User's Guide

333/334 HPLC Pumps

GILSON®

TABLE OF CONTENTS

SAFETY

IN THIS CHAPTER

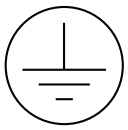
The instrument is intended to be used in a laboratory by trained technical personnel. For safe and correct use, both operating and service personnel should follow the instructions contained in this guide when installing, cleaning, and maintaining the instrument.

The following safety precautions must be observed during all phases of operation, service, and repair of the instrument. Failure to comply with these precautions or with specific warnings elsewhere in the user's guide violates safety standards of design, manufacture, and intended use of the instrument. Gilson assumes no liability for the customer's failure to comply with these requirements.

The instrument has been certified to safety standards required in Canada, Europe, and the United States. Refer to the rear panel label on the instrument or the Declaration of Conformity document for the current standards to which the instrument has been found compliant.

Electronic and Hazard Symbols

The following electronic and hazard symbols may appear on the instrument:

Symbol	Explanation
~	Alternating current
⎓	Direct current
	Protective conductor terminal
|	Electrical power ON
O	Electrical power OFF
	Caution, risk of electric shock
	Caution
	Fuse

Safety Notices

The following safety notices may appear in this document:

⚠WARNING	**WARNING indicates a potentially hazardous situation which, if not avoided, may result in serious injury**
⚠CAUTION	**CAUTION indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury**
NOTICE	**NOTICE indicates a potentially hazardous situation which, if not avoided, may result in equipment damage**

Lifting

The instrument exceeds the weight one person can lift safely. Two or more people are required to lift the instrument safely. Refer to the Technical Specifications for the weight. Always lift the instrument from the base and follow any unpacking instructions provided with the instrument.

Voltage

Access to the rear panel is necessary. The instrument must be detached from all voltage sources before service, repair, or exchange of parts. For normal operation, the instrument is to be grounded through the AC line cord and power supply provided. Failure to do so can result in a potential shock hazard that could result in serious personal injury.

Use only fuses with the rated current and of the specified type as listed on the rear panel label on the instrument. The instrument must only be operated with the voltage specified on the rear panel label of the instrument and with the grounded AC line cord and power supply provided.

Solvents

Observe safe laboratory practices when handling solvents. If working with hazardous solvents or flammable liquids, ensure that there is proper ventilation and that adequate protection such as safety glasses, gloves, and protective clothing are used.

If dangerous liquids are used, precautions should be taken to limit potential hazards from leaks and/or spillage through the use of a non-flammable tray or use of a fume hood, etc.

If there is the potential of explosive gases being developed, a fume hood or other means should be used to safely manage that risk.

Refer to the Material Safety Data Sheets for the solvents before use.

Replacement Parts

Be sure to use only replacement parts mentioned in the user's guide. Do not repair the instrument or change parts not listed in the user's guide. If it is necessary to replace parts not listed, please contact your local Gilson representative.

Chapter 1

INTRODUCTION

IN THIS CHAPTER

Description

The 333/334 HPLC Pumps are reciprocating piston pumps that provide a preparative pumping solution accommodating flow rates from 1 mL/min to 200 mL/min and pressures up to 3040 psi (210 bar). With additional pumps in parallel, you can double the flow rate up to 400 mL/min. Individually, a 333 Pump is for single solvent delivery (isocratic) under high pressure. When coupled with a 334 Pump, multi-solvent delivery (binary) is possible.

The 333/334 Pumps are controlled by either 333 Pump software using its control panel or by TRILUTION® LC Software as part of an HPLC system.

Figure 1
333/334 HPLC Pumps

Hydraulic Components

Open the removable door to view and/or access the hydraulics. Both the 333 Pump and 334 Pump include inlet and outlet tees to direct solvents and reciprocating piston pump heads to reduce flow pulsation. The 333 Pump has an integrated pressure, purge and mixing module (PPMM) that functions as a static mixer, a pressure transducer, and a purge valve. The 333 Pump also has an outlet filter to protect columns from contaminants. There is a drip tray at the bottom of the pump (see **Drip Tray on page 30**). Solvent bottles can be placed in the tray at the top of the pump (see **Solvent Bottle Tray on page 30**).

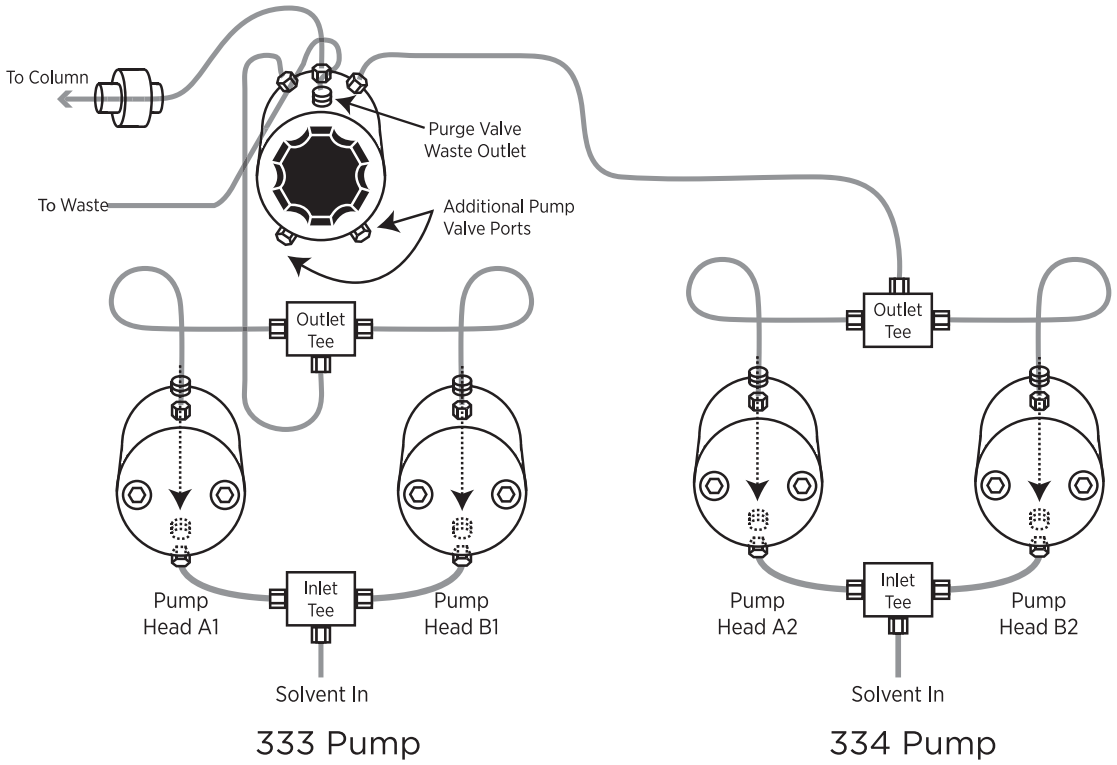


Figure 2
Hydraulic Connections between the 333/334 HPLC Pumps and the HPLC System (Side-by-Side Configuration)

PUMP HEADS

Each pump head has a solvent inlet port, a solvent outlet port, an inlet port to the rinsing chamber, an outlet port from the rinsing chamber, and a reciprocating piston.

The piston seal and the bellows are inside the pump head. The solvent inlet port and the solvent outlet port are fitted with connectors containing the check valves. All of these items can be serviced by the user.

The pump heads are pre-mounted directly onto the tops of the driving mechanisms, which have the same axis as the piston motors, so pump heads may be changed with relative ease. The pump heads must be dismounted for routine servicing purposes (for example, changing a piston seal).

Figure 3
H3 Pump Head

PPMM (333 PUMP ONLY)

The PPMM combines three functions:

- A static mixer to blend up to three solvents at high pressure.
- A built-in transducer that detects the pressure in this part of the hydraulic circuit.
- A purge valve which is used to manually direct the mobile phase towards the outlet filter or divert to the drain.

OUTLET FILTER (333 PUMP ONLY)

The outlet filter protects the injection valve and the column.

The outlet filter can be replaced by the user.

Control Panels

FRONT PANEL

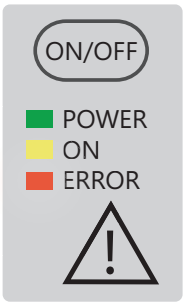


Figure 4
333 HPLC Pump Front Control Panel and Standby Panels

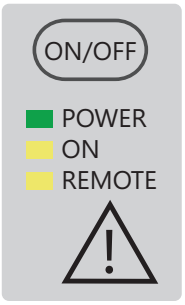


Figure 5
334 HPLC Pump Standby Panel

The 333 Pump features a front control panel with a display screen, soft keys, and fixed function keys. The 334 Pump does not have a front control panel and must be controlled by the 333 Pump or by TRILUTION® LC Software. Both pumps utilize standby panels to control power or indicate errors.

REAR PANEL

All electrical connections are made on the rear panels. The rear panel of the 333 Pump houses a main power switch, 14-pin contact output port, 10-pin contact input/output port, and GSIOC ports. The rear panel of the 334 Pump houses the main power switch, a GSIOC port, unit ID selector, and baud rate selector. The pumps have two ON/OFF switches: one on the power receptacle on the rear panel and the other on the front panel above the LEDs.

Figure 6
ON/OFF Switch on Power Receptacle

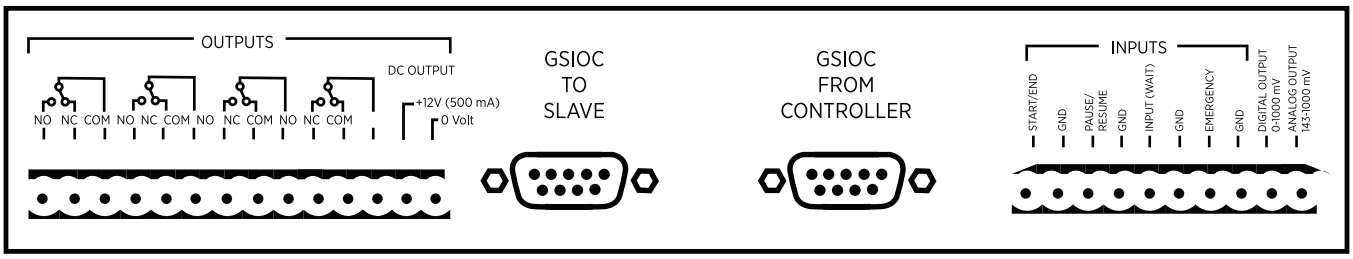


Figure 7
333 HPLC Pump Rear Panel Diagram

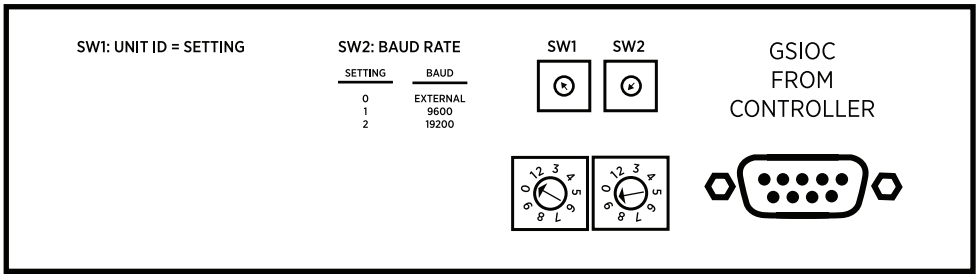


Figure 8
334 HPLC Pump Rear Panel Diagram

Unpacking

The pump is delivered with most major components already assembled. Keep the original container and packing assembly so the unit may be shipped safely, if necessary.

Carefully unpack the pump and its accessories from the carton.

⚠CAUTION **Because of their weight, you should take special care when handling the large cartons. Pump modules are heavy and should be lifted from the carton with care, by two people. Instructions describing the unpacking procedures can be found on and in the carton.**

It is necessary for two people to lift either the 333 Pump or 334 Pump out of the box, using the straps provided. The 333 Pump weighs approximately 33.1 kg (73 lbs.). The 334 Pump weighs approximately 29 kg (64 lbs.). To avoid personal injury and for general safety, if moving or lifting the system, always get another person to assist you. Always follow local health and safety regulations.

Standard Equipment

After the instrument and the accessories have been unpacked, you should have the following:

- 333 Pump and/or 334 Pump with H3 Pump Heads
- An accessory kit that includes:
  - Solvent Bottle Tray
  - GSIOC Cable (334 Pump only)
  - Wrenches
  - Glass Bottle
  - Tubing and Fittings
  - Bellows Mounting Tool
  - Extra Fuses
  - Power Cord
  - Terminal Block Connectors (333 Pump only)
  - Plumbing Kit for Piston Rinsing Chambers

DOCUMENTATION

Documents are provided on the supplied *333/334 HPLC Pumps Documentation USB*.

Optional Accessories

- Column Holder
- Manual Injection Valve Holder

For part numbers and installation instructions for the optional accessories, refer to the dedicated appendices.

Technical Specifications

Please be aware of the following before operating the pump.

NOTICE **Changes or modifications to the pump not expressly approved by Gilson could void the warranty.**

This instrument complies with part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) This instrument may not cause harmful interference, and (2) this instrument must accept any interference received, including interference that may cause undesired operation.

Shielded cables must be used with the pump to ensure compliance with the FCC Class A limits.

333/334 HPLC Pumps

Specification	Description
Pump Type	Single Solvent Pump
Hydraulic System	Reciprocating Piston Pump
Pump Head	H3: Up to 200 mL/min
Flow Rate	**Range - Single Pump** H3: 0.2–200 mL/min **Range (Recommended) - Two Pumps** H3: 2–200 mL/min **Increment** 0.01 mL/min
Flow Accuracy	± 2%
Flow Precision	≤ 0.7% RSD
Gradient	**Solvents** Two **Formation** High Pressure Mixing with Static Mixer as Part of Pressure, Purge, and Mixing Module (PPMM) **Composition Increment** 0.1%
Gradient Accuracy	± 2%
Gradient Precision	≤ 0.7% RSD
Operating Pressure	H3: 5–210 bar (70–3040 psi)
Compressibility Compensation	Settable Compensation range 0–2000 Mbar[1]
Piston Seal Wash	Pump Head Inlet and Outlet Ports to/from a Rinsing Chamber

TECHNICAL SPECIFICATIONS CONTINUED ON PAGE 17

333/334 HPLC Pumps

Specification	Description
Priming	Manual with Built-in Purge Valve via Control Software or Syringe
Liquid Contact Materials	316L Stainless Steel, Sapphire, Ceramic, UHMWPE, PTFE, Ruby, Titanium, FEP, PCTFE, ETFE For more information, refer to Liquid Contact Materials on page 93.
Control and Communication	**Communication** GSIOC (Gilson Serial Input Output Channel) **Inputs (333 Pump)** Four Digital Inputs; Start/End; Pause/Resume; Input (Wait); Emergency **Outputs (333 Pump)** Four Relay Outputs; 12V DC; One Output Channel for Pressure, Flow, or Composition; One Analog Output Channel for Pressure Sensor Reading **Software Control** TRILUTION® LC Software
Electrical	**Line Voltage** 90–260 VAC **Frequency** 50 or 60 Hz **Power Consumption** 600 W
Environmental	**Operating Temperature** 333 Pump: 10°C to 40°C 334 Pump: 4°C to 40°C **Operating Humidity** 15%–80% **Operating Altitude** Up to 2000 m (81 kPa or 604 mmHg)
Physical	**Dimensions (W x D x H)** 333 Pump: 26 x 41 x 50.7 cm (10.2 x 16.2 x 20 in.) 334 Pump: 26 x 41 x 38.7 cm (10.2 x 16.2 x 15.2 in.) **Weight** 333 Pump: 33.1 kg (73 lbs.) Shipping Weight: 36.3 kg (80 lbs.) 334 Pump: 29 kg (64 lbs.) Shipping Weight 33.1 kg (73 lbs.)
Contact **techsupport@gilson.com** for the methods and conditions that were used to obtain technical specifications.	

Customer Service

Gilson, Inc. and its worldwide network of representatives provide customers with the following types of assistance: sales, technical support, applications, and instrument repair.

If you need assistance, please contact your local Gilson representative. Specific contact information can be found at **www.gilson.com**. To help us serve you quickly and efficiently, please refer to **Repair and Return Policies on page 58.**

Chapter 2

INSTALLATION

IN THIS CHAPTER

This chapter describes the minimum connections required for operation (including procedures for preparing piston rinse lines). The pumps must be set up and installed in the order described in this chapter to avoid damaging the system.

TRILUTION® LC Software is the recommended controller for the pumps; however, manual control can be achieved by controlling the 334 HPLC Pump with the control panel atop the 333 HPLC Pump.

Both pumps are delivered with the main hydraulic components installed. Solvent bottles can be placed in the removable trays atop the pumps, on the bench, or on the floor. The pumps can be stacked to conserve bench space.

Electrical Connections

Plug the power cords into the pump and all associated devices before making the communication or hydraulic connections. Power is needed to prime the pump and piston rinse chambers prior to use.

Rear Panel

All electrical connections are made on the rear panel. Also present on the rear panel are the fan's ventilation slots, which must never be obstructed in any way.

LEGEND

NO = Normally Open
NC = Normally Closed
COM = Common

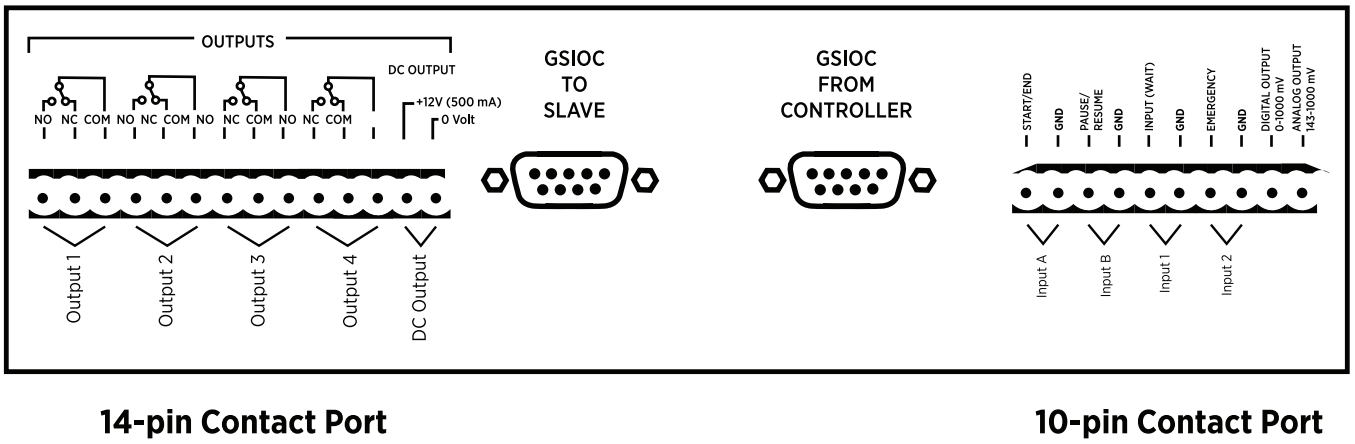


Figure 9
Rear Panel Communications Port and Legend for 333 HPLC Pump

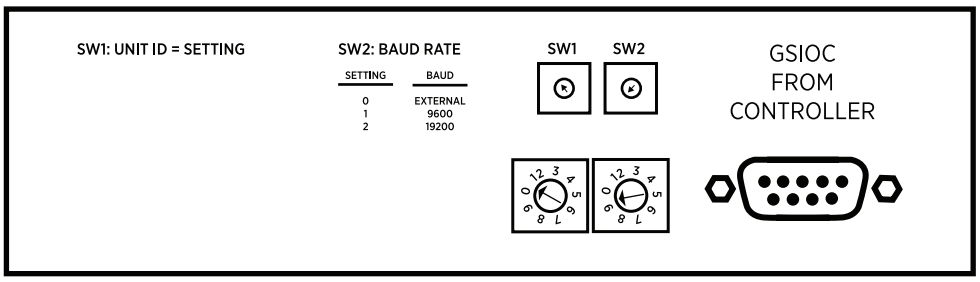


Figure 10
334 HPLC Pump Rear Panel Diagram

GSIOC ID

You must use the default values for a system where a 333 Pump is controlling other devices. By default, the controlling pump is assigned ID #1, the connected pumps are assigned IDs #2, and ID #3. If an injection pump is connected, use ID #4.

To change the GSIOC ID for the 333 Pump (0–63):

1. Switch on the pump and wait until it has initialized.
2. Press **Edit > Config > Misc.**
3. Key in the desired ID for the 333 Pump.
4. Press **ENTER.**

The GSIOC ID may be changed when the pump is being controlled from a computer. The configured ID on the pump must correspond to those set in the controlling software.

The GSIOC identification number (ID) that you set must be unique for each item of Gilson equipment.

The GSIOC ID (0–9) is set mechanically for the 334 Pump using the SW1 (left) selector on the rear panel.

Figure 11
GSIOC ID
(0–9)

BAUD RATE

You must use the default values when the 333 Pump is controlling other devices. When a 334 Pump is controlled by a 333 Pump, the baud rate must be set to 0 (External). Baud rates can also be changed to match the values of other sending and receiving devices.

