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The Volatiles Interface

Using a Volatiles Interface

The volatiles interface provides a simple, reliable way to introduce a gas sample into your gas chromatograph (GC) from an external device such as the headspace, purge and trap, or air toxics samplers. The interface has a small volume and is highly inert, thus ensuring high sensitivity and resolution for applications requiring trace level detection.

Total flow to the interface is measured by a flow sensor and is divided into two streams. One stream connects to the septum purge regulator; the other connects to a frit block. At the frit block, the flow is further divided. The first stream goes to the gas-phase sampler and from there is introduced into the interface. The second stream, called the pressure sensing line, passes through the frit block and is measured by a pressure sensor. This stream also provides a trickle flow to the interface.

There are three modes of operation—split, splitless, and direct. The pneumatics vary for each operating mode and are discussed in detail in "Split mode" ,"Splitless mode" , and "Direct mode" . Table 51 summarizes some issues to consider when choosing an operating mode. Specifications for the interface are also listed.

Table 51 Overview of volatiles interface

Mode	Sample type (concentration)	Sample to column	Comments
Split	High	Very little, most is vented	
Splitless	Low	All	Can switch to split mode electronically.
Direct	Low	All	Must physically disconnect split vent, plug the interface, and reconfigure the GC. Maximizes sample recovery and eliminates possibility of contamination to pneumatic system.
Specifications			
Silcosteel®-treated flow path			
Volume:			32 µL
Internal dimensions:			2 mm by 10 mm
Maximum total flow to interface:			100 mL/min
Split range:			Dependent on column flow Typically no split to 100:1
Temperature range:			10° C above ambient (with oven at ambient) to 400° C
Recommended temperature:			≥ transfer line temperature of the external sampling device

Split mode

When you introduce a sample in the split mode, a small amount of the sample enters the column while the major portion exits from the split vent. The ratio of split flow to column flow is controlled by the user. The split mode is primarily used for high concentration samples when you can afford to lose most of the sample out the split vent and for samples that cannot be diluted.

Understanding the pneumatics

During Pre Run, during sampling, and after sampling, total flow to the interface is measured by a flow sensor and controlled by a proportional valve. Flow at the head of the column is back-pressure regulated. Pressure is sensed upstream from the proportional valve.

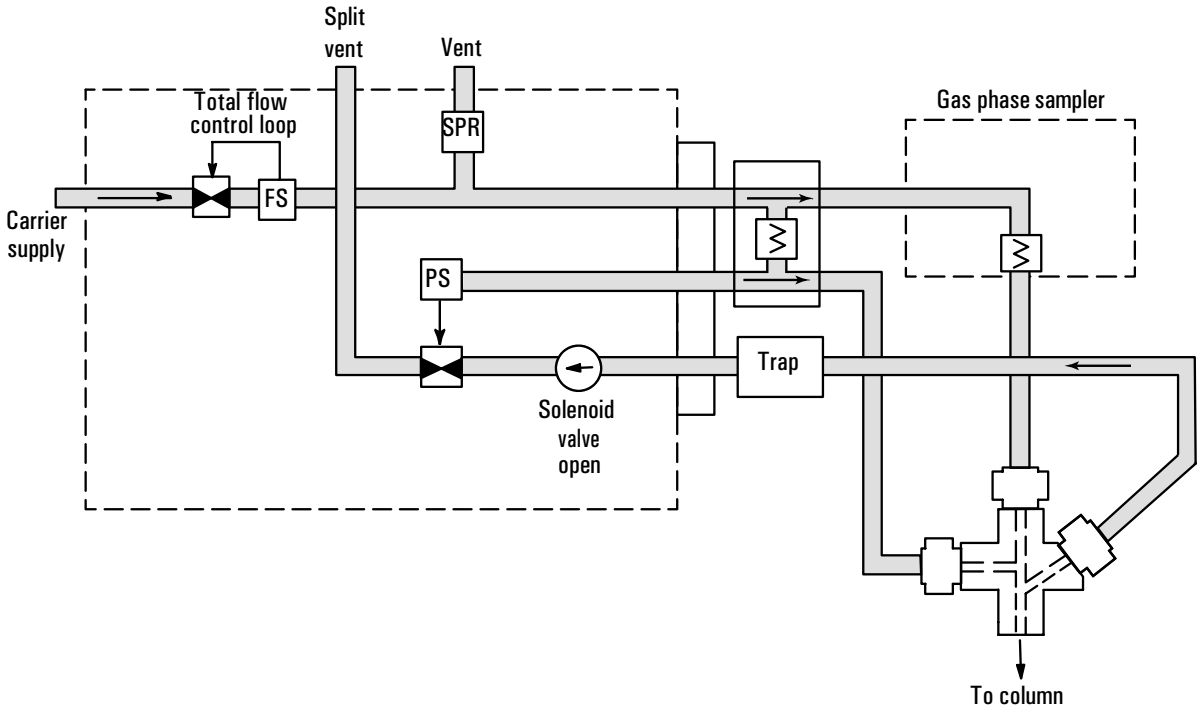


Figure 66 Pneumatics: Split mode
Splitless mode: Idle or after sampling end

Using the control table

`Mode:` The current operating mode—split

`Temp` Actual and setpoint interface temperatures

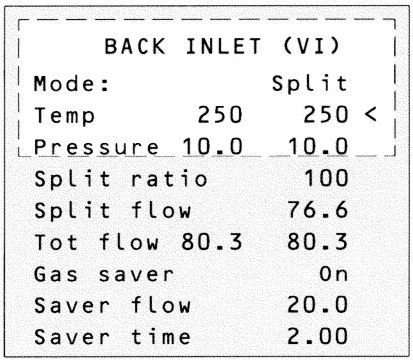
`Pressure` Actual and setpoint interface pressure

`Split ratio` The ratio of split flow to column flow. Column flow is set at the Column 1 or Column 2 control table. This parameter is not available if your column is not defined.

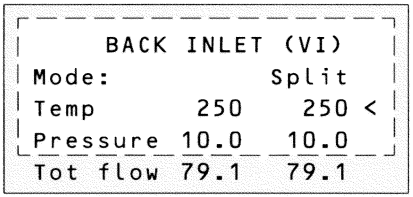
`Split flow` Flow, in mL/min, from the split vent. This parameter is not available if your column is not defined.

`Total flow` The total flow into the interface, both setpoint and actual.

Column defined



Column not defined



Some setpoints are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 52 Split mode pneumatic setpoints

Column defined	
When you change:	**These setpoints change:**
Pressure	Column flow* Split flow Total flow
Column flow*	Pressure Split flow Total flow
Split flow	Split ratio Total flow
Split ratio	Split flow Total flow
Total flow	Split flow Split ratio

*This setpoint appears in the column control table.

Column not defined

Setpoints for Column flow, Split flow, and Split ratio are not available.

You can change the setpoints for Total flow and Pressure without affecting other setpoints.

Operating parameters

Use the information in Table 53 to help you set up the operating conditions for your interface.

Table 53 Split mode operating parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	After sample on column
Interface temperature	Ambient + 10° C to 400° C	≥ Transfer line temperature
Gas saver time	0 to 999.9 minutes	After sample on column
Gas saver flow	15 to 100 mL/min	15 mL/min greater than maximum column flow

Split ratio

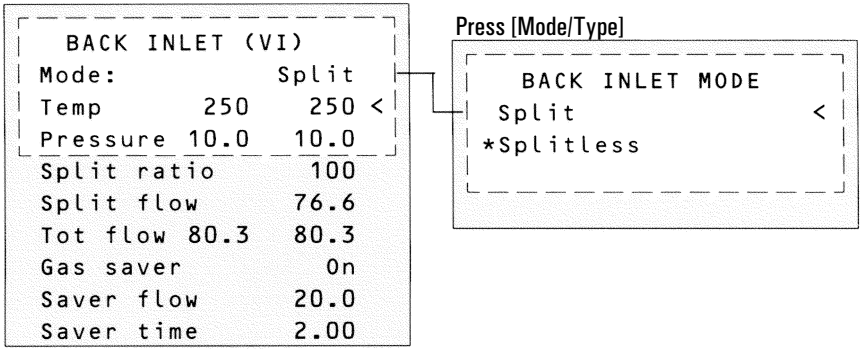
Because of the interface's small internal volume, the maximum total flow to the interface is 100 mL/min. This maximum flow puts some restriction on the split ratio you can set.

