PFT - TRACER Pump

Alltech Model 301 Pump
Operator's Manual

July 1998
Manual Part No. **301100M**

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5. Maintenance

1. INTRODUCTION

WARNING: There are potentially lethal voltages inside the pump case. Before removing the cover, disconnect the line cord. Never bypass the power grounds. Do not operate the pump from an ungrounded power outlet. Only qualified technicians should adjust or make changes to the electrical components of the system.

1.1 DESCRIPTION OF THE SERIES 301 PUMP

The Alltech Model 301 high performance liquid chromatography (HPLC) pump is engineered to perform equally well as a dependable system for routine analyses or as a sophisticated research instrument. While ideal for HPLC applications, the Model 301 pump is also useful as a metering pump for general laboratory or industrial use.

The flowrate of the Model 301 pump with a standard 10mL pump head can be set in 0.01mL increments from 0.01 to 9.99mL/min with a precision of 0.2%. The optional 40mL pump head allows flowrates from 0.1 to 39.9mL/min. Either size is available in type 316 stainless steel or Metal-free (biocompatible).

Low flow modulation of the reciprocating, single-piston pump is achieved with an advanced rapid-refill cam design, programmed stepper motor acceleration, and an internal pulse damper.

1.1.1 PUMP FEATURES

The Model 301 Pump features:

- ***Easy modification for microcolumns and semi-preparative techniques***
- ***A diaphragm-type pulse damper reduces pulsations in the system by as much as 90%***
- ***Integrated prime/purge valve***
- ***Self-flushing pump head***
- ***Front panel LED readout—shows the flowrate***
- ***Microprocessor advanced control***
- ***RS-232 control interface***
- ***Stepper motor accuracy***

1.1.2 Wetted Materials

The fluid contact surfaces on the stainless steel pump heads are 316 stainless steel, ruby, sapphire, and fluorocarbon polymer. Metal-free pump heads wetted surfaces are ruby, sapphire and inert polymers.

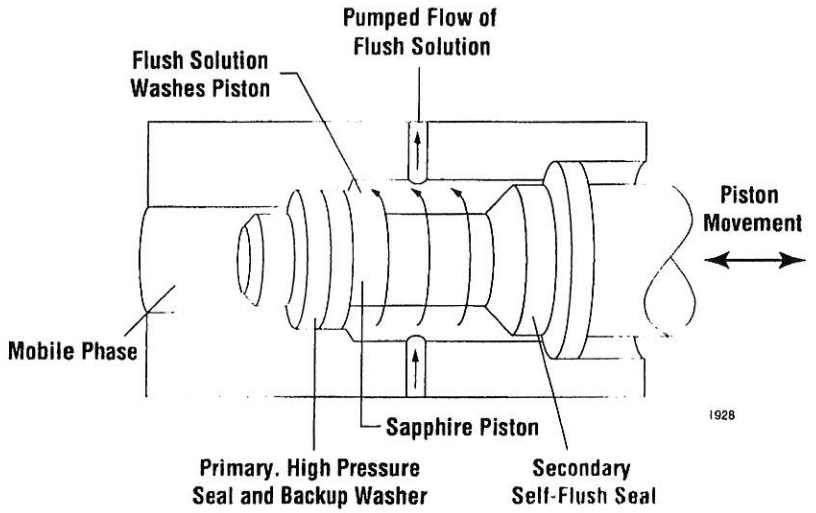

Figure 1 - Self-Flushing Pump Head

1.1.3 Self-Flushing Pump Heads

Self-flushing pump heads provide continuous washing of the region behind the primary high pressure seal without the inconvenience of a manual flush or gravity feed arrangement. A microscopic layer of fluid always wets the area between the piston and seal in an HPLC pump; when buffered mobile phases are used in a standard pump head, this layer evaporates behind the seal and deposits crystals on the piston. Those crystals abrade the pump seal and cause premature seal failure, leakage, and can possibly damage the pump. The self-flushing pump head uses a second seal and check valves to create an actively pumped flow in the area behind the high pressure pump seal, which washes away the film of mobile phase before crystals can precipitate.

1.2 SPECIFICATIONS FOR THE 301 PUMP

301 PUMP SPECIFICATIONS	
Flowrates:	0.01 to 9.99mL/min for 10mL head, 0.1 to 39.9mL/min for 40mL head
Pressure:	0 to 6000 psig for 10mL Stainless Steel (SS)pump head, 0 to 5000psig for 10mL Metal-Free (MF) head, 0 to 1500psig for 40mL heads
Accuracy:	2% measured at 1mL/min and 1000psig using 10% IPA in water
Precision:	0.2% RSD
Dimensions:	5.5"(14cm) high, 10.4"(26cm) wide, 17.5"(44.5cm) deep
Weight:	24 lbs
Power:	110-120 VAC, 50-60Hz; or 220-240 VAC, 50-60Hz
Features:	Prime purge valve Pulse damper Outlet filter Autoprime™ purging
Remote Inputs:	RS-232

2. INSTALLATION

2.1 UNPACKING AND INSPECTION

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

2.2 LOCATION/ENVIRONMENT

The preferred environment for the Model 301 pump is normal laboratory conditions. The area should be clean and have a stable temperature and humidity. The specific temperature and humidity conditions are 10 to 30°C and 20% to 90% relative humidity. The instrument should be located on a stable flat surface with surrounding space for ventilation and the necessary electrical and fluid connections.

2.3 SOLVENT PREPARATION

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

2.3.1 Solvent Outgassing and Sparging

Solvent outgassing occurs because the mobile phase contains dissolved atmospheric gases, primarily N_2 and O_2. These dissolved gases may lead to bubble formation and should be removed by degassing the mobile phase before or during use. The best practical technique for degassing is to sparge the solvent with standard laboratory grade (99.9+%) helium. Helium is sparingly soluble in HPLC solvents, so other gases dissolved in the solvent diffuse into the helium bubbles and are swept from the system. Solvent filtration is not an effective alternative to helium degassing.

Sparging the solvent vigorously for 10 to 15 minutes before use is recommended. Then, maintain a trickle sparge during use to keep atmospheric gases from dissolving into the mobile phase. The sparged solvent must be continually blanketed with helium at 2 to 3psig. Non-blanketed sparged solvents will have atmospheric gases dissolved back into the mobile phase within four hours.

Solvent mixes using water and organic solvents (like methanol or acetonitrile) hold less dissolved gas than either pure component holds. Therefore, there is a strong tendency for outgassing to occur. Sparging to reduce the amount of dissolved gas is critical in this case; but even with sparging some outgassing may be observed. Many separations will still give consistent results, however, even in the presence of these bubbles. A back pressure regulator (39020) after the detector flow cell will prevent bubbles from generating baseline noise.

2.3.2 Cavitation

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

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

2.3.3 Filtration

Solvents must always be filtered with a 0.5 micron filter prior to use. This ensures that no particles will interfere with the reliable operation of the piston seals. Note that solvents which produce precipitates, allow potential growth, or otherwise generate particles will need to be filtered more often. Strict filtering practices are especially warranted in the use of buffer solvents where insoluble impurities are a source of particles. In general, solvent filtration is good practice for the reliability of other components in the HPLC system. After filtration the solvents should be stored in a closed, particulate-free bottle.

2.3.4 Solvents With Harmful Effects

Except for the Metal-free pump heads, all portions of the Model 301 pump that contact mobile phase are manufactured of type 316 stainless steel, sapphire, ruby or fluorocarbon polymer. Some of these materials are extremely sensitive to acid chlorides. Avoid using solvents that contain any amount of hydrochloric acid. For example, the acidic form of tris-(hydroxymethyl) aminomethane HCl contains acid and must not be used. The pH conditions required to duplicate most published separations can be met with the phosphoric form, monotris-(hydroxy-methyl) aminomethane phosphate.