For a 333 Pump, the baud rate is set as follows:

1. Switch on the pump and wait until it has initialized.
2. Press **Edit > Config > Misc.**
3. Press the **Down** arrow key.
4. Press **Change** until you see the desired baud rate (1200, 2400, 4800, 9600, 19200, or External)

The baud rate (9600, 19200, or External) is set mechanically for the 334 Pump using the SW2 (right) selector on the rear panel.

External clock control should be selected for all pumps running under computer control. Internal is used when the connected device does not provide a clock source, in which case you have to select an appropriate baud rate.

Figure 12
Baud Rate
0 = External
1 = 9600 Baud
2 = 19200 Baud

VOLTAGE SELECTION

The pump can be connected to an AC power supply of 110/120V or 220/240V. Automatic selection of the operating voltage takes place on the power supply board.

POWER CONNECTION

Plug the power supply cord that you received with the pump into the socket on the power receptacle (standard 3-pin connector) and to a suitable source of power.

CONTACT CONNECTIONS

Make any single wire contact connections for input/output signals, which may be needed for communicating with other equipment.

Contact Connections (Input & Output)

The input and output contact functions are shared between two terminal blocks (14-pin and 10-pin) situated on the pump's rear panel.

NOTICE **The maximum input voltage for any electrical device connected to each input or output contact is 48 V.**

In order to connect signal wires to the terminal blocks, you must first fit the appropriate connector (as supplied in the standard accessories package). The 14-pin connector fits the left-hand socket and the 10-pin connector fits the right-hand socket. After gently pushing each connectors into its socket, you can connect wires to the appropriate terminal.

To connect a wire to the terminal you should strip back the wire's protective covering by 5 mm, undo the terminal's fixing screw, push the bared wire into the corresponding hole in the terminal block, and then tighten the fixing screw using a small screwdriver.

Powering an External Relay

A 12 V (500 mA) DC supply is available at the right-most pair of pins of the left-hand terminal. This supply can be switched manually from the control panel or automatically from a Method Program by connecting it to one of its neighboring outputs.

Output Signals

Four two-way relay-type outputs are available via the left-hand socket. Each of these outputs is electrically isolated from the others and from ground. Switching from the 'normally closed' to the 'normally open' position takes place under software control (see **Appendix D | Front Panel Control on page 67**) at pre-programmed times. You can program these contacts to 'Open', 'Close', or 'Pulse', for a specific duration.

You connect one wire from the receiving device to the common terminal of Output # 1 (2, 3, or 4) and a second wire to either the corresponding 'normally open' or 'normally closed' terminal, depending on the requirements of the receiving device.

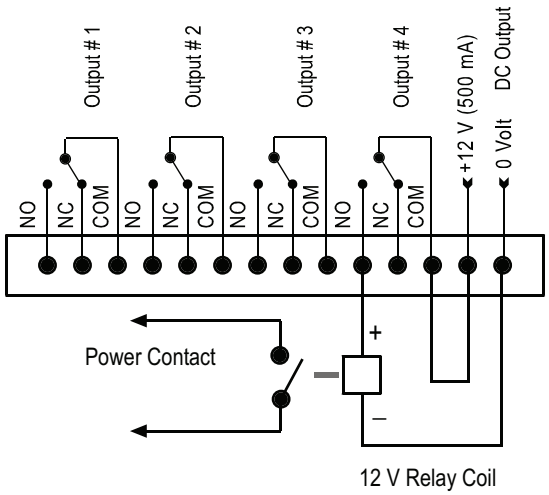


Figure 13
14-Pin (Output) Contact Connections

An output (pin # 9) is available on the right-hand socket for feeding a recorder or a PC (via a suitable interface). This output, which comes from the pump's microprocessor, can be configured to follow pressure, flow, or composition (%A, %B, or %C). It is labeled 'Digital' but outputs a series of discrete signals (0–1 V) via the digital-to-analog converter.

An output (pin # 10) is available on the right-hand socket for feeding any suitable recorder or a PC (via a suitable interface). This analog output, which comes directly from the sensor, is for pressure only (0.142–1 V for 0–60 MPa).

Input Signals

Four pairs of terminals of the right hand socket are dedicated to specific software-related input functions:

START / END
PAUSE / RESUME
INPUT (WAIT) (Input # 1)
EMERGENCY (Input #2)

You connect a pair of wires from an output (e.g., relay- type) of the external device, to a pair of terminals, for each of the functions that you want controlled externally.

Except for WAIT, to activate one of these inputs, you must change the state (close or open) of an output on the external device.

START/END

An external relay contact, connected to this input, may be used to start (OPEN ➔ CLOSE) and stop (CLOSE ➔ OPEN) program.

PAUSE/RESUME

An external relay contact, connected to this input, may be used to pause (OPEN ➔ CLOSE) and resume (OPEN ➔ CLOSE) the Method Program currently running. The pause may be configured with or without flow.

With flow means that pumping continues at the rate and composition that existed at the time the pause was initiated; when 'resume' is signaled the Method Program continues from the same point.

INPUT (WAIT) (Input # 1)

An external relay contact, connected to this input, may be used to signal to the Method Program currently running the 333 Pump that the external equipment (e.g., fraction collector, sampling injector) is ready.

EMERGENCY (Input #2)

Closing an external relay contact, connected to this input, may be used to signal to the pump software to start a Safety Program file (file 24).

Cabling for TRILUTION LC Control

Plug one end of a GSIOC cable into the port marked GSIOC FROM CONTROLLER. Plug the other end into the GSIOC port of an interface module (or other controlling device, such as a Gilson liquid handler), and then connect an RS-232 cable from the interface module (or other controlling device) to the computer.

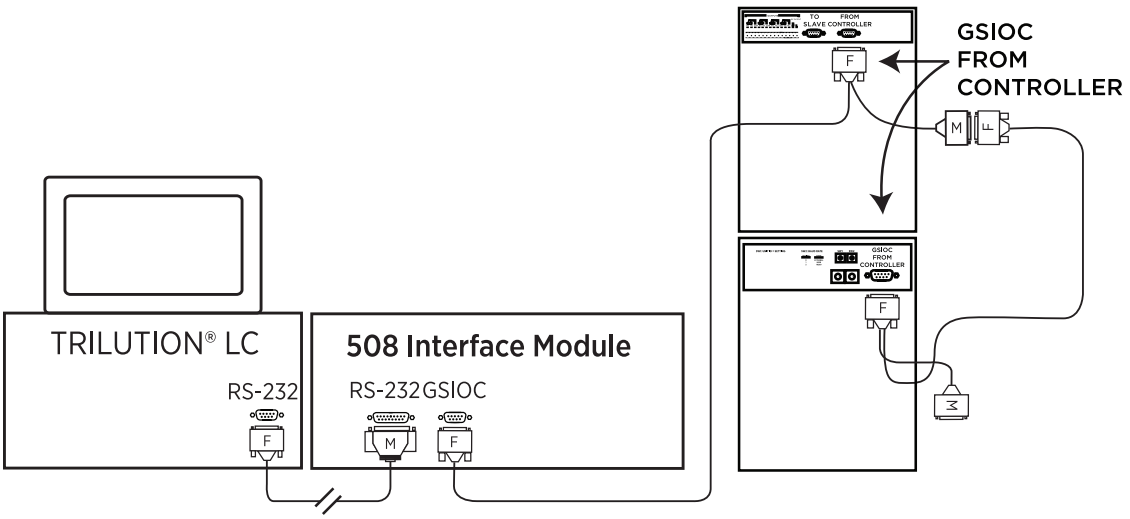


Figure 14
Cabling Connections with TRILUTION® LC Software as Controller

Cabling for Control from 333 Pump

The 333/334 Pumps connect with the GSIOC to Slave (333 Pump) and GSIOC from Controller (334 Pump) ports. For the third and subsequent instruments, connect another cable between the male end of the GSIOC cable to the female end (with only one cord) of another GSIOC cable, then out to additional instruments.

Daisy-Chaining the GSIOC Cables

Connect your instruments in a linear series. The first pair of instruments in the chain are connected using the female ends. For the third and subsequent instruments, connect another cable between the male end on the GSIOC cable and the GSIOC port on the next instrument. Thus, you can 'daisy-chain' the pumps to other Gilson GSIOC instruments.

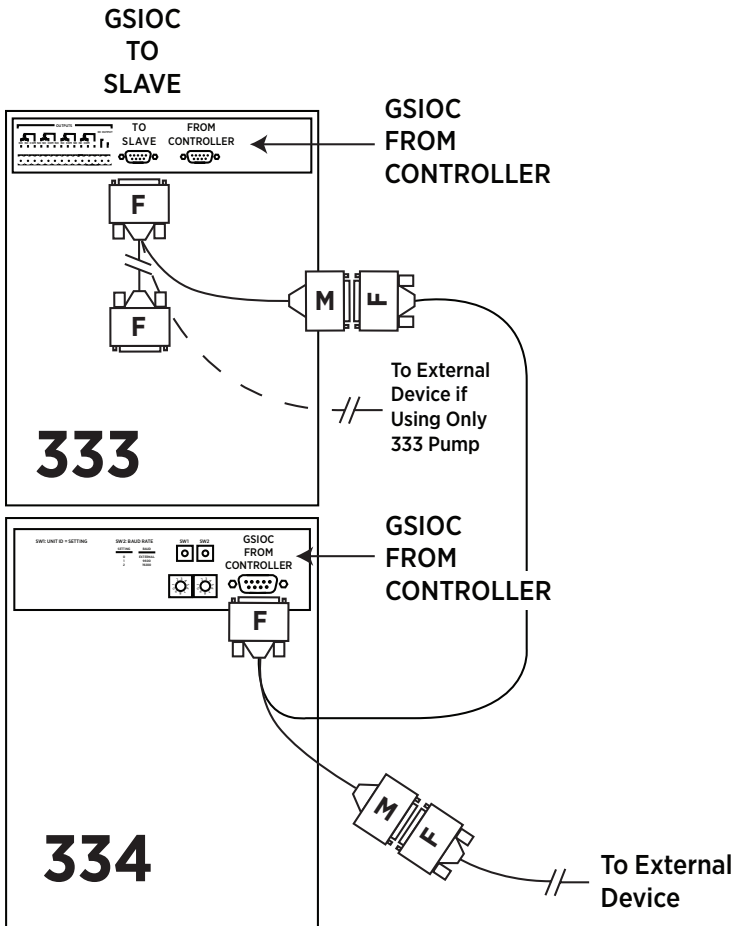


Figure 15
GSIOC Series Connection with 333 Pump as Control Device

Twin Pump Systems

Two pumps may be connected in parallel, thereby doubling the programmed flow rate. In theory, there is no limit to the number of pumps that can be connected in parallel, but the twin system with two liquid streams is the most practical. The hydraulic outlet tubings are connected together using a tee piece to increase the overall flow rate.

To set up 333/334 Pumps to operate in parallel, you must give each of the paralleled pumps the same GSIOC ID: the Master 333 Pump has an ID = 1 as does its twin; the Slave 334 Pump and its twin both have an ID = 2, and so on. The twinned pumps are connected together using GSIOC cables.

When operating a twinned system, the commands from the controlling device go to each pair of twins, so that they act in unison. The twinned pumps must have the same configuration as their master pumps.

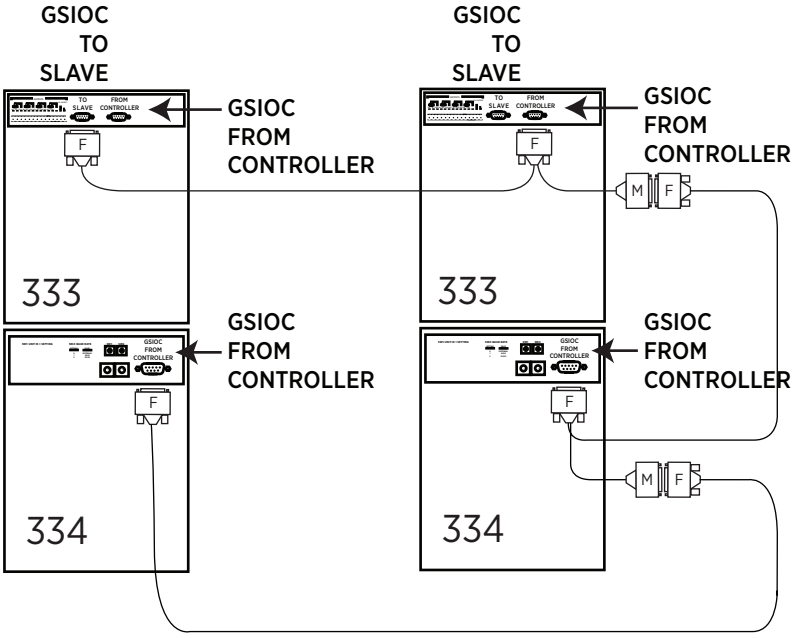


Figure 16
333/334 HPLC Pumps Used in Parallel

Positioning the Pumps

The 333/334 Pumps can be placed side-by-side or stacked to conserve bench space.

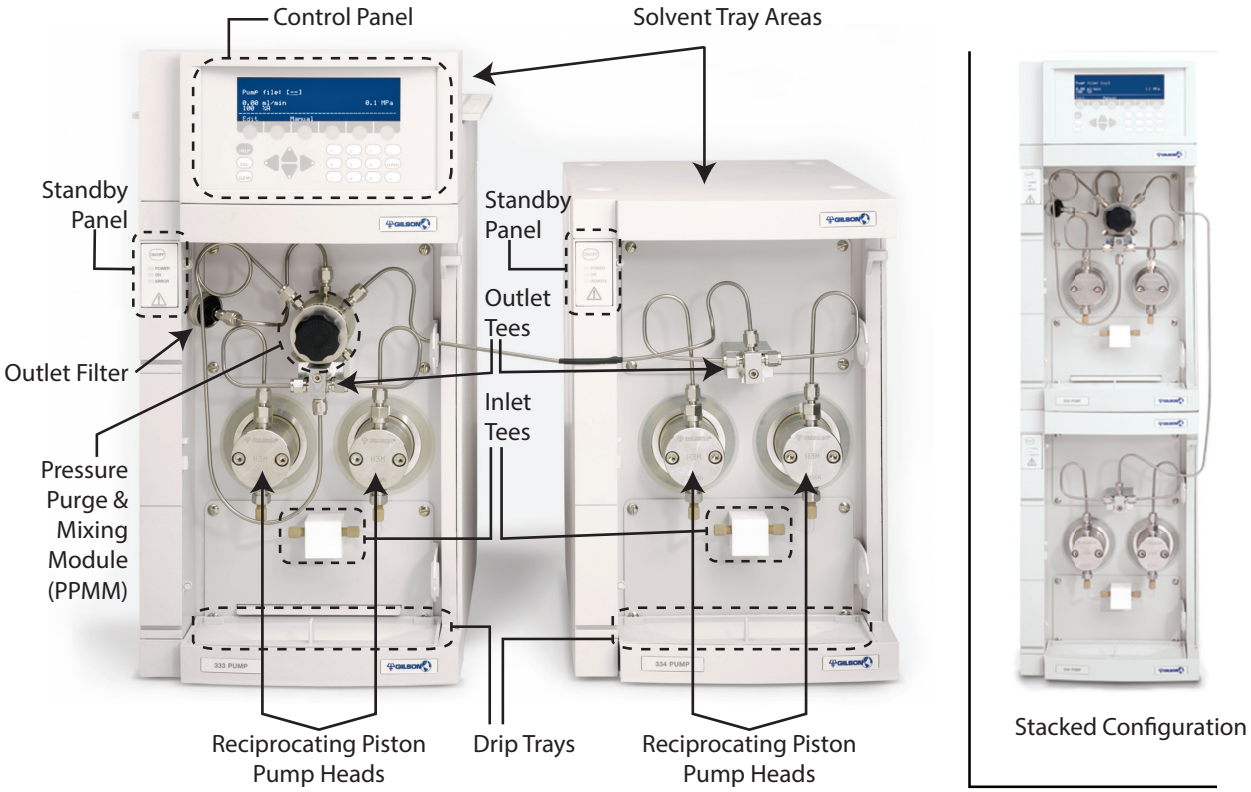


Figure 17
System Diagram for Paired 333/334 HPLC Pumps
(Side-by-Side Configuration on the Left; Stacked Configuration on the Right)

NOTE The tubing linking the two pumps goes through the tubing port, together with solvent and rinsing port tubing. To make it easy to install and to remove tubing, there is a fissure in the tubing port, which is accessed by rotating the tubing port until the fissure lines up with the opening in its housing.

NOTICE Pre-shaped steel tubing is used to connect pumps. Do not bend or attempt to reshape this tubing.

Side-by-Side Configuration

The tubing port of the 333 Pump should face the 334 Pump so that the steel tubing (part number 380133592) can properly feed from the pressure, purge, and mixing module (PPMM) on the 333 Pump to the outlet tee on the 334 Pump.

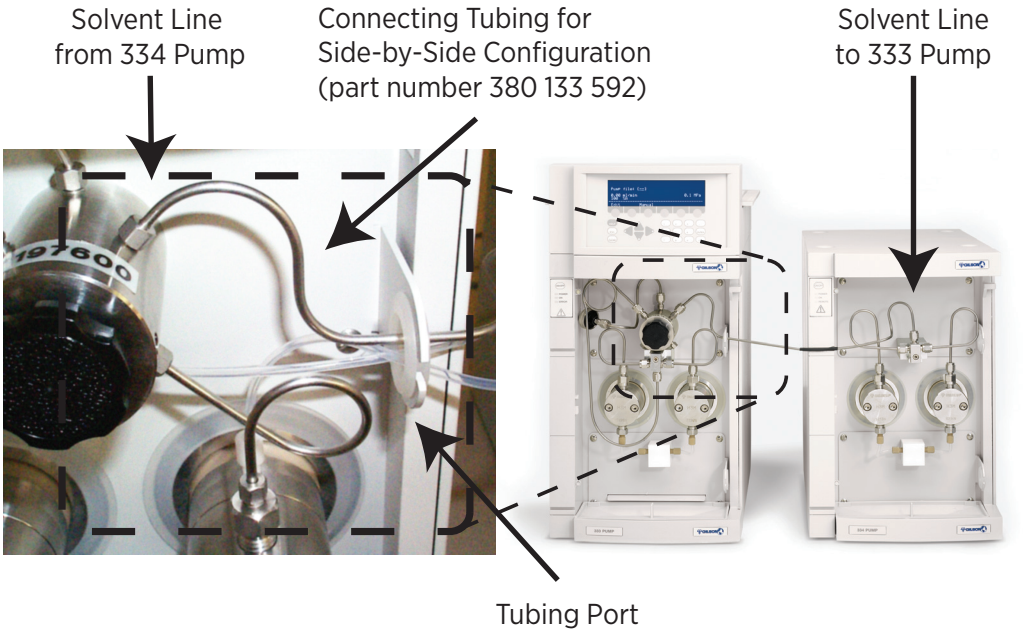


Figure 18
Side-by-Side Configuration with Tubing Port Detail

Vertical Stacking Configuration

Stack the 333 Pump on top of the 334 Pump to conserve bench space. The tubing ports on both pumps face the same side to allow the pre-shaped tubing (part number 380132582) to connect from the PPMM on the 333 Pump to the outlet tee of the 334 Pump.

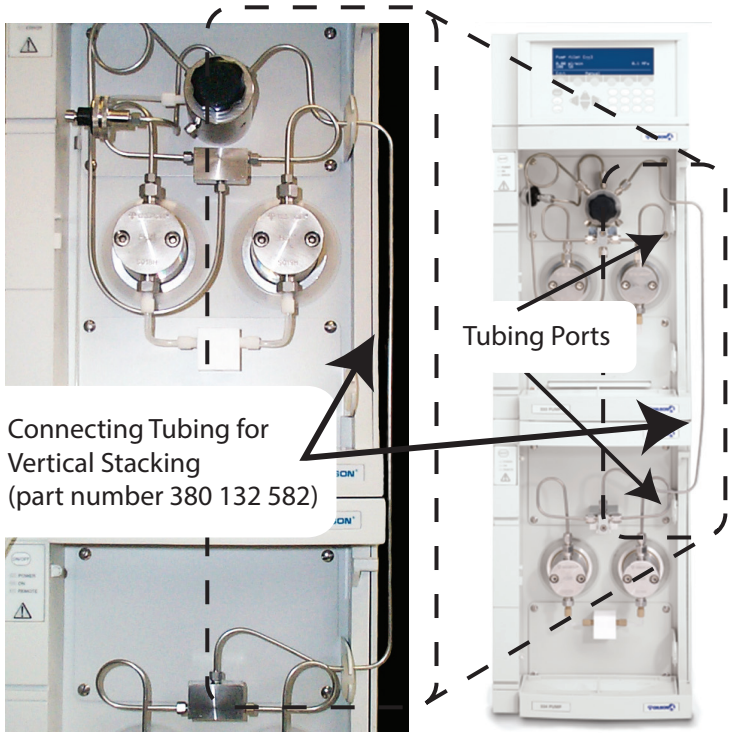


Figure 19
Vertical Stacking with Tubing Ports

Hydraulic Connections

Solvents flow through the inlet tees of the 333/334 Pumps before reaching the pump heads. Solvent flows out from the pump heads to the outlet tee, through the PPMM, and finally to the outlet filter. Additional solvents can be mixed in the PPMM via the additional ports on the bottom of the PPMM. Up to three additional solvents can be pumped into and mixed in the PPMM.

