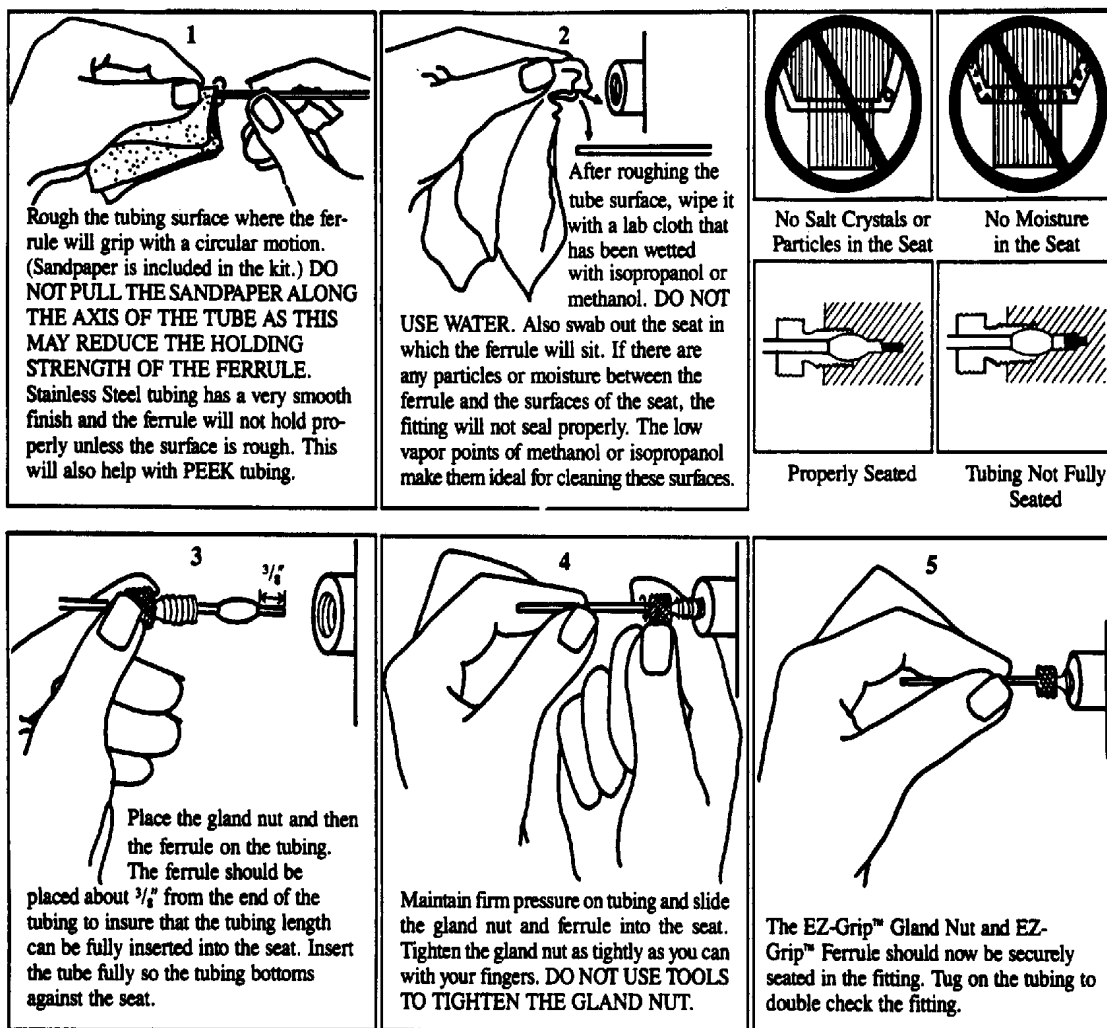


Figure 2-4. E-Z Grip Preparation

2.7 Preparation for Storage or Shipping

2.7.1 *Isopropanol Flush*

Disconnect the outlet tubing from the pump. Place the inlet filter in isopropanol. Use a syringe to draw a minimum of 50 ml through the pump. Pump a minimum of 5 ml of isopropanol to exit. Leave the inlet tubing connected to the pump. Place the inlet filter in a small plastic bag and attach it to the tubing with a rubber band. Plug the outlet port with the shipping plug or leave a length of outlet tubing on the pump or cover the outlet port with plastic film.

2.7.2 *Packaging for Shipping*

CAUTION: Reship in the original carton, if possible. If the original carton is not available, wrap the pump in several layers of bubble wrap and cushion the bottom, top, and all four sides with 2" of packaging foam. Although heavy, an HPLC pump is a delicate instrument and must be carefully packaged to withstand the shocks and vibration of shipment.

3. OPERATION

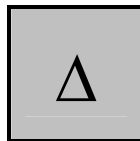
3.1 Pump Front Panel Controls and Indicators

3.1.1 Control Panel

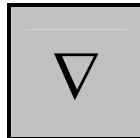
3.1.1.1 Digital Display

The 3-digit display shows the pump flow rate (mL/min), system pressure (psi), or the set upper or lower pressure limit (psi) when operating. Choice of display is selected with the MODE key.

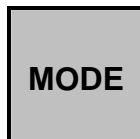
3.1.1.2 Digital Display Pump Keypad



When pressed, this button increases the flow rate.



When pressed, this button decreases the flow rate.



Use this button to cycle through the four display modes: flow rate, pressure, upper pressure limit, or lower pressure limit. A status LED to the right of the digital display indicates which mode is active.



When pressed, this button alternately starts and stops the pump.

Fast And Slow Button Repeat On The Up And Down Arrow Buttons:

If the UP-ARROW or DOWN-ARROW button is held down for more than approximately one half of a second, the button press will repeat at a slow rate of approximately 10 times a second. Once slow button repeat has begun, fast button repeat can be initiated by using a second finger to press down the second arrow button. During fast button repeat, the button press will repeat at a rate of approximately 100 times a second. Switching back and forth between repeat speeds can be accomplished by pressing and releasing the second arrow button while keeping the first arrow button held down.

3.1.1.3 Status LEDs

- **ml/min** When lit, the digital display shows flow rate in mL/min.
 - **PSI** When lit, the digital display shows system pressure in psi.
 - **HI PR** When lit, the display shows the user-set upper pressure limit in psi.
 - **LO PR** When lit, the display shows the user-set lower pressure limit in psi.
 - **RUN** Lights to indicate that the pump is running.
 - **FAULT** Lights when a fault occurs and stops the pump.
- PUMP RUN Lights to indicate that the pump is running.
- FAULT Lights when a fault occurs and stops the pump.

3.1.1.2 Power-up Configuration

Non-volatile Memory Reset: If the pump is operating erratically, there is the possibility that the memory has been corrupted. To reset the memory and restore the pump to its default parameters, press and hold the UP-ARROW button when the power is switched on. Release the button when the display reads "rES". The parameters stored in non-volatile memory, i.e., the flowrate, the pressure compensation, the voltage/frequency select, the lower pressure limit, and the upper pressure limit will be set to the factory default values. The head type setting is the only parameter not changed by the non-volatile memory reset function. If the firmware is upgraded to a newer version, a non-volatile memory reset will automatically occur the first time the power is switched on.

