Waters
Division of MILLIPORE

Waters 1000 PrepPAK Module

Operator's Manual

MILLIPORE CORPORATION
WATERS CHROMATOGRAPHY DIVISION
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34 MAPLE STREET
MILFORD, MASSACHUSETTS 01757

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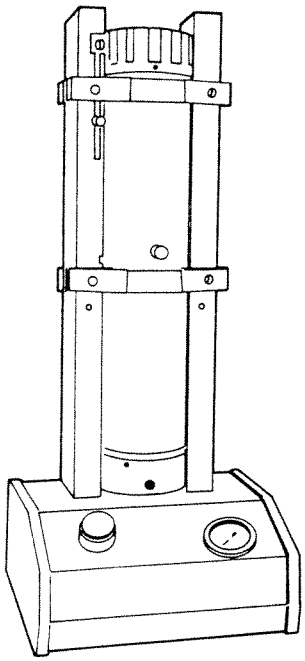

FIGURE 1-1 Waters 1000 PrepPAK MODULE

1

INTRODUCTION

The Waters™ 1000 PrepPAK® Module (referred to as the Waters 1000) is a pneumatically operated radial column compression chamber designed for use with Waters PrepPAK cartridges. The chamber applies radial pressure to the flexible walls of the Waters PrepPAK cartridge to create a stable, uniform bed. In conjunction with an appropriate Waters solvent delivery system and detector, the unit is well suited for preparative scale separation and purification.

With the control valve turned on, the system is closed by a pressure driven linear actuator that lifts the cartridge against the chamber end cap, creating a seal. Pressure then develops between the cartridge and the wall of the chamber, providing the radial compression. With the valve turned off, the radial compression is released and the actuator lowers the column, allowing the cartridge to rest on the bottom end cap.

System operating pressure is 760 psi (set at the cylinder regulator). The maximum solvent pressure delivered to the column should be set at 600 psi to allow a minimum 150 psi differential across the column wall. The maximum recommended flow rate is 200 ml per minute. Should the radial compression exceed 825 psi, a relief valve on the bottom of the unit vents the excess pressure. If the relief valve fails, and the radial compression exceeds 1100 psi, a replaceable burst disk on the bottom vents the excess pressure.

An interface connector is provided on the rear of the base of the unit, for use with a Waters Model 590 Programmable Solvent Delivery Module (referred to as the Model 590). The interface aborts further pump action should radial compression fall below 650 psi.

Options

An optional high pressure regulator (Part Number 50207) is available. The high pressure regulator delivers up to 1500 psi and accommodates a Waters 3/16″ compression screw connection.

2

INSTALLATION

The column chamber and all accessories are shipped in a single container.

After unpacking the chamber and associated hardware, check the contents against the packing list to ensure that the shipment is complete.

Inspect all items for damage.

Any damage should be reported immediately to both the shipping carrier and to Waters Chromatography Division. If any items are damaged, save the shipping container for subsequent claim purposes.

A startup kit is supplied with the Waters 1000. Check the contents of the startup kit (Part Number 89607) against the list enclosed in the startup kit.

The Waters 1000 arrives with the chamber held up by protective packing material. Prepare as follows:

1. Hold the chamber and remove the packing material. Be sure all packing material is removed from the chamber.

2. Lower the chamber to allow it to rest on the bottom end cap.

CAUTION

DO NOT SECURE THE TOP END CAP ON AN EMPTY CHAMBER. PRESSURIZING AN EMPTY CHAMBER MAY DAMAGE THE Waters 1000.

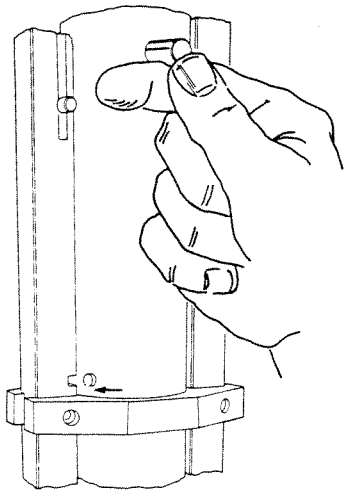

FIGURE 2-1 LOWERING CHAMBER

CONNECTIONS

Nitrogen connection

Connect the nitrogen inlet tubing assembly (Part Number 51326) from the on/off valve on the regulator to the nitrogen inlet on the back of the Waters 1000.