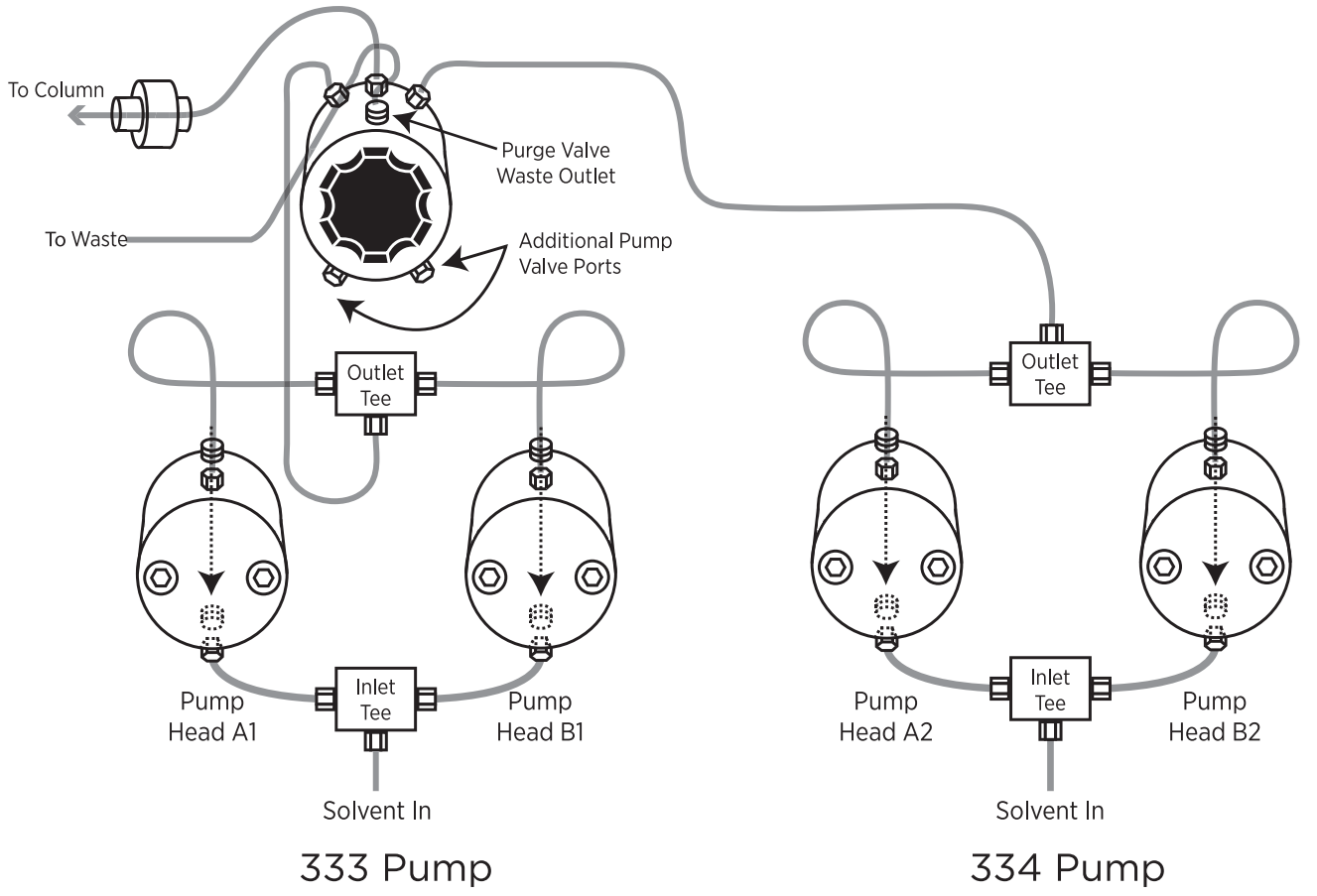


Figure 20
Hydraulic Connections between the 333/334 HPLC Pumps and the HPLC System (Side-by-Side Configuration)

Pump Heads

The pump heads are pre-installed. However, for routine servicing, you will need to remove the pump heads (refer to **Pump Head on page 41**).

Solvent Inlet Lines

For each solvent pump, the standard accessory package contains an inlet line fitted with a 20 µm inlet filter.

Connect the inlet line to the inlet tee. The filter goes in the solvent bottle.

Air entering the hydraulic circuit would adversely affect the flow rate. Make sure that all connectors are correctly seated and properly tightened.

To ensure the connectors are seated and properly tightened:

1. Remove the plastic plug from the inlet of each head.
2. Check that the reverse ferrule is correctly seated at the end of the tubing.
3. Connect the solvent inlet line to the inlet tee.

NOTICE

When screwing or unscrewing the white connector, secure the tubing with one hand and slide the connector down. Make sure the ferrule touches the female port while sliding the connector down to prevent the line from twisting.

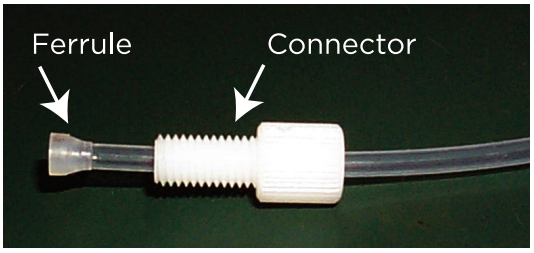


Figure 21
Ferrule and Connector Close Up

4. Check that the connectors are tight enough to prevent air from entering the hydraulic lines.
5. Thread the solvent line through the port on the door before placing the inlet filter in the appropriate solvent bottle.
6. Clip the solvent lines to the tray. Push the tubing into the fissure on the clip before placing it in the appropriate slot on the tray.

NOTE

Because of the larger diameter tubing, a different clip is supplied for the rinsing line.

7. Prime the solvent lines prior to operation. Follow the instructions provided in **Priming on page 34** for both TRILUTION LC and manual control.

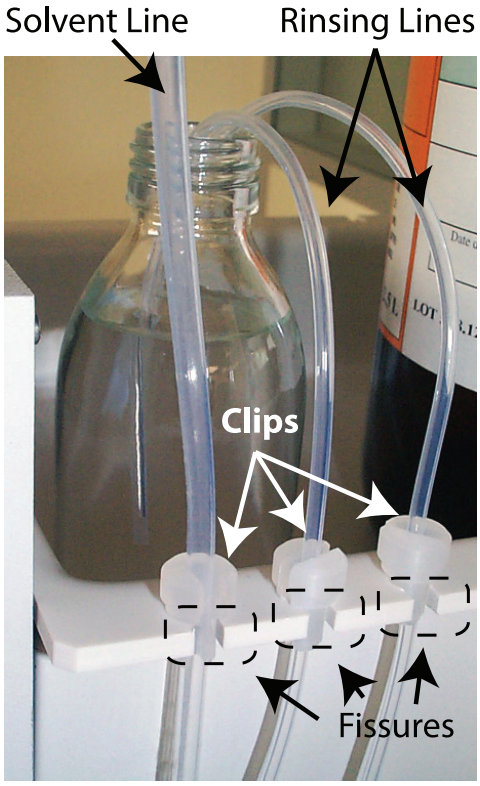


Figure 22
Solvent clips in Fissures

Drip Tray

A removable drip tray fitted to the pump is supplied.

The drip tray slots into the well at the bottom of the pump. Installation consists of lifting up the tray, fitting one end with a length of tubing. The other end of the tubing goes to the drain (or a suitable receptacle) via the drain tubing exit port. Installation is completed by replacing the drip tray.

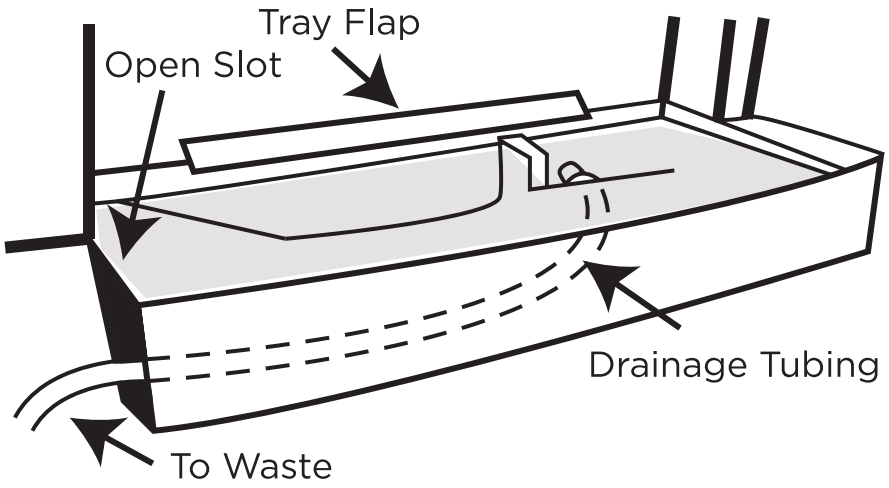


Figure 23
Drip Tray Diagram

Solvent Bottle Tray

The solvent bottle tray sits on top of the pump with its feet resting in the special recesses.

Figure 24
Solvent Bottle Tray installed on 333 HPLC Pump (Left) and 334 HPLC Pump (Right)

Piston Rinsing Chamber

When a pump is delivered, the inlet and outlet ports to the rinsing chamber are fitted with plugs, which prevent airborne particles from entering the ports. If you do not need to use piston rinsing, you should leave these plugs in place.

If the solvent is an aqueous solution containing more than 0.1 M of solute, which is solid in ambient conditions, then the piston should be rinsed continuously with water.

A plumbing kit should be installed for the piston rinsing chambers of both heads.

The plumbing kit consists of: two rinsing lines, which connect to the rinsing chamber inlets at the bottom of each head (the longer line connects to the left head) and two shaped purge lines, which connect to the rinsing chamber outlets at the top of each head.

For details about installing the piston rinse plumbing and the piston rinsing procedure, refer to **Piston Rinsing Procedure on page 36.**

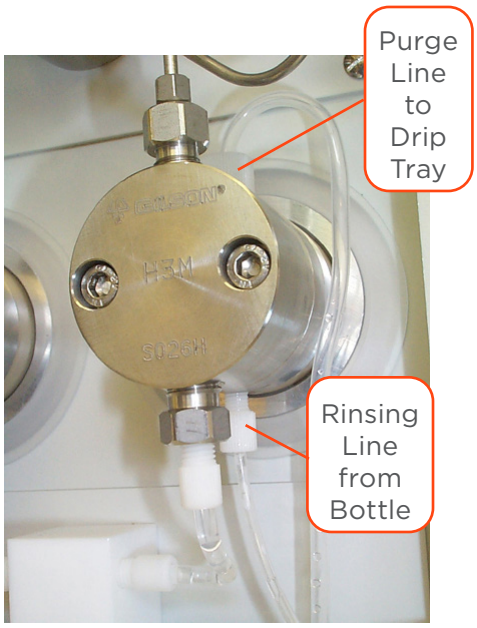


Figure 25
Close Up of Piston Rinse Lines

Chapter 3

OPERATION

IN THIS CHAPTER

TRILUTION® LC Software provides software control of the pumps for setup and operation. Refer to the *TRILUTION® LC Software User's Guide* for more information.

For information about front panel control of the pumps, refer to
Appendix D | Front Panel Control on page 67.

Front Panel and Startup

The pump has two ON/OFF switches: one on the power receptacle on the rear panel and the other on the front panel above the LEDs.

Powering the Pumps On

1. Press the switch on the power receptacle to the 'I' position; the 'Power' LED (green) on the front panel should light up; if it does not, check the fuses and power connections.
2. Open the pressure, purge, and mixing module (PPMM) valve by turning the knob on the PPMM fully counterclockwise to direct any flow to the purge outlet.
3. Press the ON/OFF button on the front panel; the ON indicator light should illuminate on the front panel and the display on the 333 Pump control panel should activate.

STARTUP SCREENS

After switching on a 333 Pump, the display briefly shows the **Initialization** screen, which displays the current software version. After a few seconds the **Startup** screen will appear.

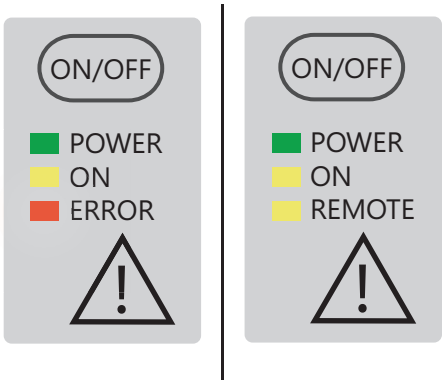


Figure 26
Front Panels with All LEDs Activated

Priming

Priming helps prevent the introduction of air bubbles into the system. It is recommended to prime the pump before using it for the first time, or if it has not been used for some time.

This is an essential step, which must be carried out before operating the pumps.

NOTICE	**Operating the pumps dry, even for a short time, can damage the equipment. Use a syringe to prime the pump if the pump does not self-prime within two minutes.**

NOTE	**Ensure that all plumbing connections have been made as described in INSTALLATION.**

Priming the Pump

1. Fill the solvent bottle(s) with degassed, high performance liquid chromatography (HPLC) grade solvent(s) and immerse the filter connected to the inlet tubing for each pump into the solvent.
2. Open the purge valve on the PPMM by turning the black knob fully counterclockwise to direct the flow to the atmospheric purge-outlet. Make sure the purge line is connected to the purge valve and directed to an appropriate waste receptacle.
3. Use the controlling software to run the pump at the maximum flow rate for the pump head. If self priming is achieved, skip to step 9. If the pump does not self-prime within two minutes, stop the pump and continue with manual priming.
4. Disconnect the waste tubing from the purge valve on the PPMM and then connect a syringe.
5. Draw liquid into the syringe. It is likely that it will first draw air, but then liquid droplets should start to appear.

6. Disconnect the syringe from the waste outlet.
7. Reconnect the waste tubing to the purge valve on the PPMM and place the other end in a waste container.
8. Run the pump to dispense at a suitable flow rate.
9. When no bubbles can be seen at the waste tubing, stop the pump to end the priming procedure.
10. Turn the knob purge valve on the PPMM all the way to the right (clockwise) to close the outlet to waste and direct flow to the outlet filter.

Depending on your solvents, ensure that the rinse plumbing kit is installed. Refer to **Piston Rinsing Procedure on page 36.**

Priming Using TRILUTION LC Control

1. Fill the solvent bottle(s) with degassed, high performance liquid chromatography (HPLC) grade solvent(s) and immerse the filter connected to the inlet tubing for each pump into the solvent.
2. Open the purge valve on the PPMM by turning the black knob fully counterclockwise to direct the flow to the atmospheric purge-outlet. Make sure the purge line is connected to the purge valve and directed to an appropriate waste receptacle.
3. Open a method in TRILUTION LC that includes the pump(s) in its configuration.
4. Select **Run**.
5. Select the **Manual Control** icon. The **Manual Control** window appears.
6. Select the **Manual Run Gradient** icon. The **Manual Run Gradient** window appears.

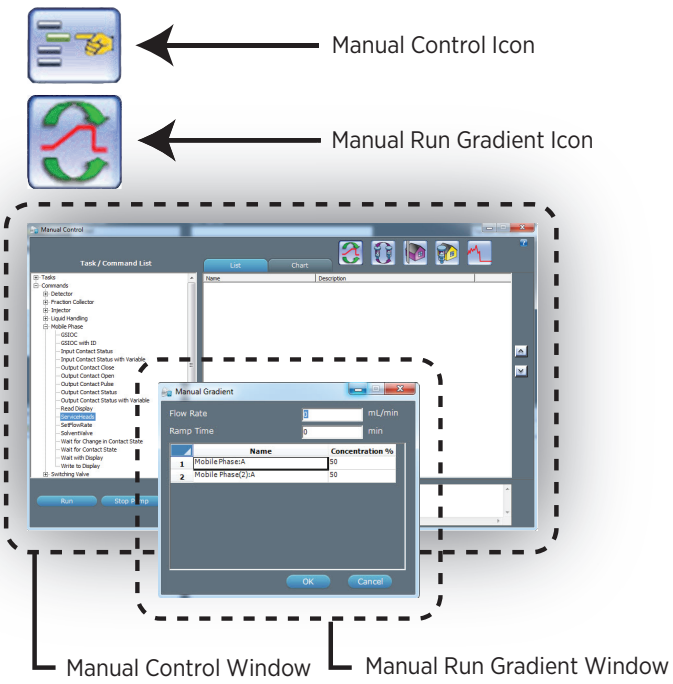


Figure 27
Priming with TRILUTION® LC Software

7. Enter a flow rate (maximum flow rate for the pump head, for example) into the **Flow Rate** field.
8. Set the **Concentration%** for the first mobile phase instrument to **100**. Values for any additional mobile phase instruments automatically set to 0.
9. Select **OK** to confirm. Allow the solvent to flow through the purge line until there are no air bubbles present.
10. Repeat steps 5–9 for any additional mobile phase instruments. This will ensure that each pump is primed individually.
11. Stop the pumps by selecting **Stop Pump** on the **Manual Control** window.
12. Close the purge valve on the PPMM.

Piston Rinsing Procedure

PLUMBING SETUP

1. Fill the glass bottle (part number 54350403) with distilled water and push the open end of each rinsing line through the bottle's pierced cap, to between 1 to 2 cm of the bottom.
2. Clip the rinsing lines to the tray. Each line is first attached to a clip by pushing it gently into the fissure, and then the clip is pushed into a slot on the tray.

PRIMING OPERATION

1. Ensure that each purge line is closed (turn each connector fully counterclockwise until finger tight), then press **Manual**, **Prime**, **Start** (or pump at an appropriate flow rate—the maximum flow rate, for example). After about 20 seconds, water should have sufficiently filled the rinsing line, even though some air is also present.
2. Stop the pump and then undo both purge line connectors. Water will run from each purge line into the drip tray; the rinsing lines and rinsing chambers will fill with water. When you see bubble-free water in the rinsing lines, and that air is no longer exiting from the head via the purge lines, close both purge lines. In use, water from the small bottle rinses the piston and although it is a closed circuit, you may need to change the water from time to time and also repeat this procedure.

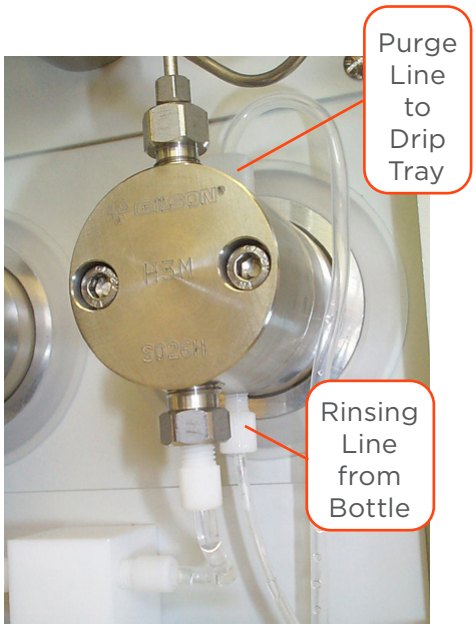


Figure 28
Close Up of Piston Rinse Lines

NOTE

To get the initial flow of water to reach the pump head, it may be necessary to use a priming syringe (part number 36460058) to pull excess air from the rinse line. Opening and closing the purge line connectors can also encourage initial rinse water flow.

NOTICE

Over time the buffer concentrate will dilute into the water bottle via entropy and laminar flow. To prevent sediment build-up in the piston chamber, change the water and clean the bottle.

Pump Operating Parameters

Refill Time

Refill time is the duration of the piston return stroke.

The refill time can be adjusted from 125–1000 ms.

Normally, you can use the default value of 125 ms. If cavitation occurs, then use a higher value.

For volatile and non-degassed solvents, better performance may be achieved by entering a higher value, up to the limits shown.

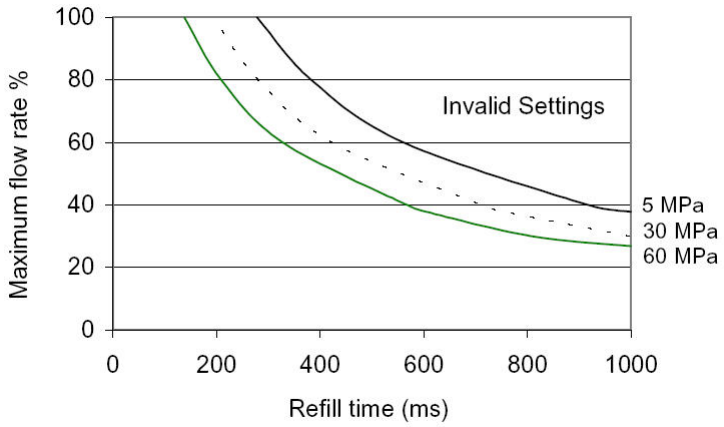


Figure 29
Refill Time

Inlet Pressure

Inlet pressure is the pressure at the inlet to the pump head.

The inlet pressure can be adjusted from 0–50 MPa.

Compressibility

Compressibility is a compensation parameter for solvent compressibility.

The compressibility can be adjusted from 0–2000 $Mbar^{-1}$. The standard compressibility values are 34 for water, 162 for methanol, and 180 for acetonitrile.