3.1.1.5 Power-Up Tests

Display Software Version Mode: The software version can be displayed during power-up by pressing and holding the RUN/STOP and the UP-ARROW buttons when the power is switched on. Release the buttons when the display reads "UEr". The decimal point number displayed on the display is the software version. To exit this mode, press the RUN/STOP button.

Align Refill Switch Mode: The signal that initiates the refill phase can be displayed during power-up by pressing and holding the PRIME and the UP-ARROW buttons when the power is switched on. Release the buttons when the display displays "rFL". When the slotted disk allows the light beam to pass from the emitter to the detector on the slotted optical switch a pulse will be generated which signals the beginning of refill. When this pulse occurs the three horizontal segments displayed at the top of the display will turn off and the three horizontal segments at the bottom of the display will turn on. To exit this mode, press the RUN/STOP button.

Serial Port Loopback Test Mode: If an external device will not communicate to the pump via the serial port, the serial port loopback test can be used to verify that the serial port is functioning properly. During power-up press and hold the UP-ARROW and the DOWN-ARROW buttons when the power is switched on and then release the buttons. The display must display "C00" for the first half of the test to pass. Plug in the serial port loop back plug (A modular plug with pins 2 & 5 jumpered together and pins 3 & 4 jumpered together.). The display must read "C11" for the second half of the test to pass. To exit this mode, press the RUN/STOP button.

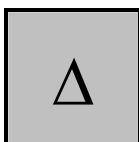
3.2 Reaction Coil Front Panel Controls and Indicators

3.2.1 Control Panel

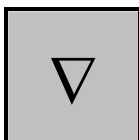
3.2.1.1 Digital Display

The 4-digit display shows the reaction coil's setpoint temperature or current temperature in degrees Celsius or Fahrenheit. It also displays an error message in the event of a fault.

3.2.1.2 Operation Keys



When pressed, this button increases the temperature set point.



When pressed, this button decreases the temperature set point.



Use this button to toggle between display of set point temperature and actual temperature readings.



When pressed, this button alternately turns on and off the coil's heating element. The HEAT light will flash to indicate the button has been pressed.

Fast And Slow Button Repeat On The Up And Down Arrow Buttons: If the UP-ARROW or DOWN-ARROW button is held down for more than approximately one half of a second, the button press will repeat at a slow rate of approximately 10 times a second. Once slow button repeat has begun, fast button repeat can be initiated by using a second finger to press down the second arrow button. During fast button repeat, the button press will repeat at a rate of approximately 100 times a second. Switching back and forth between repeat speeds can be accomplished by pressing and releasing the second arrow button while keeping the first arrow button held down.

3.2.1.3 Status Lights

- °C When lit, the temperature displayed is in degrees Celsius.
- °F When lit, the temperature displayed is in degrees Fahrenheit.
- ACTUAL When lit, the actual coil temperature is displayed. When unlit, the set point temperature is displayed.
- HEAT Lights to indicate power is being applied to the heating element. LED will flash when the RUN/STOP button is pressed. LED will remain steady as coil heats to the set point temperature and go out once set point temperature is reached.
- READY Lights to indicate the coil has reached its set point temperature.
- FAULT Lights when a fault occurs, and an error message is displayed on the digital display.

NOTE: Once temperature reaches setpoint, Ready Light will not be lit for 5 – 7 minutes. This is a stabilization time. After stabilization time has passed, Ready Light will be lit.

3.2.1.3 Power Up Configuration:

°C / °F Temperature Display: On power-up, hold both the **READ TEMP** and **DOWN ARROW** buttons on the front panel while pressing the **ON** switch on the front panel. Then press the **UP ARROW** button to toggle between displaying temperatures in degrees Celsius (**C** on display) or degrees Fahrenheit (**F** on display).

3.2 Over-Pressure Switch

The system contains an integrated mechanical over-pressure switch set at 350 - 500 p.s.i. If coil pressure exceeds this set point, the system will fault and the heating will shut down. **An audible alarm will sound.** Also, the word “coil” will appear on the keypads. Tripping the over-pressure switch likely means the coil is plugged and should be replaced (see Maintenance Section).

3.3 Nitrogen Sparge for Buffer Bottles (if equipped)

The system may include a nitrogen sparge system for the buffer bottles (optional). This set-up includes pressure regulator/manifold and gauge, two reagent bottle caps (3 valve) with tubing and mounting bracket. Install on either side of the PCR cabinet.

3.4 Low HPLC Pressure Disable (if equipped)

The system may include (optional) a low HPLC pressure cut-off switch (400 p.s.i.). Connection is made to the HPLC via a single fluid line “T”ed between the HPLC Pump and Autosampler. PCR Pumps and Reaction Coil Heater are disabled below 400 p.s.i. All three units can be placed in “Stand By” mode by pressing Run/Stop Buttons while in low HPLC pressure condition. “Stand By” will appear on keypads. When in Stand-By, units will re-activate automatically when HPLC pressure is restored. Otherwise, user must manually re-start Pumps and Heater by pressing Run/Stop after HPLC pressure is restored. If user chooses not to use this option, simply unplug all connection, including communication wiring.

3.5 Rear Panel Remote Input

An RS-232 modular jacks are provided on the back panel. A computer, with appropriate software, can be used as a remote controlling device for pump operation via this connection.

See Appendix A for details on connection and operation.

3.6 Symbols

The following symbols may appear on back panel of the unit:



Caution: To avoid chemical or electrical hazards, always observe safe laboratory practices while operating this equipment.



Caution: To avoid electrical shock and possible injury, remove the power cord from the back panel of this equipment before performing any type of service procedures.

Note: The user shall be made aware that, if equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

4. THEORY OF OPERATION

4.1 Pump Mechanical Operation

4.1.1 *Liquid System Flow Path*

The flow path of the Post Column Reactor starts at the inlet reservoir filter, passes through the inlet check valve, then through the pump head, and finally exits through the outlet check valve.

4.1.2 *Pump Cycle*

The pump cycle consists of two phases, the pumping phase, when fluid is metered out of the pump at high pressure, and the refill phase, when fluid is rapidly drawn into the pump.

During the pumping phase, the pump piston moves forward at a programmed speed; this results in a stable flow from the pump. The piston is driven by an eccentric bearing which is directly driven by the motor.

At the end of the pumping phase, the pump enters the refill phase. The piston quickly retracts, refilling the pump head with solvent, and the piston begins to move forward again as the pumping phase begins.

The motor speed is increased during refill to reduce refill time and to pre-compress the solvent at the beginning of the pumping phase. Since the output flow completely stops during refill, an optional, external pulse damper is necessary for applications requiring extremely low pulsation levels.

For optimal operation of the check valves, a back-pressure of at least 25 psi is required. Operating at lower pressures can lead to improper seating of the valves and subsequently inaccurate flow rates.

4.1.3 Pulse Damping / Back Pressure Coil

The diaphragm-type pulse damper (inside the pump drawer) consists of a compressible fluid (isopropanol) held in an isolated cavity by an inert but flexible diaphragm. During the pumping phase of the pump cycle, the fluid pressure of the mobile phase displaces the diaphragm, compressing the fluid in the cavity and storing energy. During the pump refill phase the pressure on the diaphragm is reduced and the compressed fluid expands, releasing the energy it has stored. This helps to stabilize flow rate and pressure. The amount of mobile phase in contact with the pulse damper is small, only 0.9 mL at 2,500 psi, and the geometry used insures that the flow path is completely swept, so solvent “memory effects” are virtually eliminated.

To be effective, the pulse damper requires a back-pressure of approximately 500 psi or greater. A back pressure coil (restrictor) made from small bore PEEK tubing is installed between the pulse damper and the outlet.

4.2 Electronic Control

4.2.1 Microprocessor Control

The pump is controlled by hybrid microprocessor circuitry which (1) provides control signals to the motor drive circuitry, (2) interfaces with the keyboard/display, (3) receives signals from the refill flag, and (4) provides external input/output (RS-232) interfacing. Firmware programming is stored in an EPROM.

An eccentric cam provides refill in a fraction of the full cam cycle. The remaining revolution of the cam provides piston displacement for outward flow of the mobile phase. In addition to the rapid refill characteristics of the drive, the onset of refill is detected by an infrared optical sensor. The microprocessor changes the refill speed of the motor to an optimum for the set flow rate. At 1ml/min, the refill rate is more than five times faster than if the motor operated at constant speed. The optimum refill rate minimizes the resulting pulsation while avoiding cavitation in the pump head.

The flow rate of any high pressure pump can vary depending on the operating pressure and the compressibility of the fluid being pumped. The Post Column Reactor is calibrated at 1000 psi using a 80:20 mixture of water and isopropanol.

4.2.2 DC Power Supply

Power for the pump is provided by a switching power supply which accepts voltages from 90 – 240 VAC. Output is 24 VDC for the pumps and heater. A switching 5 VDC supply is also provided to power control and display circuits.

4.2.3 Remote Interfacing

RS-232C modular jacks are provided on the back panel. See Appendix A for information on operation via this connection.

4.2.4 Motor Stall Detector

The motor can stall and create a loud buzzing sound if the flow path connected to the pump's outlet becomes plugged, if the pressure exceeds the maximum pressure rating of the pump, or if the mechanism jams. If a motor stall occurs, the electrical current being supplied to the motor is turned off and the fault light is turned on.

The Motor Stall Detector is enabled or disabled during power-up by pressing and holding the RUN/STOP and the PRIME buttons while the power is switched on. Release the buttons when the display displays "SFE". To enable the Motor Stall Detector press the UP-ARROW button and the display will display "On". To disable the Motor Stall Detector press the DOWN-ARROW button and the display will display "OFF". To exit this mode and store the current setting in non-volatile memory, press the RUN/STOP button.

The Motor Stall Detector uses a timer to determine if the camshaft has stopped turning or if the refill switch is defective. The timer begins timing after the pump accelerates or decelerates to its set point flow rate. If the Motor Stall Detector has been enabled, and the camshaft stops turning or the refill switch stops operating, the fault will be detected between the time it takes to complete 1 to 2 pump cycles. One revolution of the camshaft produces a delivery phase and a refill phase.

The fault is canceled by using one of the following methods: (1) by pressing the RUN/STOP button on the front panel, (2) by sending a stop command "ST" via the serial communications port on the back panel, or (3) by connecting the PUMP-STOP input to COM on the back panel, or removing the connection between the PUMP-RUN input and COM if the PUMP-STOP input is permanently jumpered to COM on the back panel. Note: the PUMP-RUN, PUMP-STOP, and COM are an option and do not exist on the standard pump.

5. MAINTENANCE

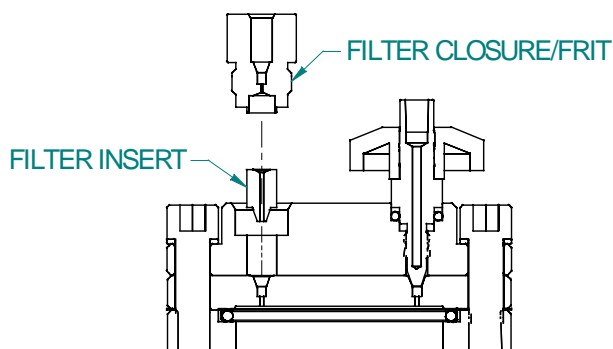
Cleaning and minor repairs of the Post Column Reactor can be performed as outlined below.

Lower than normal pressure, pressure variations, or leaks in the pumping system can all indicate possible problems with the piston seal, piston, or check valves. Piston seal replacement could be necessary after 1000 hours of running time.

5.1 Pump Filter Replacement - Inlet / Outlet Filters

Inlet filters should be checked periodically to ensure that they are clean and not restricting flow. A restriction could cause cavitation and flow loss in the pump. Two problems that can plug an inlet filter are microbial growth and impure solvents. To prevent microbial growth, use at least 10-20% organic solvent in the mobile phase or add a growth-inhibiting compound. If you pump 100% water or an aqueous solution without any inhibitors, microbes will grow in the inlet filter over time, even if you make fresh solution every day. Always use well filtered, HPLC grade solvents for your mobile phase.

Pump Head Type	Inlet Filter	Outlet Filter
10 ml PEEK™	88-0721	88-0740



Pump outlet filter replacement (below Prime-Purge Valve)

5.2 Pump Heads

5.2.1 Removing the Pump Head

As a guide to pump head assembly, the standard pump head is shown in Figure 5-1 and figure 5-2.

1. Turn OFF the power to the Post Column Reactor.
2. Remove the inlet line and filter from the mobile phase reservoir.
Be careful not to damage the inlet filter or crimp the Teflon tubing.
3. Optionally remove the outlet line from the outlet check valve.
4. Momentarily turn ON the Post Column Reactor Pump and quickly turn OFF the power upon hearing the refill stroke. This reduces the extension of the piston and decreases the possibility of piston breakage.
5. Unplug the power cord.
6. Carefully remove the two knurled nuts at the front of the pump head.

CAUTION: Be careful not to break the piston when removing the pump head. Twisting the pump head can cause the piston to break.

7. Carefully separate the pump head from the pump. Move the pump head straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the seal and seal backup washer from the piston if they did not stay in the pump head. Remove the O-ring.
8. Carefully separate the flush housing from the pump. Move the flush housing straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the self-flush diaphragm from the piston by carefully grasping the sealing flange on two sides and sliding it straight out on the piston being careful not to exert side pressure that may break the piston.

5.2.2 Replacing Piston Seals

Lower than normal pressure, pressure variations, and leaks in the pumping system can all indicate possible problems with the piston seal. Depending on the fluid or mobile phase used, piston seal replacement is often necessary after 1000 hours of running time.

The following table details the pump head types and appropriate seal kit choices. Each replacement seal kit contains one seal, one backup washer, a seal insertion/removal tool, a diaphragm and a pad to clean the piston when changing the seal.

Pump Head	Piston Seal Kit
10 ml PEEK	88-0197

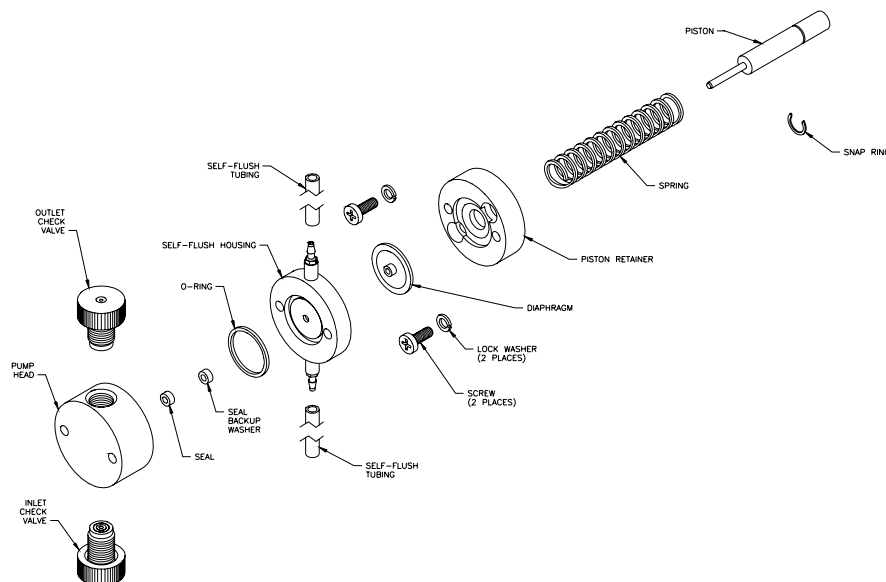
5.2.2.1 Conditioning New Seals

Note: Use only organic solvents to break-in new seals. Buffer solutions and salt solutions should never be used to break-in new seals.

Using a restrictor coil or a suitable column, run the pump with a 50:50 solution of isopropanol (or methanol) and water for 30 minutes at the back pressure and flow rate listed under PHASE 1 below and according to the pump head type.

PHASE 1

Pump Head Type	Pressure	Flow Rate
10 ml PEEK	2000 psi	<3 ml/min.



5.2.3 *Cleaning the Pump Head Assembly*

Note: If you choose to remove the piston seal or self-flush diaphragm, you should have a new set on hand to install after cleaning. It is not recommended that you reinstall the used piston seal or diaphragm since they are likely to be scratched and damaged during removal and would not provide a reliable seal if reused. If you decide to remove the seal, use only the flanged end of the plastic seal removal tool supplied with the seal replacement kit, and avoid scratching the sealing surface in the pump head. See Section 5.2.2 for seal replacement instructions.

1. Inspect the piston seal cavity in the polymer pump head. Remove any foreign material using a cotton swab or equivalent, and avoid scratching the sealing surfaces. Be sure no fibers from the cleaning swab remain in the components.
2. The pump head, check valves, and flushing housing may be further cleaned using a laboratory grade detergent solution in an ultrasonic bath for at least 30 minutes, followed by rinsing for at least 10 minutes in distilled water. Be sure that all particles loosened by the above procedures have been removed from the components before reassembly.

CAUTION: When cleaning check valves, be sure that the ball is not against the seat in the ultrasonic bath. This may destroy the precision matched sealing surface and the valve will not check.

3. If the check valves had been removed, tighten each check valve firmly by hand. Each check valve assembly contains two capsules. The sapphire seat in each capsule must be oriented downward in all cases in the final pump assembly.

Note: The inlet check valve has a larger opening (1/4"-28, flat-bottom seat) for the 1/8" inlet tubing; the outlet check valve has a smaller opening (#10-32, cone seat) for the 1/16" outlet tubing. The inlet check valve must be connected at the larger opening in the pump head.

If the piston seal has been removed, insert a new seal as described in Section 5.2.2, then continue with Section 5.2.5 to replace the pump head.

5.2.3.1 Removing the Seals

1. Remove the pump head as described in Section 5.2.1.
2. Insert the flanged end of the seal insertion/removal tool into the seal cavity on the pump head. Tilt it slightly so that flange is under the seal and pull out the seal.

CAUTION: Using any other “tool” will scratch the finish.

3. Inspect, and if necessary, clean the pump head as described in Section 5.2.3.

5.2.3.2 Cleaning the Piston

1. It is not necessary to remove the piston from the housing to clean the piston. Use the scouring pad included in the seal replacement kit to clean the piston. Gently squeeze the piston within a folded section of the pad and rub the pad along the length of the piston. Rotate the pad frequently to assure the entire surface is scrubbed. Do not exert pressure perpendicular to the length of the piston, as this may cause the piston to break. After scouring, use a lint-free cloth, dampened with alcohol, to wipe the piston clean.

5.2.3.3 Replacing the Seal

1. Place a high pressure replacement seal on the rod-shaped end of the seal insertion/removal tool so that the spring is visible when the seal is fully seated on the tool. Insert the tool into the pump head so that the open side of the seal enters first, facing the high pressure cavity of the pump head. Be careful to line up the seal with the cavity while inserting. Then withdraw the tool, leaving the seal in the pump head. When you look into the pump head cavity, only the polymer portion of the seal should be visible.
2. Attach the pump head as described in Section 5.2.5.
3. Condition the new seal as described in Section 5.3.

5.2.4 *Changing the Piston*

1. Remove the pump head as described in Section 5.2.1.
2. With your thumb pressing the piston retainer against the pump housing, remove the two Philips head screws from the retainer. Do not allow the spring pressure to force the retainer away from the housing as the screws are loosened.
3. After both screws have been removed, slowly allow the spring pressure to push the retainer out of the housing. Gently pull the retainer straight out and carefully remove it from the piston and threaded rods. Also, gently pull the spring straight out of the housing and remove.
4. Grasp the metal base of the piston assembly so that you avoid exerting any pressure perpendicular to the length of the piston, and gently pull it from the pump housing.
5. Remove the snap ring from the groove on the old piston and place it into the groove on the new piston.
6. Place a small amount of high quality grease on the back end of the metal base of the piston assembly. Grasp the metal base of the piston assembly near the front so that you avoid exerting any pressure perpendicular to the length of the piston, and gently slide it into the pump housing.
7. Gently slide the spring over the piston assembly and back into the pump housing. Carefully align the retainer and gently push it straight in against the spring force until the retainer is against the housing. If misalignment with the piston occurs, wiggle while pushing the retainer to align the piston & retainer.
8. Hold the retainer flush against the housing with your thumb. Insert and tighten the Philips head screws. Do not allow the spring pressure to force the retainer away from the housing. Insure that there are no gaps between the retainer and the housing.
9. Attach the pump head as described in Section 5.2.5.

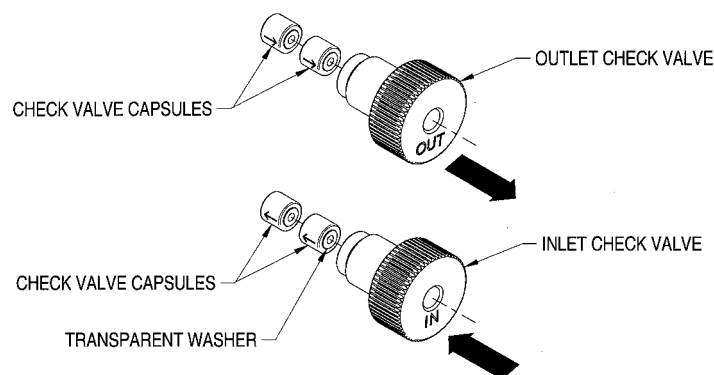
5.2.5 Replacing the Pump Head

1. Gently place diaphragm onto piston with center hub protruding towards you. Push diaphragm all the way back into recess and against metal base of piston. Do not exert pressure perpendicular to the length of the piston, as this may cause the piston to break.
2. Carefully align the flush housing and gently slide it into place on the pump. Make sure that the Inlet valve is on the bottom and the Outlet valve is on the top.
3. Line up the pump head and carefully slide it into place. Be sure that the Inlet valve is on the bottom and the Outlet valve is on the top. Do not force the pump head into place.
4. Finger tighten both knurled nuts into place. To tighten firmly, alternately turn nuts 1/4 turn while gently wiggling the pump head to center it.
5. Reattach the inlet and outlet lines. Change the flushing solution.

5.3 Pump Check Valve Cleaning and Replacement

Many check valve problems are the result of small particles interfering with the operation of the check valve. As a result, most problems can be solved by pumping a strong solution of liquid laboratory grade detergent through the check valves at a rate of 1 ml/min for one hour. After washing with detergent, pump distilled water through the pump for fifteen minutes. Always direct the output directly to a waste beaker during cleaning. If this does not work, the check valve should be replaced. Install as shown.

Replace check valve capsules as shown. Note directional arrows. Check valve kits (P/N 88-0402) contain the check valve capsules pre-loaded in the Inlet and Outlet holders (recommended).



5.4 Lubrication

The Post Column Reactor has no lubrication requirements. The bearings in the pump housing and piston carrier are permanently lubricated and require no maintenance. Keeping the interior of the pump free of dirt and dust will extend the pump's useful life.

5.5 Other Pump Maintenance

The internal components of the pump can be accessed by removing the pump drawer. To do so, turn off power at front switch. Unplug power cord. **Ensure there is no power to the system.** Disconnect the reagent inlet line and disconnect pump outlet at the "T" Connector Block above. Unscrew the two thumbscrews on the upper portion of the drawer until fully released (counterclockwise). Pull the drawer out as shown.



The Pump Control Boards, Back Pressure Coil and Pulse Damper (with Pressure Transducer) are accessible. The Pulse Damper can be re-built, if required, using the kit listed in on the Spare Parts List (Section 7 of this manual). Instructions are included with the kit. The Back Pressure Coil and Control Boards are replaceable.

5.6 Reaction Coil Replacement

Turn off power at front switch. Unplug power cord. **Ensure there is no power to the system.** Remove the “jumper line from the Column Inlet connector block to the Coil Inlet connector block. Unscrew the two thumbscrews on the upper portion of the drawer until fully released (counterclockwise). Pull the drawer out as shown.



Note: Dual channel system is shown. Single channel system will not have ambient coil (left hand). Heated coil is under the internal insulating box.

Detach the fluid connections from the front “T” connector block. For the heated coil, release the two thumbscrews holding down the internal insulating box. Remove the box. Pull the coil straight up from the mounting posts as shown.



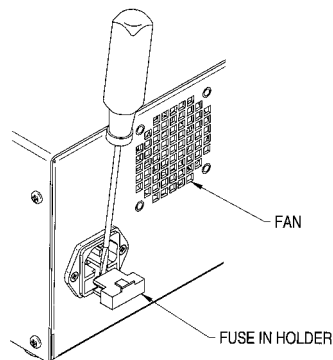
Install a new coil in reverse order.

NOTE: For the heated coil, the positioning posts are the heating elements. Also, the temperature probe (RTD) is found near the posts. Before installing a new coil, apply heat conductive grease to the posts and the RTD. Thermally conductive grease is required to ensure proper operation.

5.7 Fuse Replacement

Three fuses protect the Post Column Reactor. Two of the fuses are located in the power entry module at the rear of the cabinet and are in series with the AC input line. The other fuse is located on the circuit board and is in series with the 24 VDC supply.

Troubleshooting the fuses is straightforward. If the power cord is plugged in and the on/off power entry switch is on and the display does not light, check the two fuses in the power entry module. To gain access to these fuses, gently pry off the cover plate with a small flat-bladed screwdriver. Replace with fuses of the correct rating: 1 A slow-blo for 120 VAC systems.



5.8 Battery Replacement

The battery provides power for the memory that holds the current pump configuration. If the pump is set at a flow rate other than 1.00 or 10.0 and the power is turned off, when the power is turned back on the flow rate should appear as it was set. If this flow rate does not appear the battery will need replaced.

1. Unplug the unit.
2. Remove the cover.
3. Turn the unit so that the control panel is to the right. The battery can be seen in the lower right corner of the circuit board. The battery is circular and has a positive pole mark (+) on the top. Gently pull it from its socket.
4. With the positive mark (+) up, gently slide the new battery into the battery socket. Be sure the battery is all the way into place. It must contact the base of the battery socket.
5. Replace the cover to the unit. Plug the unit back in.