NOTE

DO NOT USE A NITROGEN INLET TUBE OF A DIFFERENT LENGTH OR INTERNAL DIAMETER. DOING SO MAY CHANGE THE COMPRESSION TIME AND INHIBIT CHAMBER SEALING.

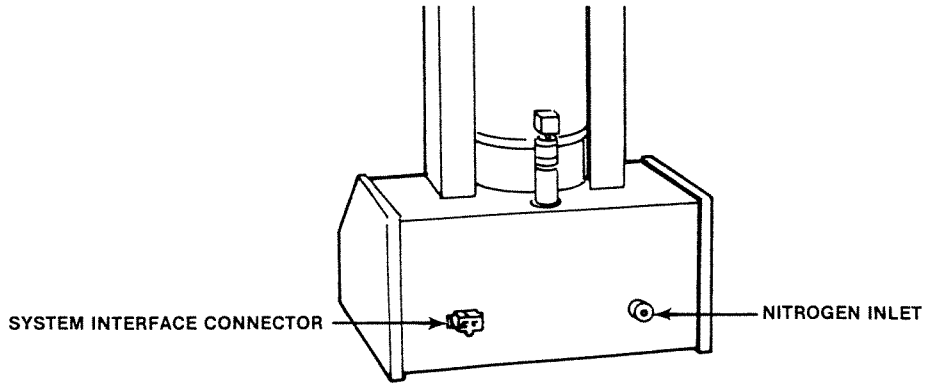

FIGURE 2-2 NITROGEN INLET AND SYSTEM INTERFACE CONNECTOR

Solvent connections

Connect the solvent inlet line (Part Number 89609) from the pump to the column inlet on the front of the bottom end cap.

Connect the solvent outlet line (Part Number 89609) from the outlet on the top end cap to the system detector.

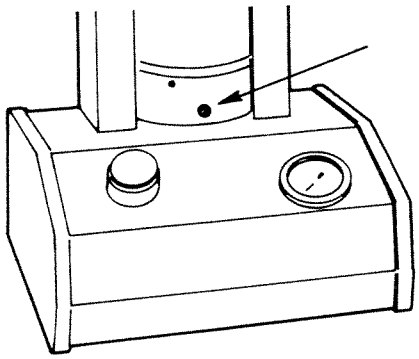

FIGURE 2-3 SOLVENT INLET

System Interface Connection

Connect the system interface cable (Part Number 89610) from the interface connector on the rear of the base of the unit (Figure 2-2) to an EVENT INPUT terminal in the event box of the Model 590. Program that event input number to shut off flow when it is turned on.

NOTE

THE Waters 1000 INTERFACES WITH THE MODEL 590 ONLY WHEN THE MODEL 590 IS IN AUTO MODE.