Table 54 Split ratio

Column diameter (µm)	Column flow (mL/min)	Maximum split ratio	Total flow (mL/min)
200	1	100:1	100
530	5	20:1	100

Procedure: Operating in the split mode with the column defined

1. Verify that the split vent line is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See “Flow and Pressure Control”.
3. Press [Front Inlet] or [Back Inlet].



   a. Scroll to `Mode`: and press [Mode/Type]. Select `Split`.

Split ratio = Split flow / Column flow

   b. Set the interface temperature.
   c. If you want a specific split ratio, scroll to `Split ratio` and enter that number. The split flow will be calculated and set for you.
   d. If you want a specific split flow, scroll to `Split flow` and enter that number. The split ratio will be calculated and set for you.
   e. If desired, turn on `Gas saver`. Set the `Saver time` after the sample has been introduced.
   f. If gas saver is on, be certain `Auto prep run` is On (see page 285) or use the [Prep Run] key before introducing the sample.

Procedure: Operating in the split mode with the column not defined

1. Verify that the split vent is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See “Flow and Pressure Control”.

3. Press [Front Inlet] or [Back Inlet].

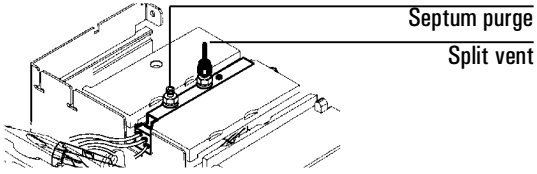
```
      BACK INLET (VI)
Mode:              Split
Temp        250     250 <
Pressure  10.0    10.0
Tot flow  79.1    79.1
```

   a. Set the temperature.

   b. Set total flow into the interface. Measure flow out of the split vent using a flow meter.

   c. Subtract the split vent flow from the `Total flow`. Subtract the septum purge flow (see "Septum purge" for nominal septum purge flows).

   d. Calculate the split ratio. Adjust as needed.



Front of GC

Splitless mode

When you introduce a sample, the solenoid valve remains closed while the sample enters the interface and is transferred to the column. At a specified time after the sample is introduced, the solenoid valve opens.

Understanding the pneumatics

Before Pre Run, when the GC is preparing for sample introduction, total flow to the interface is measured by a flow sensor and controlled by a proportional valve. Column flow is controlled via back-pressure regulation. See Figure 67.

During sampling, pressure upsets caused by switching valves in the external sampling device can cause fluctuations in column flow rates. To compensate for this, the interface is flow controlled during sampling time. The sampling flow rate is calculated from the pressure setpoint that is active when sample introduction begins. This flow control starts when the GC goes into the Pre Run state (when your system is automated and the Pre Run light is on or during manual operation when you press [Prep Run]) and ends after the interface's `Sampling end` setpoint expires.

During this user-specified sampling period, the solenoid valve is closed. Flow to the interface is measured by a flow sensor and controlled by a proportional valve. See Figure 67.

After sampling end, the solenoid valve opens. Flow to the interface is again measured by a flow sensor and controlled by a proportional valve while column flow is controlled via back-pressure regulation. The purge flow is controlled by the user. If desired, gas saver can be turned on at the end of the run. See Figure 67.

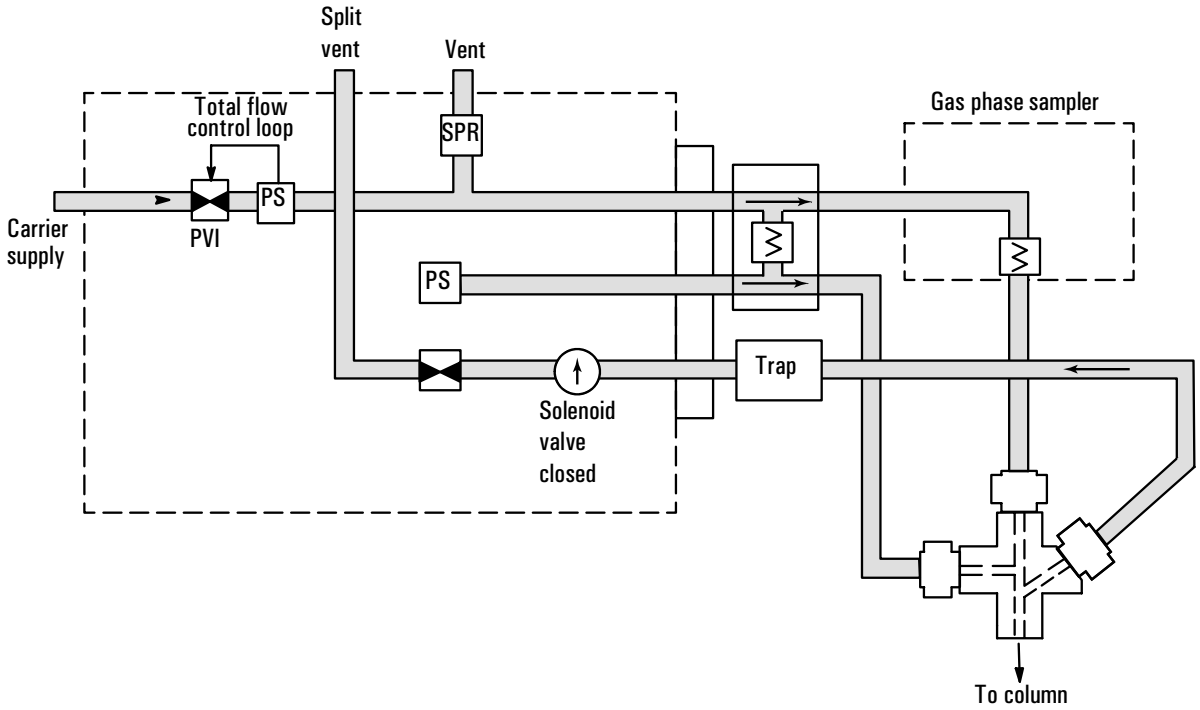


Figure 67 **Splitless mode pneumatics: beginning of pre run to sampling end *(sample introduction in progress)***

Using the control table

`Mode`: The current operating mode—splitless

`Temp` Actual and setpoint interface temperatures

`Sampl'g end` The sample introduction interval, in minutes. The flow rate is calculated from the pressure setpoint that is active at the start of sample introduction.

Set the sampling end setpoint 0.2 minutes longer than the time the sampler needs to introduce the sample. For example, the 7694 headspace sampler has an Inject time parameter which controls how long the valve remains in the inject position. If Inject time is 1 minute, the sampling end setpoint should be set to 1.2 minutes.

If you're using an 7695 Purge and Trap Concentrator, set the Sampling end setpoint 0.2 minutes longer than the Desorb time parameter.

If your column is defined and you specify a flow or pressure program for your column, the ramp does not begin until after the sampling end setpoint expires.

`Pressure` Actual and setpoint interface pressure in psi, bar, or kPa.

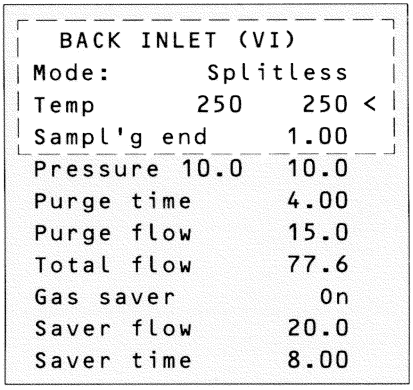
`Purge time` The time, after the beginning of the run, when purging resumes.

`Purge time` must be greater than `Sampling end`.

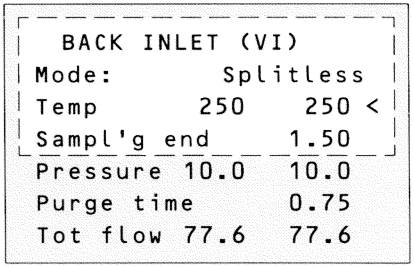
`Purge flow` The flow, in mL/min, from the split vent at `Purge time`. You will not be able to access or specify this value if operating with your *column not defined*.

