210 Split/Splitless Inlet

The theory and procedures described in this section apply to both the standard (10 to 30 psig) split/splitless inlet and the high-pressure (0 to 100 psig) split/splitless inlet.

Theory of operation

The split mode of operation is used when small quantities of sample need to be introduced into a capillary column. The injected sample is vaporized and then split between the column and an inlet vent. Typical split ratios used are 20:1 up to 500:1, where the majority of sample is vented out the inlet split vent.

The splitless mode of operation is generally used for trace analysis. In trace analysis, sample splitting would further dilute the amount of analyte entering the column and is not desired. In splitless mode, nearly all the analyte and solvent enter the column.

To avoid solvent interferences, an initial column temperature of 25 to 30°C below the solvent boiling point is recommended and an inlet purge time of 0.5 to 1.0 minute time be used. This splitless mode of operation is best for compounds with a retention index greater than 600.

EPC inlet

Split mode operation

For an EPC inlet in split mode, the proportional valve, PV1, is a mass flow controller for the total inlet flow as measured by the flow sensor, FS. V is an on/off valve which is turned on to allow flow out of the split vent. PV2 is a back pressure regulator controlling the inlet pressure as measured by the pressure sensor, PS. SPR is a calibrated regulator/frit which is used to control the septum purge flow.

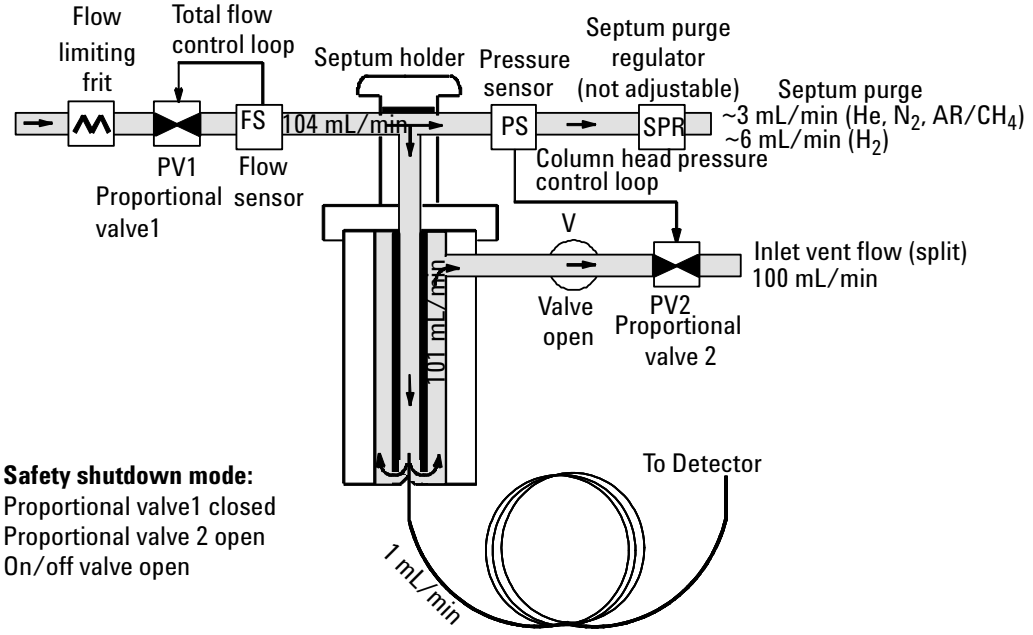


Figure 210-1 EPC control split mode flow diagram. 100:1 split ratio

Pulsed split mode

For EPC inlets in this configuration, the split flow and the column pressure (flow) are programmed up to a higher value prior to sample injection. The

split ratio remains constant due to electronic control. The actual flow rate through the inlet is much higher during the injection period, transferring the analytes quickly onto the column which minimizes sample losses that can occur in glass inlet liners. After the pulsed time expires, the split flow and column flow are returned to the lower flows selected for best chromatography.

Splitless mode

For an EPC inlet during splitless injections, PV1 is used to control the inlet pressure as measured by PS. V is an on/off valve which is turned off so there is no flow out of the split vent. PV2 is turned on at a nominal value so that there is no back pressure on V. FS will still be measuring flow, but it is no longer used to control PV1. In this configuration, the total flow into the inlet is the column flow + septum purge flow. At the user specified purge time, the inlet control is returned to split mode (see Figure 210-1) to allow the inlet to be purged.

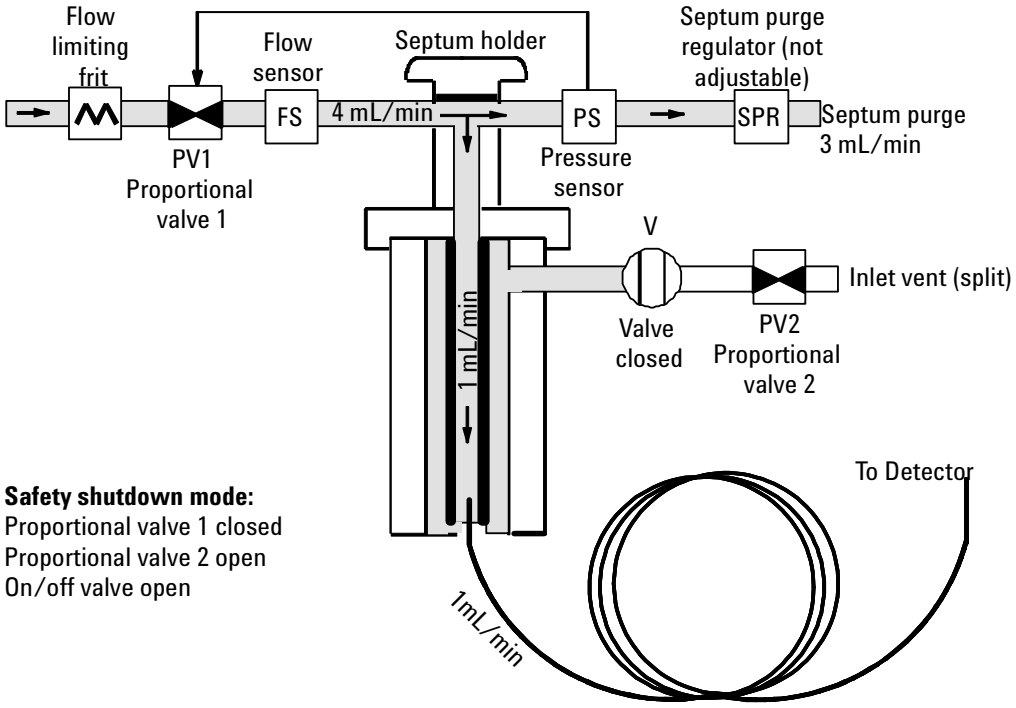


Figure 210-2 EPC control splitless mode flow diagram (pre-run to purge time).

Pulsed splitless mode

For EPC inlets set to pulsed splitless mode, the column head pressure is increased to some selected value and selected time period prior to sample introduction. Like the split mode, the increased flow through the column during the injection period minimizes sample losses in glass liners.

Gas saver mode

For EPC inlets, Gas Saver mode may be used in splitless or split mode of operation for conservation of split and inlet purge flows when not in use or in between runs. A gas saver flow rate and start time can be set independently of normal flow rates being used.

Manually controlled inlet

Split mode

For a manual inlet in split mode, V1 is a manually regulated mass flow controller that controls total inlet flow. PCV is a three-way purge control valve that remains in the normally open position to allow flow out of the split vent. V2 is a manually controlled back pressure regulator in the split vent path controlling the inlet pressure and thus, flow through the column. SPR is a manually controlled septum purge regulator used to control the septum purge flow. This flow is set to approximately 3.0 mL/minute for operation.

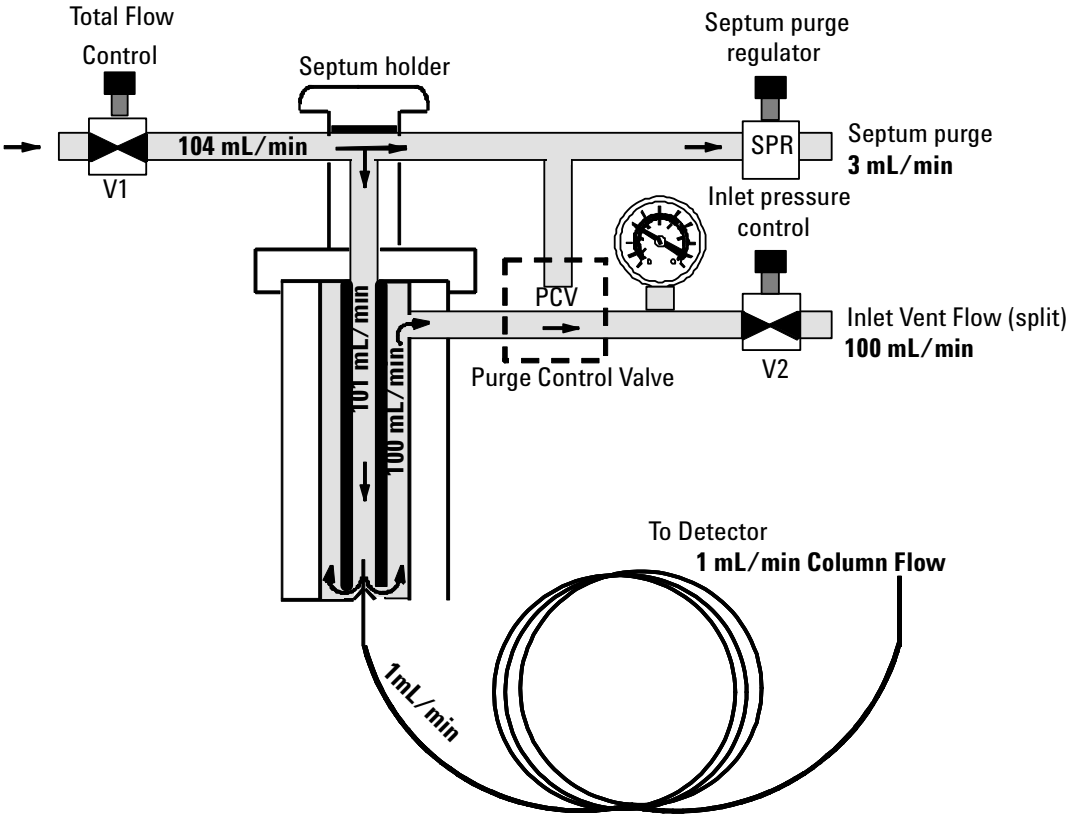


Figure 210-3 Manual control split mode flow diagram. 100:1 split ratio

Splitless mode

For a manual inlet in splitless mode, V1 is a manually regulated mass flow controller that controls total inlet flow. The Purge Control Valve (PCV) is a three-way solenoid valve that switches prior to injection to divert the higher part of the flow across the top of the liner, through the PCV and out the inlet split vent. At a user specified time after injection, the valve switches back to split mode (see Split mode) to purge remaining sample vapor out of the inlet split vent. V2 is a manually controlled back pressure regulator in the split vent path controlling the inlet pressure and thus, flow through the column. SPR is a manually controlled septum purge regulator used to control the septum purge flow.

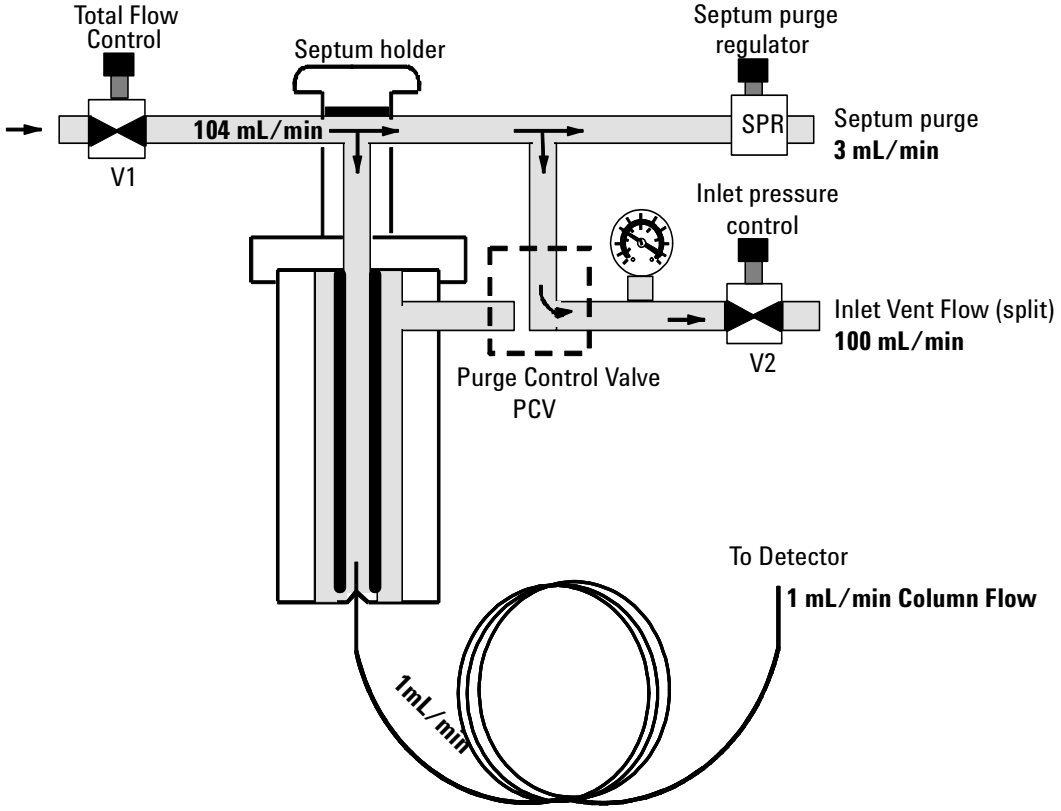


Figure 210-4 Manual control splitless mode flow diagram (pre-run to purge time).

Replacement procedures

Removing the inlet

WARNING Turn off the oven and turn off the heater of the inlet you are replacing and let them cool down. Turn off all detector flows. Turn off the carrier gas supply pressure, then turn off the main power switch and unplug the power cord.

1. From inside the oven, remove the column and column fitting from the bottom of the inlet.
2. Remove the left side cover from the GC.
3. From the top of the 6890 GC, remove the blue inlet carrier cover (or the tray bracket, if installed).
4. Unclip the heater/sensor leads from the connector to the left of the inlet carrier.
5. Unscrew the top insert assembly (large inlet nut and flow lines on the top of the inlet) that has the carrier gas and septum purge lines plumbed to it.
6. Raise this assembly up and away from the inlet

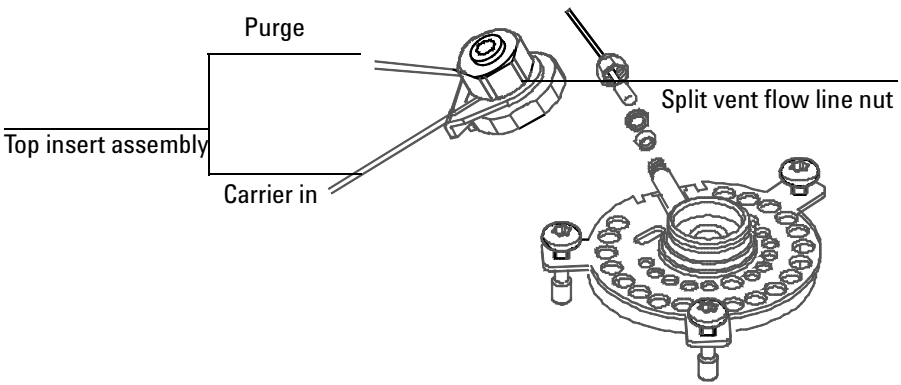


Figure 210-5 Removing the inlet

7. Remove the 1/8-inch Swagelok nut using a 7/16-inch open end wrench.
8. Use a Torx T-20 screwdriver to loosen the three captive screws that attach the inlet weldment plate to the top of the inlet carrier.
9. Pull the inlet up out of the inlet carrier. If necessary, you can also slide the insulation sleeve off of the bottom of the inlet.

Top insert assembly replacement

1. The top insert assembly is replaced as one assembly if required.

   For EPC inlets:

   a. Disconnect the pneumatics block from the front of the EPC module (one Torx T-10 screw). Be careful not to lose the three O-rings from the pneumatics module.

   b. Follow the split vent flow line back from the pneumatics block and disconnect the line from the split vent trap.

   c. Remove the carrier and septum purge lines from the left side of the GC.

   For non-EPC inlets:

   a. Unscrew the top insert assembly (large inlet nut and flow lines on the top of the inlet) that has the carrier gas and septum purge lines plumbed to it. Raise this assembly up and away from the inlet.

   b. Follow the split vent flow line back from the pneumatics block attached to the top insert assembly and disconnect the line from the split vent trap.

   c. Remove the carrier and septum purge lines from under the plastic tabs on the left side of the GC.

2. Use a 7/16-inch wrench to unscrew the split vent flow line nut from the Swagelok fitting at the top of the inlet.
3. Use a Torx T-20 screwdriver to loosen the three captive screws that attach the inlet weldment plate to the top of the inlet carrier.

4. Pull the inlet up out of the inlet carrier. If necessary, you can also slide the insulation sleeve off of the bottom of the inlet.

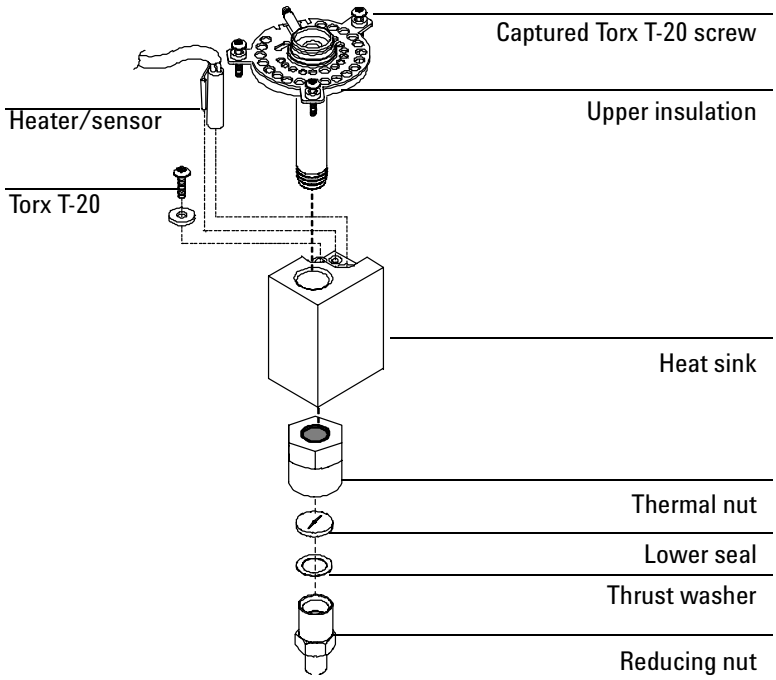


Figure 210-6 Removing the heater/sensor assembly

Heater/sensor assembly replacement

1. Follow the Removing the inlet procedure described previously in this section.
2. Remove both the bottom reducing nut (1/2-inch) and the larger thermal nut (3/4-inch) from the bottom of the inlet. Be careful not to lose the lower inlet seal and thrust washer inside the reducing nut.

Note You may want to use a vise to hold the heat sink when removing the thermal nut.

3. Slide the aluminum heat sink off of the inlet weldment.
4. Remove the Torx T-20 screw and washer from the top of the heat sink and slide the heater/sensor elements out of the heat sink.
5. Replace the heater/sensor assembly, being careful not to damage the sensor. Replace the washer/T-20 screw and reassemble the rest of the inlet.

Reinstalling the inlet

1. Make sure the heater/sensor assembly is installed and the inlet insulation sleeve is in place.
2. Install a column nut and blank ferrule on the bottom of the inlet to prevent insulation contamination, and place the inlet into the inlet carrier.

Note Make sure the insulation is properly seated around the inlet and that the heater/sensor wiring harness insulation sleeve is tucked under the top inlet plate.

3. Retighten the three screws (Torx T-20) to secure the top inlet weldment plate to the inlet carrier.
4. Reconnect the split vent flow line.

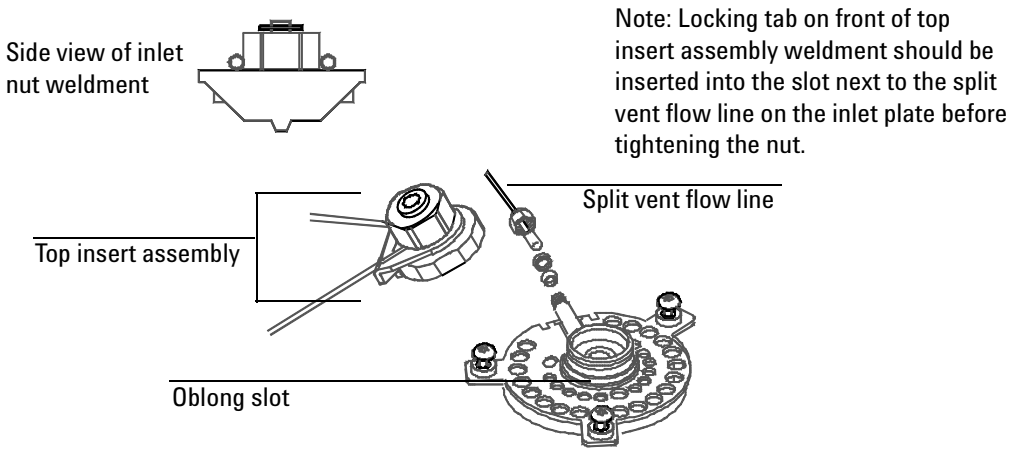


Figure 210-7 Reinstalling the inlet

5. Reinstall the top insert assembly (with septum and carrier lines attached). Make it finger tight plus a quarter turn with the inlet wrench provided in the ship kit (part number 19251-00100).

Note Make sure the locking tab fits into the oblong slot on the left side of the inlet weldment plate.

6. Tuck the “service loop” of the septum purge and carrier gas lines under the tabs on the left side of the GC.
7. Seat the heater/sensor leads into the channel on the inlet carrier.
8. Reconnect the heater/sensor assembly into the provided connector (front or back) on the left side of the GC.
9. Reinstall the insulated thermal cup and insulation in the GC oven.

10. Reinstall the blue inlet plastic cover or tray bracket to the top of the inlet area.

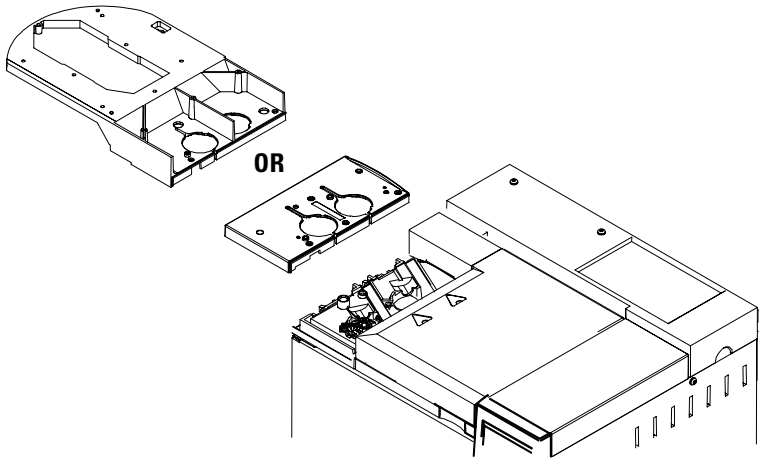


Figure 210-8 Reinstalling the inlet cover or tray bracket

Replacing the split vent trap

For GCs manufactured before May 1997, the split vent trap (part no. G1544-80550) or EPC units can be replaced by the Replacement Split Vent Trap kit, part no. G1544-60610. The new split vent trap uses a replaceable filter cartridge, which is sold in a package of 2 each (part no. G1544-80530).

To replace the filter cartridge in the trap, see Replacing the split vent trap filter cartridge below.

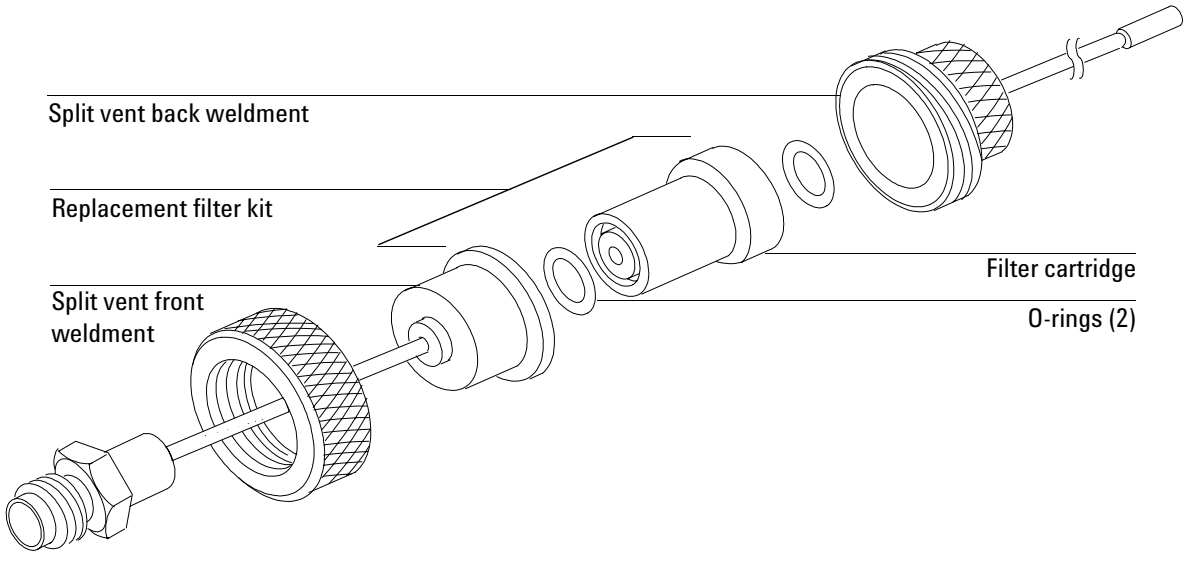


Figure 210-9 Replacement split vent filter trap kit, part no. G1544-60610

WARNING Turn off the oven and turn off the heater of the inlet you are replacing and let them cool down. Turn off all detector flows. Turn off the carrier gas supply pressure, then turn off the main power switch and unplug the power cord.

1. Disconnect the spit vent trap line from the inlet assembly.

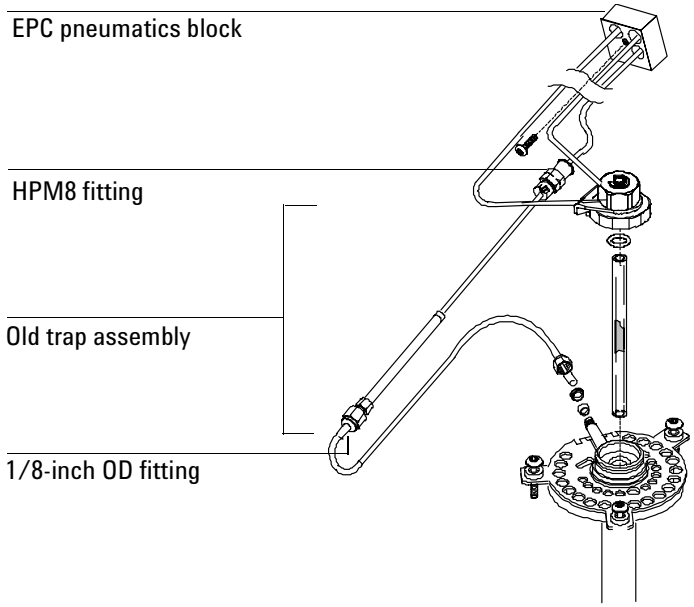


Figure 210-10 EPC manifold configuration

2. For EPC inlets, follow the split vent line from the trap to the HPM8 connector from the pneumatics block on the EPC manifold. Disconnect the plastic fitting and remove the old trap assembly and connected plumbing.

Replacing the split vent trap filter cartridge

1. Turn off the inlet and the oven and allow to cool.
2. Set all GC flows to zero.
3. Remove the pneumatics cover.
4. Lift the filter trap assembly form the mounting bracket and unscrew the filter trap assembly.
5. Remove the old filter cartridge and O-rings and replace them.

6. Reassemble the trap.

Replacing the inlet EPC flow manifold

WARNING Before proceeding, cool the heated zones, then turn off the main power switch. Set pressure/flow to 0.0. Follow ESD precautions.

All EPC inlets (and the ECD detector) in the 6890 GC use Type 1 flow manifolds.

1. Shut off the main gas supply to the manifold and remove the 1/8-inch Swagelok fitting for the gas supply.
2. Remove the plastic detector cover and the plastic pneumatics cover.
3. Remove the metal RFI shield and the rear top cover on the back of the GC.
4. Disconnect the ribbon cable for the module from the main EPC board. The adjacent ribbon cable may have to be disconnected as well.

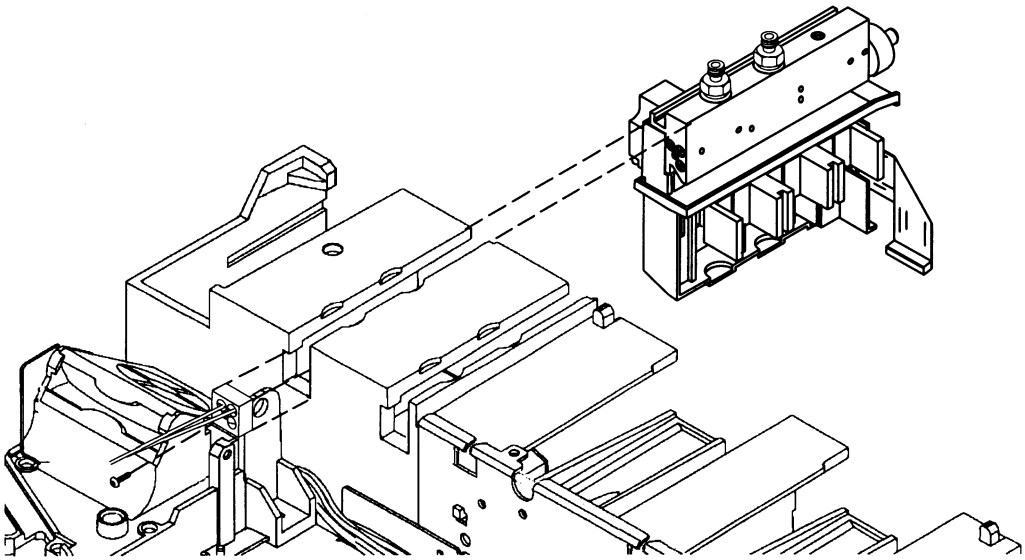


Figure 210-11 Removing the Type 1 EPC flow manifold

5. Remove the plumbing block from the front of the manifold (one captured Torx T-10 screw). Replace the three rubber O-rings behind the block if they are worn or damaged.
6. Remove the long screw (Torx T-20) from the top of the manifold and slide the manifold out of the back of the GC.
7. Reinstallation is the reverse of removal.

Replacing the supply fitting on a Type 1 flow manifold

Carrier gas enters an inlet EPC flow manifold through a fritted, stainless steel fitting. If the frit in this fitting clogs and cannot be cleaned, replace the assembly as a whole. Remove the two screws attaching the fitting to the manifold, replace the O-ring behind the fitting, and install a new fitting.

To clean the supply fitting frit, use solvents and/or an ultrasonic bath, then, dry the supply fitting with an N_2 or air stream.

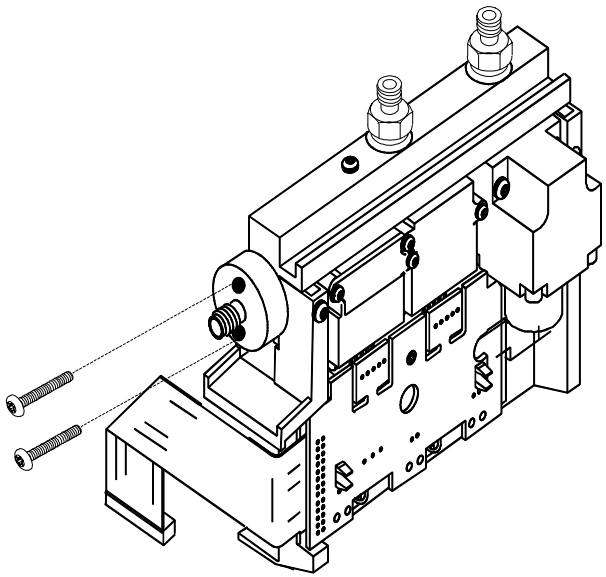


Figure 210-12 Replacing the supply fitting

Leak testing—EPC and manual inlets

Preparation

1. Cool the column to ambient, and cool inlets to below 75°C to avoid damage to deactivated glass liners.
2. Remove the column from the inlet fitting on the inside of the oven.
3. If the quality of the septum, the O-ring on the glass liner and the lower inlet seal are unknown, replace them now.

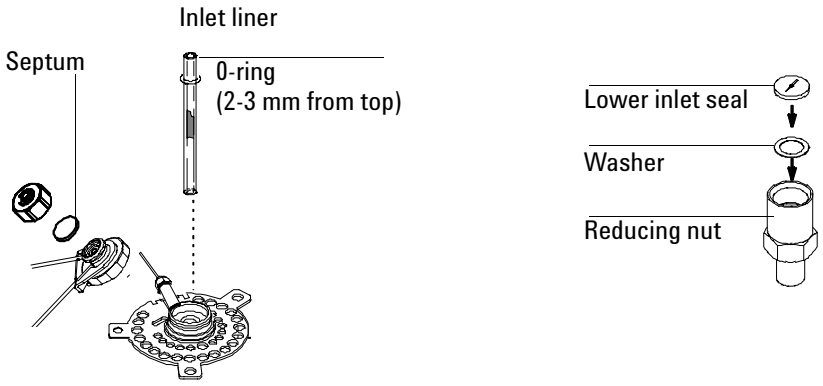


Figure 210-13 Location of septum, liner, O-ring, and lower inlet seal

4. Cap the septum purge vent and the inlet's column fitting. Use solid (no hole) Vespel type ferrules 1/8-inch (part no. 0100-1372) and 1/16-inch (part no. 5181-7458) with a 1/8-inch Swagelok nut (part no. 5180-4103) and a capillary column nut.

Note As alternate capping devices, a 1/8-inch Swagelok cap can be used for the septum purge vent. A capillary column nut with a solid piece of wire the size of a paper clip and a 0.5 mm ID graphite ferrule may be used for the inlet column fitting.

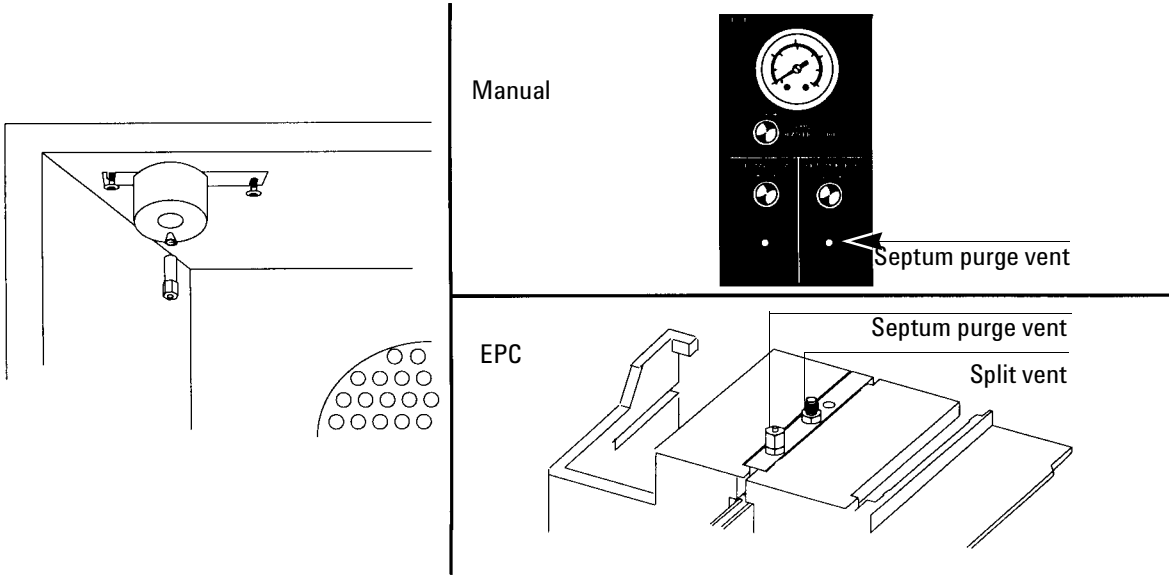


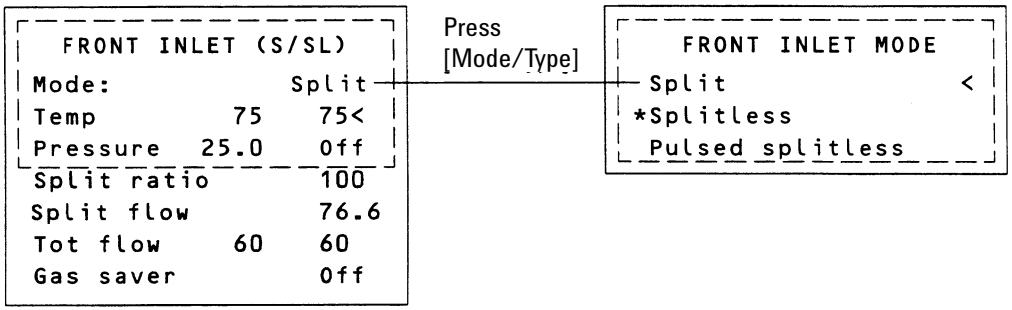
Figure 210-14 Capping the bottom of the inlet and septum purge vent

Note Make sure that the carrier gas source pressure is at least 35 psi. Carrier source pressure should always be at least 10 psi greater than the desired inlet pressure.

Performing the leak test—EPC inlets

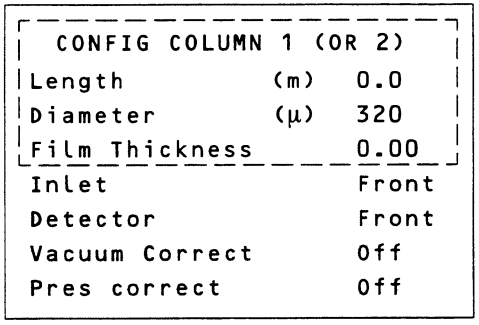
Note Be sure to complete all of the preparation steps on pages 20 and 21 before continuing.

1. Set the inlet to "Split Mode."



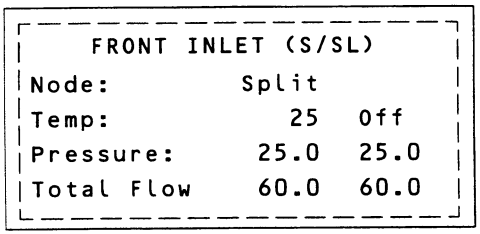
2. Configure the column as 0 length.

Press [Config] [Column 1] or [Config][Column 2] and enter "0" in the first column of the "Dim" field.



3. Set the inlet's Total Flow to 60 mL/min.

Press [Front Inlet] or [Back Inlet] and enter "60" in the "Tot flow" field.



4. Set the pressure to 25 psi.

   Scroll to Pressure and enter "25" in the "Pressure" field.

5. Wait 10 minutes for pressure equilibration.

   If pressure cannot be achieved, either a very large leak is present in the system, or the supply pressure is not high enough.

6. After 10 minutes, turn the inlet pressure "Off."

   Press [Front Inlet] (or [Back Inlet]), scroll to the "Pressure" field, and press [Off]. Both the flow controller and the back pressure valves will close.

Note When the inlet pressure or flow is turned off, the safety shutdown feature, including the audible alarm, is not functional. The inlet will not automatically shut down. This will provide you with unlimited time to locate leaks.

7. Note the “Actual” reading on the display and monitor the pressure for 10 minutes.

   You can use the stopwatch feature of the 6890 GC to monitor the time. Press [Time] and then [Enter] to start timing, then toggle between the time and the pressure reading with the [Time] and the [Front Inlet]/[Back Inlet] keys.

   - If there is less than 0.5 psi pressure loss (approximately 0.05 psi/min), consider the system leak tight.
   - If pressure loss is much greater than 0.5 psi, there is a leak that must be found and corrected. Note, however, that you may want to slightly decrease the leak test time based on the internal inlet volume, which changes with the liner type used (smaller volumes = shorter acceptable leak test times). See Correcting leaks later in this section.
   - If there is a rise in pressure, see Forward pressure valve leaks below.

8. When the system is considered leak tight, the caps may be removed, the column reinstalled, its dimensions configured at keyboard, and the desired pressure and flow rate set.

Performing the leak test—manual inlets

Note Be sure to complete all of the preparation steps on pages 18 and 19 before continuing.

1. Turn the Total Flow controller counter-clockwise to obtain approximately 60 mL/min. flow at the split vent. Use a flow meter to verify this flow rate.
2. Turn the column head pressure controller clockwise until the column head pressure reaches 25 psi.

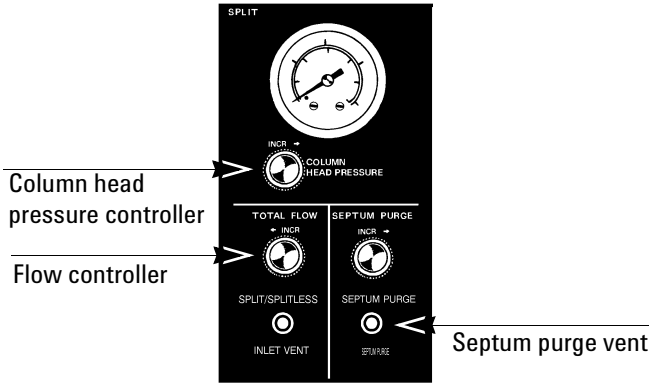


Figure 210-15 Manual flow panel

3. Wait approximately 15 seconds for equilibration.

   If pressure cannot be achieved, either a very large leak is present in the system, or the supply pressure is not high enough.
4. Shut off the flow controller by turning it fully clockwise. Do not overtighten. Use a flow meter to verify that the split flow is off.

5. Turn the column head pressure controller an additional half turn clockwise to assure that it will be closed and monitor the column head pressure gauge for 10 minutes.
   - If there is less than 0.5 psi pressure loss (approximately 0.05 psi/min), consider the system leak tight.
   - If pressure loss is much greater than 0.5 psi, there is a leak that must be found and corrected. See Correcting leaks later in this section.
6. When the system is considered leak tight, the caps may be removed, the column reinstalled, and the split flow and column pressure may be set.

Leak testing the EPC module only

Occasionally, to locate small leaks, you will need to isolate the EPC module from the inlet weldment and leak test the EPC module separately.

1. On the keyboard, turn off pressure to the inlet being tested.

   Press `[Front Inlet]` or `[Back Inlet]`, scroll to the Pressure field and press `[Off]`.

2. Use a Torx T-10 screwdriver to remove the screw in the plumbing block on the front of the module. Remove the plumbing block from the EPC module, being careful not to lose the O-rings between the block and the module.

3. Replace the inlet's plumbing block with the leak test block (part no. G1530-20660) from the leak test kit (part no. G1530-60960). Make sure you install O-rings (if needed) between the block and the EPC module to create a seal.

   The leak test block is a special fitting that plumbs the carrier gas coming out of the module directly back into the septum purge and split vent flow paths on the module. It allows the carrier gas, septum purge, and split vent line to function normally as if an inlet were present.

4. Perform the normal leak test for the EPC inlet as described previously in this section. With the inlet removed, the internal volume is quite small and a pressure loss of 1.0 psi or less in *10* minutes time is considered to be leak free.

5. If there is a leak, you will probably need to remove the flow manifold to try to isolate the leak as described in the following Locating leaks on the flow manifold procedure.

6. If there is an *increase* in pressure, see the following Forward pressure valve leaks procedure.

Forward pressure valve leaks

Occasionally an increase in pressure, rather than a decrease may be observed. This is usually due to slight leakage into the module across the forward pressure control proportional valve. Although slight leaks of this nature do not create chromatographic problems, they may obscure other small leaks that do cause problems by allowing air into the system. The valves can leak at about
0.2 mL/min and be within specification.

To check for internal valve leakage (when leak testing the EPC module only):

1. Remove the supply pressure at the carrier inlet fitting, and quickly cap the fitting with a solid 1/8-inch Vespel plug and a Swagelok nut.
2. Check the actual pressure on the display and monitor it for 5 minutes. Pressure loss should not be greater than 0.5 psi.

Locating leaks on the flow manifold

If the EPC module appears to have a leak, you can remove it to locate the leaky component. The leak test kit (part no. G1530-60960) contains a longer ribbon cable to allow you to lay the EPC module on the benchtop for testing.

Caution Be sure to wear an ESD strap grounded to the 6890 GC chassis while performing this procedure.

1. Turn off the main power switch.
2. Remove the top plastic pneumatics cover and the detector cover.
3. Remove the top rear cover on the GC.
4. Disconnect the ribbon cable for the module from the main EPC board. You may have to remove the adjacent ribbon cable also.
5. Use a Torx T-20 screwdriver to remove the screw from the top of the module and slide the module out of the back of the GC.

6. Connect one end of the leak test ribbon cable (G1530-61370) to the ribbon cable connector on the EPC module and connect the other end to the appropriate connector on the EPC board. Reconnect the gas supply and turn on the main power switch. Set the pressure to 25 psi and the flow to 60 mL/min.

7. Lay the EPC module on the lab bench and use an electronic leak detector to locate the leaky component on the module.

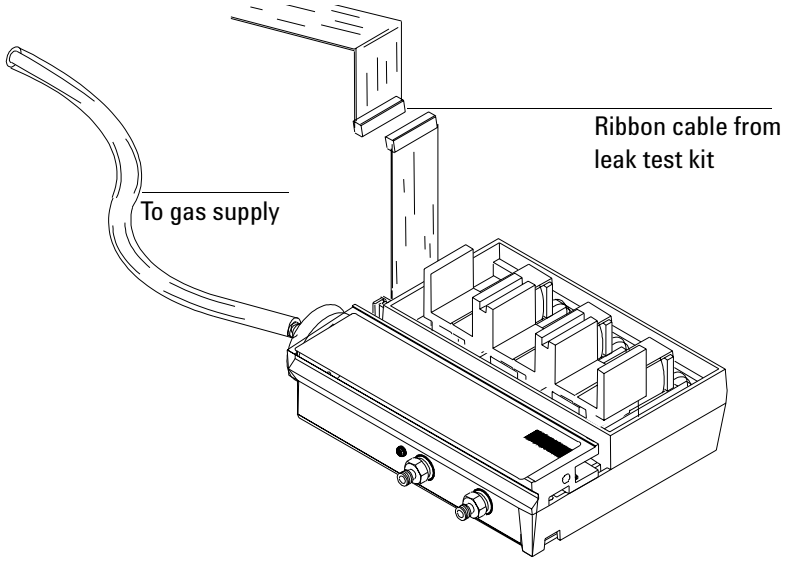


Figure 210-16 Leak testing the EPC module on the lab bench

8. If the leaky component is serviceable, such as a vent/inlet fitting (see diagram of serviceable parts, Figure 210-17), replace it. Otherwise, replace the EPC module.

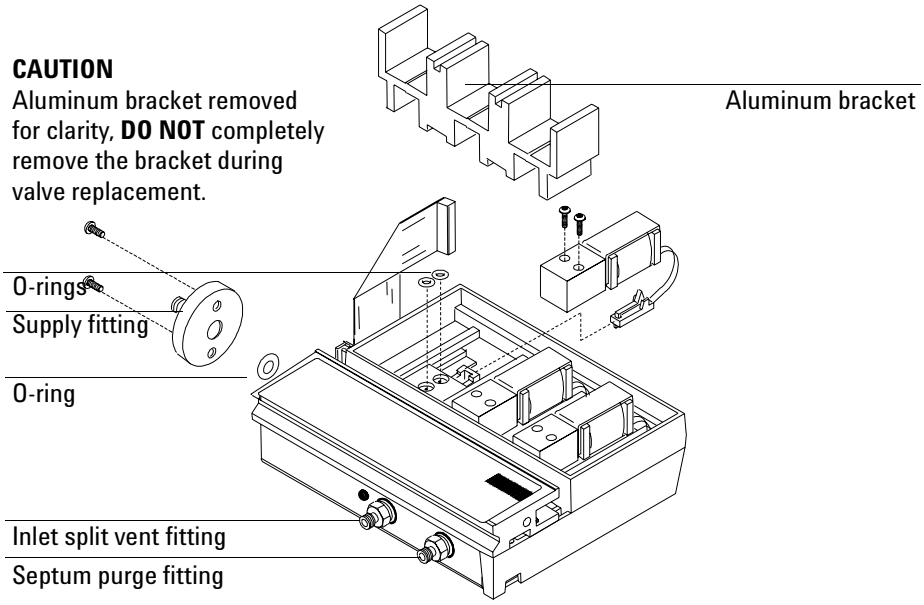


Figure 210-17 EPC module serviceable parts

Correcting leaks

1. Use an electronic leak detector to check all areas of the inlet and plumbing that are potential sources of a leak.
2. Tighten loose connections to correct leaks, if necessary. You may need to repeat the leak test.
3. If the pressure drop is now 1.0 or less, you can consider the inlet system leak-free. If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test.

Potential leak points

Check the following areas when checking an inlet system for leaks.

In the oven

Make sure the bottom of the inlet is correctly capped.

On the inlet

- Septum
- O-ring in top of inlet
- Lower inlet seal at bottom of inlet

At EPC module

- Three O-rings behind block where the inlet's pneumatic lines enter module
- Two O-rings for each valve
- Septum purge cap