Chapter 4

MAINTENANCE

IN THIS CHAPTER

To obtain optimum performance and maximum life from the pump, it is important to keep the instrument well-maintained.

User maintenance is generally limited to the following:

- Cleaning check valves and filters (clean outlet filter after changing piston seal)
- Replacing parts subject to wear and tear in each pump head: piston seal, check valves, and piston assembly
- Run-in the pump head and/or seal

Maintenance Overview

General guidelines for the periodic replacement of the 'wear parts' are indicated in the table below, according to the amount of use: intensive, regular, or occasional. This schedule should be regarded as a guide; changes in performance, or visible leaks, give an indication that a part should be changed.

Component	Intensive (168 h/week)	Regular (40 h/week)	Occasional (10 h/week)
Piston seals	4.5 months (3000 h)	1–1.5 years (2500 h)	2 years (1000 h)
Check valves	7 months (4500 h)	1.5 years (3000 h)	3 years (1500 h)
Piston assembly	9 months (6000 h)	2–3 years (5000 h)	4 years (2000 h)

NOTE These recommendations are based on the assumption that the pump is working at half its maximum flow rate and pressure.

⚠CAUTION To prevent injury, observe good laboratory practices when handling solvents. Know the physical and chemical properties. Refer to the Material Safety Data Sheets for the solvents used.

Cleaning

Keep the pumps clean for peak performance. Always turn the power off to the pumps before cleaning. Wipe the pumps with a soft cloth dampened with a mild detergent and disinfect as needed.

Pump Head

NOTICE

Before the pump head can be physically removed from the pump, you must disengage the pump head from the pump motor using the dismount procedure.

Removal of the pump head is required for all maintenance described in this chapter. Refer to the instructions in this section when removing or installing the pump head.

Do not autoclave the pump head.

Dismount Pump Heads

DISMOUNTING WITH TRILUTION LC

1. Open a method that includes the pump in its configuration.
2. Select **Run**. The **Application Run** window will appear.
3. Select the method name from the **Method Configuration** drop-down box.
4. Select the **Method Configuration** icon.
5. Select the **Manual Control** icon. The **Manual Control** window will appear.
6. Click **'+'** next to the **Commands** field to expand the list, and then again next to **Mobile Phase**.
7. Drag and then drop the **ServiceHeads** command into the **List** space. The **ServiceHeads Properties** window appears.
8. Change the **MountHeads** value to **False**.
9. Select **OK** and then select **Run**.
10. Wait for the pistons to fully retract.

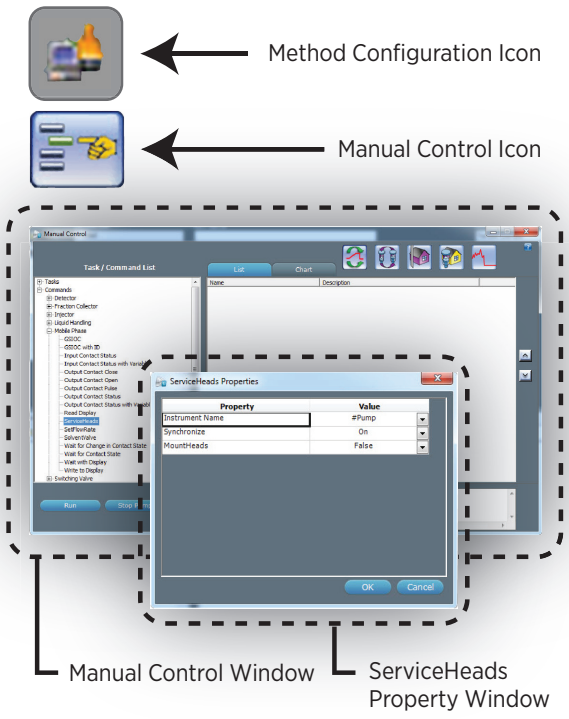


Figure 30
Manual Control Windows and Icon

NOTE

Repeat steps 1-10 to mount the pump heads, but select TRUE for the MountHeads property in the ServiceHeads command.

MANUAL DISMOUNTING PROCEDURE

1. Open the purge valve on the PPMM by turning the black knob counterclockwise.
2. Switch off the pump and disconnect it from the power supply.
3. Remove the lower back panel (to which the fan is attached) by removing the retaining screws. The mounting screws for both heads are now visible.
4. Use the 3 mm Allen wrench to turn the mounting screw in the clockwise direction, until you reach a stop (you will hear a 'thump' as you hit the stop). At this point, the piston engaging mechanism will be withdrawn to a maximum distance into the interior of the pump motor. Turn the wrench two turns counterclockwise.

Figure 31
Removing the Lower Rear Panel

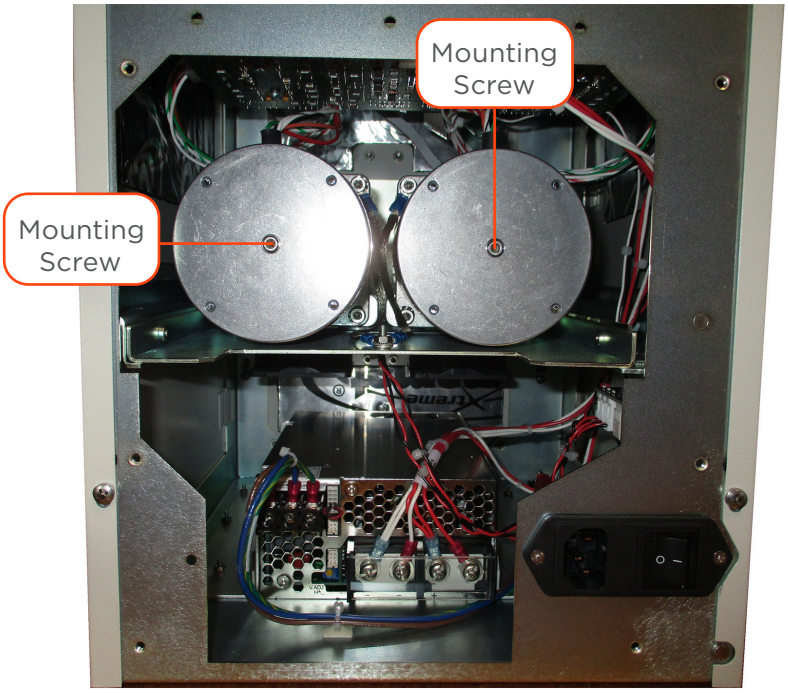


Figure 32
Loosen Mounting Screws

Remove Pump Head

1. Disconnect all solvent lines from the pump head.
2. Remove the two screws securing the pump head with the supplied 5 mm Allen wrench. Support the pump head with your other hand while loosening the screws, alternating equally between the two. Moderate force may be required to remove the pump head.

Figure 33
Loosening Pump Head Screws

Disassemble Pump Head

The following steps should be carried out on a clean, dry surface. No special tools are required.

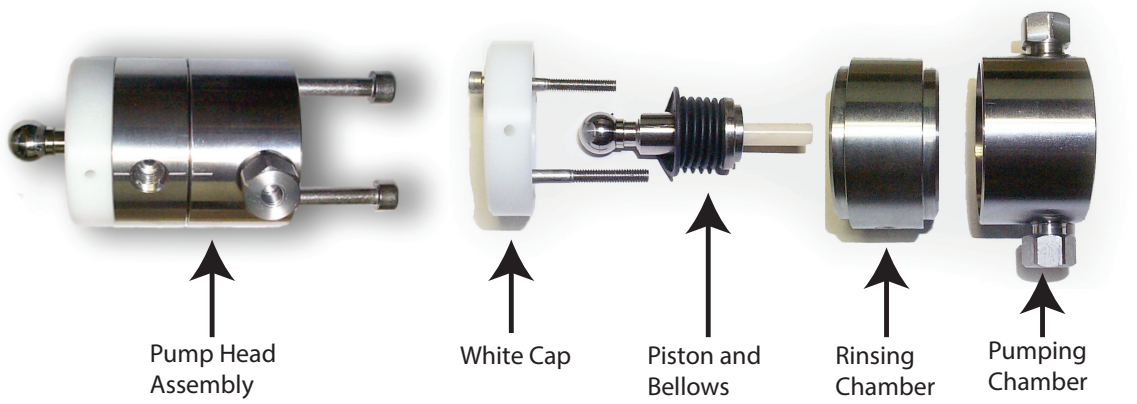


Figure 34
Complete Pump Head Assembly (left) and Disassembled Pump Head (right)

1. Undo the two retaining screws using a 3 mm Allen wrench (part number 4320302).

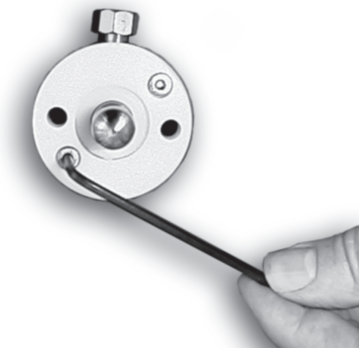


Figure 35
Initial Steps for Pump Head Disassembly

2. Remove the white cap and retaining screws.

Figure 36
Remove the White Cap and Retaining Screws

3. Pull the piston and bellows out of the body of the pump head.

Figure 37
Pull the Piston and Bellows Out of Pump Head

4. Pull apart the two halves of the pump head using a slight twisting motion.

Piston Seals

The nature of the liquid pumped is a major factor affecting longevity of the piston seal. The piston seal consists of a seal ring made of either graphite reinforced PTFE (black) or UHMWPE (yellow) and a spring made of Titanium (wire). It must be changed whenever a piston seal leak occurs.

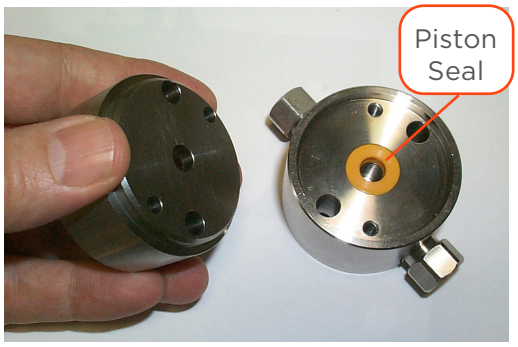


Figure 38
Piston Seal Location

Choose piston seals based on the solvents used:

- The black, polytetrafluoroethylene (PTFE) piston seals are generally best suited for use with tetrahydrofuran, hexane, methylene chloride, carbon dioxide, and low polarity solvents (part number 38013253 for H3 pump head).
- The yellow ultra-high-weight polyethylene (UHMWPE) piston seals are generally best suited for use with water, aqueous solvents, alcohols, acetonitrile, and polar solvents. Pump heads are supplied from the factory with the yellow seal (part number 38013261 for H3 pump head).

The piston, bellows, and spacer can also be replaced while the pump head is disassembled.

Replace a Piston Seal

The following steps should be carried out on a clean, dry surface. No special tools are required. These instructions assume that the pump head has been dismounted, removed and disassembled per the instructions in the previous section.

1. Remove the piston seal by carefully levering it out with the end of the Allen wrench. Discard the seal.
2. Clean out any debris from the seal recess using a soft cloth or an air line.
3. Push a new seal carefully into the recess.
4. Initialize and run-in the new seal(s) as described in **Run-In Procedure on page 52.**

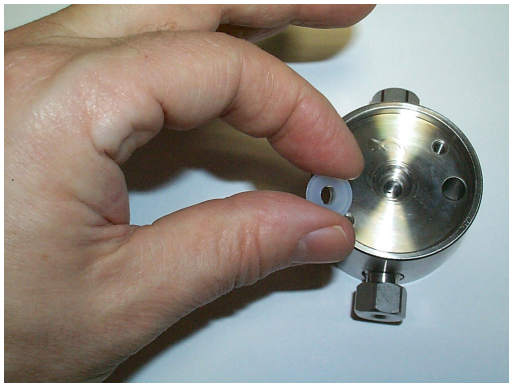


Figure 39
Piston Seal Removal

Piston and Bellows

The following steps should be carried out on a clean, dry surface. These instructions assume that the pump head has been dismounted, removed and disassembled per the instructions in the **Pump Head** section in this chapter.

Inspect the bellows for damage. If the piston bellows are cracked, damaged, corroded, or degraded by use, follow these instructions for removal and replacement.

> **NOTE** **Although piston shafts vary in size, the procedure for bellows replacement is identical.**

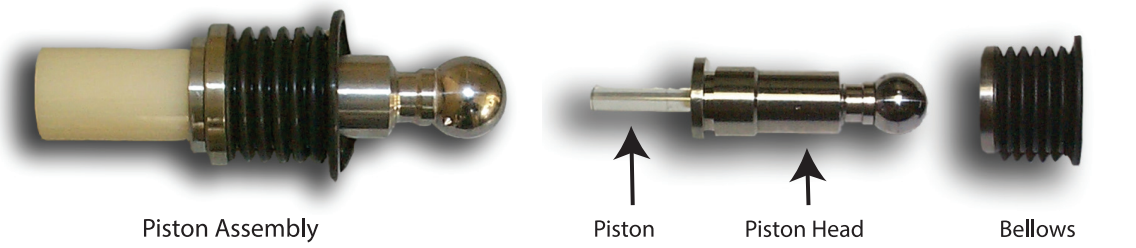


Figure 40
Intact H3 Piston Assembly (left) and Disassembled H1 Piston Assembly (right)

Remove Bellows

Use your thumbs to remove the bellows; moderate force is required. When removing the bellows, support the piston by the head, not by the shaft.

Figure 41
Removing the Bellows

Figure 42
Individual Components (Piston and Bellows)

Replace Bellows

1. Insert the bellows tool (part number 38013235) into the open end of the bellows. This tool helps ensure that the orifice at the end of the bellows remains open while you work.

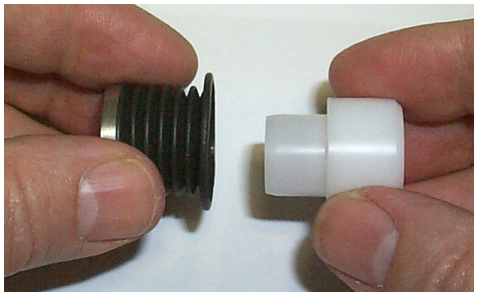


Figure 43
Inserting the Tool

Figure 44
Opening the Orifice

2. Place the tool and bellows on a firm, flat surface.
3. Push down on the metallic ring using four fingers until the orifice grips the end of the tool on its own. The tool, which keeps the end of the bellows open, enables the bellows to be slid back into position.
4. Pull on the metallic ring at the end of the bellows until it can go no further; moderate force is required. When refitting the bellows, support the piston by the head, not by the shaft.

Figure 45
Refitting

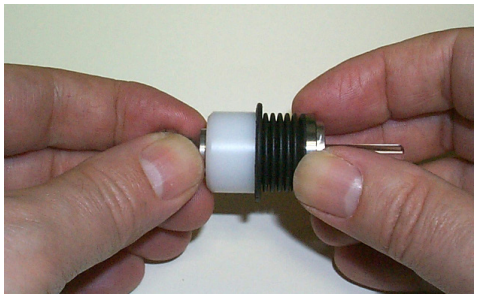


Figure 46
Pull on the Metallic Ring

5. Remove the tool and check that end of the bellows (with the metallic ring) is correctly seated against the piston collar.

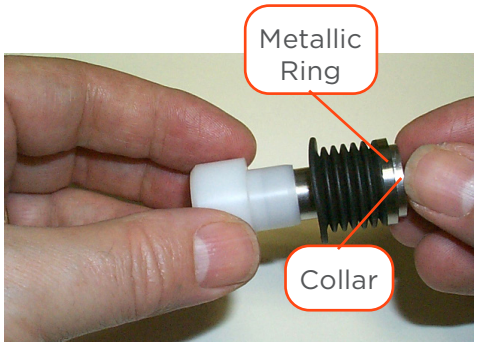


Figure 47
Metallic Ring and Piston Collar Seating

Piston Rinsing Chamber

Change the rinsing liquid at least once a week.

For details, refer to **Piston Rinsing Procedure on page 36.**

Reassemble Pump Head

The following steps should be carried out on a clean, dry surface.

1. Push the two halves of the pump head (rinsing and pumping chambers) together with a slight twisting motion.
2. Turn the rinsing chamber relative to the pumping chamber, until holes for the pump head retaining screws are aligned (because of the asymmetry of the holes there is only one correct position).
3. Check that the marks on the rinsing and pumping chambers are aligned.
4. Refit the bellows to a clean piston and insert the assembly fully into the rinsing chamber.

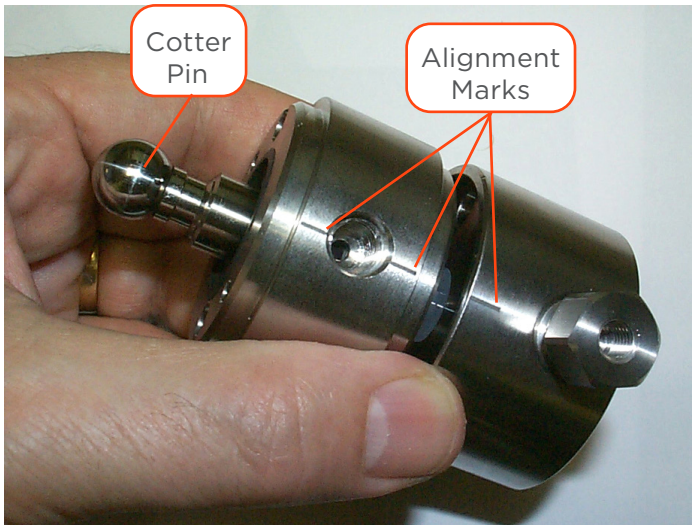


Figure 48
Correct Alignment of Chambers and Pistons

Figure 49
Correctly Seated Piston Assembly

5. Turn the piston until the small cotter-pin at the rounded end of the piston is in line (approximately) with the alignment marks on the pump head.
6. Refit the white cap, reinsert the screws, and progressively tighten them, making sure that the alignment marks are still correctly aligned (do not overtighten the screws).

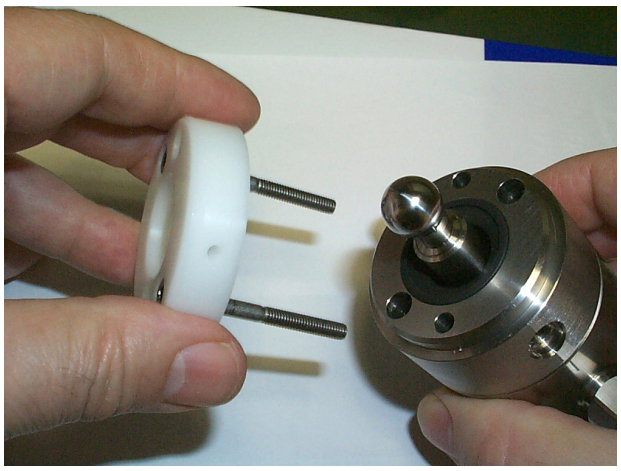


Figure 50
Refitting the White Cap

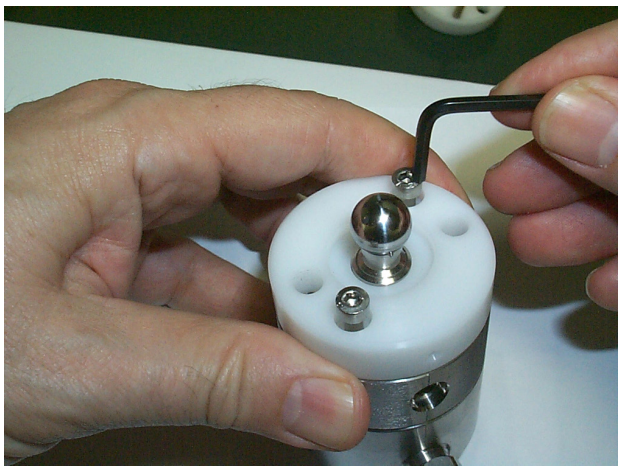


Figure 51
Tightening the Screws

NOTICE **Do not turn the piston after the head is reassembled, because it is possible to damage the bellows.**

Check Valves

The check valves are supplied as cartridges: one for the inlet connector and one for the outlet connector. The two check valves, inlet and outlet, should be cleaned periodically to ensure reliable flow rates. Reliable flow rates will be achieved only if the check valves are kept in good operating condition by proper care and maintenance.

NOTE **Check valve cartridges are identical for H3 pump heads, but the connectors for inlets and outlets are different for each. Because the dimensions of the threaded parts of the connectors are different, neither the connectors nor the cartridges are interchangeable.**

The check valves must not be disassembled into sub-components. No check valve sub-component is available from Gilson.

Cleaning a Check Valve

This procedure is carried out with the check valves installed:

1. Open the purge valve on the PPMM to the drain position. Make sure the purge line is connected and directed to an appropriate waste receptacle.
2. Pump isopropanol (provided the current solvent and isopropanol are miscible).

⚠CAUTION **To prevent injury, observe good laboratory practices when handling solvents. Know the physical and chemical properties. Refer to the Material Safety Data Sheets for the solvents used.**

3. When the pump head is full of isopropanol, stop the flow for at least 15 minutes, to dissolve any sticky deposits.
4. Reconnect the previous solvent and then pump the isopropanol to waste.
5. Check the flow rate. If the flow rate is still low, remove the check valve and then clean the check valve by blowing compressed air though it. If the flow rate is still low, replace the check valve.