3

OPERATION

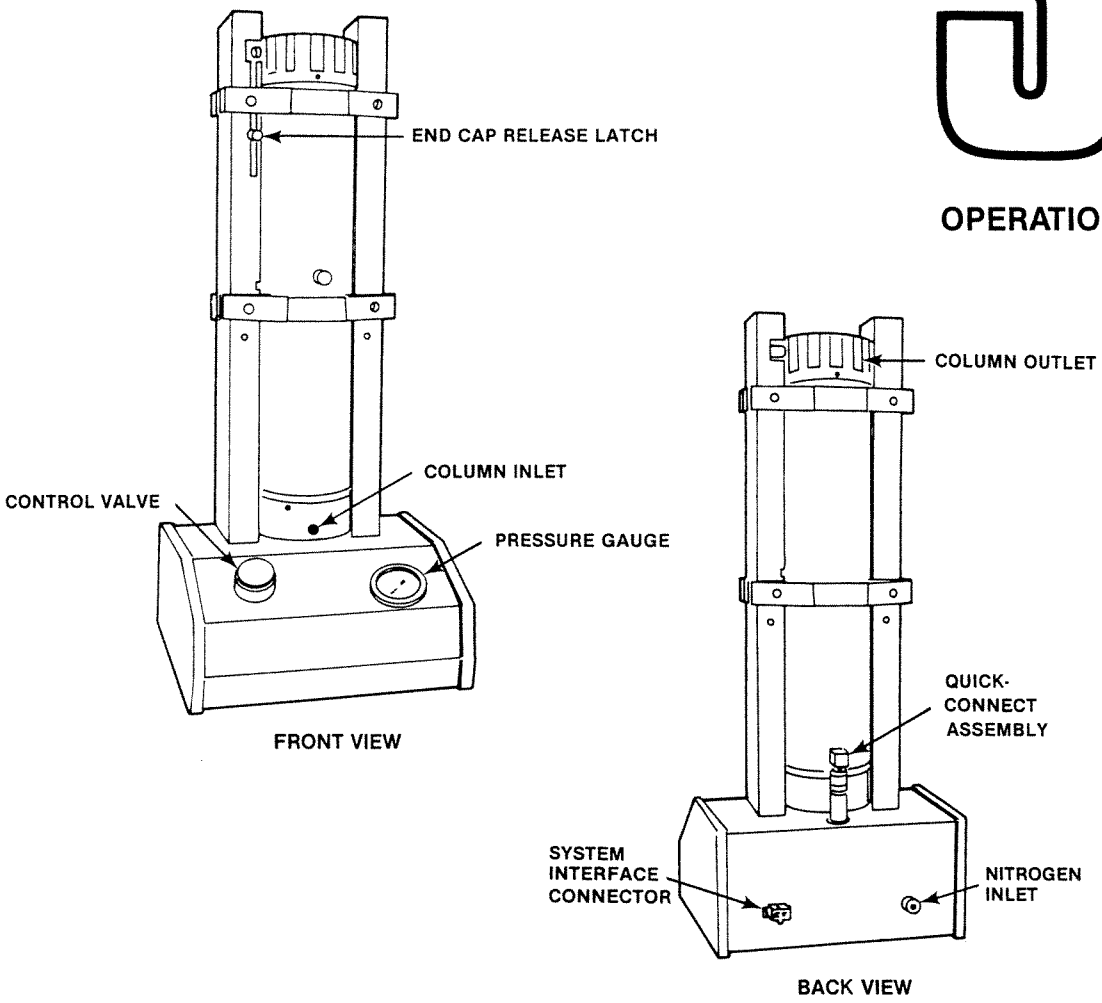

FIGURE 3-1 Waters 1000 PrepPAK MODULE

Column chamber control valve — Turn all the way to the right (clockwise) to pressurize the column chamber. Turn all the way to the left (counterclockwise) to vent the chamber. Approximately two minutes are required to fully pressurize or depressurize the chamber when using a PrepPAK cartridge.

Column chamber pressure gauge — Indicates radial compression. Maintain radial compression at 760 psi. Adjust at the gas cylinder regulator.

Column inlet — Bottom end cap

Column outlet — Top end cap

Nitrogen inlet — Provides connection from nitrogen source

Quick-connect assembly — The quick-connect stem on the chamber fits into the quick-connect fitting on the base to create a closed nitrogen pathway from the inlet to the chamber.

System interface connection — Connect to Model 590 via the event box for automatic flow shutoff if radial compression falls below 650 psi. The Waters 1000 can be operated without using this connection, but a cartridge could rupture if solvent flow is allowed to continue with radial compression below 650 psi.