`Total flow` When your column is defined, `Total flow` displays the actual flow to the interface. You cannot enter a setpoint. If your column is not defined, `Total flow` will have both setpoint and actual values during purge time. All other times, the actual flow to the interface is displayed.

Column defined



Column not defined



Some setpoints in the flow system are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 55 Splitless Mode Pneumatic Setpoints

Column defined	
When you change:	**These setpoint change:**
Purging	
Purge flow	Total flow**
Pressure	Total flow** Column flow*
Column flow*	Pressure Total flow**
Before and after sampling, not purging	
Pressure	Column flow* Total flow**
Column flow*	Pressure Total flow**
During sampling: You cannot change pressure and flow setpoints during sampling time.	

*This setpoint appears in the column control table.
**This value is actual only.

Column not defined
Purging: You can change the Pressure and Total flow setpoints; other setpoints are not affected.
Before and after sampling, not purging: You can change the Pressure setpoint; other setpoints are not affected.
During sampling: You cannot change pressure and flow setpoints during sampling time.

Operating parameters

A successful splitless injection consists of these steps:

1. Introduce a gas sample into the heated interface.
2. Use a low oven temperature while the sample collects at the head of the column.
3. Set your sampling end time to allow the entire sample to be swept out the sampler.
4. Set the purge time so that all the sample has collected on the column.
5. Begin your oven temperature program.

Table 56 Splitless Mode Operating Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	≥ Interface purge time
Interface temperature	Ambient + 10° C to 400° C	≥ Transfer line temperature
Interface sampling end	0 to 999.9 minutes	0.2 Minutes longer than introduction time
Interface purge time	0 to 999.9 minutes	
Gas saver time	0 to 999.9 minutes	Must be after purge time
Gas saver flow	15 to 100 mL/min	15 mL/min greater than maximum column flow

Procedure: Operating in the splitless mode

These instructions apply to both column *defined* and *not defined*.

1. Verify that the split vent line is connected to your interface. Verify that the [Config][Inlet] control table displays “split plumbed”.
2. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
3. Press [Front Inlet] or [Back Inlet].
   a. Scroll to `Mode`: and press [Mode/Type]. Select `Splitless`.
   b. Set the interface temperature and a sampling end time.

Column defined

```
  BACK INLET (VI)
Mode:       Splitless
Temp       250    250 <
Sampl'g end        1.5
Pressure 10.0    10.0
Purge time         1.75
Purge flow         15.0
Total flow         77.6
Gas saver           On
Saver flow         20.0
Saver time         2.00
```

Column not defined

```
  BACK INLET (VI)
Mode:       Splitless
Temp       250    250 <
Sampl'g end       1.50
Pressure 10.0    10.0
Purge time         0.75
Tot flow 77.6    77.6
```

If using gas saver, set time after purge flow time.

   c. If your column is defined, enter a purge time and purge flow. Turn Gas saver on if desired. Set the `Gas saver` time *after* the purge time and enter a Gas saver flow.
   d. If your column is not defined, enter a purge time (purge flow is not available). Set total flow greater than column flow plus septum purge flow (about 6 mL/min) to guarantee adequate column flow.
4. Make certain `Auto Prep Run` is On (see page 285) or use the [Prep Run] key before introducing a sample.

Direct mode

Direct sample introduction permits a quantitative transfer of analyte without risking contamination to the pneumatic system. It provides the sensitivity required for air toxics analyses. The interface's minimal dead volume also eliminates the potential interaction of solutes with poorly swept, active surfaces.

To operate in the direct mode, you must physically disconnect the split vent and reconfigure the GC. Instructions for performing these procedures are discussed in "Connecting to an External Gas Sampler".

Understanding the pneumatics

Before Pre Run, the interface is forward pressure controlled; pressure is sensed downstream from the flow proportional valve. See Figure 68a.

During sampling, pressure upsets caused by switching valves in the external sampler can cause fluctuations in column flow rates. To compensate for this, the interface is flow controlled during sampling time. The sampling flow rate is calculated from the pressure setpoint that is active when sample introduction begins. This flow control starts when the GC goes into the Pre Run state (when your system is automated and the Pre Run light is on or during manual operation when you press [Prep Run]) and ends after the interface's `Sampling end` setpoint expires.

Flow to the interface is measured by a flow sensor and controlled by a proportional valve. See Figure 68b.

After sampling end, the interface is forward pressure controlled; pressure is sensed downstream from the proportional valve. See Figure 68a.

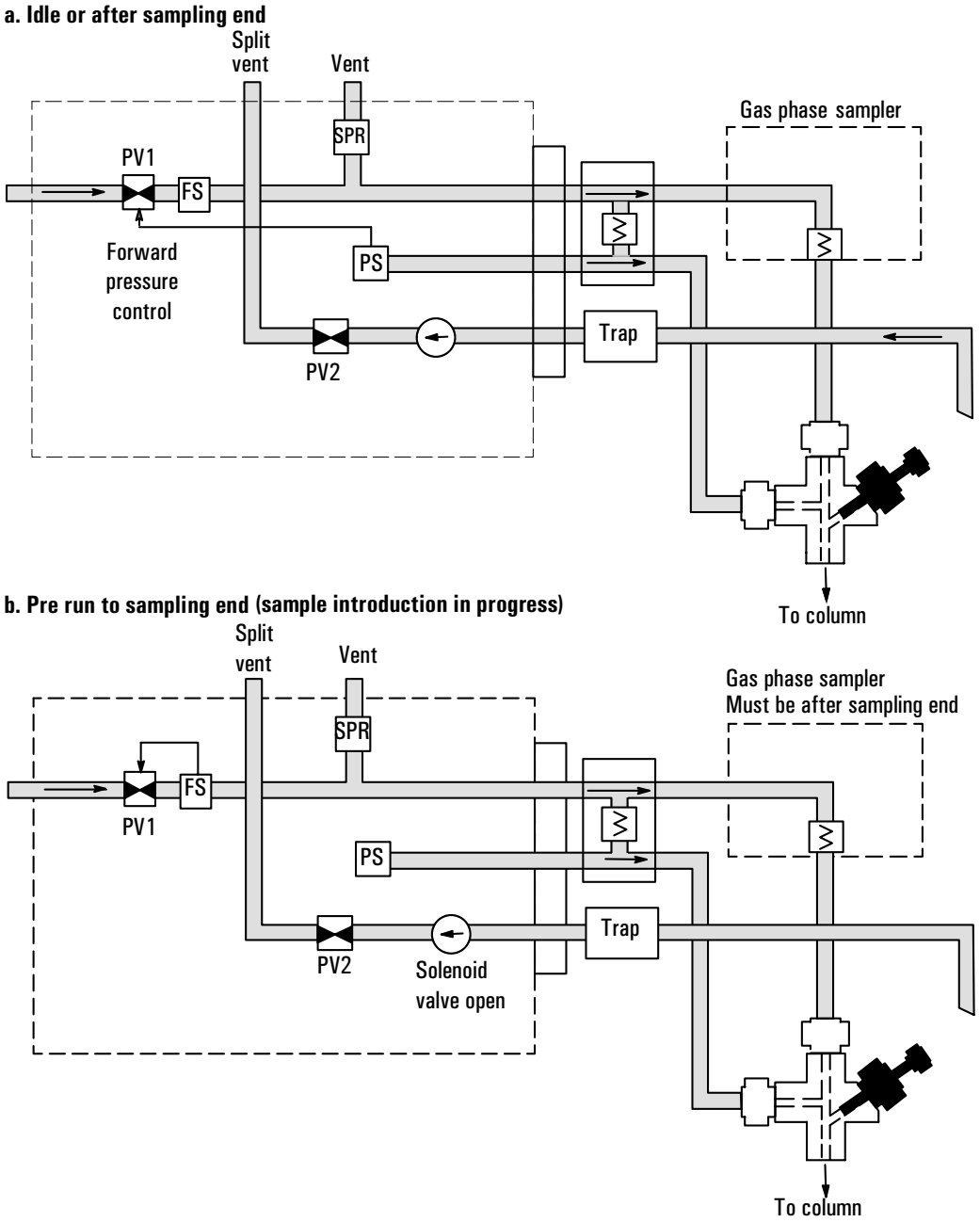


Figure 68 Pneumatics for direct mode

Preparing your interface for direct sample introduction

Before you can operate your interface in direct mode, you must:

- Disconnect the split vent line
- Configure the GC for a direct injection

Procedure: Disconnecting the split vent line

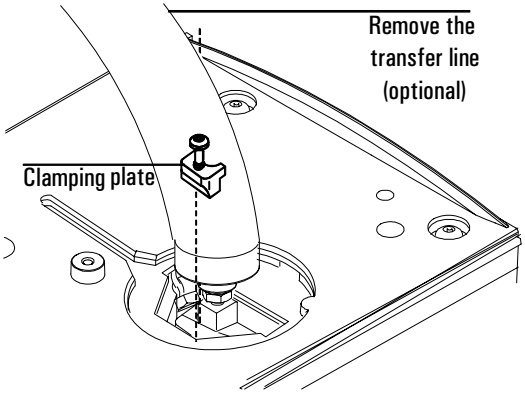
WARNING Be careful! The interface may be hot enough to cause burns.

Materials needed:

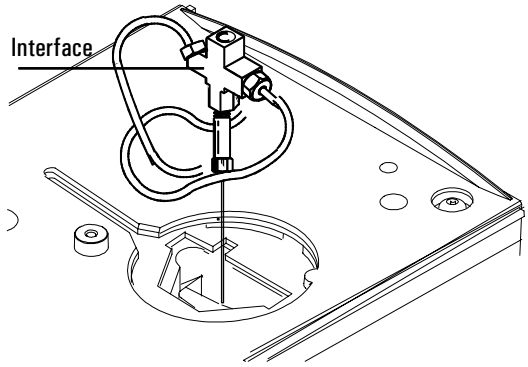
- Blanking nut
- 1/4-inch wrench
- 5/16-inch or adjustable wrench
- T-20 Torx screwdriver

1. Press [Front Inlet] or [Back Inlet] and turn off the interface temperature and pressure. Allow the interface to cool.
2. If desired, remove the transfer line by loosening the hex nut with a 1/4-inch wrench. Remove the clamping plate from the interface by

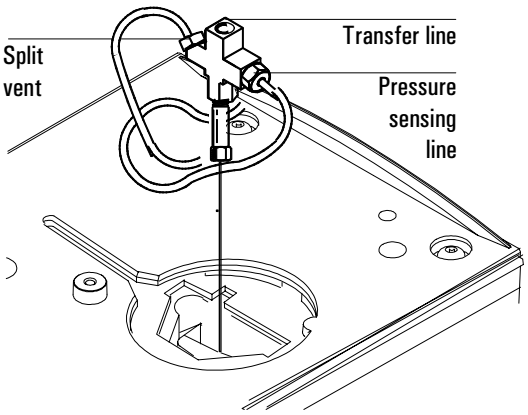
   loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



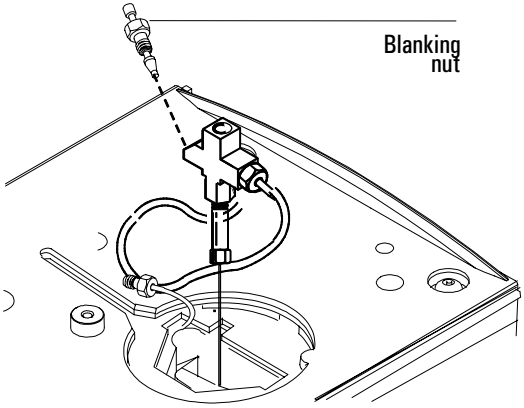
3. Carefully lift the interface out of the heater block.



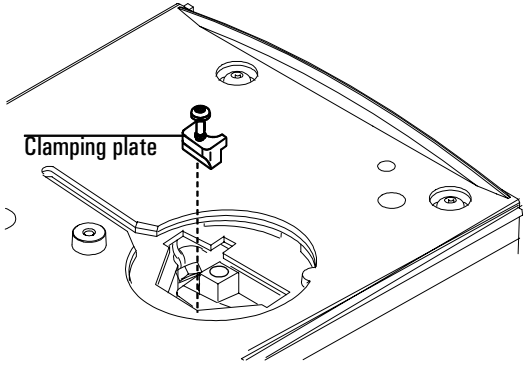
4. Loosen the hex nut connecting the split vent line to the interface until you can remove the line. Put the line aside. You do not need to plug it.



5. Install a blanking nut into the split line port and finger tighten the nut. Tighten the nut an additional 1/4-turn using two wrenches in opposition, the adjustable wrench on the interface and the 1/4-inch wrench on the nut.



6. Place the interface in the heater block. Replace the clamping plate you removed in Step no. 2 and tighten the screw until snug. Do not overtighten. If you removed the transfer line, replace it.

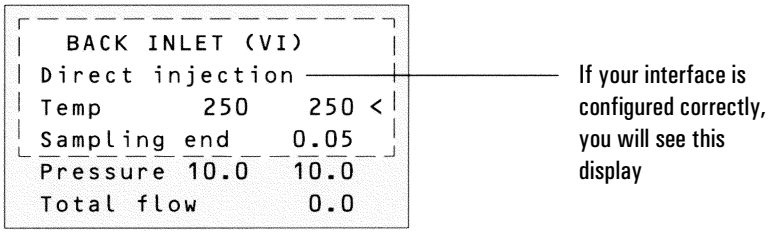


7. Restore the GC to normal operating conditions. Perform a leak test on the interface fittings.

Procedure: Configuring for a direct injection

The GC cannot sense the presence of the split vent. When you disconnect or reconnect the vent, you must configure the GC so that the pneumatics work properly.

1. Press [Config] [Back Inlet] or [Config] [Front Inlet].
2. Press [Mode/Type].
3. Choose `Split removed`.
4. Press [Back Inlet] or [Front Inlet]. If your GC is correctly configured, you will see the following display:



Using the control table

`Direct injection` If your GC is configured correctly, you will see above display.

`Temp` Actual and setpoint interface temperatures

`Sampl'g end` The sample introduction interval, in minutes. The flow rate is calculated from the pressure setpoint that is active at the start of sample introduction.

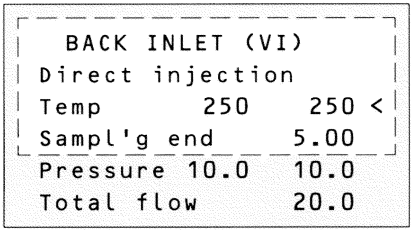
Set the sampling end setpoint 0.2 minutes longer than the time the sampler needs to introduce the sample. For example, the 7694 headspace sampler has an Inject time parameter which controls how long the valve remains in the inject position. If Inject time is 1 minute, the sampling end setpoint should be set to 1.2 minutes. If you're using an 7695 Purge and Trap Concentrator, set the Sampling end setpoint 0.2 minutes longer than the Desorb time parameter.

If your column is defined and you specify a flow or pressure program for your column, the ramp does not begin until after the sampling end setpoint expires.

`Pressure` Actual and setpoint interface pressure before a run and after sampling time.

`Total flow` The actual flow to the interface. This is a reported value, not a setpoint.

Column defined or column not defined



Some setpoints in the flow system are interdependent. If you change one setpoint, other setpoints may change to compensate.

Table 57 Direct Mode Pneumatic Setpoints

Column defined	
When you change:	**These setpoints change:**
Before and after sampling	
Pressure	Column flow* Total flow**
Column flow*	Pressure Total flow**
During sampling	
You cannot change pressure and flow setpoints during sampling time.	
Column not defined	
Before and after sampling	
The Column flow* setpoint is not available. You can change the pressure setpoint; other setpoints are not affected.	
During sampling	
You cannot change pressure and flow setpoints during sampling time.	

*This setpoint appears on the column control table.
**This value is actual only.

Operating parameters

Use the information in Table 58 to help you set up the operating conditions for your interface.

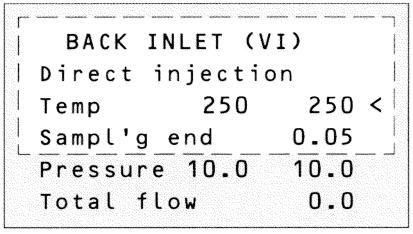
Table 58 Direct Mode Operating Parameters

Parameter	Allowed setpoint range	Suggested starting value
Oven initial time	0 to 999.9 minutes	≥ interface sampling end
Interface temperature	Ambient + 10° C to 400° C	≥ transfer line temperature
Interface sampling end	0 to 999.9 minutes	0.2 minutes longer than actual sampling time

Procedure: Operating in direct mode

These instructions apply to both column *defined* and *not defined*.

1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See "Flow and Pressure Control".
2. Press [Front Inlet] or [Back Inlet].
   a. Verify that your GC is configured for a direct injection.
   b. Set the interface temperature.
   c. Set sampling end. Set 0.2 minutes longer than the sample introduction time.



3. Make certain `Auto Prep Run` is On (see page 285) or use the [Prep Run] key before introducing a sample.

Maintaining a Volatiles Interface

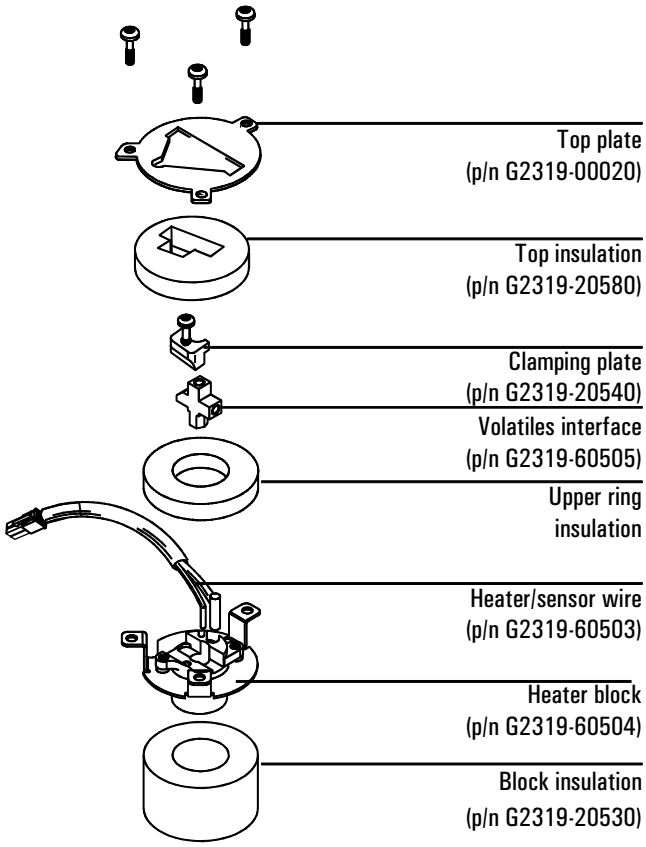


Figure 69 The volatiles interface parts breakdown

Procedure: Installing columns

WARNING Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing columns. Use care in handling these columns to prevent puncture wounds.

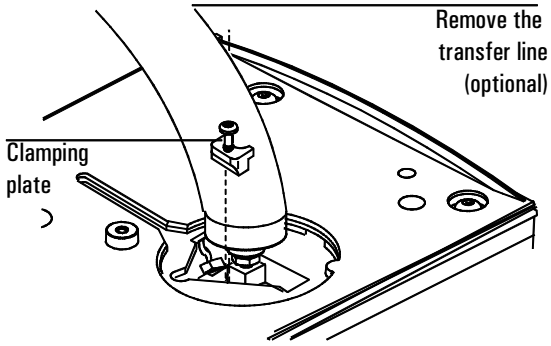
WARNING Be careful! The interface may be hot enough to cause burns.

Materials needed**:**

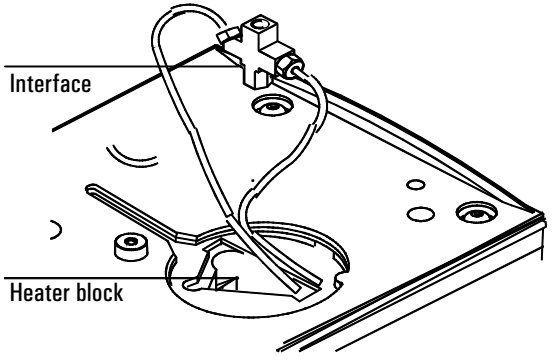
- Column nut and ferrule
- Column cutter
- Tissue
- Typewriter correction fluid
- 1/4-inch wrench
- 5/16-inch or adjustable wrench
- Metric ruler
- T-20 Torx screwdriver

1. Press [Oven] and set the oven to 35°C. Press [Front Inlet] or [Back Inlet] and turn off the interface temperature and pressure. Allow the interface to cool. When the oven temperature reaches setpoint, turn the oven off.
2. Disconnect the transfer line, if desired. Loosen the nut with a 1/4-inch wrench and remove the line. Remove the clamping plate from the interface

by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.

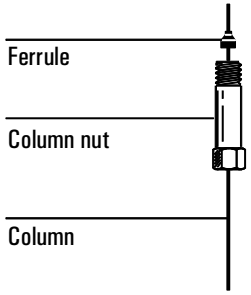


3. Lift the interface out of the heater block.

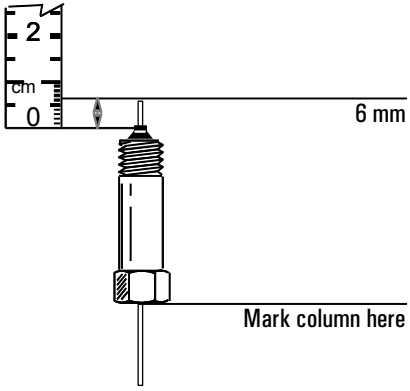


4. From inside the oven, push the column through the opening in the oven top. Grab the column from the oven top.

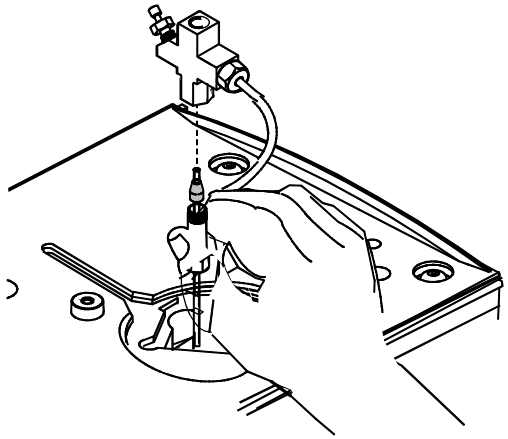
5. Place a capillary column nut and ferrule on the column and prepare the column end. If you need help with this step, "Procedure: Preparing capillary columns".



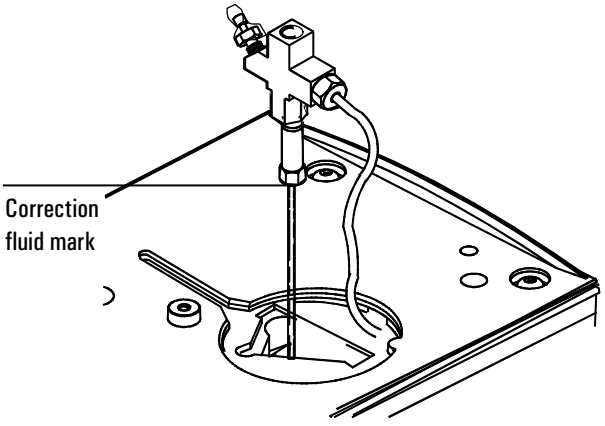
6. Position the column so it extends 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



7. Insert the prepared column in the interface and finger tighten the column nut.

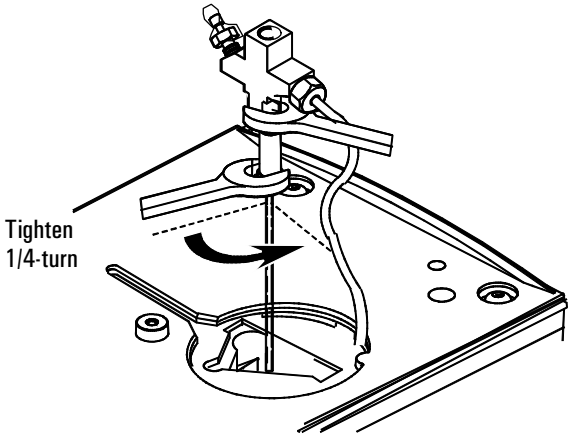


8. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.

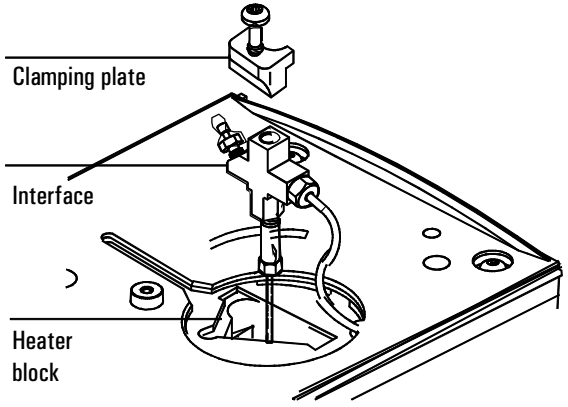


9. Tighten the column nut an additional 1/4- to 1/2-turn. Use the adjustable wrench to hold the interface while you tighten the column nut with the

1/4-inch wrench until the column cannot be pulled from the fitting with gentle pressure.



10. Replace the interface in the heater block. Replace the clamping plate and tighten the screw until snug. If you removed the transfer line, reinstall it.



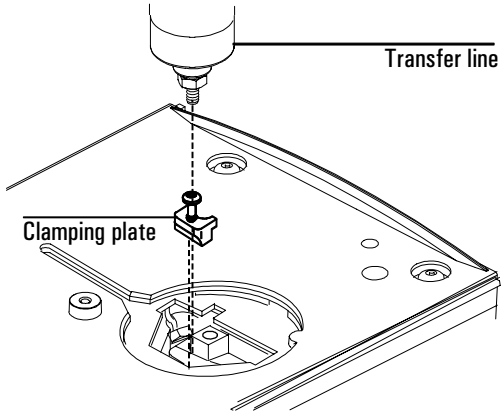
11. After the column is installed at both interface and detector, establish a flow of carrier gas through the interface. Heat the interface to operating temperature. Retighten the fittings, if necessary.

Procedure: Replacing or cleaning the interface

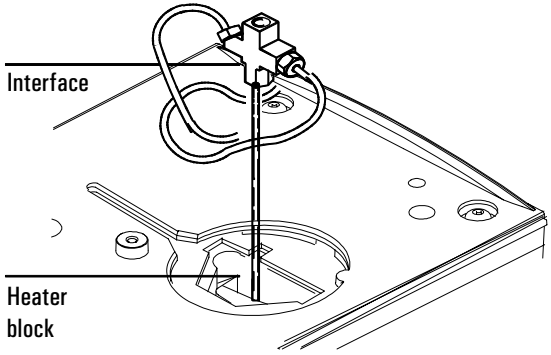
Materials needed:

- 1/4-inch or 7-mm wrench
- Sonicator or new interface
- T-20 Torx screwdriver

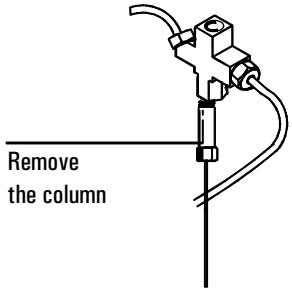
1. If you have entered parameters that you do not want to lose, store them as a method. Allow the oven and interface to cool. Turn off all flows at the initial gas supply or set the flows to 0 in the inlet control table.
2. Disconnect the transfer line. Loosen the nut with a 1/4-inch wrench and remove the line. Remove the clamping plate from the interface by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



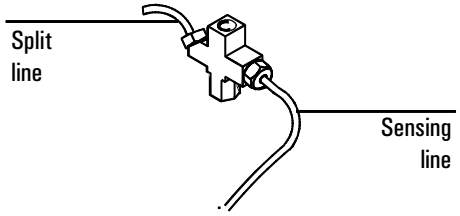
3. Lift the interface out of the heater block.



4. If a column is installed, remove it.



5. Remove the split and pressure sensing lines by loosening the hex nuts with the wrench.

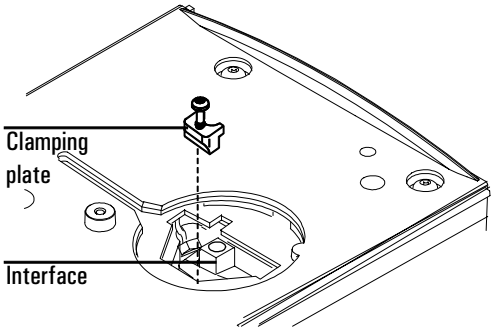


6. Clean or replace the interface. If you are cleaning the interface, sonicate it twice and then rinse.

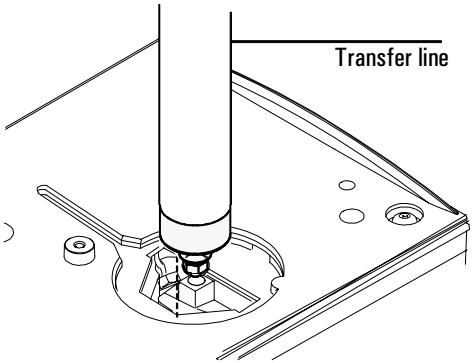
   Reinstall the split line and pressure sensing lines and finger tighten the hex nuts. Tighten the hex nuts an additional 1/4-turn with the wrench.

7. Reinstall the column in the interface. See "Procedure: Installing columns".

8. Place the interface in the heater block. Replace the clamping plate you removed earlier and tighten the screw until snug. Do not overtighten.



9. Reinstall the transfer line. Finger tighten the nut and then tighten an additional 1/4-turn with the wrench.



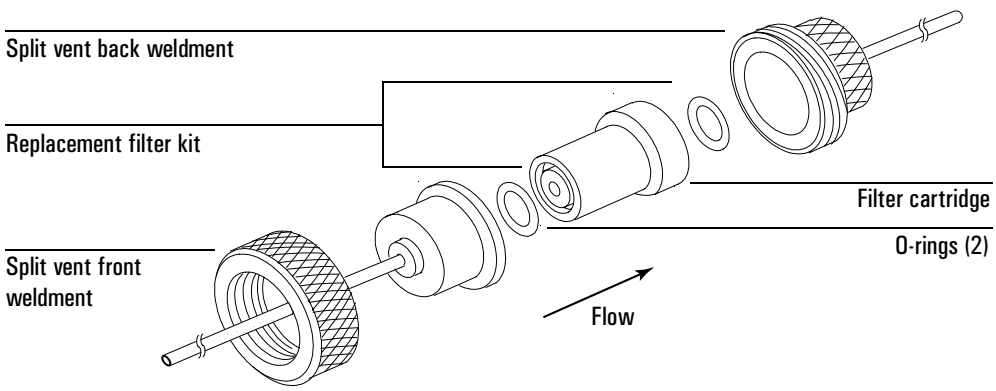
10. After the column is installed at both interface and detector, establish a flow of carrier gas through the interface and maintain it for 10 to 15 minutes. Check for leaks. Heat the interface to operating temperature and retighten the fittings, if necessary.

Replacing the split vent trap filter cartridge

WARNING Turn off the oven and turn off the heater for the inlet that uses the split vent trap and let them cool down. Turn off the carrier gas supply pressure.

The split vent trap may contain residual amounts of any samples or other chemicals you have run through the GC. Follow appropriate safety procedures for handling these types of substances while replacing the 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.
7. Check for leaks.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the interface flow module. If this portion of the system proves to be leak-free, refer to the next procedure to check the interface and interface module.

Liquid leak detectors are not recommended, especially in areas where cleanliness is very important. If you do use leak detection fluid, immediately rinse the fluid off to remove the soapy film.

WARNING To avoid a potential shock hazard when using liquid detection fluid, be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector capable of detecting your gas type or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-inch wrenches

1. Using the leak detector, check each connection you have made for leaks.
2. Correct leaks by tightening the connections with the wrenches. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing the system

There are several places in the interface-sampler system that can leak. This procedure helps you determine, in general, if there is an unacceptable leak in the system. If there is a leak, you should use an electronic leak detector to pinpoint the component that is leaking.

WARNING Be careful! The oven and interface may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-inch wrench
- Two, 1/8-inch SWAGELOK caps
- Gloves (if the interface is hot)
- 1/4-inch or 7 mm wrench

1. Complete the following preliminary steps:
   a. If you have entered parameters that you do not want to lose, store them as a method.
   b. Cool the oven to room temperature and then turn it off.
   c. When the oven is cool, turn off the interface pressure from the keyboard.
   d. Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
2. Cap the septum purge and split vent fittings located on the flow module with 1/8-inch Swagelok caps.
3. Press [Front Inlet] or [Back Inlet] to open the control table. Enter a pressure setpoint between 20 and 25 psi, or enter your normal operating pressure if it is greater. Make sure that the pressure at the initial gas supply is at least 10 psi higher than the interface pressure. Wait a few minutes for the pressure to equilibrate.
4. Turn the pressure off. Because the septum purge, split vent, and column fittings are capped, gas should be trapped in the system and the pressure

should remain fairly constant. Turn the pressure off at the source if you want to isolate the pneumatic system completely.

5. Continue to monitor pressure for 10 to 15 minutes. The pressure should drop approximately 1 psi during the first 1 to 2 minutes. After an initial pressure drop of about 1 psi, the pressure should not drop more than 0.03 psi/min.

   If the pressure drop is 0.03 psi/min or less, you can consider the interface-gas sampler system leak-free.

   If the pressure drops faster than the acceptable rate, you must check the interface and sampler systems separately to determine the source of the leak. See "Procedure: Preparing the interface for a leak test" to create a closed flow system, then return to this section and complete Steps 3 to 5 again.

   If you find a leak in the interface, refer to "Procedure: Correcting leaks".

   If the interface is leak-free, pressure check the sampling device. See the operating manual for your sampler for instructions.

Procedure: Preparing the interface for a leak test

To leak check the interface independent of the gas sampling device, you must disconnect the sampler from the interface to isolate the interface flow system from the sampler.

WARNING Be careful! The oven and interface may be hot enough to cause burns.

Materials needed:

- 1/16-inch male GC nut
- Graphite/Vespel ferrule

1. Disconnect the transfer line from the interface (see page 478).
2. Disconnect the carrier line from the sampler (see page 479 if you have a Headspace sampler or page 483 if you have a Purge and Trap Concentrator).
3. Prepare the end of the carrier line using the 1/16-inch male GC nut and the graphite/vespel ferrule.
4. Connect the carrier line to the interface where you removed the transfer line and tighten the nut finger tight and then tighten 1/4 to 1/2 turn with the 1/4-inch wrench.
5. Return to “Leak testing the system” and repeat steps 3 to 5.

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- Tool that will tighten leaking fittings — 1/4-inch, 5/16-inch, or 7-mm wrench

1. Use the electronic leak detector to check all areas of the interface that are potential sources of a leak. Potential leak areas are:
   - The capped purge vent
   - The capped split vent
   - The plugged column connection
   - The area where the gas lines are plumbed to the interface
2. Correct leaks using the correct size wrench to tighten connections. You may need to repeat the leak test again to check for leaks.

   If the pressure drop is now 0.03 psi/min or less, you can consider the interface system leak-free.

   If the pressure drops faster than this, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak-free but the interface system is still losing too much pressure, you may need to replace the interface module. Contact your Agilent service representative.

Connecting to an External Gas Sampler

Figure 70 shows a gas sampling device connected to the volatiles interface.

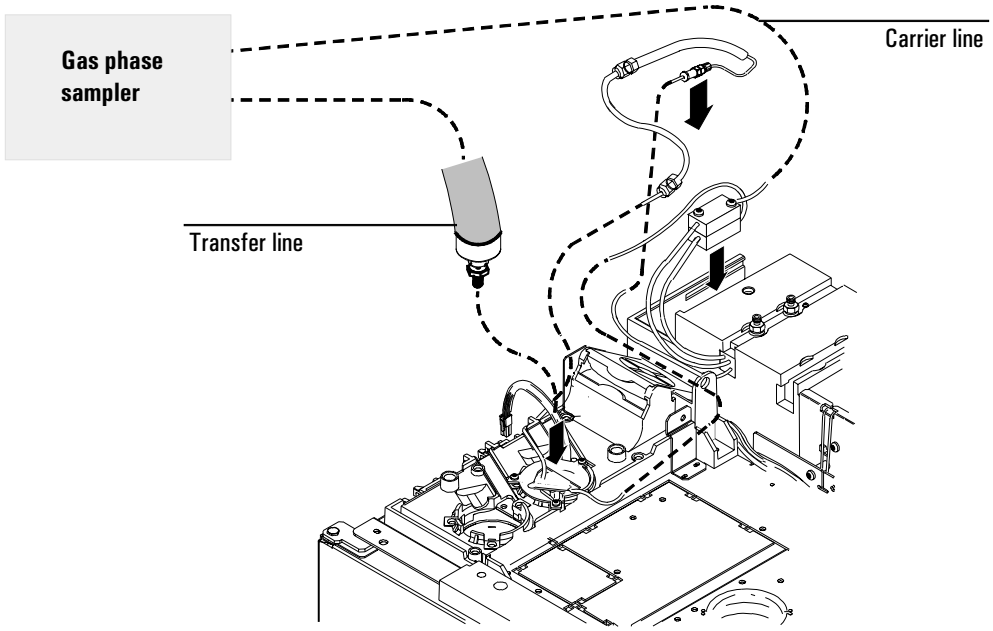


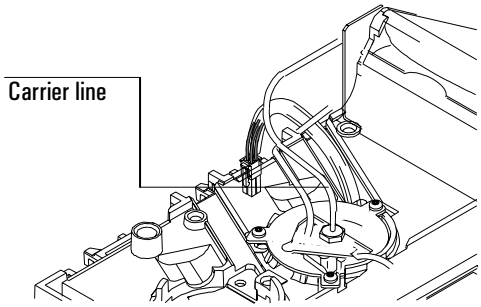
Figure 70 Flow diagram of an external sampling device

Procedure: Connecting the 7694 headspace sampler

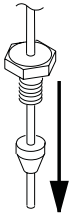
Materials needed:

- 1/8-inch Swagelok nut
- 1/16-inch to 1/8-inch reducer
- 1/8-inch ferrule set
- Wrenches
  — One 7/16-inch
  — Two 5/16-inch
  — One 1/4-inch
  — One 7-mm

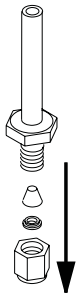
1. Remove carrier line tubing labeled “supply” attached to the volatiles interface using a 1/4-inch wrench.



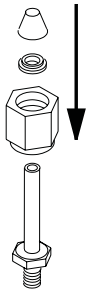
2. Remove the male fitting and Vespel/graphite ferrule from the carrier line. Keep the parts for later use.



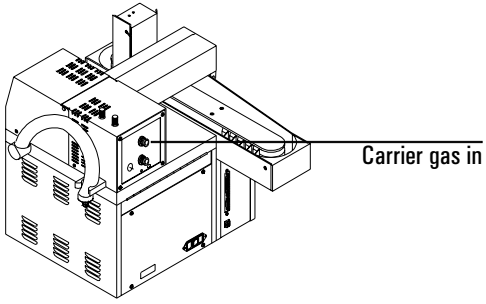
3. Remove the nut and the metal ferrules from a 1/16-inch to 1/8-inch reducer. Keep the parts for later use.



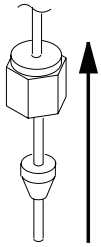
4. Slide a 1/8-inch Swagelok nut, a 1/8-inch back ferrule, and a 1/8-inch front ferrule onto the unthreaded end of the reducer.



5. Connect the reducer to the gas supply port labeled “Carrier” on the back of the headspace sampler by tightening the 1/8-inch Swagelok nut using a 7/16-inch wrench. Tighten the nut 1/4-turn past finger tight.

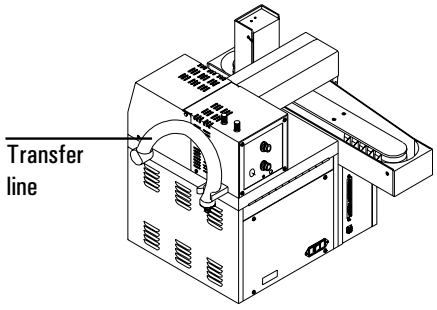


6. Slide the 1/16-inch female nut from step 3 and then the 1/16-inch Vespel/graphite ferrule from step 2 onto the end of the carrier line.

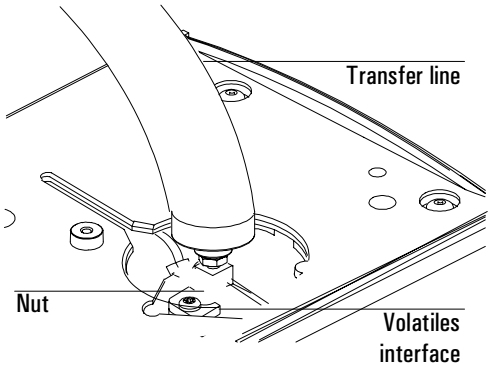


7. Connect the carrier line to the gas supply port. Use two wrenches to tighten the 1/16-inch Swagelok nut 1/4-turn past finger tight. Do not overtighten. If the fitting leaks, tighten an additional 1/8-turn.

8. Locate the headspace sampler's transfer line tubing.



9. Connect the transfer line (with the pre-attached nut and steel ferrule) to the interface. Tighten the nut 1/4-turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8-turn.

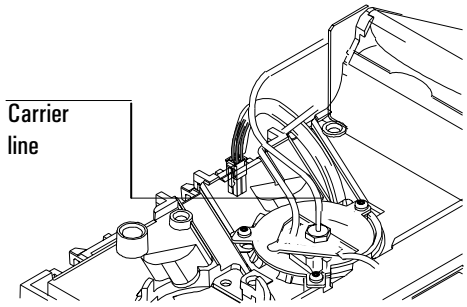


Procedure: Connecting the 7695 purge and trap concentrator

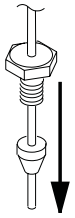
Materials needed:

- 1/16-inch Swagelok nut
- Vespel/graphite ferrule of the appropriate size for the transfer line
- Column cutter (fused silica)
- 5/16-inch and 1/4-inch wrenches
- Typewriter correction fluid
- Metric ruler

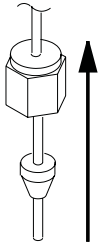
1. Remove the GC carrier line tubing labeled “supply” attached to the volatiles interface using a 1/4-inch wrench.



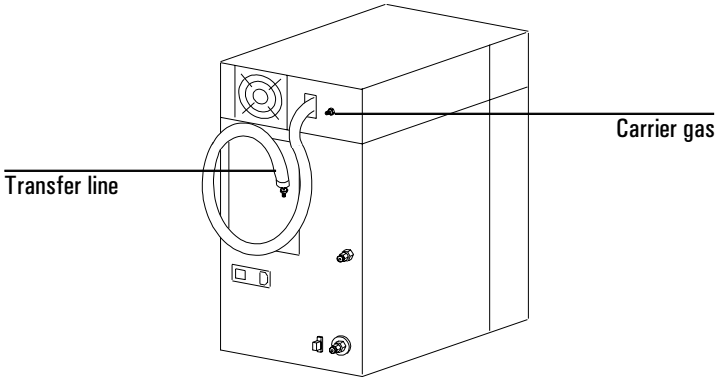
2. Remove the nut and Vespel/graphite ferrule from the carrier line. Keep the parts for later use.



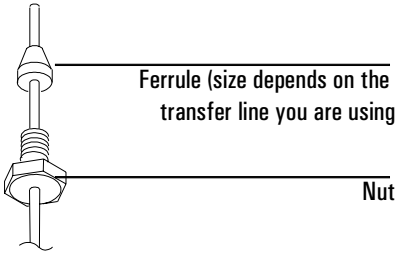
3. Slide a 1/16-inch Swagelok nut and then the Vespel/graphite ferrule from step 2 onto the end of the carrier line.



4. Connect the carrier line to the gas supply port labeled “Carrier Gas” on the back of the P&T concentrator using a 5/16-inch wrench. Tighten the nut 1/4-turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8-turn until it seals.

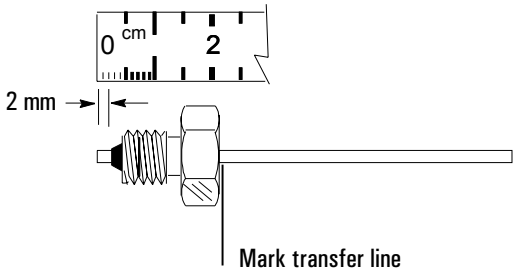


5. Slide the 1/16-inch male nut from step 2 and an appropriate Vespel/graphite ferrule onto the end of the P&T transfer line.

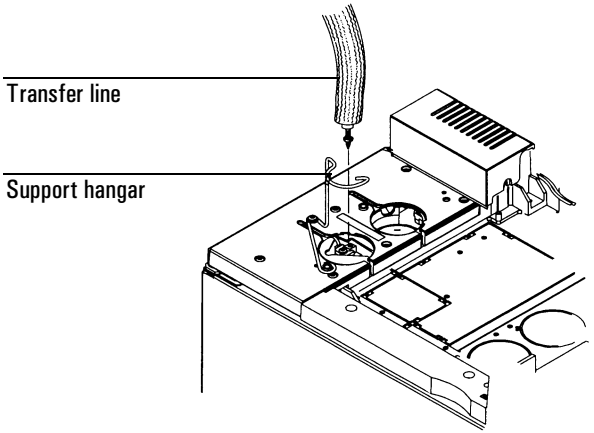


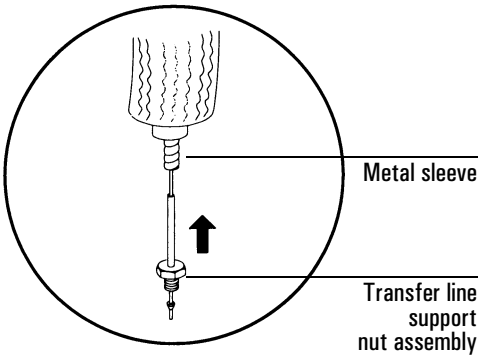
6. If you are using a *nickel-plated* transfer line, proceed to step 8. If you are using a *fused-silica transfer* line, prepare the end of the fused silica line.

7. Position the transfer line so that 2 mm of tubing is exposed in front of the ferrule, and mark the transfer line with typewriter correction fluid at a point even with the nut.



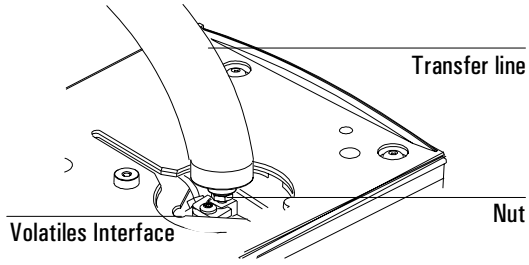
8. To connect the transfer line to the volatiles interface, first install the transfer line support nut assembly up and inside the metal sleeve of the heated transfer line assembly.





Then, connect the transfer line to the volatiles interface by finger tightening the 1/16-inch male nut while adjusting the transfer line's position so that the correction fluid mark stays aligned with the nut. Using a 1/4-inch wrench,

tighten the nut 1/4-turn past finger tight. Do not overtighten. If the fitting leaks, tighten an additional 1/8-turn until it seals.



9. After the column is installed at both the interface and the detector, establish a flow of carrier gas through the interface and maintain it for 10 to 15 minutes. Check for leaks. Heat the interface to operating temperatures and retighten the fittings, if necessary.