Replacing a Check Valve

A check valve can be replaced without dismounting the head; however, the pump must first be powered off and the hydraulic tubing must be disconnected from the pump head.

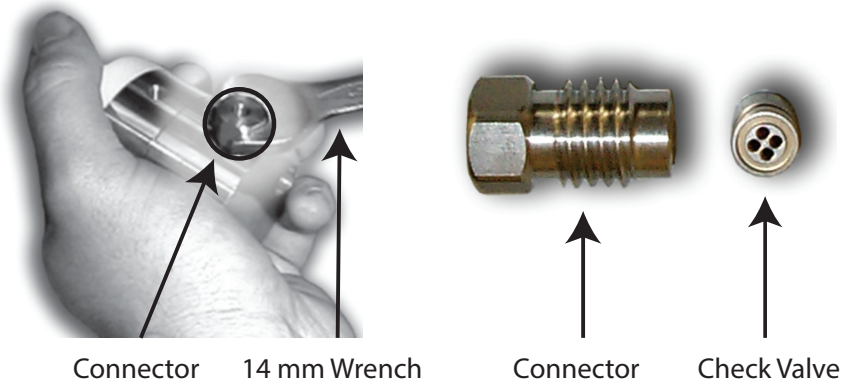


Figure 52
Check Valve Components

1. Loosen the connector with a 14 mm wrench, and then unscrew completely by hand.
2. Remove the check valve from the connector.
3. Make sure that the connector and pump head housing are clean.
4. Slide a new check valve into the connector.

> **NOTE** The arrow on the cartridge must point in direction of solvent flow.

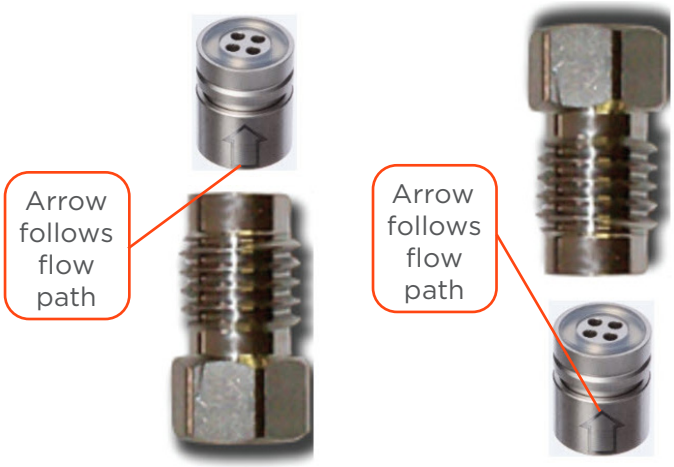


Figure 53
Inlet Check Valve (Left) and Outlet Check Valve (Right)

5. Screw the connector into the pump head housing.
6. Carefully tighten the connector using a torque wrench set to 7 Nm. Or, turn the connector using the 14 mm wrench until there is contact, then tighten the connector by turning it a further 30° in the clockwise direction. If leakage is observed, tighten the connector progressively until the leakage stops.
7. Run the pump and perform the **Leak Test Procedure on page 52**.

Filters

Inlet Filters

To protect the check valves, an inlet filter (part number 4957231) must be used with all solvents. Inlet filters must be in good condition for the pump to operate efficiently.

Clean the inlet filter using a suitable solvent. Change the inlet filter if you suspect that it has become plugged.

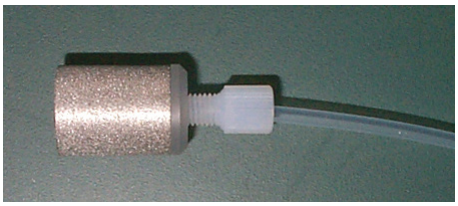


Figure 54
Inlet Filter

Outlet Filter

The outlet filter (part number 49571579) protects the injection valve and the column. A plugged outlet filter may cause pressure buildup and leaks. Check and clean the interior of the outlet filter routinely and, when necessary, replace the filter cartridge.

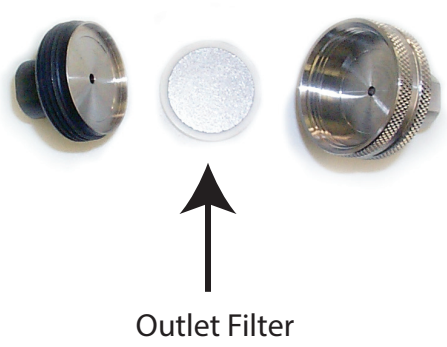


Figure 55
Outlet Filter

CLEANING THE OUTLET FILTER

1. Unfasten the outlet filter from the steel tubing with the supplied 11 mm wrench (part number 4340431).
2. Unfasten the filter from the column or injector tubing with the supplied 11 mm wrench.
3. Separate the two halves of the filter casing with your hands.
4. Rinse the interior of both halves of the casing using a suitable solvent.
5. Replace the filter cartridge.
6. When you reinstall the filter cartridge, place it in the black (downstream) half of the casing to ensure that the cartridge is properly seated before reassembling the filter casing.
7. Reassemble the filter casing, using moderate force and then refit the filter to the pump and re-prime the system, and check for leaks.

Maintenance Procedures

Leak Test Procedure

This test consists of pressurizing the pump to a user selectable pressure in a closed hydraulic circuit. The outlet from the pump (filter) should be sealed with the supplied plug before running the test. Use manual control in TRILUTION LC to start the pump at a slow flow rate and allow the pump to reach a pressure of 150 bar (2176 psi). Stop the pump once the pressure is reached. (Alternatively, use error handling in TRILUTION LC to stop the pump once the pressure is reached as part of an application run.) If during the next five minutes the pressure decay is less than 10%, then the test is successful.

Run-In Procedure

Whenever using a new pump head or a new piston seal, it is strongly recommended to follow this run-in procedure.

1. Run the pump unloaded for 1–2 minutes at 20% of nominal flow rate with methanol or isopropanol.
2. Run the pump at maximum operating pressure for 4–5 minutes. While running, check for any leaks.
3. Repeat the first step, but for 30 minutes.

Resetting Maintenance Logs

1. Select **Edit > GLP > Maint** from the **Startup** Screen on the 333 Pump control panel.
2. Select **Reset** to clear values.
3. Navigate the screen with directional arrows to set hour limits for the following values: Seal, In CV, Out CV, and Piston.

Fuse Replacement

1. Power off the instrument and disconnect the power cord.
2. Locate or order replacement fuses. (Extras were provided with the instrument.)
3. Place a small screwdriver or a fingernail under the tab on the fuse drawer to detach it from its receptacle on the rear panel. The fuse drawer will remain hinged to the instrument.

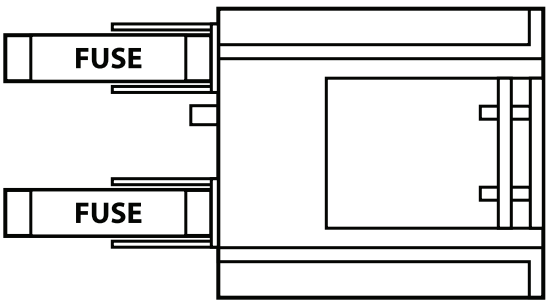


Figure 56
Fuse Replacement

4. Replace both fuses. Use only fuses with the rated current and specified type as listed on the rear panel of the instrument.
5. Insert the fuse drawer into its receptacle on the rear panel.

Chapter 5

TROUBLESHOOTING

IN THIS CHAPTER

When troubleshooting, try to check each part of the system independently. Check the solvent bottles, the connections between the bottles and pump heads, inlet filters, outlet filters, and so on. Check each component in the circuit, even if it is new.

To receive notification of and to designate instructions for response to error states in TRILUTION LC, users must define parameters in the **Method - Error Handling** tab.

Error messages appear on the control panel display or are indicated by the ERROR light.

Error LED (333 Pump)

The ERROR light may activate (steady red) for any of the following reasons:

- A pump motor has ceased to function or has been asked to function in a way that is not possible.
- Communication has failed with a connected pump.
- Pressure limits have been exceeded (high, File 21 and low, File 22). Any attempt to run a file that does not exist will activate the error LED.
- Restart after a power failure has attempted to start File 23. If the file does not exist, the software will stop the pump and activate the ERROR LED.
- Input #2 was closed, attempting to start File 23. If the file does not exist, the software will stop the pump and activate the ERROR LED.
- A maintenance counter limit has been reached. See **Resetting Maintenance Logs on page 52.**
- Pump operation will cease when the maintenance counter limit is reached, even if a method program is running.
- The audit trail is saturated.
- There is an invalid setting.

Error Messages (333 Pump)

Many types of messages are displayed on the screen; for instance, errors that occur during the entry of a method program or configuration parameters. For this type of error, the software displays messages that explain the required corrective action. However, there are some critical error situations that can occur during or after a run.

Message	Corrective Action
Pressure lower than 'X' Pressure higher than 'Y' Input 2 was activated	Check for leaks Check configuration parameters Check method parameters
Power restored after failure	The latest run could be invalid. Check the system before a possible rerun of this file
Channel 'Z' failing	Failure may be caused by a blockage (tubing, check valve, etc.) Check the hydraulics, especially Channel 'Z' Check configuration parameters Check method parameters
Communication fails with 'N'	Check that pump 'N' is switched on Check GSIOC ID and connections
File does not exist	Check that you are trying to run a valid file Check that any linked files are valid
Maintenance limit is passed for 'M'	Service item 'M' and reset the counter See **Resetting Maintenance Logs on page 52**
Validation process has restored default parameter(s)	Check all method program and configuration parameters
RAM error contact your Gilson representative	Contact your local Gilson representative

Troubleshooting

When troubleshooting, try to check each part of the system independently; try one solution at a time and proceed in a systematic way.

Electrical Problems

Problem	Possible Causes	Solutions
Pump does not operate—no POWER indicator LED	No power or fuse blown	Check fuses, plug in power cord, switch on at rear
Pump does not operate—no ON indicator LED	Pump is not operational	Press the ON/OFF key on the indicator panel
Pump does not stop at the end of a run	Not programmed	Program a flow rate of zero and a composition of zero at the desired end time for all solvents
Error LED lights up	Various	See text
Slave pump does not operate	No GSIOC communication	Check GSIOC cable(s) are connected correctly between pumps and that GSIOC ID #s are correctly set
Pump 'X' is missing message appears on the screen	No GSIOC communication or the Slave Pump ('X') is not switched on	Switch on the Slave Pump, check GSIOC connectors, the baud rate and ID of the slave

Hydraulic Problems

Problem	Possible Causes	Solutions
Air bubbles in both the inlet and the outlet tubings	Inlet tubing is loose Nut and/or ferrule damaged	Tighten the connectors Replace the nut and/or ferrule
	Inlet filter is plugged	Clean or replace the inlet filter(s)
	Refill time is too short	Increase the refill time; refer to page 37
Air bubbles in the outlet tubing only	Loose connection of outlet	Tighten connectors
Leaks from a pump head	Defective piston seal	Replace the defective seal
Abnormally low flow rate	Leaks	Check the all plumbing for leaks
	Air entering upstream from the head	Check the upstream connections
	Plugged inlet filter	Replace inlet filter
	Defective check valve	Clean or replace the check valve
	Incorrectly mounted pump head	Remount the pump head
Abnormally high pressure	Plugged outlet filter	Clean or replace outlet filter
	Column particle size too small or plugged column	Change or flush column
	Mobile phase viscosity too high	Use lower viscosity solvents or increase temperature
Baseline noise, periodic pulses	Mixer volume too small	Increase mixer volume
	Air in the hydraulics	Prime the pump, degas the solvent
	Faulty pressure module	Contact your Gilson representative

NOTE **Contact your local Gilson representative or techsupport@gilson.com for assistance resolving problems described in this chapter.**

Repair and Return Policies

Before Calling Us

Your local Gilson representative will be able to serve you more efficiently if you have the following information:

- Serial number and model number of the instruments involved
  - The serial number is located inside the door on the right side of the pump.
- Installation procedure you used
- List of concise symptoms
- List of operating procedures and conditions you were using when the problem arose
- List of other devices connected to the instrument and a description of those connections
- List of other electrical connections in the room

Warranty Repair

Units covered under warranty will be repaired and returned to you at no charge. If you have any questions about applicability, please contact your local Gilson representative.

Non-Warranty Repair

For out-of-warranty repairs, contact your local Gilson representative who will discuss service options with you and can assist in making arrangements to return the equipment, if necessary.

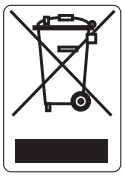
Return Procedure

Contact your local Gilson representative to obtain authorization before returning any Gilson equipment. To return a piece of equipment:

- Carefully pack the unit to prevent damage in transit. Check with your local Gilson representative regarding proper method of shipment. No responsibility is assumed by Gilson or your local Gilson representative for damage caused by improperly packaged instruments. Indicate the authorization on the carton and on the packing slip.
- Always insure for the replacement value of the unit.
- Include a description of symptoms, your name, address, phone number, and purchase order to cover repair costs, return and shipping charges, if your institution requires it.

Unit End-of-Life

When a unit reaches the end of its useful life, refer to **www.gilson.com** for directions and information on the end-of-life policy. This is in accordance with the European Union Directive on Waste Electrical and Electronic Equipment (WEEE).



Appendix A

REPLACEMENT PARTS

IN THIS CHAPTER

333/334 HPLC Pumps

PART NUMBER	DESCRIPTION
38103331	333 Pump (110 and 220V), Primary Solvent, Dual-Piston, Reciprocating Master Pump
38103341	334 Pump (110 and 220V), Secondary Solvent, Dual-Piston, Reciprocating Remote Controlled Pump

Hydraulic

Tubing and Fittings

PART NUMBER	DESCRIPTION
38013363	333/334 Rinse Plumbing Kit
38013364	Inlet Tubing Assembly
4957231	Inlet Filter, 20 µm, 5/16-24, 3/16 (A-231A)
49571579	Outlet Filter Assembly, Prep, 2.1mm Through, 10µm, 333 Pump
4957331	Outlet Filter, Stainless Steel, 10 µm, 1.6 mm (ID) x 19 mm (OD)
49041040	Nut, 316 steel, 10-32 TPI, for 1.6 mm (1/16") tubing (Upchurch U400)
49041045	Ferrule, 316 steel, 10-32 TPI, for 1.6 mm (1/16") tubing (Upchurch U401)

TUBING AND FITTINGS CONTINUED ON PAGE 60

PART NUMBER	DESCRIPTION
380133552	Tubing, Stainless Steel, Left Pump to Outlet Tee, 33X Pumps
380133562	Tubing, Stainless Steel, PPMM to Outlet Filter
380133542	Tubing, Stainless Steel, Right Pump to Outlet Tee, 33X Pumps
380133742	Tubing, Stainless Steel, Outlet Tee to Pressure, Purge, and Mixing Module (PPMM)
38013357	Tubing, FEP, Inlet Tee to Right Pump Head, 33X Pumps
38013358	Tubing, FEP, Inlet Tee to Left Pump Head, 33X Pumps
380133592	Tubing, Stainless Steel, Horizontal, 334 Tee to 333 PPMM
380132582	Tubing, Stainless Steel, Vertical, 334 Tee to 333 PPMM
49090000	Tee, HOKE, P2TTT-316
38013319	Adapters, Male, 1/4-28 (TPI), Female, 10-32 (TPI), 1mm Through, 11 µL
490310236	Ferrule, 316 Stainless Steel for 3.2 mm (1/8”) Tubing
490310235	Nut, Stainless Steel, 1/4-28 (TPI) for 3.2 mm (1/8”) Tubing
38013367	Tubing, 2.1 X 3.2, 500 mm, SS
49934609	Tubing, 1 x 1.6 x 600 mm, 471 µL
38013372	OQ Plumbing line, 333/334
38013368	Tubing, 0.13 X 1.6 X 185 mm, SS, 2 µL (seal replacement)
471078702	Tubing, FEP, 0.085 ID X 0.124 OD (Waste tubing)
49041015N	Ferrule, 1/8", ETFE, Natural (P-300N)
490410315	Nut, 1/4-28 ETFE (P-315)
490310478	Plug, Stainless Steel, 1/4-28 (TPI), 333/334
490410557	Plug, White, Delrin, 5/16-24 (TPI), P-557
4904101381	Nut, White, Delrin, 5/16-24 (TPI) with 1 mm Through
490410133N	Ferrule, Tefzel®, 3/16", P-133N
38013251	Drain Nut, 5/16-24, 4.8mm Tubing, TFE (Rinse Plumbing)

Pump Head

PART NUMBER	DESCRIPTION
38013257	H3 Pump Head

PUMP HEAD PARTS

PART NUMBER	DESCRIPTION
38013311	Spacer, Rear, PEEK
3801335301	Chamber Body H3
3801335302	Rinsing Body Chamber H3
38001000	Outlet Check Valve Chamber H3
3801335304	Inlet Check Valve Chamber H3

Piston Seals

PART NUMBER	DESCRIPTION
38013253	Piston Seal, Black, PTFE, Ti spring, for H3 Pump Head
38013261	Piston Seal, Yellow, UHMWPE, Ti spring, for H3 Pump Heads

Piston Assembly and Bellows

PART NUMBER	DESCRIPTION
38013262	Piston assembly with bellows, for H3 pump head
38013238	Bellows, fluoroelastomer, piston rinsing compartment, for H3 pump head

Check Valve Cartridge

PART NUMBER	DESCRIPTION
38013254	Inlet/Outlet Check valve cartridge, for H3 pump head

Electrical

PART NUMBER	DESCRIPTION
6736004007	Fuse, 6.0A

Miscellaneous (Tools, Power Cords, Cables, etc.)

PART NUMBER	DESCRIPTION
38013280	Solvent Bottle tray
38013228	Tubing clip, PP, for 3.2 mm, 1/8” for bottle tray
38013229	Tubing clip, PP, for 4.8 mm, 3/16” for bottle tray
38073202	Drip pan, PP, with drain tubing (silicone, 4 x 6 mm, 1000 mm)
470223639	Tubing, silicone, 4 x 6 mm, 1 meter (drip pan tray)
7080318107	Power Cord for 110V
7080316106	Power Cord for 220V
36610101	Spanner for tubing nuts, 6.3–7.9 mm (1/4”–5/16”)
38013236	Spanner for check valve holder, 14 mm
4320302	Allen wrench, 3 mm (rear-end, to release pump head)
4320502	Allen wrench, 5 mm (front-end, to remove pump head)
38013235	Bellows mounting tool, POM, white, piston assembly, for pump heads
4340431	Wrench, Open End, 3/8 and 7/16” (9 and 11 mm), Double Ended
36078143	GSIOC Cable, Shielded, 30”
638310512	10-pin Terminal Block Connector for Input and Output Contacts
638314512	14-pin Terminal Block Connector for Output Contacts
36460058	Priming Syringe, Glass, 10 mL
38013234	Glass bottle (Piston Rinse)

Service Part

Replacing this part may require assistance from your technical service representative.

PART NUMBER	DESCRIPTION
38013342	333 Pressure, Purge and Mixing Module (PPMM)

Appendix B

COLUMN HOLDER

IN THIS CHAPTER

Installation

The column holder attaches to the left side of the pump. Three pairs of column support, fitted with clips for different diameter columns, are provided in the kit.

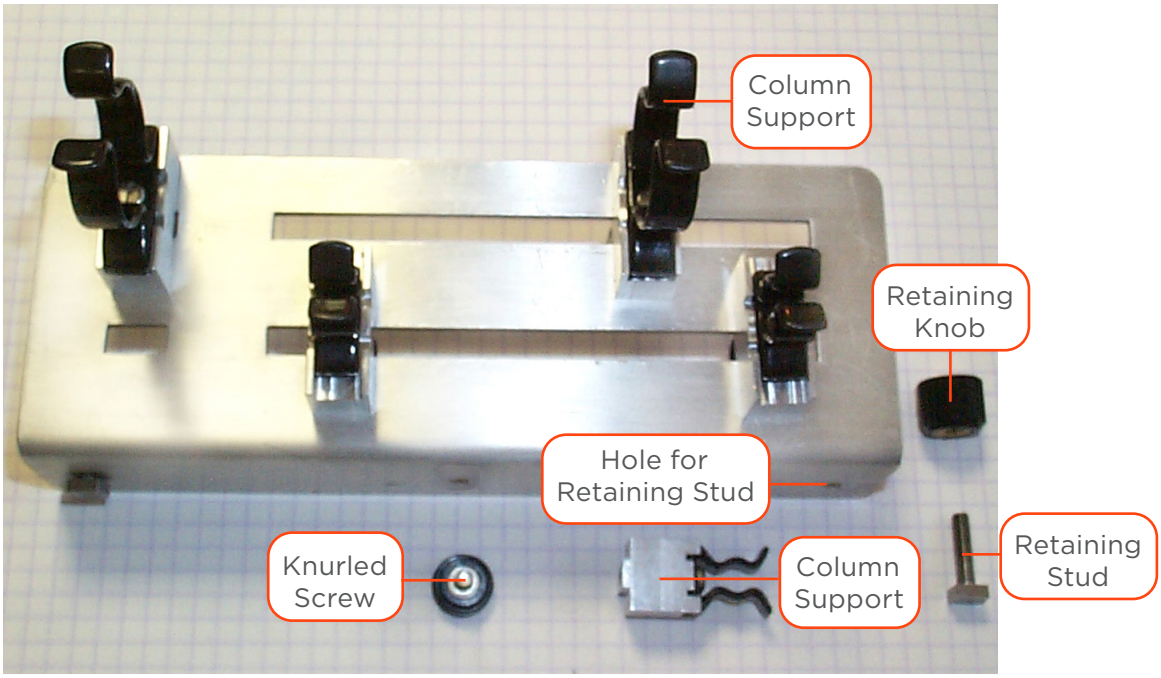


Figure 57
Column Holder

Holder Installation

1. Loosely fit the retaining stud.
2. Slide the studs into the tops of the retaining slots at the side of the pump.
3. When the holder is correctly positioned, fully tighten the retaining knobs.