End cap release latch — Secures the top end cap

TABLE 3-1 OPERATION PARAMETERS

Radial compression	760 psi
Maximum solvent pressure	600 psi
Maximum flow rate	200 ml/min

Leave the PrepPAK cartridge under radial compression when not in use to maintain uniformity of packing material.

CAUTION

NEVER PRESSURIZE THE Waters 1000 WITHOUT A PURGE CARTRIDGE OR PrepPAK CARTRIDGE INSTALLED, AS THIS COULD DAMAGE THE FERRULES OR END CAPS.

Insertion and Removal of PrepPAK Cartridges

If a PrepPAK C_{18} cartridge is to be removed and stored for future use, pump methanol or isopropanol through it and store in its container.

1. Turn the control valve counterclockwise to OFF to decompress the Waters 1000. Allow approximately two minutes for the chamber to vent and for retraction of the linear actuator.
2. Open the end cap release latch. Rotate the top end cap to release the cap pins from their slots, and lift the top end cap off.

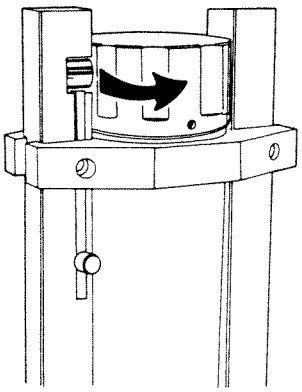

FIGURE 3-2 RELEASING TOP END CAP

3. Lift the chamber by its front handle. Rotate the chamber to the right to allow the guide pins to rest on the upright supports to hold the chamber up.

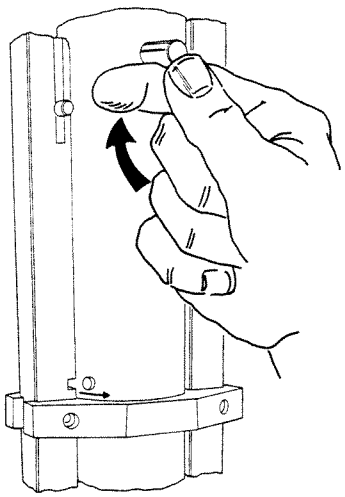

FIGURE 3-3 LIFTING COLUMN CHAMBER

4. Disengage the cartridge from the lower end cap ferrule and push it up through the chamber for removal. The cartridge may have a wrinkled appearance; this is a normal occurrence.

5. Rotate the chamber to the left to allow the guide pins to return to the side slots. Lower the chamber gently until it rests on the bottom end cap.

CAUTION

DO NOT SECURE THE TOP END CAP ON AN EMPTY CHAMBER. PRESSURIZING AN EMPTY CHAMBER MAY DAMAGE THE Waters 1000.

6. If purging is required before inserting the new cartridge, see the following section on system purging.

7. Insert the new PrepPAK cartridge into the chamber and seat it on the bottom end cap. Replace the top end cap. Open the end cap release latch and rotate the end cap clockwise to insert the end cap pins into their slots. Close the end cap release latch.

CAUTION

BE SURE TO FULLY ENGAGE THE TOP END CAP PINS INTO THEIR SLOTS AND TO PROPERLY CLOSE THE END CAP RELEASE LATCH.

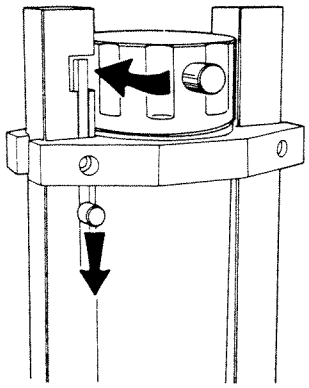

FIGURE 3-4 SECURING TOP END CAP

System Purging

Purge if there is any concern about cross contamination from the previous separation or to wash the previous mobile phase from the system.

1. Decompress the chamber by turning the control valve to OFF. Allow approximately two minutes for the chamber to fully vent. Unlatch the end cap. Rotate the cap to the right to release the pins from their slot, and remove the end cap. Raise the chamber and turn it to the right to latch the guide pins on the upright supports. Disengage the cartridge from the lower end cap ferrule and push it up through the jacket for removal.

2. Slide the PrepPAK purge cartridge (Part Number 50981) through the chamber and allow it to rest on the ferrule of the bottom end cap. Replace the top end cap. Open the end cap release latch and rotate the end cap clockwise to insert the end cap pins into their slots. Close the end cap release latch.

CAUTION

BE SURE TO FULLY ENGAGE THE END CAP PIN INTO THE SLOT AND TO PROPERLY CLOSE THE END CAP RELEASE LATCH.

3. Apply radial compression to the PrepPAK purge cartridge by turning the control valve clockwise to the ON position. Allow two minutes for compression to reach operating range in the chamber.

4. Purge the system with flushing solvent. The solvent should be miscible with both the previous solvent and the new solvent to be used. (If the instrument will be shut down, the flushing solvent should be chosen to dissolve any residual sample material or solvent remaining in the system, especially if the residue is toxic or corrosive.)

4

MAINTENANCE

4.1 PLASTIC FERRULE REPLACEMENT (Part Number 50247, Included in Startup Kit)

Change the plastic ferrules on the chamber end caps as needed. The replacement frequency varies according to the cumulative length of time the chamber is under compression. The stress of compression causes the plastic ferrule to gradually extrude and lose its effective sealing shape.

1. Remove the column as described in Chapter 3, under "Insertion and Removal of PrepPAK Cartridges."

2. Loosen the small set screws on the sides of each end cap with a spline wrench from the tool kit (included in the startup kit). This frees the ferrule in each end cap.

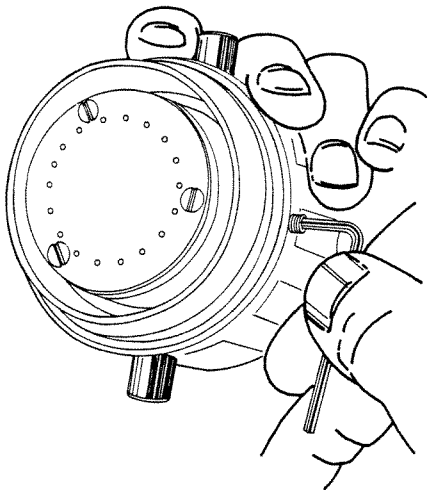

FIGURE 4-1 RELEASING THE FERRULE FROM CHAMBER END CAP

3. Grip the ferrule with a pair of pliers and pry it out of the end cap. Do not score the end cap with the pliers.

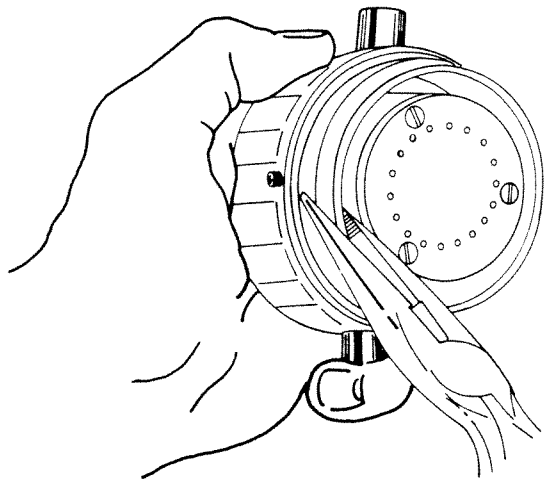

FIGURE 4-2 REMOVING EXTRUDED FERRULE FROM TOP END CAP

4. Seat the new ferrules evenly into each end cap with hand pressure only.

5. Secure the ferrule in each end cap by tightening the set screws.

4.2 SEAL REPLACEMENT

4.2.1 Bottom End Cap Seals (Part Number 89745, Included in Startup Kit)

Replace the bottom end cap seals if solvent is observed leaking from beneath the bottom end cap, or whenever the bottom end cap is removed for any reason.

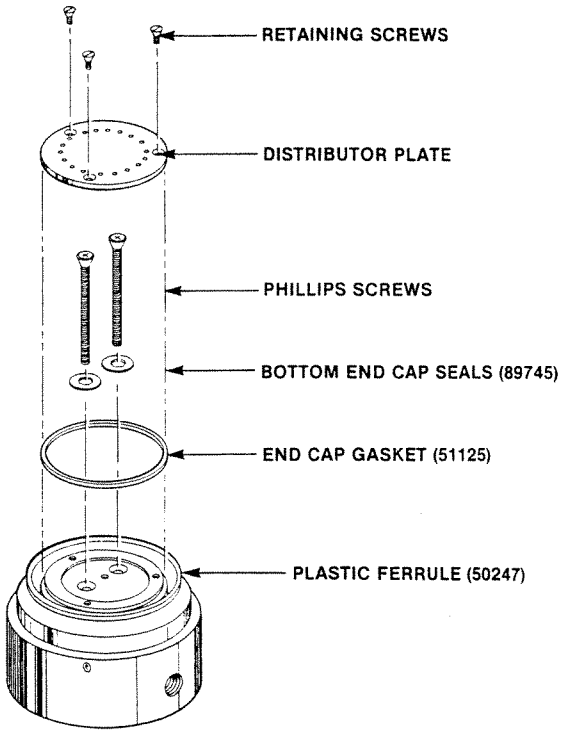

FIGURE 4-3 BOTTOM END CAP ASSEMBLY

1. Remove the cartridge as described in Chapter 3, under "Insertion and Removal of PrepPAK Cartridges." Leave the chamber up.
2. Remove the distributor plate (Figure 4-3) from the bottom end cap by removing the three retaining screws.
3. Remove the two Phillips screws from the bottom end cap.
4. The seals may come out with the Phillips screws. If not, remove them from the beveled screw holes with a small screwdriver or pair of tweezers. The old seals are funnel shaped.
5. Install new seals, one on each end of the Phillips screws.
6. Replace the two Phillips screws.
7. Replace the distributor plate with the retaining screws.
8. Lower the chamber and install a PrepPAK cartridge.

4.2.2 Quick-connect Fitting Seal (Part Number 75464, Included in Startup Kit)

Replace this O-ring seal if nitrogen leaks from the quick-connect fitting. Leaks may be detected with water or a commercial leak detector liquid.

1. Turn the control valve counterclockwise to OFF to decompress the Waters 1000. Allow approximately two minutes for the chamber to fully vent.

2. Raise the chamber and turn to the right to allow the guide pins to rest on the upright supports to hold the chamber up.

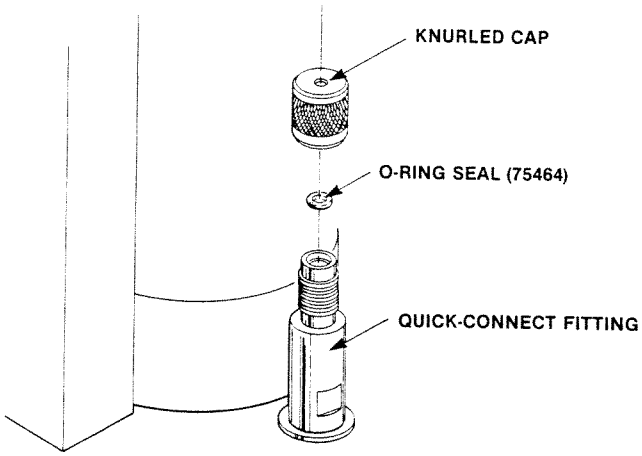

FIGURE 4-4 QUICK-CONNECT FITTING ASSEMBLY

3. Hold the bottom part of the quick-connect fitting stationary (with a 7/16″ wrench if necessary) and turn the knurled knob of the quick-connect fitting counterclockwise to remove.

4. With the top of the quick-connect fitting removed, the O-ring is exposed. Remove the O-ring with a pair of tweezers.

5. Apply a thin coat of silicone grease to the O-ring seal and install so it remains flat in the fitting. The O-ring will seat itself when the top of fitting is replaced.

> **NOTE**
>
> **TO MAINTAIN LUBRICATION OF THE SEAL, OCCASIONALLY APPLY A THIN COAT OF SILICONE GREASE TO THE QUICK-CONNECT ASSEMBLY PIN ON THE REAR OF THE COMPRESSION CHAMBER.**

6. Hold the fitting stationary and replace the knurled knob by screwing it clockwise onto the fitting, finger tight.

4.3 FILTER REPLACEMENT

Replacement of the cup filter and its O-ring in either the linear actuator line or the chamber line is indicated if the chamber does not decompress properly when the valve is turned off.

The cup filter (Part Number 25495) and accompanying O-ring (Part Number 75490) are included in the startup kit.

4.3.1 Cup Filter Replacement (Linear Actuator Line)

1. Turn the control valve counterclockwise to OFF to decompress the chamber. Allow two minutes for the chamber to fully vent.

2. Remove the right end cover (on the side of the pressure gauge). The end cover is held by four retaining screws.

3. The filter is located in a filter fitting in the chamber manifold, as shown in Figure 4-5. Disconnect the compression screw from the filter fitting with a 3/16″ wrench.

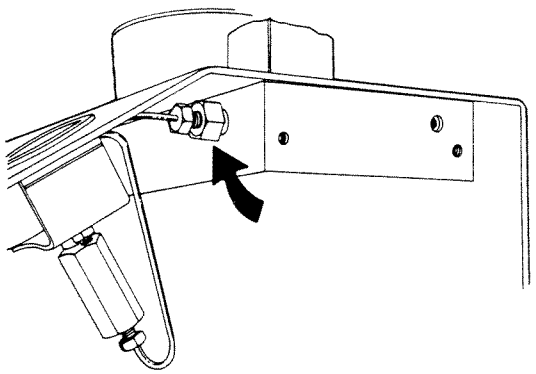

FIGURE 4-5 LINEAR ACTUATOR FILTER FITTING (RIGHT END COVER REMOVED)

4. Remove the filter fitting (Figure 4-6) with a 7/16″ wrench.

5. The cup filter (Part Number 25495) and O-ring (Part Number 75490) are in the end of the filter fitting. Remove the filter and O-ring with a pair of tweezers.

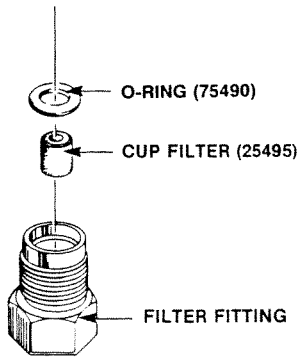

FIGURE 4-6 FILTER FITTING ASSEMBLY

6. Place the new O-ring around the open end of the new cup filter.
7. Place the new filter into the filter fitting, open end showing (Figure 4-6). The opening of the cup filter must face up to utilize the full surface area of the filter. Be sure the filter is seated into the fitting.
8. Screw the filter fitting into the chamber manifold, and tighten.
9. Connect the compression screw and ferrule, and tighten.
10. Replace the right end cover and secure with four retaining screws.

4.3.2 Cup Filter Replacement (Chamber Line)

1. Turn the control valve counterclockwise to OFF to decompress the Waters 1000. Allow approximately two minutes for the chamber to vent and for the linear actuator to retract.
2. Remove the left end cover (on the side of the control valve). The end cover is held by four retaining screws.
3. The chamber filter is located in a filter fitting in the chamber manifold as shown in Figure 4-7. The tubing from the filter fitting leads to the quick-connect fitting.
4. To access the filter fitting, remove the compression screw and ferrule (3/16″ wrench) from the tubing assembly leading to the radial compression gauge (see Figure 4-7).
5. Remove the compression screw and ferrule from the filter fitting with a 3/16″ wrench.