<p>WARNING: Unplug power cord before removing cabinet lid.</p>

6. PROBLEM SOLVING

Quick Guide to Problem Solving

You Notice	This May Mean	Possible Cause	You Should
<ol style="list-style-type: none"> 1. Uneven pressure trace. 2. Pressure drops. 3. No flow out the outlet check valve. 	<ol style="list-style-type: none"> 1. Bubble in check valve. 2. Leaks in system. 3. Dirty check valve. 4. Bad check valve. 	<ol style="list-style-type: none"> 1. Solvent not properly degassed. 2. Fittings are not tight. 3. Mobile phase not properly filtered. 4. Particles from worn piston seal caught in check valve. 5. Plugged inlet filter. 	<ol style="list-style-type: none"> 1. Check to be certain that mobile phase is properly degassed. 2. Check connections for leaks by tightening fittings. 3. Prime the system directly from the outlet check valve. 4. Clean or replace the check valves. See Section 5.4. 5. Clean or replace inlet filter. See Section 5.1.1.
<ol style="list-style-type: none"> 1. Uneven pressure trace. 2. Pressure drops. 3. Fluid between the pump head and the retainer. 	<ol style="list-style-type: none"> 1. Leaks in system. 2. The piston seal or diaphragm is worn. 	<ol style="list-style-type: none"> 1. Fittings not tight. 2. Long usage time since last seal / diaphragm change. 3. Salt deposits on seal or diaphragm (especially if buffered aqueous mobile phases are used). 	<ol style="list-style-type: none"> 1. Check all connections for leaks. 2. Replace piston seal & diaphragm. See Sections 5.2 and 5.3. 3. Check the piston for salt deposits. Clean as necessary. See Section 5.2.4.
Pump makes a loud clanging or slapping noise (intermittent contact with cam).	Piston carrier is catching in piston guide.	<ol style="list-style-type: none"> 1. Cap nut screws on the pump head are loose. 2. Seal(s) are worn. 3. Piston guide is worn 	<ol style="list-style-type: none"> 1. Check cap nut screws on pump head. Tighten if necessary. 2. Replace seals. 3. Replace piston guide and seals. See Sections 5.2 and 5.3.
No power when pump turned ON.	Blown fuses in the power entry module.	<ol style="list-style-type: none"> 1. Power surge. 2. Internal short. 	<ol style="list-style-type: none"> 1. Replace only with the appropriate fuses (1A for 100/110 Vac or 1/2A for 220/240 Vac). 2. Contact service technician if problem persists.
Blue dye in mobile phase.	Pulse damper diaphragm has burst.	Sudden pressure drop when purging system.	Replace pulse damper. See Section 5.5.
Pump runs for 50 pump strokes, then shuts down.	Lower pressure limit is activating.	<ol style="list-style-type: none"> 1. Mobile phase is not properly filtered. 2. Particles from worn seal trapped in the system (e.g., tubing, filters, injection valve, column inlet). 	<ol style="list-style-type: none"> 1. Check to be certain the low pressure limit is set to 0 psi. 2. Only increase the low pressure limit after the pump attains operating pressure. 3. Contact service technician.
<ol style="list-style-type: none"> 1. Pump shuts down after run is called even with no column connected. 2. Pump runs to maximum pressure and shuts down. 	Clog in fluid system.		<ol style="list-style-type: none"> 1. Remove and clean both the inlet and bulkhead filters. See Section 5.2. 2. If the problem persists, remove tubing from system one piece at a time until you find the clogged piece. Most clogs occur outside the pump itself.
No power when pump turned ON. Fan does not run.	Blown fuses in the power entry module.	<ol style="list-style-type: none"> 1. Power surge. 2. Internal short. 	<ol style="list-style-type: none"> 1. Replace only with the appropriate fuses (1A for 100-120 Vac or 1/2A for 220-240 Vac). 2. Contact service technician if problem persists.
Front panel appears OK but pump motor does not run.	Blown fuse on the motor power circuit board.	<ol style="list-style-type: none"> 1. Power surge. 2. Internal short. 	<ol style="list-style-type: none"> 1. Replace only with the appropriate fuse . 2. Contact service technician if problem persists.
PEEK fittings or components leak.	You cannot force PEEK parts with interference to seal by brute force tightening.	<ol style="list-style-type: none"> 1. Film of fluid between surfaces. 2. Salt crystals between surfaces. 3. Scratches in mating surfaces. 	<ol style="list-style-type: none"> 1. Clean and dry mating surfaces. 2. If scratched, replace defective part.

7. LIST OF REPLACEMENT PARTS

All available from **Chrom Tech** . . . “your chromatography specialists”

Tel: 800-822-5242 ■ in MN 952-431-6000 ■ Fax: 952-431-6345

Email: sales @chromtech.com

Website: www.chromtech.com

<u>Part No.</u>	<u>Description</u>
88-0402	PEEK™ Check Valve Kit
88-0197	Seal Kit, Aqueous, 10 ml
88-0740	Outlet Filter Integrated PPV/PD
88-0660	Prime Purge Valve Stem
88-0721	Replacement Inlet Filter Elements (2)
88-0603	Pulse Damper Rebuild Kit
88-0301	Head & Self Flush Kit, 10 ml
88-0411	Self Flush Assembly, 10 ml
88-0351	Series I Piston, 10 ml
12-0957	Integrated Pulse Damper / PPV
88-0509	Series I Drive Assembly, 10 ml, PCR
88-0132	Series III SMT Board Set
12-0963	Front Panel PC Board
16-0482	Pump Back Pressure Coil
88-0960	0.15 ml Reaction Coil
88-0961	0.5 ml Reaction Coil
88-0962	1.4 ml Reaction Coil
88-0963	Reactor Heater Assembly (heating elements & temperature probe)
88-0964	Heater PC Board Assembly
88-0806	Fuse, 2 Amp, 5x20 mm (10 pack)

APPENDIX A

A.1 Rear Panel Serial Communications Port - Pump

An RS-232C modular jack is provided on the back panel. A computer, with appropriate software, can be used as a remote controlling device for pump operation via this connection.

A.1.1 Hardware Implementation

The REMOTE INPUT serial communications port is configured for 9600 baud, 8 data bits, 1 stop bit, and no parity. The connector is a standard RJ-11 modular telephone type jack. When looking at the connector on the rear panel of the pump, pin 1 is at the top and pin 6 is at the bottom. The pin-out is:

<u>Pin</u>	<u>Function</u>
1, 6	Ground
2	DSR (Handshaking input to pump)
3	RXD (Serial data input to pump)
4	TXD (Serial data output from pump)
5	DTR (Handshaking output from pump)

Special wiring considerations: Use the following chart for interfacing the pump's serial communications port to either a 25-pin or a 9-pin COM port on an IBM-PC type computer.

<u>Pump (RJ11)</u>	<u>Signal</u>	<u>IBM (DB25)^a</u>	<u>IBM (DB9)^b</u>
1, 6	Ground	7	5
2	DSR	20	4
3	RXD	2	3
4	TXD	3	2
5	DTR	6	6

^a Jumper pins 4, 5, and 8 on DB25.
^b Jumper pins 1, 7, and 8 on DB9.

Part Description	Part Number
Modular Cable	12-0677
Adapter RJ-11 to DB9	12-0672
Adapter RJ-11 to DB-25	12-0671

A.1.2 Hand-Shaking

The pump uses hardware handshaking. The pump will not transmit on the TXD output if the DSR input is at a low logic level. And, the pump will not receive on the RXD input when the DTR

output is at a low logic level. A low logic level is -3.0 to -15 volts and a high logic level is 3.0 to 15 volts.

A.1.3 Command Interpreter

The pump's high-level command interpreter receives and responds to command packets. The pump will not send a message except when prompted, and it will send a response to every valid command as described below. The response to an invalid command is "Er/".

Each command is characterized by a unique two-letter command code, and only one command can be issued per line. Case is not important; that is, the command codes "PR" "Pr" "pR" and "pr" are all equivalent. Response strings sent by the pump are terminated by the "/" character.

If the pump's response is "Er/", send a "#" to clear any characters which may be remaining in the command buffer. The pump will automatically clear all characters in the command buffer after one second elapses from the time at which the last character of an incomplete command was sent.

The command packets are as follows:

Command	Response	Comments
RU	OK/	Sets the pump to the RUN state.
ST	OK/	Sets the pump to the STOP state.
FLxxx	OK/	Sets the flowrate to x.xx or xx.x mL/min where the range is fixed for the pump head size, i.e., for 0.01 to 9.99 mL/min xxx = 001 to 999, for 0.1 to 99.9 mL/min xxx = 001 to 999.
FOxxxx	OK/	Sets the flowrate to xxx.x mL/min, i.e., for 0.1 to 100.0 mL/min xxxx = 0001 to 1000.
PR	OK,x/ (x, xx, or xxx)	Reads the pump's current pressure, where: x, xx, or xxx = Current pressure in PSI
CC	OK,x,yyy.y/ (x, xx, or xxx) (y.y, yy.y, or yyy.y)	Reads the pump's current pressure and flowrate, where: x, xx, or xxx = Current pressure in PSI y.y, yy.y, or yyy.y = Flowrate in mL/min
CS	OK,xxx.x,y,z,PSI,w,v,u/ (x.x, xx.x, or xxx.x) (y, yy, or yyy) (z, zz, or zzz)	Reads the current pump setup, where: x.x, xx.x, or xxx.x = Flowrate in mL/min y, yy, or yyy = Upper pressure limit z, zz, or zzz = Lower pressure limit PSI = Units (PSI, ATM, MPA, BAR, or KGC) w = Pump head size (0 = standard, 1 = macro) v = Run status (0 = stopped, 1 = running) u = Pressure Board present = 0; otherwise 1
ID	OK,vx.xx SR3P firmware/	Identifies the pump type and EPROM revision x.xx

UPxxxx	OK/	Sets the upper pressure limit in PSI. The maximum value is 500; the minimum value is the lower limit plus 10. The value must be expressed as four digits, i.e., for 400 PSI xxxx = 0400.
LPxxxx	OK/	Sets the lower pressure limit in PSI. The maximum value for xxxx is the current upper pressure limit setting minus 10; the minimum value is 0. The value must be expressed as four digits, i.e., for 50 PSI xxxx = 0050.
SF	OK/	Puts the pump in fault mode. Turns on the FAULT LED and stops the pump immediately.
RF	OK,x,y,z/	Reads the fault status, where: x = Motor stall fault (0 = no, 1 = yes) y = Upper pressure limit fault (0 = no, 1 = yes) z = Lower pressure limit fault (0 = no, 1 = yes)
KD	OK/	Disables the keypad. (Default status at power-up is enabled.)
KE	OK/	Enables the keypad.
PCxx	OK/	Sets the pressure compensation value, where xx = the operating pressure (in PSI divided by 100), i.e., for 0 PSI xx = 00, for 0500 PSI xx = 05.
RC	OK,x/	Reads the pressure compensation value in hundreds of PSI, i.e., for 0 PSI x = 0, for 0500 PSI x = 5.
HTx	OK/	Sets the pump head type, where: x = 3 for a stainless steel 50 mL/min pump head x = 4 for a plastic 50 mL/min pump head The pump is stopped; and, the pressure compensation and pressure limits are initialized, when the head type is changed.
RH	OK,x/	Reads the pump head type, where: x = 3 for a stainless steel 50 mL/min pump head x = 4 for a plastic 50 mL/min pump head
PI	OK,a.aa,b,c,d,e,f,g,h,i,j,k,l,m,n,o,p,q/ (a.a, aa.a, or aaa.a) (c or cc)	Reads the current pump setup, where: a.a, aa.a, or aaa.a = Flowrate in mL/min b = Run status (0 = stopped, 1 = running) c or cc = Pressure compensation d = Pump head type (see RH command) e = Pressure Board present = 0; otherwise 1 f = External control mode (0 = frequency, 1 = voltage) g = 1 if pump started and frequency controlled, else 0 h = 1 if pump started and voltage controlled, else 0 i = Upper pressure limit fault (0 = no, 1 = yes) j = Lower pressure limit fault (0 = no, 1 = yes) k = Priming (0 = no, 1 = yes) l = Keypad lockout (0 = no, 1 = yes) m = PUMP-RUN input (0 = inactive, 1 = active) n = PUMP-STOP input (0 = inactive, 1 = active) o = ENABLE IN input (0 = inactive, 1 = active) p = Always 0 q = Motor stall fault (0 = no, 1 = yes)
RE	OK/	Resets the pump configuration to its default power-up state.
#	(no response)	Clears all characters from the command buffer.

A.2 Pump Rear Panel 6-Pin and 10-Pin Terminal Board Connectors

A 6-pin terminal board connector and a 10-pin terminal board connector are provided on the back panel. Any device capable of providing the proper run/stop logic level, flowrate control frequency, or flowrate control voltage can be used as a remote controlling device for pump operation via this connection. The terminal board connectors can be removed for ease of connecting wires, if desired, by pulling firmly rearward and should be reinserted firmly afterward.

A.2.1 Pressure Fault and Motor Stall Fault Output

The pump's output is on the 6-pin terminal board connector. The pinout is:

<u>Pin</u>	<u>Function</u>
6	EVENT 1
5	No connection
4	EVENT 2
3	No connection
2	EVENT 3
1	No connection

This output is produced internally by a reed relay which has SPDT contacts with a 0.25 amp maximum, 50 VDC maximum, 0.2 ohm rating. The 6-pin connector allows wires to be connected to the EVENT 1 (Pole), EVENT 2 (NC), and EVENT 3 (NO) terminals. When the pump stops due to the sensed pressure exceeding the set pressure limits or if a motor stall fault occurs, the connection between the EVENT 1 terminal and the EVENT 2 and EVENT 3 terminals is affected. EVENT 2 is Normally Closed (connected to EVENT 1) until a fault occurs and then opens. EVENT 3 is Normally Open (not connected to EVENT 1) until a fault occurs and then closes.

A.2.1.1 Upper and Lower Pressure Limit Range

The pressure sensing transducer provides accurate, wide range pressure monitoring. Because of the sensitivity of the transducer, the zero reading may shift up to 0.1% of the full pressure scale over years of operational use. The user should also be aware that the resistance to flow of the fluid being pumped through the tubing and fittings may cause the pressure to vary with the flow rate and the viscosity of the mobile phase employed.

If absolute accuracy is needed for the pressure safety limits:

1. Disconnect the column from pumping system and operate the pump with the mobile phase and flow rate to be used in the

analysis. Observe the resulting pressure displayed on the pump readout. The column will cause a pressure reading that adds to this basic reading due to system flow resistance.

2. Set the upper limit shut-off to a pressure equal to the basic reading *plus* the safe operating pressure for the column to be used. For example, if the basic pressure reading (without the column) is 7 PSI and the safe limit for the column is 25 PSI, set the maximum pressure limit to 32 PSI or less.
3. If the mobile phase or flow rate is changed, reset the pressure limit as appropriate.
4. Note that a lower pressure limit is available to prevent continued operation in the event of a leak. For proper operation, this must be set to a pressure higher than the basic pressure or it may not sense the reduced pressure.

A.2.2 General Information on Inputs

The pump's inputs are on the 10-pin terminal board connector. The pinout is:

<u>Pin</u>	<u>Function</u>
10	VOLTAGE COM
9	VOLTAGE IN
8	FREQ IN
7	ENABLE IN
6	PUMP-RUN
5	PUMP-STOP
4	No connection
3	No connection
2	No connection
1	COM

A.2.3 General Information on Run, Stop, and Enable Inputs

The PUMP-RUN, PUMP-STOP, and ENABLE IN inputs operate from an internal 5 VDC source and each one draws approximately 0.008 amps when connected to COM. To activate either the PUMP-RUN, PUMP-STOP, or ENABLE IN input connect it to COM. Any device capable of switching 0.008 amps can be connected between the PUMP-RUN, PUMP-STOP, or ENABLE IN input and COM, such as: a switch contact, a relay contact, an open collector output, an open drain output, or any output with a high logic level output of 3.8 to 6.0 volts and a low logic level output of 0.0 to 0.5 volts. A switch contact or a relay contact is preferred since this type of connection will

provide isolation between the pump and the controlling device. The COM terminal is internally connected to the pump's chassis ground and should be connected to the controlling device's ground or zero volt terminal when the controlling device has an open collector output, an open drain output, or any output with logic level output.

A.2.4 Run and Stop Inputs

The pump's motor can be commanded to run or stop from the back panel inputs when the pump's flowrate is controlled from the front panel or when the pump's flowrate is controlled by the voltage or frequency input. There two modes of operation for the run and stop inputs which are described below:

Dual Signal Pulse: In this mode of operation both the PUMP-RUN and PUMP-STOP inputs are normally at a high logic level. To start the pump, pulse the PUMP-RUN input to a low logic level for a minimum of 500 mS. To stop the pump, pulse the PUMP-STOP input to a low logic level for a minimum of 500 mS.

Single Signal Level: To enable this mode of operation the PUMP-STOP input must be permanently connected to COM with a jumper wire. To start the pump, put a low logic level on the PUMP-RUN input. To stop the pump, put a high logic level on the PUMP-RUN input.

A.2.4 Enable Input

When activated (ENABLE IN is at a low logic level), the ENABLE IN input disables flowrate control on the front panel and enables flowrate control on the back panel.

A.2.5 General Information on Voltage and Frequency Inputs

Special programming and circuitry allows this pump to be operated remotely with the flowrate controlled by voltage or frequency inputs. To select the remote mode of operation:

- a.) With the pump plugged in and the rear panel power switch OFF, press in and hold the "DOWN ARROW" button while turning the power switch ON.
- b.) Release the "DOWN ARROW" button and either a U (closest approximation to V for voltage) or an F (for frequency) will be displayed.
- c.) Select the desired remote operating mode by pressing the "DOWN ARROW" button to toggle between the voltage and frequency mode.

- d.) Press the "RUN/STOP" button to place the pump in normal operating mode.
- e.) To enable the currently selected remote mode (voltage or frequency), connect the rear panel ENABLE IN connection to the COM connection.
- f.) When in the remote mode (ENABLE IN at a low logic level) all front panel buttons remain active except the flow setting increase/decrease capability.

A.2.6 Voltage Input

The remote voltage flow control is implemented by connecting a negative input to the rear panel VOLTAGE COM connection and a positive input to the VOLTAGE IN connection. A 0-10 VDC input corresponds to a 0 to 10 mL/min flow rate. Any device capable of sourcing at least 0.0005 amps will work. Also, the voltage control mode must be selected and enabled as described in section "A.2.5" above. The voltage source which drives the VOLTAGE IN and VOLTAGE COM connections must be isolated from the safety ground to prevent a ground loop current. If the pump's displayed flowrate jumps up and down erratically, suspect a ground loop problem.

A.2.7 Frequency Input

The remote frequency flow control is implemented by connecting a negative input to the COM connection and +5 VDC square wave input to the FREQ IN connection. Any device capable of sinking and sourcing at least 0.008 amps will work. A 0 to 10,000 Hertz input frequency will correspond to a 0 to 10 mL/min flowrate. Also, the frequency control mode must be selected and enabled as described in section "A.2.5" above.

A.3 Rear Panel Serial Communications Port – Heated Reaction Coil

An RS-232C modular jack is provided on the back panel. A computer, with appropriate software, can be used as a remote controlling device for reaction coil operation via this connection.

A.3.1 Serial Communications Protocol

9600 baud
8 data bits
no parity
1 stop bit

A.3.2 Serial Port Connector

Connector: RJ11, 6-pin, modular telephone jack

Pinout:

Pin	Description
1	Ground
2	DSR (input)
3	RXD (input)
4	TXD (output)
5	DTR (output)
6	Ground

A.3.3 Handshaking

DSR input: logic high (3 to 15 volts); reaction coil can transmit.
logic low (-3 to -15 volts); reaction coil will not transmit.

DTR output: logic high (9.0 volts typical); reaction coil ready to receive.
logic low (-9.0 volts typical); reaction coil busy.

A.3.4 System to Computer Wiring

Use the following chart for interfacing the Heated Reaction Coil Heated coil REMOTE INPUT serial I/O port to either a 25-pin or 9-pin serial I/O COM port on the computer.

<u>Reaction coil (RJ11)</u>		<u>Function</u>	<u>IBM (DB25)^a</u>
<u>IBM (DB9)^b</u>			
1, 6	Ground	7	5
2	DSR (Input to reaction coil)	20	4
3	RXD (Input to reaction coil)	2	3
4	TXD (Output from reaction coil)	3	2
5	DTR (Output from reaction coil)	6	6

^a Jumper pins 4, 5, and 8 on DB25.
^b Jumper pins 1, 7, and 8 on DB9.

A.3.5 Command Interpreter

The Heated Reaction Coil's high level command interpreter receives and responds to ASCII commands. The reaction coil will not send a message except when prompted, and it will send a response to every valid command as described below. The response to an invalid command is "Er/".

Each command is characterized by a unique two-letter command code, and only one command can be issued per line. Case is not important; that is, the command codes "PR" "Pr" "pR" and "pr" are all equivalent. Response strings sent by the reaction coil are terminated by the "/" character. The command packets are as follows:

Command	Response	Comments
CR	OK/	Clear Ready: Turns off the READY indicator on the front panel, opens the “ready” relay contact, and resets the “ready” timer.
ID	OK,vx.xx Heated Reaction Coil/	Identifies the EPROM revision x.xx and reaction coil type.
RS	K,s,xxx.x,u/	Read status: s = 0 Idle s = 1 Trying to achieve setpoint s = 2 Ready xxx.x = The current temperature u = C Temperature in Celsius u = F Temperature in Fahrenheit
RT	OK,xxx.x/	Read Temperature: xxx.x = The temperature setpoint
SI	OK/	Set Idle: Sets the reaction coil to 0°C (or 32°F) and disables the keypad.
SR	OK/	Set Ready: Turns on the ready indicator on the front panel and closes the “ready” relay contact.
SS,x	OK/	Sets the temperature units: x = 0 for degrees Celsius x = 1 for degrees Fahrenheit
TT,xxxx	OK/	Sets the temperature setpoint to xxx.x degrees. The temperature units must first be set with the SSx command.
KD	OK/	Disables the keypad.
KE	OK/	Enables the keypad. (Default status at power-up is enabled.)