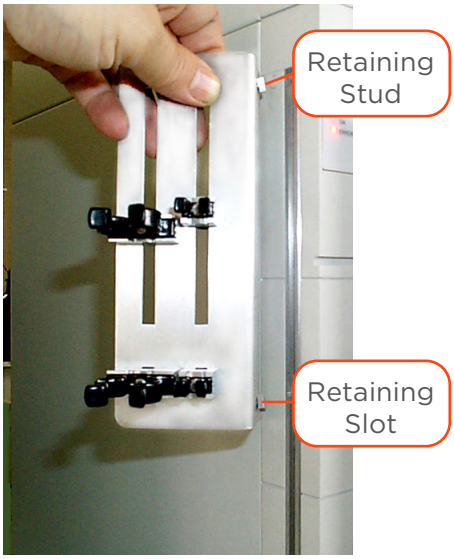


Figure 58
Holder Installation

Support Installation

1. Push one of a pair of column supports into either of the slots.
2. Fix the column support in position using a knurled screw.
3. Repeat for the second column support but don't fully tighten the screw.
4. Slide the second column support along the long slot to match the length of the column.
5. Fully tighten the screws.

Figure 59
Support Installation

Part Numbers

PART NUMBER	DESCRIPTION
38013203	Column holder, equipped for 2 to 20 mm ID column
380132661	Column holder clips, 1/4" (2 mm), 2/pk
380132662	Column holder clips, 1/2" (10 mm), 2/pk
380132663	Column holder clips, 1" (20 mm), 2/pk

Appendix C

INJECTOR

IN THIS CHAPTER

Injection Valve Holder

The injection valve holder has two pairs of holes, one pair is for mounting the valve in the vertical position and the other is for mounting the valve in the horizontal position.

Holder Installation

1. Loosely fit the retaining studs to either pair of holes.
2. Slide the feet into the top of the retaining slot at the side of the pump.
3. When the holder is securely positioned, fully tighten the retaining feet knobs.

Figure 60
Holder Installation

Valve Installation

1. Secure the valve body to the holder using two screws.
2. Fit the valve handle, as described in the documentation supplied by the valve manufacturer.

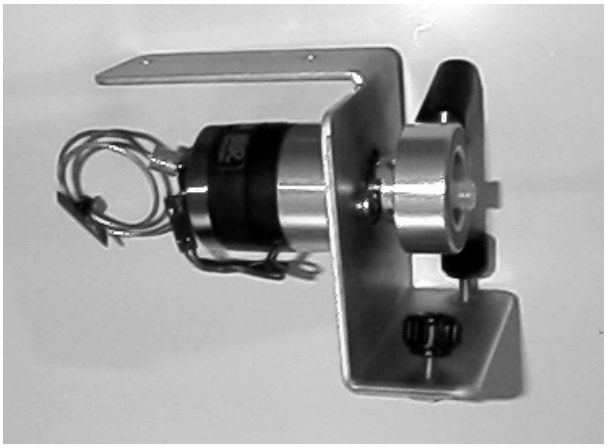


Figure 61
Valve Installation

Connection to Injector

For flow rates lower than 10 mL/min, use the 0.25 x 1.6 x 550 mm tubing (part number 49931559). For flow rates higher than 10 mL/min, use the 0.5 x 1.6 x 500 mm tubing (part number 49933509).

Part Numbers

PART NUMBER	DESCRIPTION
38013205	Valve holder with manual Rheodyne 7725i injection valve
38013206	Valve holder without valve
49931559	Tubing, 316 steel, 0.25 x 1.6 x 550 mm, 27 µL (to injector, <10 mL/min)
49933509	Tubing, 316 steel, 0.5 x 1.6 x 500 mm, 98 µL (to injector, >10 mL/min)

Appendix D

FRONT PANEL CONTROL

IN THIS CHAPTER

This appendix chapter details the steps involved in using the front control panel for the 333/334 HPLC Pumps.

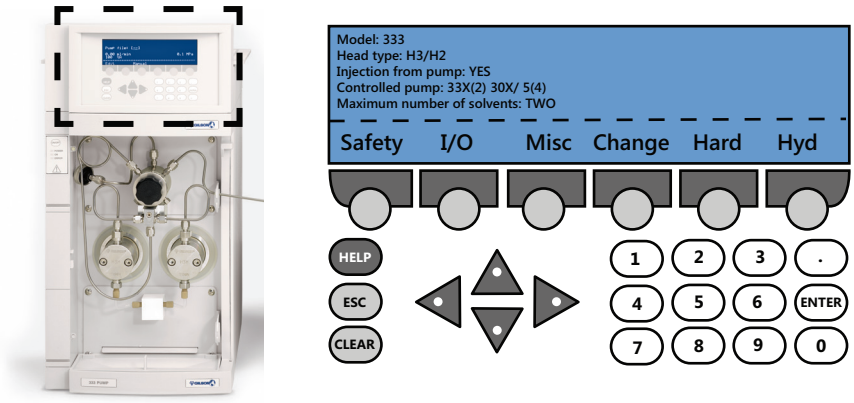


Figure 62
333 HPLC Pump Control Panel and Diagram

Using the Control Panel

Display Unit

The top of the screen displays input fields and configuration details. The bottom of the screen displays up to six menu items above the soft key buttons.

Keypad

The keypad consists of the entire pressable area of the control panel.

SOFT KEYS

Six circular buttons below the menu items on the display unit. Pressing a soft key selects the function displayed directly above it, such as **Run, Edit, Misc**, etc.

ARROW KEYS

Directional buttons used to navigate the screen.

NUMERIC KEYS

0 through 9 and a decimal (.)

ENTER

Used to confirm selections and store values into memory.

HELP

Displays detailed help messages and indicates the screen number.

CLEAR

Cancels values before they are entered into memory.

ESC

Used to return to the previous screen.

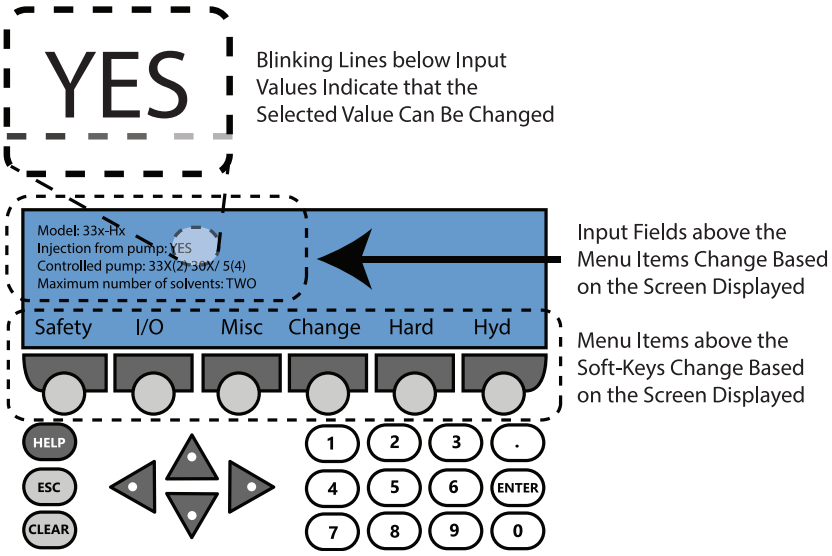


Figure 63
Control Panel Diagram

Electrical Connections

For instructions to make input/output contact connections, refer to Contact Connections on page 22.

Input Signals

Four pairs of terminals of the right-hand socket are dedicated to specific software-related input functions:

Contact	Function	Pin Location
START/END	Used to start (Open -> Close) and stop (Close -> Open) a program.	1
PAUSE/RESUME	Used to pause (Open -> Close) and resume (Open -> Close) a program currently running. The pause may be configured with or without flow.	3
INPUT (WAIT)	Used to signal to the method program currently running that the external equipment (e.g., fraction collector, sampling injector) is ready.	5
EMERGENCY	Used to signal to the pump software to start a Safety Program File (24).	7

Output Signals

Two outputs on the right-hand socket are dedicated to outputting signals:

Contact	Function	Pin Location
Digital Output Converted to Analog (0–1V)	Used to connect to analog recorders.	9
Analog Output (143–1000mV)	Used for pressure.	10

Basic Control Configurations

A 333 Pump can be used alone or as a master pump to control up to two other solvent pumps (333 Pump or 334 Pump). It may also be used to command an injection pump.

NOTE **Additional solvent pumps are always connected hydraulically to the pressure, purge, and mixing module (PPMM) of the 333 Pump.**

Hydraulic Connections for Gradient or Isocratic Mixtures

The 333 Pump can be configured to control the flow and composition of the mobile phase on a time variant (gradient) or a time invariant (isocratic) basis, when associated with a 334 Pump. Thus, you can pump gradients using two solvents, or you can pump in isocratic mode after mixing Solvent A (from one pump) and Solvent B (from the other).

For a binary gradient configuration, connect a 333 Pump to a 334 Pump. Solvent B is routed via the outlet tee of the 334 Pump to an inlet on the PPMM of the 333 Pump, where the two solvents are mixed. When three solvents (ternary) are to be combined, a second slave pump can be connected to the PPMM.

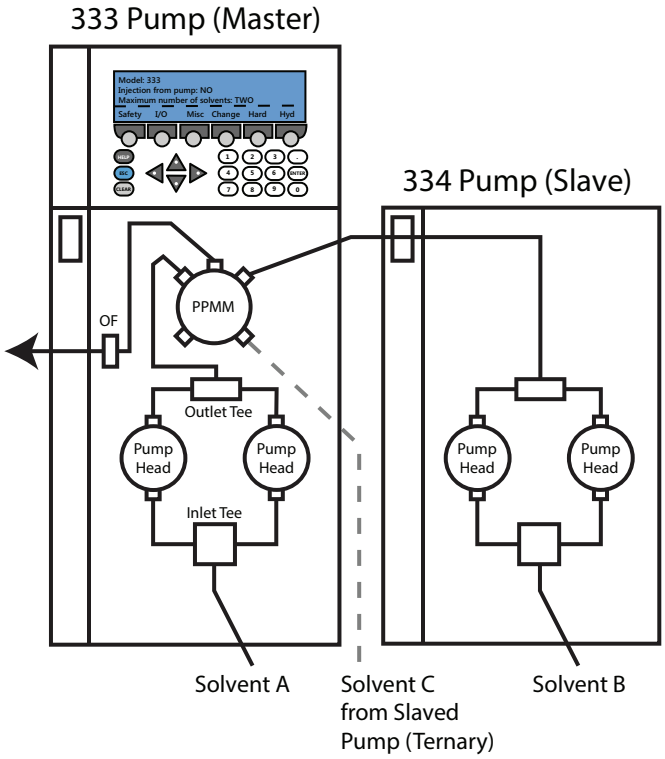


Figure 64
Hydraulic Configuration Diagram

MAKING THE HYDRAULIC CONNECTIONS TO AN EXTERNAL INJECTOR

1. Connect from the outlet filter of the 333 Pump to the downstream injector.
2. Connect solvent line A to the inlet tee piece on the 333 Pump.
3. Connect solvent line B to the inlet tee piece on the 334 Pump.
4. Connect horizontal (part number 380133592) or vertical (part number 380132582) tubing from the tee piece of the 334 Pump to the inlet of the PPMM of the 333 Pump.
5. Prime the system. See Hydraulic Priming on page 78.

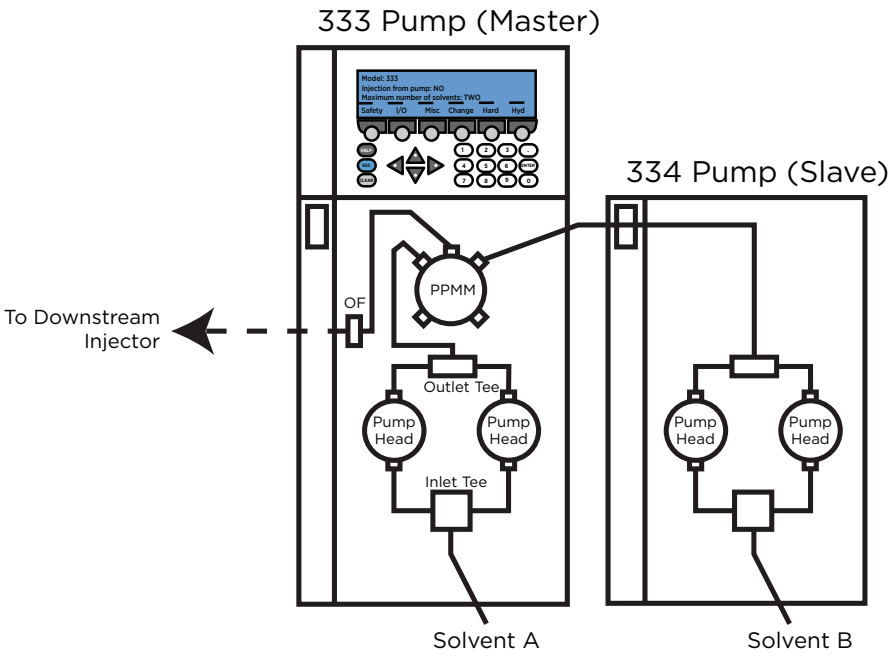


Figure 65
Hydraulic Connections

6. Initialize and run-in the piston seals (if new). See Run-In Procedure on page 52.

Software Setup

1. Switch on the pump(s). The initialization screen will appear briefly.
2. Check or set the GSIOC IDs for the devices. See GSIOC ID on page 21
3. Make the electrical connections between the instruments, including the GSIOC cable linking the 333 Pump to controlled devices.
4. Check the software configuration. Makes changes as needed.
   a. Press **Edit > Config > Hard.**
   b. Make sure the correct device is selected before advancing to the next step. The selected ID will blink. Use the **Up** and **Down** arrows to navigate.
   c. Press **Change** at the **Hardware Configuration** screen.
   d. Press **Esc** to leave the **Hardware Configuration** screen and advance to the **Configuration** screen. The screen displays whether injection from pump is available, the names of controlled devices, and the maximum number of solvents.

   Alternatively, press **Scan** at the **Hardware Configuration** screen to identify any connected pumps. Their configured IDs will appear on the **Hardware Configuration** screen.

You are now ready to create your method program file(s).

Hardware Configuration

Adding Solvent and Injection Pumps

A 333 Pump can control one or two additional solvent pumps and an injection pump. The controlled pumps must be assigned as follows:

Extra Solvent (id2): 333 Pump or 334 Pump

Extra Solvent (id3): 333 Pump or 334 Pump

Injection (id4): 30X Pump if an injection (sample) pump is present.

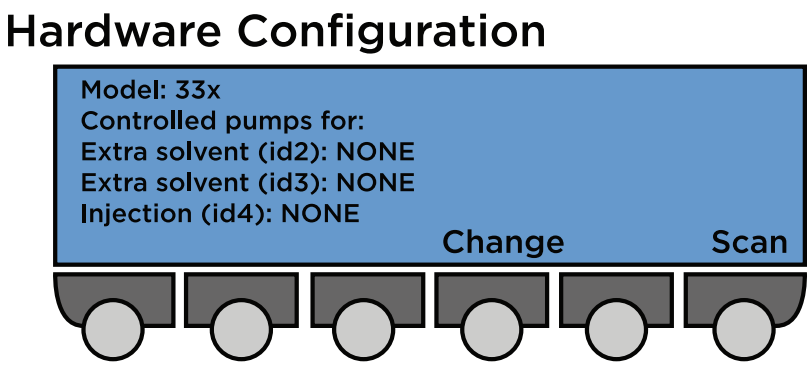


Figure 66
Hardware Configuration Screen

Navigate from the **Start** screen to the **Hardware Configuration** screen by pressing: **Edit > Config > Hard.**

1. Press **Change** to switch the value of **Extra Solvent (id2)** from **NONE** to **33X.**
2. Press **Enter**. An additional Extra Solvent (id3) line will appear.
3. Press the **Up** or **Down** arrows to navigate between Extra Solvent and Injection lines. A blinking line will appear below the selected value.
4. Press **Change** to add or remove solvent or injection pumps, as needed.
5. Press **Enter** to confirm your selection.
6. Press **Esc** when finished to return to the **Configuration** screen.

Configuring a Solvent Pump

1. Check that the head corresponds to the type fitted; press **Change** to modify the type configured, press **ENTER** to confirm. For 333/334 Pumps the head type must be H3.
2. You will see one solvent channel per pump (A, B, or C). For each select the solvent type and enter a value for **Inlet Pressure** (0-50 MPa); that is the pressure at the inlet to the pump head.
3. For each solvent channel (A, B, C), use the arrow keys to select a solvent name (or enter a value for compressibility), press **Change** to select another solvent, press **ENTER** to confirm.

Configuring an Injection Pump

From the **Start** screen, press **Edit > Config** to reach the **Configuration** screen.

1. Ensure that the **Injection from Pump** line is selected. If an injection pump was not selected at the **Hardware Configuration** screen, this option will not be available.
2. Press **Change** to switch the value from NO to YES.

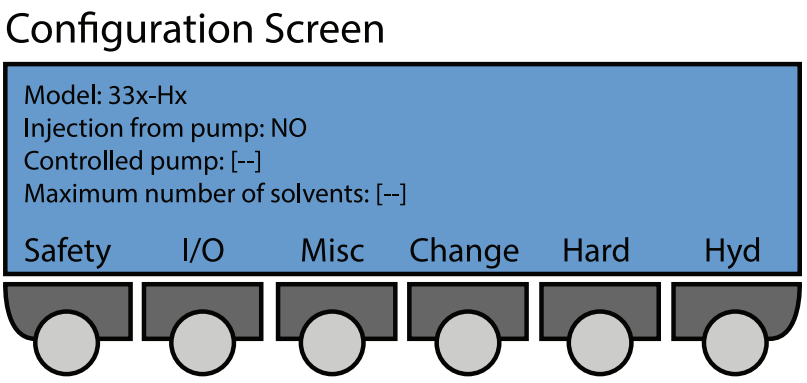


Figure 67
Configuration Screen

3. Press **ENTER** to confirm selection.
4. From the **Configuration** screen press **Hyd > Inject** to navigate to the **Injection** screen.
5. Press the **Down** or **Up** arrows to select one of the following parameters:
   - **Refill Time**: The duration of the piston return stroke (125–1000 ms). If cavitation occurs, enter a higher value.
   - **Inlet Pressure**: Use the keypad to enter the desired value.
   - **Head Type**: Select the installed pump head.
   - **Sample**: Identical to the **Solvent Selection** screens. The compressibility values will be automatically selected for the solvents listed; however, selecting **OTHER** will require the manual entry of compressibility in $Mbar^{-1}$.

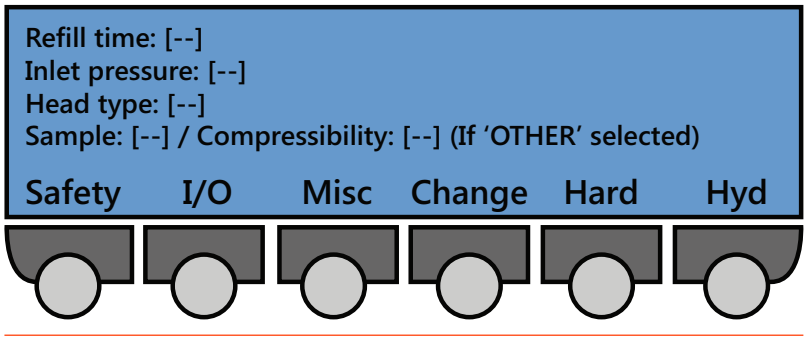


Figure 68
Injection Screen

6. Key in the desired value, then press **ENTER** to confirm the selection.

Adjusting the Hydraulics

Initializing the piston of the injection pump can improve injection precision at low flow rates.

From the **Start** screen, press **Edit > Config > Hyd > Adjust** to reach the **Adjust Hydraulics** screen.

Press **Change** to switch the value of the **Initialize Piston before Injection** line from **NO** to **YES**.