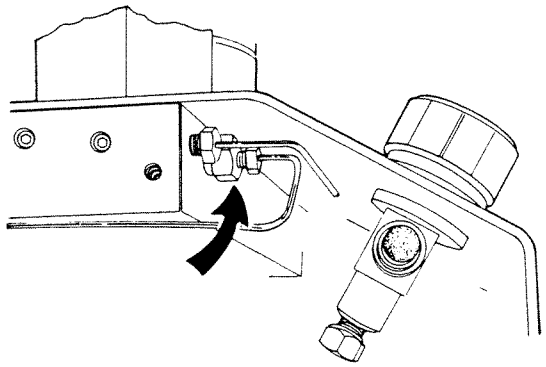

FIGURE 4-7 CHAMBER FILTER FITTING (LEFT END COVER REMOVED)

6. Remove the filter fitting with a 7/16" wrench.
7. The filter (Part Number 25495) and O-ring (Part Number 75490) are in the end of the filter fitting. Remove the filter and O-ring with a small screwdriver or pair of tweezers.
8. Place the new O-ring around the open end of the new cup filter.
9. Place the new filter into the filter fitting, open end showing (Figure 4-6). Be sure the filter is seated into the fitting.
10. Screw the filter fitting into the chamber manifold and tighten.
11. Connect the compression screw and ferrule and tighten.

NOTE

REFER TO FIGURE 4-7 TO PROPERLY PERFORM STEP 10 OF THIS PROCEDURE. BE SURE THE TUBING ASSEMBLY CONNECTED TO THE FILTER FITTING LEADS TO THE QUICK-CONNECT FITTING.

12. Connect the tubing from the radial compression gauge to the proper connection as shown in Figure 4-7.
13. Replace the left end cover and secure with four retaining screws.

4.4 END CAP GASKETS REPLACEMENT (Part Number 51125)

The end cap gaskets are beneath the distributor plate on each end cap (see Figure 4-3).

1. Remove the PrepPAK cartridge as described in Chapter 3 under "Insertion and Removal of PrepPAK Cartridges."

2. Remove the top end cap. Lift the chamber up and turn it to the right to engage the guide pins and keep it up.

3. Remove the distributor plate on each end cap. The distributor plate is held by three retaining screws. Then remove the end cap gasket. Use a pair of tweezers, if necessary, but do not score the end cap.

4. Install the new gaskets by fitting them in place. No tool is necessary.

5. Reinstall the distributor plate on each end cap and secure each with three retaining screws.

4.5 TROUBLESHOOTING

TABLE 4-1 TROUBLESHOOTING GUIDE

Condition	Possible Cause	Corrective Action
Air bubbles in detector wasteline	Solvent delivery pump	Turn pump off and observe if air bubbles are still moving. If they are, the problem lies with the radial compression chamber or cartridge
	Chamber leaking from around end cap ferrules	Check for leaks* around end cap ferrules and replace if leaking occurs per Section 4.1
	Cartridge not seated properly	Reseat cartridge and recheck for leaks
	Cracked cartridge	Replace cartridge
Failure to achieve radial compression	Nitrogen cylinder is at low pressure	Adjust regulator or replace nitrogen cylinder
	Cartridge not seating on end cap ferrules	Reseat on end cap ferrules. Replace ferrules if failure persists
	Leak at quick-connect fitting	Check with liquid around quick-connect fitting. Replace quick-connect fitting seal if necessary
	Filter in linear actuator line plugged	Replace per Section 4.3.1
Failure to decompress	Plugged filter (either chamber or linear actuator)	Remove filters per Sections 4.3.1 and 4.3.2. Replace if necessary. If neither filter is plugged, call your Waters Service Representative for assistance.

*To check for leaks disconnect the chamber inlet line from the pump and the chamber outlet line from the detector and submerge both lines in the mobile phase. Observe for bubbles.