Some solvents you should specifically avoid are

Aqua Regia	Hydrochloric Acid (20%)
Bromine	Hydrochloric Acid (37%)
Chlorine Anhydrous	Hydrofluoric Acid (50%)
Copper Chloride	Hydrofluoric Acid (20%)
Ferric Chloride	Hydrofluoric Acid (75%)
Ferrous Chloride	Hydrofluorosilicic Acid (20%)
Freon 12 (wet)	Hydrogen Peroxide
Guanidine HCl 6M	Iodine
Hydrobromic Acid (20%)	Mercuric Chloride (Dilute)

In addition, some users of HPLC systems have observed that chloroform and carbon tetrachloride slowly decompose to liberate hydrochloric acid, which, as noted above, attacks stainless steel. Do not leave these solvents in the systems for a prolonged period. Ammonium hydroxide should also be avoided. Although ammonium hydroxide will not harm the pump itself, it is likely to damage the stator and rotor in the injection valve.

2.4 INSTRUMENT INSTALLATION

2.4.1 Mobile Phase Reservoirs

The mobile phase reservoir should be placed at the same level or slightly higher than the pump, never below the pump, and the inlet tubing should be as short as practical. These steps minimize pressure losses on the inlet side of the pump during refill and help to avoid bubble formation. These steps are particularly important when using high vapor pressure solvents (hexane, methylene chloride, etc.). Mobile phases should be degassed, filtered and covered. (See Section 2.3).

2.4.2 Self-Flush Solution

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

2.4.3 Inlet Tubing and Filters

The inlet tubing used in the Model 301 standard and 40mL MF pump head has 1/8" outer diameter. The inlet tubing for 40mL SS pump head has 3/16" outer diameter. The inlet tubing (Teflon®) is supplied in a 36" (91cm) length; an inlet filter (Part No. **32170**) is also supplied with the pump.

2.4.4 Outlet Tubing

Outlet tubing (not supplied with the pump) should have a 1/16" outer diameter. It is available in type 316 stainless steel, or Metal-free. Tubing with a 0.020" inner diameter is normally used before the injection valve. Tubing with a 0.010" inner diameter is normally used after the injection valve. The tubing must be cut squarely with no burrs. The tube itself should not be crimped, and the center hole must be open. A tubing cutter is recommended for cutting stainless steel tubing. Metal-free tubing should be cut with a plastic tubing cutter (Part No. **3206**).

Part No.	Description	OD(in)	ID(in)
	TUBING		
35702	PEEK tubing, 10ft	1/16	0.010
35708	PEEK tubing, 10ft	1/16	0.020
35710	PEEK tubing, 10ft	1/16	0.030
35712	PEEK tubing, 10ft	1/16	0.007
30051	SS tubing	1/16	0.010
30171	SS tubing	1/16	0.020
30161	SS tubing	1/16	0.030
30141	SS tubing	1/16	0.007

2.4.5 Priming the Pump and Flushing the Lines

Be sure all of the connections downstream of the prime/purge valve are closed. Connect a syringe (included with the pump) to the prime/purge valve. Open the prime/purge valve 1 to 2 turns (counter clockwise). Run the pump at a flowrate of 3 to 5ml/min. Prime the pump by pulling mobile phase and any air bubbles through the system and into the syringe (a minimum of 20mL). Close the prime/purge valve and stop the pump.

To prime the flush lines for a self-flush head, connect the small Luer to barb fitting (included with the pump - it is attached to the outlet line (at the top of the pump head) of the self flush tubing) to a syringe and pull 10-20mL of flush solution through the outlet line.

2.5 PREPARATION FOR STORAGE OR SHIPPING

2.5.1 Isopropanol Flush

Disconnect the outlet tubing from the pump. Insert the inlet filter in isopropanol. Open the prime/purge valve and use a syringe to draw a minimum of 50mL. Close the prime/purge valve and pump a minimum of 5mL of isopropanol to exit. Leave the inlet tubing connected to the pump. Place the inlet filter in a small plastic bag and attach it to the tubing with a rubber band. Plug the outlet port with the shipping plug, leave a length of outlet tubing on the pump, or cover the outlet port with plastic film.

2.5.2 Packaging for Shipping

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

3. OPERATION

3.1 FRONT PANEL CONTROLS AND INDICATORS

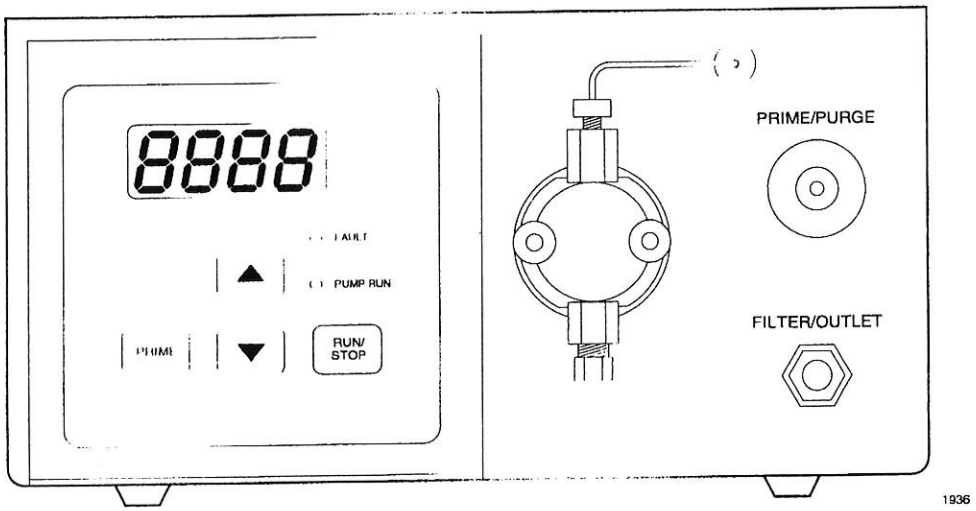

Figure 2 - Model 301 Pump Front Panel

3.1.1 Prime/Purge Valve

CAUTION: When the PRIME key is pressed, the pump will run at the maximum flowrate. Be sure the prime/purge valve is open.

The prime/purge valve vents the flow to atmosphere and permits efficient priming of the Model 301 pump. When the valve is closed firmly (fully CW), high-pressure flow is directed to the Filter/Outlet port. When the valve is open one-half to one full turn (CCW), pressure is vented and flow exits through the drain port in the prime/purge valve stem assembly. Suction with a 1 mL tip syringe at the drain port will purge air bubbles from the pump and reservoir lines (provided there are no open valves to lines downstream at the injector/column interface). To prime the pump, draw about 20 to 30mL of mobile phase.

3.1.2 Filter/Outlet

A high pressure in line filter (0.5 micron)(Part No. **250163**) is included at the output of the Model 301 pump. The Filter/Outlet port is the high pressure filter closure and is designed for a 1/16" OD tubing connection.

3.1.3 Control Panel

3.1.3.1 Digital Display

The 3-digit display shows the pump flowrate (mL/min) when operating.

3.1.3.2 Operation Keys

RUN/STOP — When pressed, this key alternately starts and stops the pump.

▲ — When pressed, this key increases the flowrate.

▼ — When pressed, this key decreases the flowrate.

PRIME — When the PRIME key is pressed, the pump runs at the maximum flowrate for the pump head. It will stop when any key is pressed.

3.1.3.3 Status LEDs

STATUS LEDS	
PUMP/RUN	Lights to indicate that the pump is running.
FAULT	Lights when a fault occurs and stops the pump.

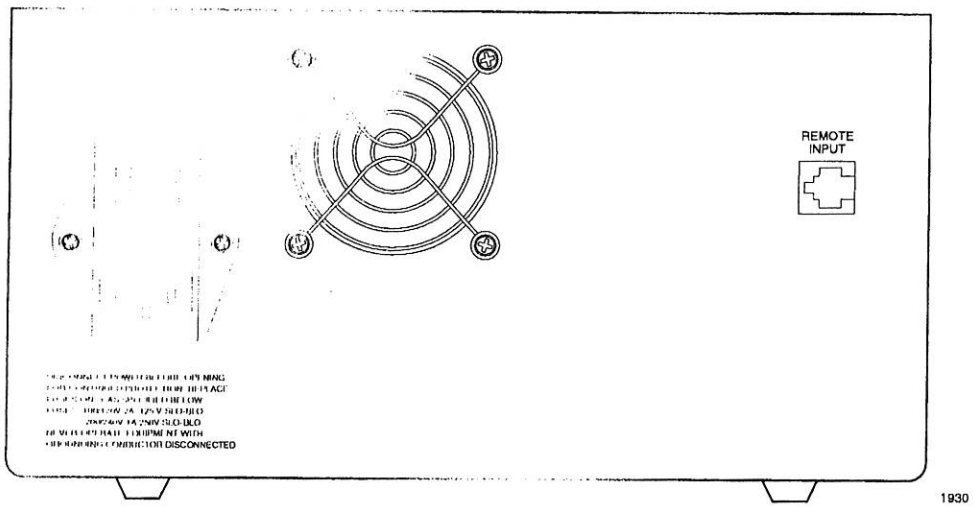

Figure 3 - Model 301 Pump Rear Panel

3.2 REAR PANEL REMOTE INPUT

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

3.2.1 Hardware Implementation

The REMOTE INPUT serial I/O port is configured for 9600 baud, 8 data bits, 1 stop bit, and no parity. The connector is a standard RJ-11 modular telephone type jack. Use the following chart for interfacing the Model 301 pump serial I/O port to either a 25 pin or 9-pin serial I/O COM port on the computer.

Pump (RJ11)	Function	IBM (DB25)[a]	IBM (DB9)[b]
HARDWARE IMPLEMENTATION			
1, 6	Ground	7	5
2	DSR (Input to pump)	20	4
3	RXD (Input to pump)	2	3
4	TXD (Output from pump)	3	2
5	DTR (Output from pump)	6	6

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

3.2.2 Hand-Shaking

The Model 301 pump uses hardware handshaking. The pump monitors the DSR input and disables the DTR output when the pump is in refill. In addition, the pump will not transmit if the DSR input becomes active.

3.2.3 Command Interpreter

The Model 301 pump's high level command interpreter receives and responds to command packets. The pump will not send a message except when prompted, and it will send a response to every valid command as described below. The response to an invalid command is "ER/".

Each command is characterized by a unique two-letter command code, and only one command can be issued per line. Case is not important; that is, the command codes "CC" "Cc" "cC" and "cc" are all equivalent. Response strings sent by the pump are terminated by the "/" character. The command packets are as follows:

Command	Response	Comments
COMMAND INTERPRETER		
CC	OK, y.yy/	Reads the pump flowrate (y.yy) in mL/min
ID	OK, version x.xx	Identifies the pump type and EPROM revision x.xx
FL, x.xx	OK/	Sets the flowrate to x.xx mL/min where the range is fixed for the pump head type, i.e., 0.01 to 9.99 or 00.1 to 39.9.
RU	OK/	Sets the pump to the RUN state.
ST	OK/	Sets the pump to the STOP state.
CS	OK, x.xx,w, v /0/	Reads the current pump setup, where: x.xx = Flowrate in mL/min w = Pump head type (0 = micro, 1 = macro) v = Run status (0 = stopped, 1 = running)
KD	OK/	Disables the keypad. (Default status at power-up is enabled.)
KE	OK/	Enables the keypad.
RE	OK/	Resets the pump configuration to its default power-up state.

4. THEORY OF OPERATION

4.1 MECHANICAL OPERATION

4.1.1 Liquid System Flow Path

The flow path of the Model 301 pump is from the pump head through the pulse damper, to the prime/purge valve, through the bulkhead filter, and out the front of the pump.

4.1.2 Pump Cycle

The pump cycle consists of two phases, the pumping phase and the refill phase. During the pumping phase, the pump piston moves at a constant linear speed, driven by a specially shaped cam which is driven by the motor using a toothed-belt drive. This results in a constant, stable flow from the pump at high pressure.

At the end of the pumping phase, the pump enters the refill phase. The cam is shaped so that the piston quickly retracts, refilling the pump head with solvent. The piston then begins to move forward again as the pumping phase begins. Since the output flow completely stops during refill, a pulse damper is necessary to provide some of the lost flow (see 4.1.3 below). In addition, the motor speed is adjusted by the microprocessor to facilitate an efficient refill phase.

The combination of increased motor speed and the rapid refill design of the cam generates refill times of less than 12.5% of the pump cycle (the refill time at 1 mL/min is less than 5% of the pump cycle).

4.1.3 Pulse Damping

The patented diaphragm pulse damper consists of a compressible fluid (isopropanol) held in an isolated cavity by an inert but flexible diaphragm. During the pumping phase of the pump cycle, the fluid pressure of the mobile phase displaces the diaphragm, compressing the fluid in the cavity and storing energy. During the pump refill phase the pressure on the diaphragm is reduced and the compressed fluid expands, releasing the energy it has stored. This helps to maintain the flowrate and pressure. The amount of mobile phase in contact with the pulse damper is small, only 1.2mL at 6000 psi, and the geometry used insures that the flow path is completely swept, so solvent "memory effects" are virtually eliminated.

4.2 ELECTRONIC CONTROL

4.2.1 Microprocessor Control

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

The motor power board contains programmed logic components which (1) provide suitable motor micro-stepping modes, (2) allow appropriate motor power adjustment, (3) maximize motor power output, (4) reduce motor resonance effects, and (5) customize motor stepping uniformity. MOSFET power transistors efficiently control the motor power provided by a 38 VDC linear power supply. This board also provides the 12 VDC (linear power supply) and the 5 VDC (switching power supply) used by the pump circuits.

A specially shaped cam provides refill in a fraction of the full cam revolution. The remaining revolution of the cam provides a linear piston displacement for constant flow of the mobile phase. In addition to the rapid refill characteristics of the cam, the onset of refill is detected by an infrared optical sensor as a slotted disc rotates through it. The microprocessor changes the refill speed of the motor to an optimum for the set flowrate. At 1mL/min, the refill is more than five times faster than with the motor operating at constant speed. The optimum refill minimizes the resulting pulsation while avoiding cavitation effects in the solvent entering the pump head.

The flowrate of any high pressure pump can vary depending on the operating pressure and the compressibility of the fluid being pumped. The pump is calibrated at 1000psig using a 90:10 mixture of water and isopropanol at 1 milliliter per minute. Adjustment can be made for other operating pressures. See Section 4.2.2 for information on pressure compensation.

4.2.2 Pressure Compensation

To improve the flow accuracy of your pump, you can compensate for the operating pressure of your analysis. On power-up, press the PRIME button on the front panel while pressing the POWER ON switch on the rear of the pump. The pump will display a number from 0 to 60 (10mL head) or 0 to 16 (40mL head). This represents the running pressure of the pump from 0 to 6000psig or 0 to 1600psig. Each digit represents 100psig. As an example, the pump is factory calibrated at 1000psig and therefore the number displayed is 10. For operating pressures higher or lower than 1000psig, change this number to equal the pressure. To change the pressure compensation number, use the ⇑ and ⇓ arrows. When you have selected the correct pressure compensation, press the RUN button to return to normal pump operation.

4.2.3 DC Power Supply

Power for the pump is provided by an isolation transformer which has taps to accommodate voltages of 115/120 or 220/230 volts AC. Selection is accomplished by changing the voltage selector (see Section 5.9). The transformer input is fused for line current. A fused linear rectifier circuit provides 38 VDC to drive the stepping motor. A linear 12 VDC supply and switching 5 VDC supply are also provided to power control and display circuits.

4.2.4 Remote Interfacing

An RS-232C modular jack is provided on the back panel. See Section 3.2 for information on pump operation via this connection.

5. MAINTENANCE

Cleaning and minor repairs of the Model 301 pump can be performed as outlined below

NOTE: Lower than normal pressure, pressure variations, or leaks in the pumping system can all indicate possible problems with the piston seal, piston, or check valves. Piston seal replacement could be necessary after 1000 hours of running time. See Section 5.2.3.

5.1 FILTER REPLACEMENT

5.1.1 Inlet Filters

Inlet filters should be checked periodically to ensure that they are clean and not restricting the flow (This could cause cavitation and flow loss in the pump). Two common causes of plugged inlet filter are microbe growth and dirty solvents. To prevent microbe growth, try to use at least 10-20% organic solvent in the mobile phase, or add a growth-inhibiting compound. If 100% water or an aqueous solution (without any inhibitors), microbes will grow in the inlet filter over time, even if a fresh solution is made every day. Always use well-filtered, HPLC grade solvents for your mobile phase.

5.1.2 Outlet Filter

To service the outlet filter on standard steel pumps

1. Unscrew the filter closure from the filter housing

   CAUTION: Do not use a metal object such as a screwdriver or paperclip to remove the seal. Doing so can scratch the precision surface of the seat and may cause the filter to leak.

2. Use a seal insertion/removal tool or a non metallic object (such as a wooden toothpick) to remove the large seal that remains in the housing
3. Unscrew the old filter and remove the small seal from the filter closure
4. Place one of the small seals included in the replacement element kit over one of the new filters from the kit. Screw the new filter into the filter closure (finger tight)
5. Place one of the large seals from the replacement kit on the filter closure. Insert the filter closure into the housing and tighten

To service the Metal-free outlet filter, simply open the filter housing and clean or replace the filter element inside.

5.2 CHANGING PUMP HEADS

5.2.1 Removing the Pump Head

As a guide to pump head assembly, the standard pump heads are shown in Figures 4 through 7. All of the Model 301 pump heads have a similar arrangement.

1. Turn OFF the power to the Model 301 pump.
2. Remove the inlet line and filter from the mobile phase reservoir. Be careful not to damage the inlet filter or crimp the Teflon® tubing.
3. Remove the inlet line from the inlet check valve.
4. Remove the outlet line from the outlet check valve.
5. Remove inlet and outlet flushing check valves.
6. Momentarily turn ON the Model 301 pump and quickly turn OFF the power upon hearing the refill stroke. This reduces the extension of the piston and decreases the possibility of piston breakage.
7. Unplug the power cord.
8. Carefully remove the two knurled nuts at the front of the pump head.

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

9. Carefully separate the pump head from the pump. Move the pump head straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the seal and seal backup washer from the piston if they did not stay in the pump head.
10. Carefully separate the flush housing from the pump. Move the flush housing straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the self flush seal from the piston if it did not stay in the flush housing.

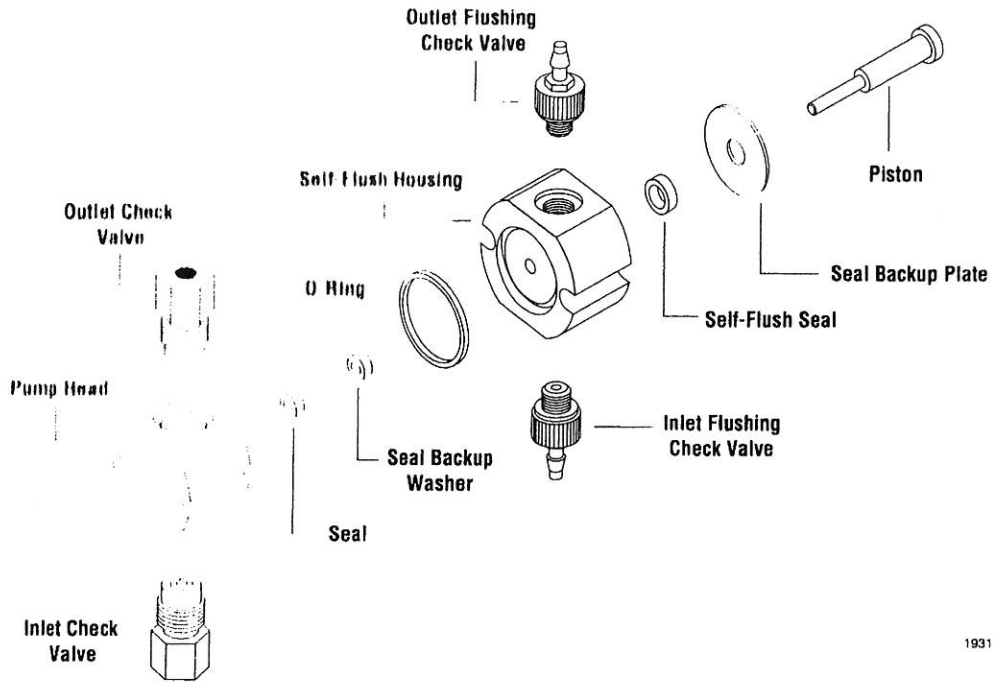

Figure 4 - Stainless Steel Self-Flushing Pump Head Assembly

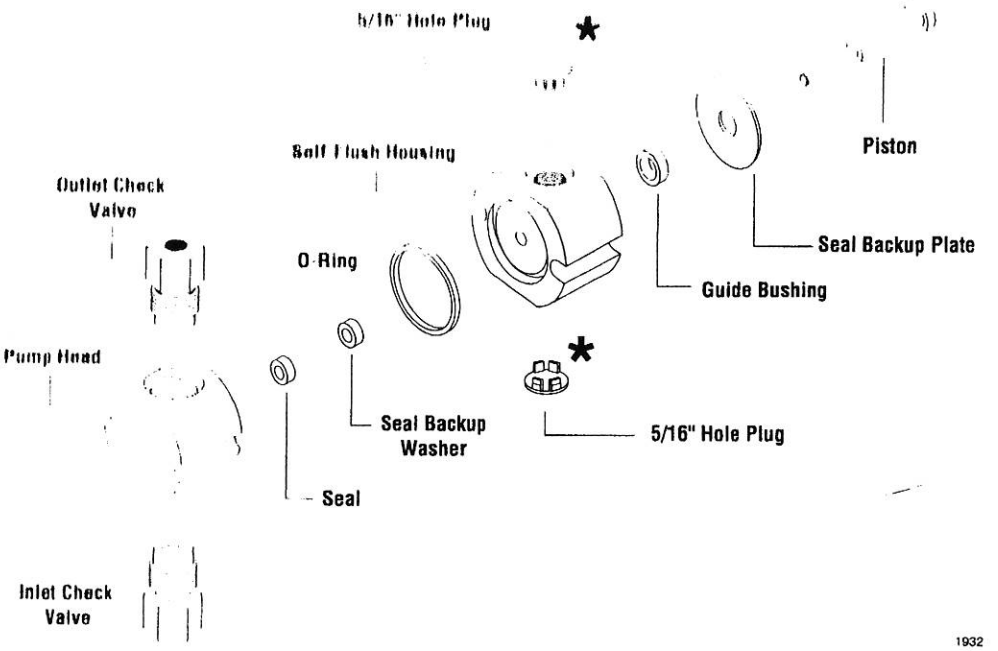

Figure 5 - Stainless Steel Non-Self-Flushing Pump Head Assembly

* Not included. Check valves can be used as plugs.

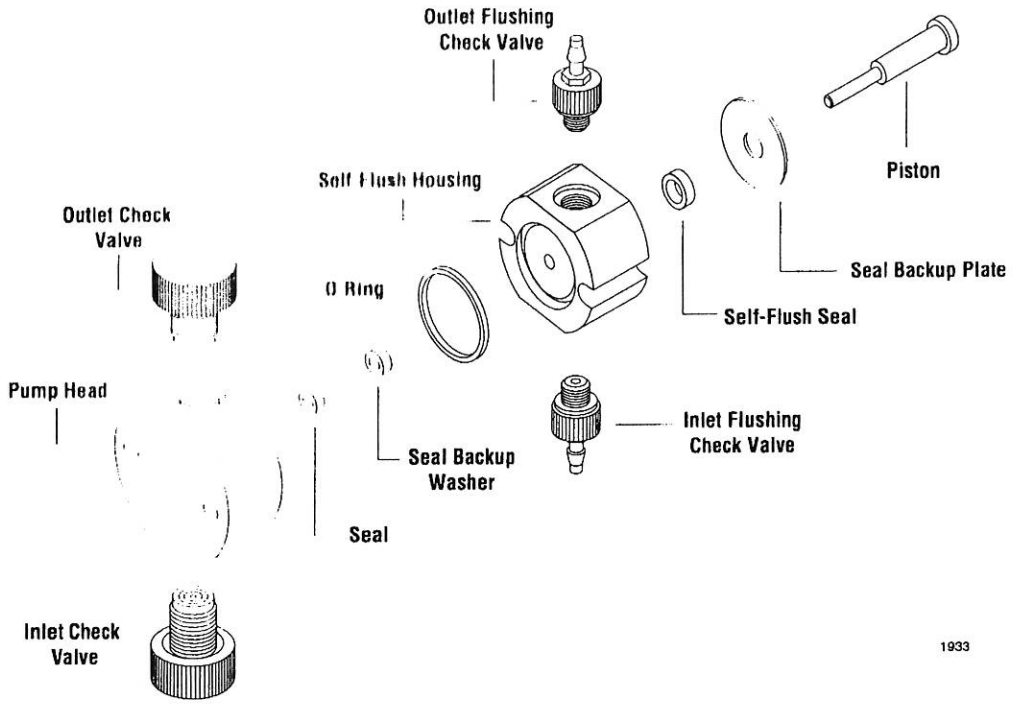

Figure 6 - Metal-free Self-Flushing Pump Head Assembly

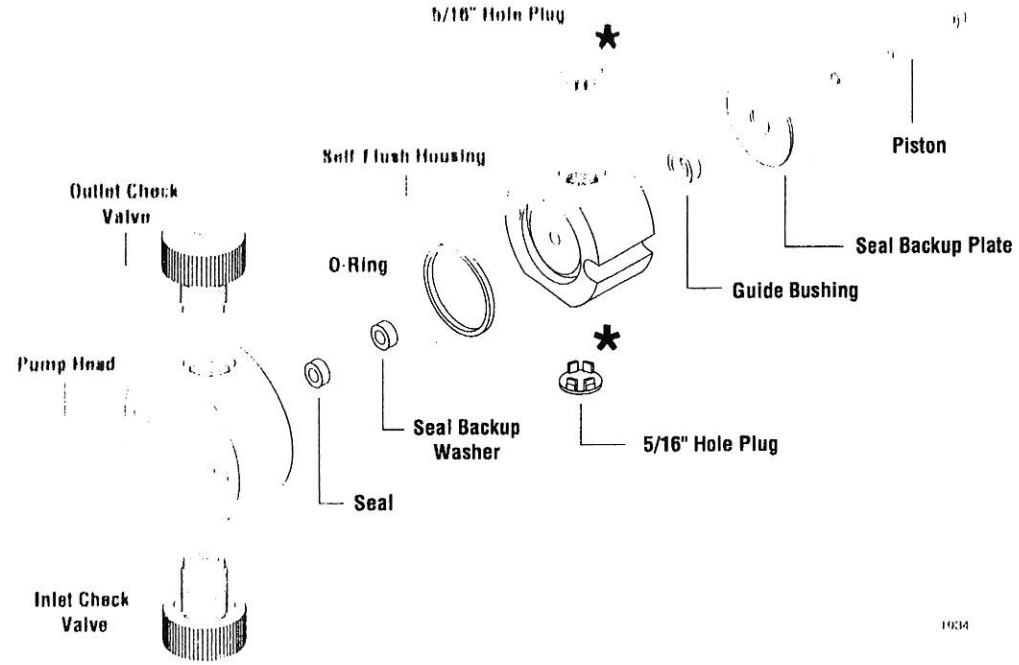

Figure 7 - Metal-free NonSelf-Flushing Pump Head Assembly

* Not included. Check valves can be used as plugs.

5.2.2 Cleaning the Pump Head Assembly

Note: Reinstallation of used piston seals is not recommended since they are likely to be scratched and damaged during removal and therefore would not provide a reliable seal if reused. Always keep a new set on hand. While removing the seals, use only the flanged end of the plastic seal removal tool supplied with the seal replacement kit, and avoid scratching the sealing surface in the pump head. See Section 5.2.3 for seal replacement instructions.

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

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

3. If the check valves had been removed, tighten the check valves on stainless steel pumps to 75psig or enough to seal at maximum pressure. For Metal free pumps, tighten each check valve firmly by hand.

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

   If the piston and flushing seals have been removed, insert new seals as described in Section 5.2.3, then continue with Section 5.2.5 to replace the pump head.

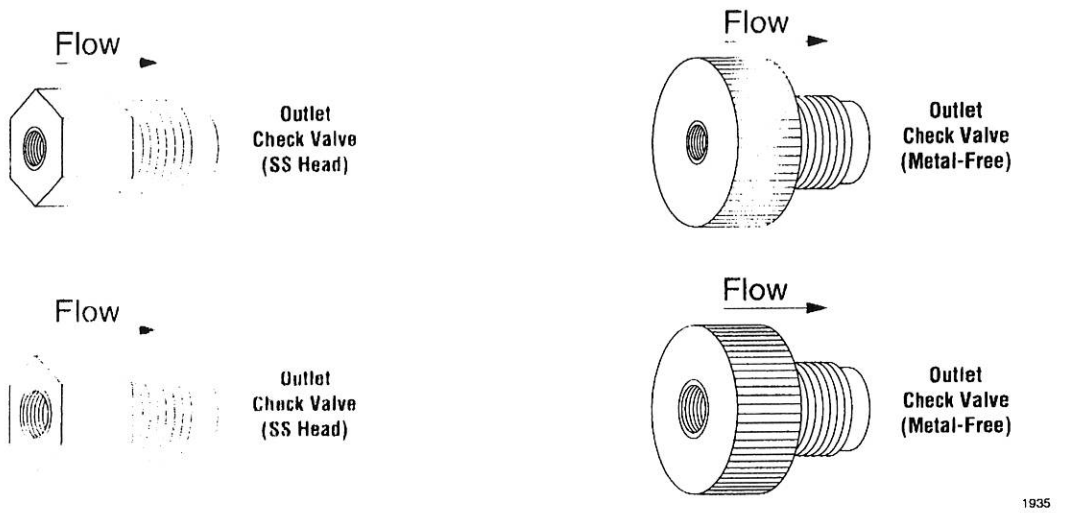

Figure 8 - Check Valves

5.2.3 Replacing Piston Seals

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

Each replacement seal kit contains one seal, one backup washer, a seal insertion/removal tool, and a pad to clean the piston when changing the seal.

5.2.3.1 Removing the Seals

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

CAUTION: Using any other "tool" will scratch the finish.

3. Repeat the procedure for the low pressure seal in the flush housing.
4. Inspect, and if necessary, clean the pump head as described in Section 5.2.2.

5.2.3.2 Cleaning the Piston

1. Gently remove the backup seal washer from the pump housing, using either a toothpick or small screwdriver in the slot on top of the pump housing.
2. Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up.
3. Use the scouring pad included in the seal replacement kit to clean the piston. Gently squeeze the piston within a folded section of the pad and rub the pad along the length of the piston. Rotate the piston frequently to assure the entire surface is scrubbed. Do not exert pressure perpendicular to the length of the piston, as this may cause the piston to break. After scouring, use a lint-free cloth, dampened with alcohol, to wipe the piston clean.
4. Grasp the metal base of the piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot.

5.2.3.3 Replacing the Seals

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

5.2.4 Changing the Piston

1. Remove the pump head as described in Section 5.2.1.
2. Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up
3. Grasp the metal base of the replacement piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot
4. Attach the pump head as described in Section 5.2.5.

5.2.5 Replacing the Pump Head

1. Make sure that the Inlet valve is on the bottom and the Outlet valve is on the top. Carefully align the flush housing and gently slide it into place on the pump. If misalignment with the piston occurs, gently push up on the piston holder.
2. Line up the pump head and carefully slide it into place. Be sure that the Inlet valve is on the bottom and the Outlet valve is on the top. Do not force the pump head into place.
3. Finger tighten both knurled nuts into place. To tighten firmly, alternately turn nuts 1/4 turn while gently wiggling the pump head to center it.
4. Reattach the inlet and outlet lines. Reconnect the self flush lines and fittings to the self-flush check valves. Change the flushing solution.

5.2.6 Changing Head Type

1. Remove the pump head as described in Section 5.2.1.
2. If appropriate, change the piston as described in Section 5.2.4.
3. Replace the pump head as described in Section 5.2.5.
4. Press and hold the RUN button on the front panel while turning on the pump. The pump will display S10 (stainless steel head, 10mL/min maximum flowrate), P10 (PEEK head, 10mL/min maximum flowrate), S40 (stainless steel head, 40mL/min maximum flowrate), P40 (PEEK head, 40mL/min maximum flowrate).
5. Press the UP or DOWN arrow buttons to change the head type
6. Press the PRIME button to return to normal operation.

5.3 CONDITIONING NEW SEALS

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

Using a restrictor coil or a suitable column, run the pump with a 50:50 solution of isopropanol (or methanol) and water for 30 minutes at the back pressure and flowrate listed under PHASE 1 below according to the pump head type. Then run the pump for 15 minutes at a back pressure and flowrate listed under PHASE 2 below.

	PHASE 1	
Pump Head Type	**Pressure**	**Flowrate**
10mL SS/MF	2000psig	<3mL/min
40mL SS/MF	1000psig	<3mL/min

	PHASE 2	
Pump Head Type	**Pressure**	**Flowrate**
10mL SS/MF	3000-4000psig	3-4mL/min
40mL SS/MF	1500psig	<6 mL/min

5.4 CHECK VALVE CLEANING AND REPLACEMENT

Many check valve problems are the result of small particles interfering with the operation of the check valve. Most problems can be solved by pumping a strong solution of liquid laboratory grade detergent through the check valves at a rate of 1mL/min (3mL/min for the 40mL pump head) for one hour. After washing with detergent, pump distilled water through the pump for fifteen minutes. Always direct the output directly to a waste beaker during cleaning. If this does not work, the check valve should be replaced.

5.5 PULSE DAMPER REPLACEMENT

5.5.1 Removing the Pulse Damper

WARNING: There are potentially lethal voltages inside the pump case. Disconnect the line cord before removing the cover. Never bypass the power grounds.

1. Make certain that the system has been depressurized. Unplug the power cord and remove the cover
2. Disconnect the tubing from the pulse damper.
3. Remove the four screws that secure the pulse damper from the underside of the pump.
4. Remove the pulse damper.

5.5.2 Pulse Damper Refurbishing

Refurbishing the pulse damper is a time-consuming procedure. The easiest method is returning the pulse damper to Alltech to have it rebuilt. Do not attempt to refill or refurbish the pulse damper without a refurbishing kit. Instructions are furnished with the kit.

5.5.3 Pulse Damper Installation

1. Position the pulse damper, aligning it with the four mounting holes in the bottom of the cabinet. Direct the port to the rear of the cabinet.
2. Secure the pulse damper by tightening the four screws located in the underside of the pump cabinet.
3. Connect the tubing from the pump head to the port at the rear of the pulse damper (i.e., toward the rear of the cabinet). Connect the line from the prime/purge valve to the other port, toward the front panel.
4. Replace the cover on the pump.

5.6 CLEANING THE PUMP

1. Disconnect the column inlet tube and from the column.
2. Direct the column inlet tube (the tube from the injector outlet) to a waste beaker.
3. Set the flowrate to maximum.
4. Turn the injector to the INJECT position.
5. Pump 100% isopropanol through the pump and injector for 3 minutes.
6. Pump 100% filtered, distilled water through the pump and injector for 3 minutes.

WARNING: Use standard laboratory procedures and extreme care when handling strong acids and bases.

7. Pump a 20% nitric acid/water solution through the pump and injector for 3 minutes.
8. Flush the pump and injector with 100% filtered, distilled water for at least 3 minutes.
9. Pump 100% isopropanol through the pump and injector for 3 minutes.

The pump is now prepared for any mobile phase or short-term or long-term shutdown.

5.7 LUBRICATION

The Model 301 pump has modest lubrication requirements. The bearings in the pump housing and piston carrier are permanently lubricated and require no maintenance. A small dab of a light grease such as Lubriplate 630-AA on the cam is the only recommended lubrication. Be sure not to get lubricant on the body of the piston carrier, as this can retard its movement and interfere with proper pumping.

NOTE: Keeping the interior of the pump free of dirt and dust will extend the pump's useful life.

5.8 FUSE REPLACEMENT

Three fuses protect the Alltech 301 pump. Two of the fuses are located in the power entry module at the rear of the cabinet and are in series with the AC input line. The other fuse is located on the motor power circuit board and is in series with the 38 VDC supply.

Troubleshooting the fuses is straightforward. If the power cord is plugged in and the on/off power entry switch is on and the fan does not run, check the two fuses in the power entry module. To gain access to these fuses, gently pry off the cover plate with a small flat-bladed screwdriver. Replace with fuses of the correct rating: 1 A slow-blo for 120 VAC pumps, or two 0.5Amp slow-blo for 240 VAC pumps.

If the front panel appears to function normally but the pump motor does not run, check the fuse located on the motor power circuit board. Replace it with a 5Amp fast-blo fuse.

5.9 VOLTAGE SELECTION

The Alltech 301 Pump operates with 120/240 VAC at 50 or 60Hz. The operating voltage is selected using the Corcom connector located on the rear of the unit. Check the voltage block next to the power cord connector on the rear of the unit. The value which appears in the window indicates the voltage for which the instrument has been configured. The voltage should be appropriately set for use with the local power supply. If the indicated voltage is not correct, reset it as follows:

1. Unplug the power cord.
2. Insert the blade of a small screwdriver into the slot on top of the power connector and open the cover (see **Figure 9**).
3. Insert the screwdriver into the slot on top of the Corcom and gently pull it out.

4a. For 120 VAC operation, the Corcom requires a single 1Amp fuse mounted on the left side with the bridge installed (see **Figure 10**).

4b. For 240 VAC operation, the Corcom requires two 0.5Amp fuses, one mounted on each side, with the bridge removed.

5. Re-install the Corcom into the voltage block with the proper voltage up, and close the cover.

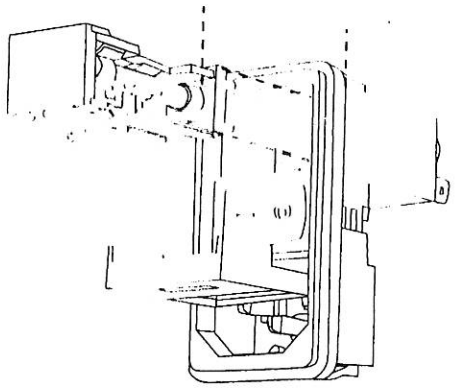

Figure 9

BRIDGE (Pt. No. **301100B**)

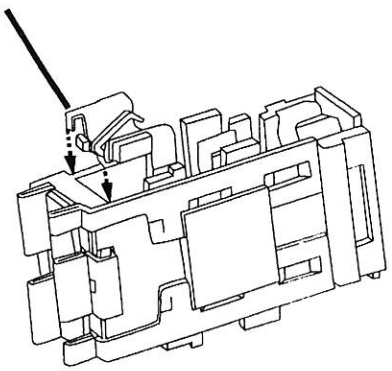

Figure 10

6. QUICK GUIDE TO PROBLEM SOLVING

QUICK GUIDE TO PROBLEM SOLVING			
You Notice	**This May Mean**	**Possible Cause**	**You Should**
1. Uneven pressure trace. 2. Pressure drops. 3. Pump shuts OFF. 4. No flow out the outlet check valve.	1. Bubble in check valve. 2. Leaks in system. 3. Dirty check valve. 4. Bad check valve.	1. Solvent not properly degassed. 2. Fittings are not tight. 3. Mobile phase not properly filtered. 4. Particles from worn piston seal caught in check valve. 5. Plugged inlet filter.	1. Check to be certain that mobile phase is properly degassed. 2. Check connections for leaks by tightening fittings. 3. Prime the system directly from the outlet check valve. 4. Clean or replace the check valves. See Section 5.4. 5. Clean or replace inlet filter. See Section 5.1.1.
1. Uneven pressure trace. 2. Pressure drops. 3. Fluid between the pump head and the chassis.	1. Leaks in system. 2. The piston seal(s) are worn.	1. Fittings not tight. 2. Long usage time since last seal change. 3. Salt deposits on seal (especially if buffered aqueous mobile phases are used without the self-flush head).	1. Check all connections for leaks. 2. Replace piston seal. See Sections 5.2 and 5.3. 3. Check the piston for salt deposits. Clean as necessary. See Section 5.2.4.
1. Pump makes a loud clanging or slapping noise (intermittent contact with cam)	1. Piston carrier is catching in piston guide.	1. Cap nut screws on the pump head are loose. 2. Seal(s) are worn. 3. Piston guide is worn 4. Salt build up on piston carrier from use of buffers 5. Excess lubricant on piston carrier.	1. Check cap nut screws on pump head. Tighten if necessary. 2. Replace seals. 3. Replace piston guide and seals. See Sections 5.2 and 5.3. 4. Consider changing to a self-flushing pump head if using buffers. 5. Clean excess lubricant and dirt off piston carrier. See Section 5.7.
1. Blue dye in mobile phase.	1. Pulse dampener diaphragm has burst.	1. Sudden pressure drop when purging system.	1. Replace pulse damper. See Section 5.5.
1. No power when pump turned ON. Fan does not run.	1. Blown fuses in the power entry module.	1. Power surge. 2. Internal short.	1. Replace only with the appropriate fuses (2Amp for 100/110 VAC or 1Amp for 220/240 VAC). 2. Contact service technician. if problem persists.
1. Front panel appears OK but pump motor does not run.	1. Blown fuse on the motor power circuit board.	1. Power surge. 2. Internal short.	1. Replace only with the appropriate fuse. 2. Contact service technician if problem persists.

QUICK GUIDE TO PROBLEM SOLVING CONTINUED			
You Notice	**This May Mean**	**Possible Cause**	**You Should**
1. PEEK fittings or components leak.	1. You cannot force PEEK parts with interferences to seal by brute force tightening	1. Film of fluid between surfaces. 2. Salt crystals between surfaces. 3. Scratches in mating surfaces	1. Clean and dry mating surfaces. 2. If scratched, replace defective part.
1. Self-flush heads leak flush solution.	1. Flush area not sealed	1. Large (Size 016) O-ring is flattened and no longer seals. 2. Head not sufficiently tightened. 3. Scratches in mating surfaces. 4. Leaky self-flush seal.	1. Replace O-ring. 2. Tighten head. 3. Replace leaky parts.

7. LIST OF REPLACEMENT PARTS

7.1 STAINLESS STEEL

Part No.	Qty	Description
STAINLESS STEEL		
120679	Ea	Check valve kit
30051	Ea	0.010" ID x 1/16" OD 316 SS tubing
30161	Ea	0.030" ID x 1/16" OD 316 SS tubing
286335	5/pkg	1/16" long male nut (for outlet tubing)
286075	5/pkg	1/16" ferrule (for outlet tubing)
20-0218	Ea	LO-Pulse damper
220292	Ea	Integrated prime/purge valve assembly
020294	Ea	Replacement seal kit for prime/purge valve
250163	Ea	High pressure outlet filter, 0.5µm
050106	2/pkg	Replacement filter elements for 250163

7.2 METAL-FREE

Part No.	Qty	Description
METAL-FREE		
060141	Ea	Metal free check valve kit
35702	Ea	0.010" ID x 1/16" OD PEEK tubing, 10ft
35708	Ea	0.030" ID x 1/16" OD PEEK tubing, 10ft
32141	Ea	PEEK 1/16" Union with fittings (10-32)
35753	10/pkg	Wrench/f ingertight nuts w/ ferrules (10-32 Thread)
32293	10/pkg	No-slip one piece fitting
35740	10/pkg	Double-sided ferrule (for 1/16" OD Tubing)
120312	Ea	Metal-free LO-Pulse damper
060115	Ea	Metal-free integrated prime/ purge valve assembly
060116	Ea	Replacement seal kit for MF prime/purge valve
060127	Ea	Metal free high pressure outlet filter, 4µm
060128	Ea	Replacement glass frit for 060127, 4µm

7.3 SEALS

Part No.	Qty	Description
SEALS		
120680	Ea	Piston seal kit, 10mL
902029	Ea	Self-flushing seal, 10mL
120861	Ea	Piston seal kit, 40mL

7.4 GENERAL

Part No.	Qty	Description
GENERAL		
32170	Ea	MF Solvent Inlet filter for 1/8" tubing, 20µm
32173	5/pkg	Replacement filter elements for 32170
120682	Ea	Flushing check valve kit
021015	Ea	Sealing washer, check valve
121486	Ea	Piston assembly, 10mL
121504	Ea	Piston assembly, 40mL
120139	Ea	Pulse damper refurbishing kit
901402	Ea	Timing belt
931617	Ea	1Amp slow-blo fuse (120 V)
931618	Ea	0.5Amp slow-blo fuse (240 V)
931619	Ea	5Amp fast-blo fuse (motor drive)
931790	Ea	Battery, 3V
021197	Ea	Injector mounting bracket
120322	Ea bracket	Column holder and mounting
931979	Ea	Power cord (North America)
931986	Ea	Power cord (Europe)
39653	Ea	Pump inlet kit (tubing, filter, 3-way valve, adapter)
447018	2/pkg	20cc pump priming syringe
20063	Ea	1/8" OD x 1.5mm ID Teflon® tubing (for pump inlet),10ft
901892	Ea	Self-flush tubing, two 24" pieces
20116	5/pkg	1/8" PEEK flange-free nut (for pump inlet tubing)
20125	5/pkg	1/8" KEL-F flange-free ferrule (for pump inlet tubing)
120800	Ea	Pump head Kit, Standard, Metal-Free
120799	Ea	Pump head Kit, Standard, SS
120802	Ea	Pump head Kit, Macro, Metal-Free
120801	Ea	Pump head Kit, Macro, SS
19255	Ea	Pressure Gauge, 0-100psig
19256	Ea	Pressure Gauge, 0-300psig
19257	Ea	Pressure Gauge, 0-1000psig
19258	Ea	Pressure Gauge, 0-3000psig
19259	Ea	Pressure Gauge, 0-5000psig

WARRANTY

Alltech warrants its products against defects in workmanship or material under normal use or service for three years. The first year of this warranty covers the cost of parts and labor, the second and third year covers the cost of parts only. All obligations or liabilities under this warranty are limited to repair or replacement, at Alltech's option, F.O.B. Deerfield, IL., of parts that are returned, freight prepaid and which are accepted as being defective upon inspection by Alltech Associates, Inc.

Components that are subject to normal wear and/or are scheduled for routine replacement within the warranty period, and/or parts, which are subjected to effects of corrosion or deterioration by chemical or other action are excluded from the above warranty. Repair or replacement will not be made under warranty for malfunction because of inadequate facilities, operating conditions or utilities. The valve, which is warranted for one year, is also excluded from the three year warranty.

Equipment and components may only be returned with Alltech's prior approval and must bear an Alltech Return Authorization Number. Call Alltech Customer Service to obtain a Return Authorization Number.

Guarantees/Warranties on accessories and equipment included by Alltech from other manufacturers are limited to the guarantees given on such equipment by the respective manufacturers.

Any modifications made to equipment covered by this warranty, without written permission from Alltech Associates, Inc. will void the warranty. Alltech reserves the right not to honor this warranty if the products are obviously mishandled by the user.

Alltech Associates, Inc. assumes no responsibility for consequential, economic or incidental damages of any nature or on-site reinstallation costs arising out of future alleged failure of any of its products or their accessories.

This warranty supersedes any and all previous warranties unless otherwise agreed upon at the time of sale, such as for customized equipment.

DAMAGED SHIPMENTS - CLAIMS - RETURNS - REPAIRS

SHIPMENTS

All shipments are made F.O.B. Deerfield, IL.

DAMAGED SHIPMENTS

The Interstate Commerce Commission has held that carriers are responsible for both concealed and visible damage occurring during transit. Unpack the shipment upon receipt and check for concealed damage even if no visible damage is apparent. If concealed damage is discovered, stop unpacking the unit, request an immediate inspection by the local carrier agent, and obtain a written report of the findings to support a claim. This request must be made within 15 days of receipt, otherwise, the claim will not be honored by the carrier. Do not return damaged goods to Alltech without first obtaining an inspection report and calling Alltech for a Return Authorization Number.

FILING OF CLAIMS

After a damage inspection report has been obtained, Alltech will cooperate in replacing damaged goods and in handling of claims, which have been initiated by either party.

RETURNS

If it is necessary to return any material to Alltech, please call Alltech's Customer Service Department for a Return Authorization Number and forwarding instructions. No returns may be made without a Return Authorization Number.

REPAIRS

Alltech Associates, Inc., is the only organization authorized to service or repair the 301 Pump. Any repairs performed without notifying Alltech Associates, Inc. will void the warranty. To obtain repair service, call Alltech's Customer Service Department for instructions and a Return Authorization Number.

Table T1. Chart used to calculate (1) travel time for PFT to drill bit based on mud pumping rate and length of drill string, and (2) HPLC pumping rate to maintain an ~1 mg/L PFT solution in the drill string.

Strokes* (min^{-1})	Liter (min^{-1})	HPLC Pump ($mL\ min^{-1}$)	Time of PFT to drill bit with pipe length (m) 5900 (min)	6000 (min)	6100 (min)	6200 (min)	6300 (min)
5	98	0.05	602	612	622	633	643
10	196	0.11	301	306	311	316	321
15	294	0.16	201	204	207	211	214
20	392	0.22	151	153	156	158	161
25	490	0.27	120	122	124	127	129
30	588	0.33	100	102	104	105	107
35	686	0.38	86	87	89	90	92
40	784	0.44	75	77	78	79	80
45	882	0.49	67	68	69	70	71
50	980	0.54	60	61	62	63	64
55	1078	0.60	55	56	57	58	58
60	1176	0.65	50	51	52	53	54
65	1274	0.71	46	47	48	49	49
70	1372	0.76	43	44	44	45	46
75	1470	0.82	40	41	41	42	43
80	1568	0.87	38	38	39	40	40
85	1666	0.93	35	36	37	37	38
90	1764	0.98	33	34	35	35	36
95	1862	1.03	[redacted]				
100	1960	1.09					

Notes: PFT = perfluorocarbc
phy; ppm = parts per m
rig floor. 1 stroke = 19.6

PTF PUMPING
CORES 5 + 22

START PUMPING WHEN PULLING 4
STOP " WHEN 4 IS ON DECK
START " " LOWERING 5
STOP " " SHOOTING 5
START " " PULLING 5
STOP " " 5 IS ON DECK

3000 M drill pipe
Time = 1/2 6000 M

CORE

HPLC pump mL/MIN	At 1750m drill string length pump strokes	minutes to fill with PFT
0.05	5.00	178.56
0.11	10.00	89.28
0.16	15.00	59.62
0.22	20.00	44.79
0.27	25.00	35.59
0.33	30.00	29.66
0.38	35.00	25.51
0.44	40.00	22.25
0.49	45.00	19.87
USE ME → 0.54	50.00	17.80
0.6	55.00	16.31
0.65	60.00	14.83
0.71	65.00	13.64
0.76	70.00	12.75
0.82	75.00	11.86
0.87	80.00	11.27
0.93	85.00	10.38
0.98	90.00	9.79
1.03	95.00	9.49
1.09	100.00	8.90

Log 201

Assume: 30 kg perfluoro carbon

50 strokes min^{-1} (= 0.54 mL/min)

$$\left(\frac{1\ \text{min}}{0.54 \times 10^{-3}\ \text{L}}\right)\left(\frac{1 \times 10^{-3}\ \text{L}}{1.76\ \text{g}}\right)\left(30 \times 10^{3}\ \text{g}\right) = 3.16 \times 10^{4}\ \text{min} = 526.1\ \text{hours}$$

pump rate — Density — Stock

= 21.92 days

for 30 strokes/min 35.9 days

Ray

30 kg PFC will last [illegible]

21.9 days [illegible]

@ 50 [illegible]