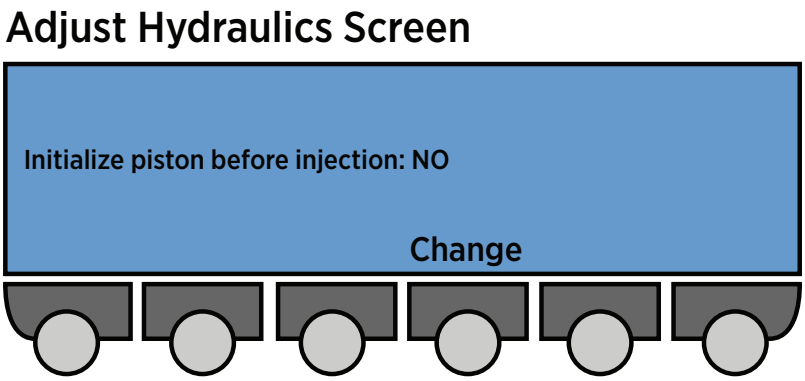


Figure 69
Adjust Hydraulics Screen

Safety Functions

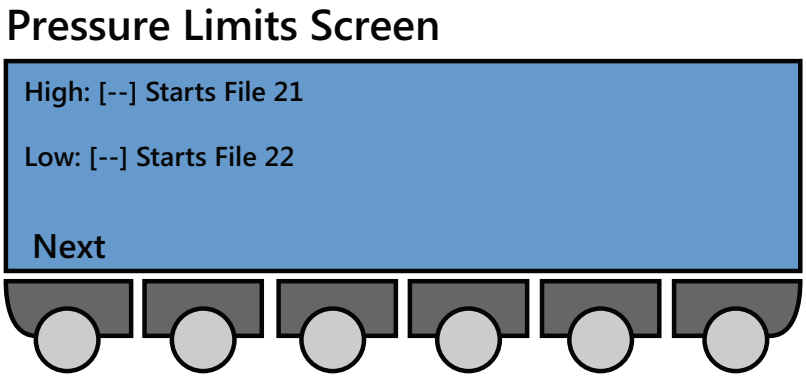


Figure 70
Pressure Limits Screen

From the **Start** screen press **Edit > Config > Safety** to reach the **Pressure Limits** screen that shows the high and low pressure limits and the associated safety files.

1. Use the **Up** or **Down** arrows to navigate to the High or Low lines, then key in the desired values. The range is 0–21 MPa for the high and low limits.
2. Press **ENTER** to confirm your choice.

NOTICE	**Pressure limits are model and configuration dependent and subject to automated validation. Ensure that the pressure limits entered are suitable for the safe and correct operation of your system.**

NOTE	**If File 21 is being executed, the pressure limit is automatically raised by 10%; then if the new pressure limit is passed, the pump stops. When the low pressure limit is crossed by decreasing values, the effect is delayed by 0.3 minute. The delay is to allow the Method Program to continue if an air bubble creates a brief and abrupt pressure decrease.**

3. Press **Next** to advance to the **Other Safety Functions** screen.

The following options will be available:

- When power restored after failure:
  - Start File 23
  - Continue from Same File
  - Stop
- Input 2 close starts file [--]
- Malfunction sounds beeper alarm: YES / NO

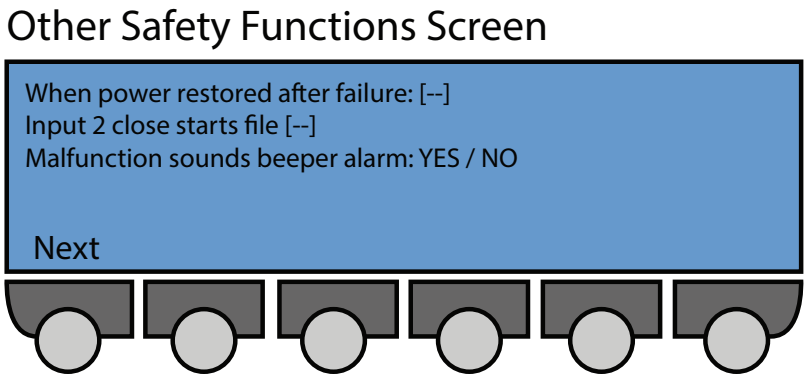


Figure 71
Other Safety Functions Screen

NOTICE

Ensure that safety files started as the result of values entered here perform the desired functions, such as stopping the pump. Files 21 through 24 are reserved for safety files. Do not assign method programs to these file numbers. If you do not create the file, an error message will display and the pump will stop.

File numbers 21 through 24 do not exist until you create the safety programs. Check that these programs function correctly to ensure the desired level of protection.

4. Use the **Up** or **Down** arrows to navigate to each option. Press **Change** to cycle through available selections for each option.
5. Press **ENTER** to confirm your selection.

Electrical Contacts

From the **Start** screen select **Edit > Config > I/O** to navigate to the **Electrical Contacts** screen.

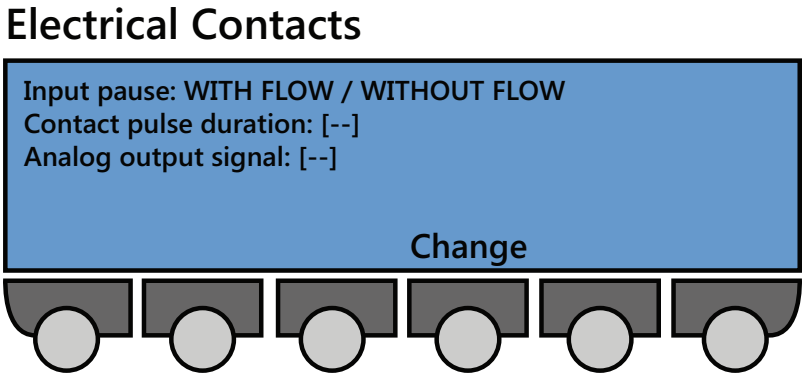


Figure 72
Electrical Contacts Screen

1. Press the **Up** or **Down** arrows to navigate to one of the following options:
   - **Input pause**: WITH FLOW or WITHOUT FLOW. When an input signal is received, this option either continues or stops flow.
   - **Contact pulse duration**: Set the length of an output pulse to a value between 0.01 and 1.00 minutes.
   - **Analog output signal**: Set the output signal to follow: pressure, flow rate, composition (%A, %B, or %C, depending on hardware setup). If you select one of the composition parameters, the **Delay Volume** line will appear with options (0 to 90mL) to get the best synchronization between the programmed profile and the detected profile.
2. Press **Change** on the **Input pause** line to cycle through the options.
3. Press **ENTER** to confirm.
4. Key in values for the **Contact Pulse Duration** line.
5. Press **ENTER** to confirm.
6. Press **Change** on the **Analog Output Signal** line to cycle through the options FLOW, PRESSURE, or composition (%A, %B, or %C).
7. Press **ENTER** to confirm.
8. If %A, %B, or %C is selected, key in values for the **Delay Volume** line that appears once you've selected the composition parameters.
9. Press **ENTER** to confirm.

GSIOC ID and Miscellaneous

From the **Start** screen press **Edit > Config > Misc** to navigate to the **Miscellaneous** screen.

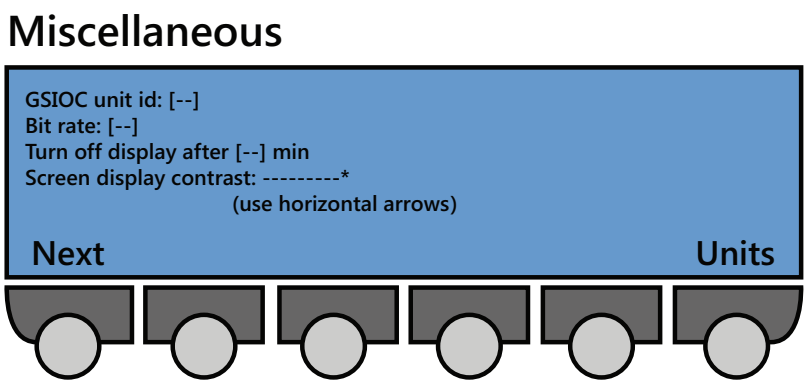


Figure 73
Miscellaneous Screen

1. Press the **Up** or **Down** arrows to navigate to one of the following options:
   - **GSIOC unit id**: (0-63). This must be done if the 333 Pump is slaved to another pump or if the default ID (#1) conflicts with other equipment.
   - **Bit (Baud) rate**: 333 Pump (External, 1200, 2400, 4800, 9600, 19200)
   - **Turn off display after 'X' minutes**: Not used.
   - **Screen contrast**
2. Key in the value for the **GSIOC Unit ID** line.
3. Press **ENTER** to confirm.
4. Press **Change** on the **Bit Rate** line and then select **External**, **1200**, **2400**, **4800**, **9600**, or **19200**. External is the recommended setting.
5. Press **ENTER** to confirm your selection.
6. Press **Next** to view the second **Miscellaneous** screen.
7. Press **Change** to switch the value of **Turn Off Keypad Beeper** (NO or YES).
8. Press **Prev** to return to the first **Miscellaneous** screen.
9. Press **Units** to navigate to the **Select Units** screen.
10. Use the **Up** or **Down** arrows to navigate to the following options:
    - Flow rate: ml/min or liter/h
    - Pressure: MPa, bar, or Psi
    - Injection volume: µl, ml, or l
11. Press **Change** to alter values for the **Flow Rate**, **Pressure**, or **Injection Volume** line.
12. Press **ENTER** to confirm.

Manual Operations

Hydraulic Priming

From the **Start** screen press **Manual > Prime** to reach the Prime Screen.

Priming is an essential precursor to operating any pump. Any actively associated pump (333, 334, or 30X) can be primed from the 333 Pump (master).

NOTICE **Running a pump head dry, even for a short time, will damage the instrument.**

⚠CAUTION **Ensure the PPMM is fitted with drain tubing before priming the pump.**

1. Fill the solvent bottle(s) with degassed, high pressure liquid chromatography (HPLC) grade solvent(s) and immerse the filter connected to the inlet tubing for each pump into the solvent.
2. Open the purge valve on the PPMM.
3. Press the **Start** soft key to prime the corresponding channel. Self-priming is generally achieved after two minutes. If self priming is achieved, skip to step 8. If not, press **Stop**, and then perform steps 4–7.
4. Disconnect the drain tubing from the purge valve, then replace it with a priming syringe (part number 36460058) equipped with its adapter.
5. Press **Start**, then draw solvent into the syringe while continuing to pump. When solvent appears in the syringe, press **Stop**.
6. Reconnect the tubing and continue priming for as long as is necessary. No bubbles should be present in the solvent flowing through the tubing. Press **Stop** to stop pumping.
7. Repeat the procedure for other pumps in the system as needed.
8. Press **ESC** to return to previous screen. All pumps must be stopped before you can leave the **Prime** screen.

NOTICE **While priming, the low pressure limit is inactive. To protect the column, the high pressure limit is automatically set to 30 bar (435 psi). If the purge valve was not opened, the screen displays the following message: Open Purge Valve.**

9. After priming, use the GLP function **Seal** to initialize the piston seals (if new). See Seal Test Procedure on page 91.

Manual Flow Control

From the **Start** screen press **Manual > Flow** to reach the **Manual Flow Control** screen. Use the **Up** or **Down** arrows to change values for the following options:

- **Ramping time**: (0 to 9999 minutes) The time given to changing the current flow to the desired flow.
- **Flow rate**: (0 to 200 ml/min)
- **Composition**: (0 to 100) for %B or %A (if more than one pump is configured)

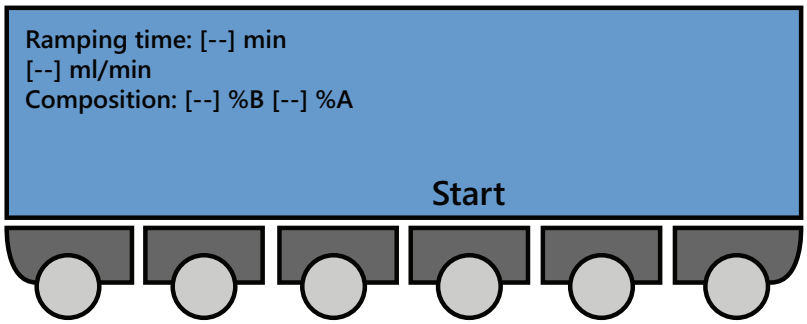


Figure 74
Manual Flow Control Screen

1. Key in the values for the **Ramping time**, **Flow rate**, and **Composition** lines.
2. Press **ENTER** to confirm.
3. Press **Start** to begin pumping after ramping to the desired flow rate and composition. The **Start** button is available only if the values demanded are different from the actual values.
4. Press **Stop** to immediately stop pumping. The **Stop** button is available only if the flow rate is not equal to zero.
5. Press **End** to interrupt the ramp. The **Manual Flow** screen appears; however, pumping still continues.

Output Contacts

From the **Start** screen press **Manual > Output** to reach the **Operate Output Contacts** screen. Use the **Right** or **Left** arrows to navigate to outputs 1 through 4. Press the **Open**, **Close**, or **Pulse** soft keys to change the state of each contact:

- Open to set the contact to normally open.
- Close to set the contact to normally closed.
- Pulse to change the state (open or closed) of the contact.

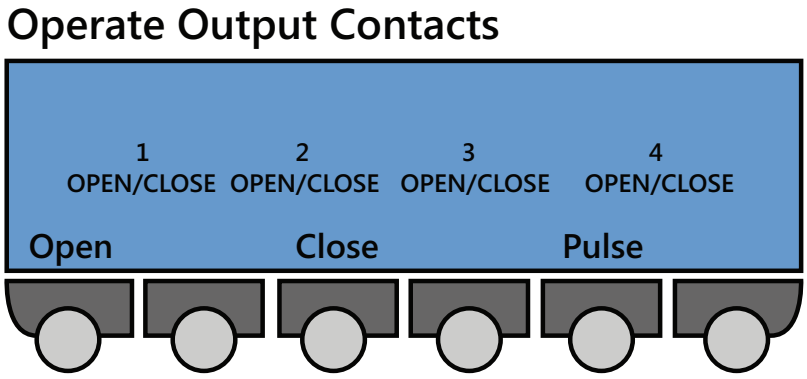


Figure 75
Operate Output Contacts Screen

Entering Method Programs

Creating and editing method program files is detailed in the following sections. You are advised to check and correct the configuration parameters before using the edit functions to create a list of events and the related parameters (flow rates, volumes, composition, etc.)

Creating a method program file consists of two different steps: 1) assigning a file number and 2) entering a list of events that you want to occur at the times that you specify. However, it is recommended that you list the events required by your method program, on paper, before attempting to create the file. See **Method Program Worksheet on page 84** for a sample.

Method program files (1–20) can be looped and linked to other files.

Safety program files (21–24) are created in exactly the same way as method program files.

Creating and Editing Method Program Files

From the Start Screen press **Edit > File** to access the **File Directory** screen.

1. Use the **Up**, **Down**, **Right** and **Left** arrow keys to select a file number. Unassigned files will appear as '--,' whereas pre-assigned files will appear in brackets.
2. Press the **Edit** soft key to confirm and edit the selected file and navigate to the **Edit Method** screen. Alternatively, press the **Create** soft key on an unassigned space to create a new file. The following options will be available:
   - **Method File**: Displays the current file and is not selectable.
   - **Number of Loops**: Indicates the number of times the method file is run before it completes or links to a separate method program file.
   - **When finished, link file to**: Indicates the file to be run once the current program file is complete.

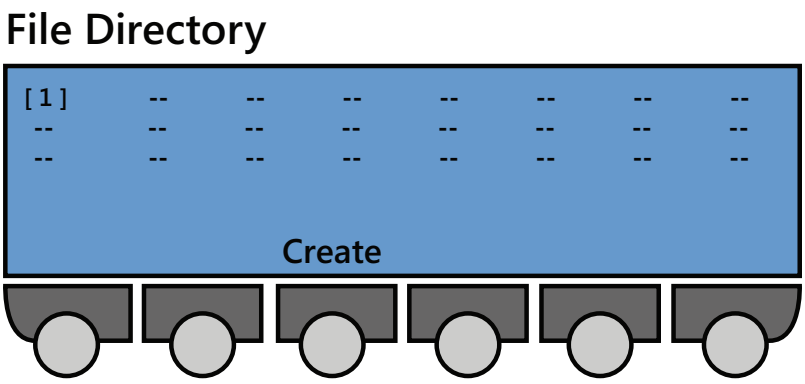


Figure 76
File Directory Screen

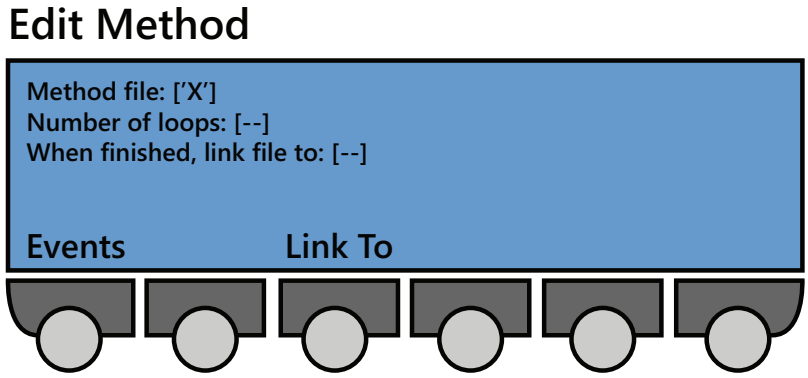


Figure 77
Edit Method Screen

3. Key in values for the **Number of Loops** and **When Finished, Link File To** line.
4. Press **ENTER** to confirm your selection(s). The **Link To** soft key will only appear after the **When Finished, Link File To** line has been selected.
5. Press the **Events** soft key to reach the **Events List** screen.

6. Press the **Up** or **Down** arrows to navigate to an event. If no events exists for that file, the **Edit Method File X** screen will appear to create an event.
7. Press the **Select** soft key to advance to the **Edit Method File X** screen. The following soft keys will be appear, allowing users to precisely define the parameters of their method program on various Events screens: **Flow**, **Comp**, **Wait**, **Output**, **Other**, and **Inj**,

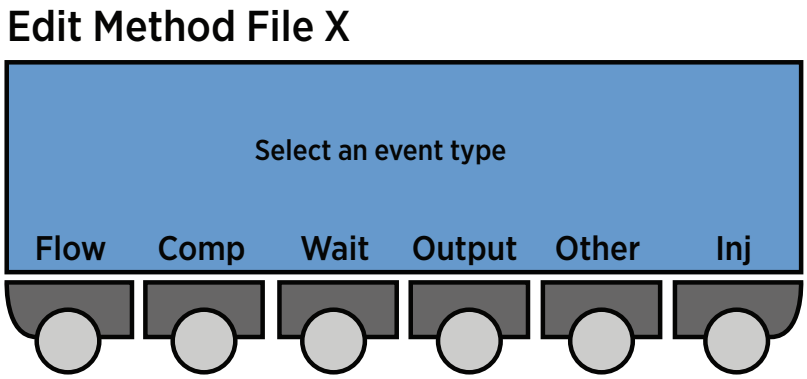


Figure 78
Edit Method File X Screen

NAVIGATION

- **Delete**: Press the **Delete** soft key to remove an event from a file.
- **Create**: Press the **Create** soft key to create the first event in a new file or the first event of this type in an existing file.
- **Time (Flow)**: Press the **Time (Flow)** soft key to toggle between injection flow rate and duration.
- **Add**: Press the **Add** soft key to add an event to a file.
- **List**: Press the **List** soft key to review the events currently present in a file.
- **Select**: Press the **Select** soft key to choose an event type to add to the file.
- **Confirm**: Press the **Confirm** soft key to affirm the value entered.
- **Consump**: Press the **Consump** soft key from the **Edit method** screen to display the solvent consumption (per loop).

INPUTTING FLOW EVENTS

Users must define the flow parameters for each event. The software will automatically ramp the mobile phase flow rate up or down based on the start and end values, which are updated and sorted as the user enters new events.

1. Press the **Flow** soft key on the **Edit Method File X** screen.
2. Key in the event time (0–9999 minutes) and flow rate. Press **ENTER** after each selection is keyed-in.
3. Press the **Confirm** soft key once the event values are set.
4. Press the **Add** soft key to add a new event.
5. Repeat Steps 2–4, as needed.

INPUTTING COMPOSITION EVENTS

1. Users must define the composition parameters for each event. The software will automatically ramp the mobile phase composition up or down based on the start and end values.
2. Press the **Comp** soft key on the **Edit Method File X** screen.
3. Key in the start time value (0–9999 minutes) and compositions (e.g., 3% B and 97% C) for the event. Press **ENTER** after each selection is keyed-in.
4. Press the **Confirm** soft key.
5. Press the **Add** soft key to add a new event.
6. Repeat Steps 2–5, as needed.

INPUTTING WAIT EVENTS

1. Press the **Wait** soft key on the **Edit Method File X** screen.
2. Key in the start time value (0–9999 minutes), and then press **ENTER**.
3. Press **Change** to switch the value of the operation between OPEN and CLOSE.
4. Press **ENTER** to confirm your selection.
5. Press the **Confirm** soft key.
6. Press the **Add** soft key to add a new event.
7. Repeat Steps 2–6, as needed.

INPUTTING OUTPUT CONTACT EVENTS

1. Press the **Output** soft key on the **Edit Method File X** screen.
2. Key in the start time value (0–9999 minutes), and then press **ENTER**.
3. Key in the output contact number (1 through 4).
4. Press **ENTER**.
5. Press the **Change** soft key to switch the operation of the output to OPEN, CLOSE, or PULSE, and then press **ENTER**.
6. Press the **Confirm** soft key.
7. Press the **Add** soft key to add another event.
8. Repeat Steps 2–7, as needed.

INPUTTING INJECTION EVENTS

1. Press the **Inj** soft key on the **Edit Method File X** screen.
2. Key in the start time value (0–9999 minutes), and then press **ENTER**.
3. Key in injection volume.
4. Press **ENTER**.
5. Key in flow rate or injection duration.
6. Press **ENTER**.

> ***NOTE*** **Press the Flow/Time soft key to toggle between time and flow views.**

7. Press the **Confirm** soft key.

Looping Method Files

Method files can be looped to repeat operations. From the **Start** screen press **Edit > File >** "Select File Number" **> Edit** to reach the **Edit Method** screen.

Key in the value (1–999) for the **Number of loops** line.

Press **ENTER**.

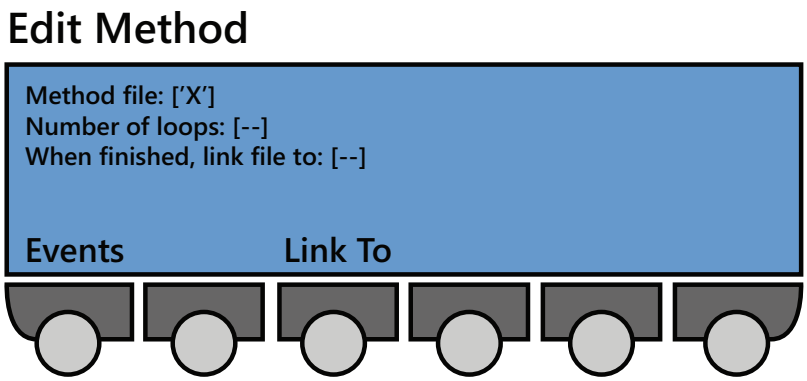


Figure 79
Edit Method Screen

Linking Method Files

Method files can be linked to each other to operate as a chain. From the **Start** screen press **Edit > File >** "Select File Number" **> Edit** to reach the **Edit Method** screen.

1. Use the **Down** arrow key to navigate to the **When Finished, Link to File** field.
2. Press the **Link To** soft key.
3. Key in the file number.
4. Press **ENTER**.

Listing Events in Method Files

From the Start Screen press **Edit > File >** "Select File Number" **> Edit** to reach the **Edit Method** screen.

1. Press the **Events** soft key.
2. Use the **Up** or **Down** arrows to navigate the list of events.

NOTE	Parameters may be changed from the list of events. Use the Up or Down arrows to navigate the list of events and use the Left and Right arrows to move the cursor from one parameter field to another. After keying in each new value, press ENTER to confirm the value.

Navigation

- Press the **Delete** soft key to erase an event. Press **Yes** to confirm the deletion or **No** to return to the previous screen.
- Press the **Time/Flow** soft key to toggle between the injection flow rate and duration view.
- Press the **Change** soft key to switch the state of an output.
- Press the **Select** soft key to edit an event.

Method Program Worksheet

File Number:	**Method Name**

Setup Parameters						
Pressure			High limit=		Low limit=	
Number of pumps=			Loops=		Link file=	
Pump Line	Model	Solvent	ID	Refill	Comp	Head Size
A						
B						
C						
D or Inj						

I/O Operations			
Time	Contact	State	Function

Program Steps			
Step No	Time	Event	Operation Event
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

File Management

From the **Start** screen press **Edit > File** to reach the **File Directory** screen. The soft keys on this screen will change based on the locked [*x] or unlocked status [x] of the file number highlighted.

File Directory [Locked File]

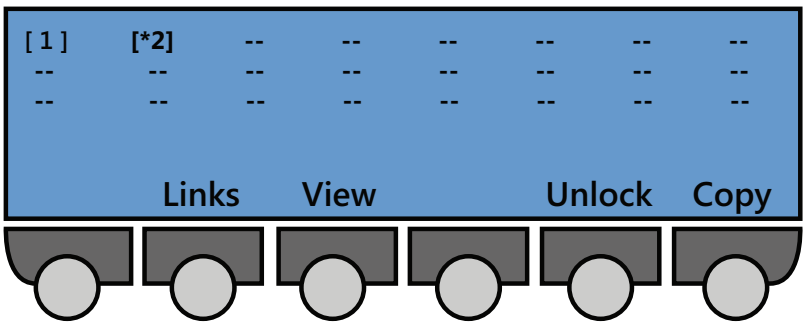


Figure 80
File Directory Screen [Locked File]

File Directory [Unlocked File]

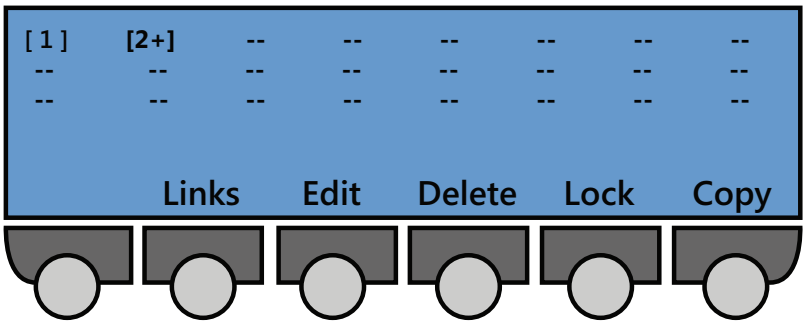


Figure 81
File Directory Screen [Unlocked, Linked]

Create File

Navigate to an empty file space on the **File Directory** screen using the arrow keys and press the **Create** soft key.

Edit File

Navigate to a file number using the arrow keys and press the **Edit** soft key. File numbers will appear as '[#]' when the file is unedited, '[*#]' when the file is locked, and '[#+]' when the file contains a link to another file.

View File

Navigate to a file number using the arrow keys and press the **View** soft key. This options allows users to view a locked file, using the same screen as the Edit Method, but without the possibility of using the **Delete** soft key to remove the events from the file currently selected, or to delete the file itself. However, you may copy a locked file, then edit the copied file.

Delete File

Navigate to a file number using the arrow keys and press the **Delete** soft key. Press **Yes** to confirm the deletion of the file or **No** to return to the previous screen.

Lock File

Navigate to a file number using the arrow keys and press the **Lock** soft key. Press **Yes** to confirm the locking of the file or **No** to return to the previous screen. A locked file cannot be edited or deleted; however, it may be copied or unlocked.

Unlock File

Navigate to a file number using the arrow keys and press the **Unlock** soft key. Press **Yes** to confirm the unlocking of the file or **No** to return to the previous screen.

Copy File

Navigate to a file number using the arrow keys and press the **Copy** soft key. Key in the file number for an unused file space and press **ENTER**. Press **Yes** to confirm the copying of the file or **No** to return to the previous screen. It is possible to copy a file to an existing file location. Overwritten files cannot be recovered.

Links

Navigate to a file number using the arrow keys and press the **Links** soft key. The screen that appears shows the order in which files are linked and will be run. 'Chain circular' means that you have cross linked files resulting in more than one occurrence of one or more file numbers in the series. Take care when linking files. Bad links can lead to unforeseen consequences.

Good Laboratory Practice (GLP Functions)

All of the following functions are available when control is from the 333 Pump.

From the Start Screen press **Edit > GLP** to reach the **GLP** screen.

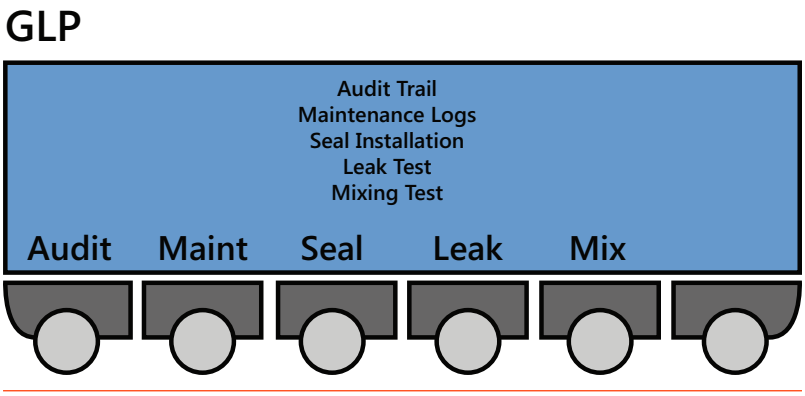


Figure 82
Good Laboratory Practices (GLP) Screen

Audit Trail

The sequence of events that takes place when you execute one or more method program files. From the control panel of the 333 Pump, you can activate or deactivate the audit trail and adjust the date and time.

From the **Start** screen press **Edit > GLP > Audit** to reach the **Audit Trail** screen.

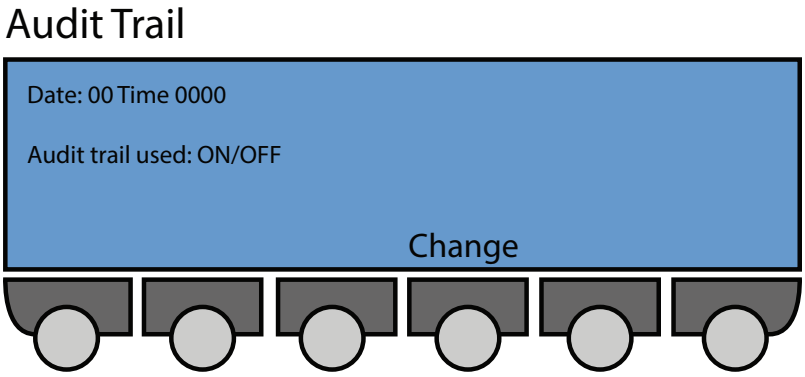


Figure 83
Audit Trail Screen

1. Navigate to the Date field with the **Directional Arrow** keys.
2. Key in the day, month, and year as a six digit number, e.g. 140220 for February 14, 2020.
3. Press **ENTER**.
4. Navigate to the **Time** field with the arrow keys.
5. Key in the hour and minutes as a four digit number with a decimal point, e.g. 22.35 for 10:35PM.
6. Press **ENTER**.
7. Select the **Audit trail used** field and then press the **Change** soft key to select **On** or **Off**.
8. Press **ENTER**.

NOTE The date and time are saved when you switch off the 333 Pump.

Maintenance Logs

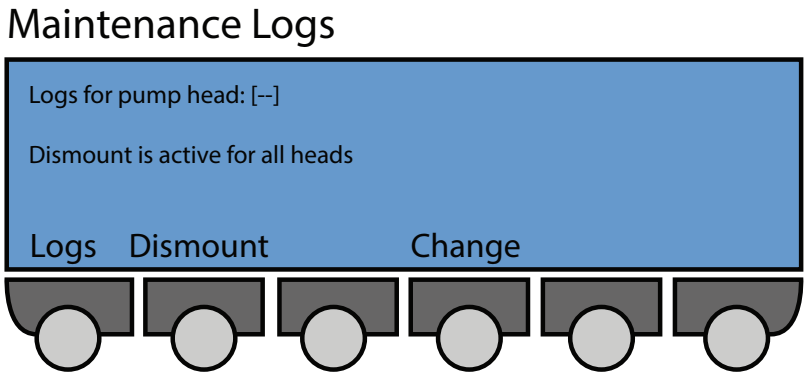


Figure 84
Maintenance Logs Screen

Use this function to view the log of wear parts for any configured 333 Pump or 334 Pump. The log contains the usage of the part to date (hours), the user set upper limit (hours), or the number of operational piston cycles that the part has completed (megacycle = one million cycles). The parts logged are the piston, piston seal, inlet check valve ('In CV'), outlet check valve ('Out CV'). After the specified usage, parts in question are normally serviced or replaced. Specify limit values that are suitable for the application and working environment.

From the Start Screen press **Edit > GLP > Maint** to reach the **Maintenance Logs** screen.

1. Press **Change** to cycle through the available pump heads: 1R, 1L, 2R, 2L, etc.
2. Press **ENTER**.
3. Press the **Logs** soft key to reset a logged value.
4. Use the **Up** or **Down** arrows to select the part.
5. Key in the desired values.
6. Press **ENTER** to confirm.
7. Press the **Reset** soft key to reset the values.
8. Press **ENTER**.
9. Repeat procedures, as needed.

DISMOUNTING PUMP HEADS

This procedure is required to change a piston seal or piston assembly. The pistons must go to the dismount position before you can physically remove the head(s) from the pump(s). Reset the maintenance logs once the components have been replaced. For manual pump head dismount instructions, see Manual Dismounting Procedure on page 42.

1. Press the **Change** soft key on the **Maintenance Logs** screen to cycle through to the appropriate pump head.
2. Press the **Dismount** soft key to begin the dismount procedure and then press **ENTER.**

> **⚠CAUTION** **Before pressing Yes, check to see that it is safe to set the piston in motion!**

3. Wait while the piston moves to the dismount position, until the message '**Change heads**' is displayed. Remove the pump head according to the directions in Remove Pump Head on page 42.
4. Press the **End** soft key to finish the dismount procedure.
5. Press the **Logs** soft key to reset the pump head logs.

Seal Test

From the Start Screen press **Edit > GLP > Seal**.

This procedure is designed for installing new piston seals with isopropanol. For a different solvent, adapt the flow rate in proportion to the viscosity of the solvent used. The seal installation procedure is divided into two parts:

Priming for two minutes at a fixed flow rate to the drain. See the table below.

Pumping at a preprogrammed gradient (ascending, then descending flow rate) against the back pressure provided by the supplied capillary tubing, for 14 minutes. If the pressure indicated in the table is reached before 2 minutes, then the software interrupts the positive flow rate gradient, and maintains, during 10 minutes, the flow rate at the value that generated this pressure, otherwise it goes to the flow rate indicated in the table and maintains it during 10 minutes. At the end of this time, the flow rate linearly decreases to zero in 2 minutes.

Pump Head	Prime	GLP/Seal			GLP/Leak	
	Flow Rate Fixed mL/min	Purge Flow Rate Fixed mL/min	Procedure Flow Rate Default mL/min	Procedure Pressure Default MPa	Procedure Pressure Default MPa	Procedure Flow Rate Default mL/min
33X-H3	50	50	15	15	15	0.5
33X-H2	35	35	25	45	45	0.5
30X-5	5	3.75	5	45	45	-
30X-10	10	7.5	10	45	45	-
30X-25	25	18.75	12	21	28	-
30X-50	50	22.5	12	10.5	14	-
30X-100	100	22.5	12	5.2	7	-
30X-200	200	22.5	12	2.6	3.5	-

SEAL TEST COMPONENT

The seal test uses the supplied steel capillary tubing (part number 38013368), which is to be connected at the outlet filter with water as the recommended solvent. In these conditions, the pressure is generally reached before the default flow rate.

You can select any configured pump. The parameters are adjusted automatically by the software.

Seal Test Procedure

1. Connect the supplied capillary tubing to the outlet of the outlet filter.
2. Open the purge valve on the PPMM.
3. Press the **Change** soft key to select the pump head.
4. Press **ENTER**.
5. Enter a flow rate for the **Installation Pressure Reached for Flow** line.
6. Press **ENTER** to confirm selection.
7. Press the **Start** soft key to start the pump. The pump will stop automatically after two minutes.
8. Press the **End** soft key to stop the pump, in case of emergency.
9. Close the purge valve on the PPMM.
10. Press the **Continue** soft key. The pump will run a preprogrammed sequence to finish the procedure.

Leak Test

From the **Start** screen press **Edit > GLP > Leak**.

This test consists of pressurizing the pump at a user selectable pressure in a closed hydraulic circuit. When this pressure is reached, the pump stops. If the pressure decay is less than 10% over five minutes, the test is deemed successful. A successful test will result in **OK** being displayed on the screen; otherwise, a **FAILURE** message is displayed. Check for leaks before repeating the test.

LEAK TEST COMPONENT

The leak test uses the supplied plug (part number 490310478), which must be connected at the outlet filter.

LEAK TEST PROCEDURE

1. Connect the leak test plug to the outlet filter.
2. Maintain or modify the flow rate (0.5 mL/min) and pressure (15 MPa) values by keying-in the numbers for the **Test Flow** and **Test Pressure** fields and pressing **ENTER**.
3. Press the **Start** soft key to start the pump. In case of emergency, press the **End** soft key to stop the pump.

Mixing Test

This test is based on an ASTM method for determining the linearity, accuracy, and repeatability of a binary composition gradient. The test automatically implements a program consisting of three loops.

1. From the **Start** screen press **Edit > GLP > Mix**.
2. Navigate to the **Create Test File to File** field with the **Down** arrow.
3. Key in the file number.
4. Press **ENTER**.
5. Run the file assigned as a method program.

Running a Method Program

Enter the file number (if necessary) and then press the **Run** soft key from the **Start** screen. The current method file displayed on the **Start** screen will begin. If you wish to select a different method file, visit the **File Directory** screen by pressing **Edit > File**.

After pressing **Run**, screen displays the flow rate and composition of the mobile phase and the pressure detected by the pressure sensor.

Press the **Edit** soft key to view or modify any file during a run.

Press the **Pause** soft key to interrupt the method program; however, the pump will continue to operate.

Press the **Resume** soft key to restart the method program from the point it was interrupted.

Press the **End** soft key to stop the method program; however, the pump will continue to operate.

Press the **Stop** soft key to stop pumping; however, the method program will continue to run.

When a method program file comes to an end, the pump continues to pump at the flow rate and composition that were applicable at the last programmed event; unless the flow rate is set to 0 at the end of the program or the file links to another method program file to stop the pump.

Post Run Information

Press the **Watch** soft key at the **Pump Control** screen to view the following:

- The minimum and maximum pressures attained during the last run.
- The time taken per cycle for each piston.

For a complete log of events, use the **Audit Trail** feature.

Appendix E

REFERENCE INFORMATION

IN THIS CHAPTER

Liquid Contact Materials

The information provided in the following table is accurate to the best of our knowledge and belief, but it is intended for general information only (classified by alphabetical order).

Liquid Contact Materials

Material	Description
ETFE	Ethyltrifluoroethylene (ETFE) is the generic name for the material such as Tefzel®. A fluoropolymer used for sealing surfaces, it is resistant to most chemical attack; however, some chlorinated chemicals will cause a physical swelling of ETFE tubing.
FEP	Fluorinated ethylene propylene (FEP) is a member of the fluorocarbon family with similar chemical properties as PTFE. It is generally more rigid than PTFE, with somewhat increased tensile strength. It is typically more transparent than PTFE, slightly less porous, and less permeable to oxygen. FEP is not as subject to compressive creep at room temperature as PTFE, and because of its slightly higher coefficient of friction is easier to retain in a compression fitting.
PCTFE	This material is a homopolymer of chlorotrifluoroethylene which has many of the properties similar to other fluoropolymers such as PTFE or FEP, but is mechanically superior in rigidity (does not deform easily), and has very low gas permeability. Its dimensional stability makes it attractive for use as a component of a structural part where the high temperature and chemical resistance of fluoropolymers is required. PCTFE shows high compressive strength and low deformation under load.
PTFE	Polytetrafluoroethylene is the generic name for the class of materials such as Teflon®. It offers superior chemical resistance but is limited in pressure and temperature capabilities. Because it's so easy to handle, it is often used in low pressure situations where stainless steel might cause adsorption. PTFE tubing is relatively porous, and compounds of low molecular weight can diffuse through the tubing wall. Use the black PTFE piston seals with organic solvents.

LIQUID CONTACT MATERIALS CONTINUED ON PAGE 94

Liquid Contact Materials

Material	Description
Ruby / Sapphire	Synthetic rubies and sapphires are single-crystal aluminum oxides, practically pure for the sapphire (+99,99% Al_2O_3). The color of the ruby is produced by adding a few ppm (parts per million) of chromium oxide (CrO_3). Synthetic rubies and sapphires have a hexagonal-rhombic crystal structure, density of 3.99 g/cm^3 and a water absorption coefficient of 0%. The principal properties of synthetic rubies and sapphires include a hardness and high mechanical strength, excellent resistance to wear, very low friction coefficient, chemically inert, good thermal conductivity, ideal electrical insulation.
UHMWPE	UHMWPE (Ultra-high molecular-weight polyethylene) piston seals are yellow and provide longer service with water, aqueous solutions, alcohols and acetonitrile.
Stainless Steel, Type 316L	Type 316L is an extra low carbon alloy that offer better corrosion resistance adjacent to brazes. This alloy contains a maximum of only 0.03% carbon. This amount of carbon is small enough to eliminate harmful carbon precipitation adjacent to brazes during the brazing operation. This extra low carbon grade is only recommended for equipment made for service below the lower sensitizing temperature of 800 deg. F, especially when corrosive conditions are severe. It is not recommended for use at high temperature. This grade can be highly polished with no surface blemishes.

TRADEMARK DESCRIPTION REFERENCES

PTFE, PEEK, FEP, ETFE and Titanium descriptions provided by Valco Instruments Co. Inc. (www.vici.com)

PCTFE description provided by Fluorotherm (www.fluorotherm.com)

Stainless Steel, Type 316L description provided by New England Small Tube Corporation (www.nesmalltube.com)

Ruby/Sapphire description provided by Ceramaret SA (www.ceramaret.ch)

Solvent Miscibility

Miscibility means that solvents should mix with each other in all proportions. Solvent miscibility is important both during elution and when switching from one solvent to another.

Refer to the table below when selecting solvents. For some solvents, lower toxicity alternatives are indicated [(1), (2), (3)], as follows: