Operating Manual
Volume 2. Inlets

HP 6890 Series
Gas Chromatograph

HP part number G1530-90450

First Edition, Oct 1996

Replaces parts of G153090310 Operating Manual and parts of G153090320 Maintenance and Troubleshooting Manual

Printed in USA

Warranty

The information contained in this document is subject to change without notice.

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Safety Information

The HP 6890 Gas Chromatograph meets the following IEC (International Electrotechnical Commission) classifications: Safety Class 1, Transient Overvoltage Category II, and Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the HP 6890 has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard. Disconnect the AC power cord before removing covers. The customer should not attempt to replace the battery or fuses in this instrument. The battery contained in this instrument is recyclable.

Safety Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Hewlett-Packard Company assumes no liability for the customer's failure to comply with these requirements.

WARNING

A warning calls attention to a condition or possible situation that could cause injury to the user.

CAUTION

A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.


Caution. Refer to accompanying documents.


Indicates a hot surface.


Indicates hazardous voltages.


Indicates earth (ground) terminal.


Indicates radio-activity hazard.


Indicates explosion hazard.

Important User Information for In Vitro Diagnostic Applications

This is a multipurpose product that may be used for qualitative or quantitative analyses in many applications. If used in conjunction with proven procedures (methodology) by qualified operator, one of these applications may be In Vitro Diagnostic Procedures.

Generalized instrument performance characteristics and instructions are included in this manual. Specific In Vitro Diagnostic procedures and methodology remain the choice and the responsibility of the user, and are not included.

Sound Emission Certification for Federal Republic of Germany

Sound pressure Lp < 65 dB(A)

During normal operation

At the operator position

According to ISO 7779 (Type Test)

When operating the HP 6890 with cryo valve option, the sound pressure 74.6 dB(A) during cryo valve operation for short burst pulses.

Schallemission

Schalldruckpegel LP < 65 dB(A)

Am Arbeitsplatz

Normaler Betrieb

Nach DIN 45635 T. 19 (Typprüfung)

Bei Betrieb des HP 6890 mit Cryo Ventil Option treten beim Oeffnen des Ventils impulsfoermig Schalldrucke Lp bis ca. 74.6 dB(A) auf.

Little Falls Operation
Hewlett-Packard Company
2850 Centerville Road
Wilmington, DE 19808-1610

Contents

Chapter 1. Introduction to Inlets

Chapter 2. The Split/Splitless Inlet

Part 1. Using a Split/Splitless Inlet

Part 2. Maintaining a Split/Splitless Inlet

Chapter 3. The Purged Packed Inlet

Part 1. Using a Purged Packed Inlet

Part 2. Maintaining a Purged Packed Inlet

Chapter 4. The Cool On-Column Inlet

Part 1. Using a Cool On-Column Inlet

Part 2. Maintaining a Cool On-Column Inlet

Chapter 5. The Programmable Temperature Vaporization Inlet

Part 1. Introducing the HP PTV

Part 2. Using the Split Modes

Part 3. Using the Splitless Modes

Part 4. Using the Solvent Vent Mode

Part 5. Maintaining a PTV

Chapter 6. The Volatiles Interface

Part 1. Using a Volatiles Interface

Part 2. Maintaining a Volatiles Interface

Part 3. Connecting to an External Gas Sampler

Chapter 7. NonEPC Inlets

Appendix A: Configuration Information

1

Introduction to Inlets

Chapter 1.
Introduction to Inlets

Inlet types

The HP 6890 GC has five types of inlets available. All are offered with electronic pneumatics control (EPC) and two are offered without.

Table 1. Inlet Types

Inlet Type	Gas Control
Split/splitless	EPC and nonEPC
Purged packed	EPC and nonEPC
Cool on-column	EPC only
Programmed temperature vaporization	EPC only
Volatiles interface	EPC only

Using hydrogen

When using hydrogen (H_2), as the carrier gas, be aware that hydrogen (H_2) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen (H_2) gas is supplied to the instrument.

WARNING

Hydrogen (H_2) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H_2), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H_2) supply at its source before working on the instrument.

Table 2. An Overview of Inlets

Inlet Type	Column type	Mode	Sample type	Comments	Sample to Column
Split/splitless	Capillary	Split	High concentration		Very little, most is vented
		Pulsed split	High concentration	New technique; may be useful with large (>2 μL) injections	
		Splitless	Low concentration		All
		Pulsed splitless	Low concentration	Useful with large (>2 μL) injections	
Cool on-column	Capillary	n/a	Low concentration or thermally labile	Minimal sample discrimination and decomposition	
Purged packed	Packed; 1/8- and 1/4-in. metal, 1/8-in. glass	n/a	Any		
	Large bore capillary	n/a	Any	Satisfactory if resolution is not an issue	
Programmed Temperature vaporization	Capillary	Split	High concentration		Very little, most is vented
		Pulsed split	High concentration		
		Splitless	Low concentration		All
		Pulsed splitless	Low concentration		
		Solvent vent	Low concentration	For large (>x μL) injections; multiple injections concentrate analytes while venting solvent	
Volatiles interface	Capillary	Direct	Low concentration	Lowest possible dead volume	All
		Split	High concentration	Max total flow = 100 mL/min	Very little
		Splitless	Low concentration		All

Table 3. Column Size and Carrier Gas Flow Rate

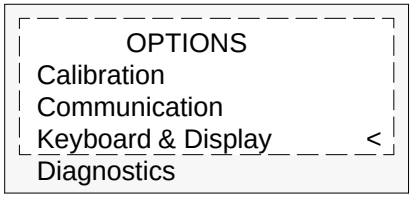
Column Type	Column Size	Carrier Gas Flow Rate	
		Hydrogen	Helium
Packed	1/8 in.		30
	1/4 in.		60
Capillary	50 µ id	0.5	0.4
	100 µ id	1.0	0.8
	200 µ id	2.0	1.6
	250 µ id	2.5	2.0
	320 µ id	3.2	2.6
	530 µ id	5.3	4.2

These flow rates, in mL/min at normal temperature and pressure (25°C and 1 atm) are recommended for all column temperatures.
For capillary columns, flow rates are proportional to column diameter and are 20% lower for helium than for hydrogen.

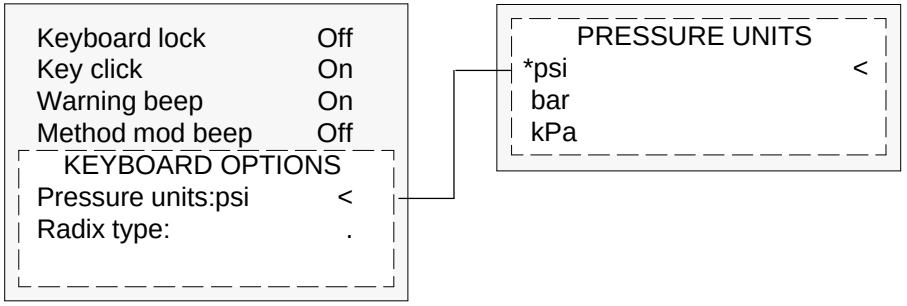
Procedure: Select pressure units—psi, bar, kPa

You can display pressure in psi, bar, or kPa. To check the units you are using, pressing the [Info] key while the cursor is on the Pressure line of a control table. To change the display units:

1. Press [Options].
2. Scroll to Keyboard & Display and press [Enter].



3. Scroll to Pressure units: and press [Mode/Type].



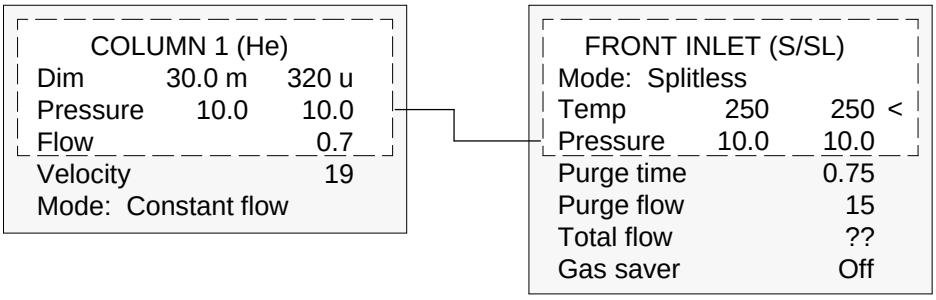
4. Choose a new pressure unit and press [Enter].

Table 4. Pressure Unit Conversions

To Convert	to	Multiply by
psi	bar	0.0689476
	kPa	6.89476
bar	psi	14.5038
	kPa	100
kPa	psi	0.145038
	bar	0.01

The inlet and column control tables

The tables for the inlet and column are interrelated. If you set a pressure at the column control table, that same pressure setting is active on the inlet control table, and vice versa. Although pneumatics can be controlled from either the column or the inlet, the column should be considered first.



The pressure readings—both setpoint and actual—are identical on the column and inlet control tables.

The column control tables

The control tables change depending on your column configuration. The next few pages describe the column control tables for the two types of columns, capillary and packed.

The column control table—defined capillary columns

If your column is defined, your control table will be similar to Figure 1.

The title This heading identifies the column—Column 1 or Column 2—and the type of carrier gas configured to the inlet (in parentheses).

Dim This line shows the column dimensions you have specified. Column length is in meters (m) and column inside diameter is in microns (µ).

Pressure, flow, and velocity are related. If the column is defined, enter any one of them and the GC computes and displays the other two.

Pressure The setpoint appears at the far right. The number at the left shows the actual pressure value. When you enter a pressure value, the values for flow and average linear velocity are calculated and displayed.

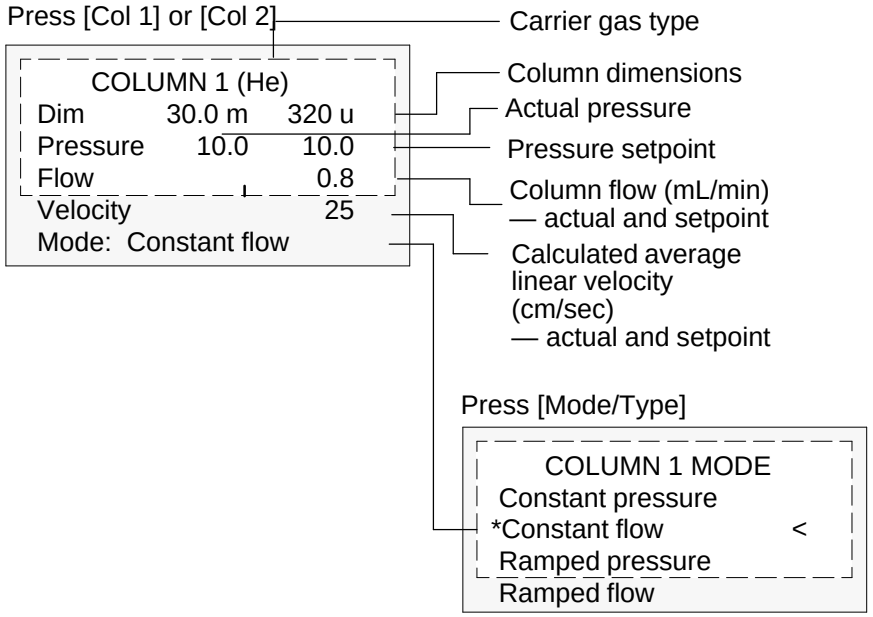
Flow If you enter a flow (in mL/min) here, pressure and velocity are calculated and adjusted.

Velocity If you enter average linear velocity (in cm/sec), pressure and flow are calculated.

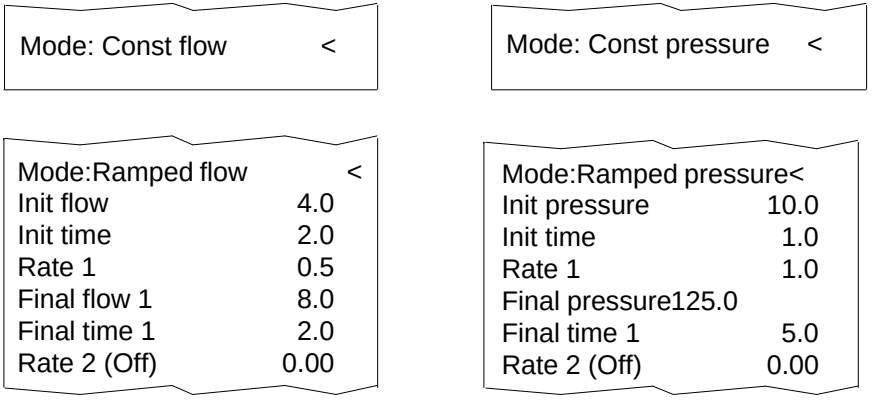
Mode: There are four column modes: constant flow, constant pressure, ramped flow, and ramped pressure. To change the mode, scroll to Mode : and press [Mode/Type].

The “Flow and Pressure Control” chapter of the General Information volume explains how to set pressure and flow programs.

Figure 1. Column display — defined capillary columns



Mode: Your control table also has one of these, depending on Mode:



The column control table—packed or undefined capillary columns

If you have not defined your column or if your inlet selection is Unspecified, your column control table will be similar to Figure 2.

The title This heading identifies the column—Column 1 or Column 2—and the type of carrier gas configured to the inlet (in parentheses).

Dimensions unknown This line tells you that you have not defined your column.

Pressure The split/splitless inlet and the cool on-column inlet are pressure controlled. Because the column is unknown, flow and average linear velocity cannot be computed.

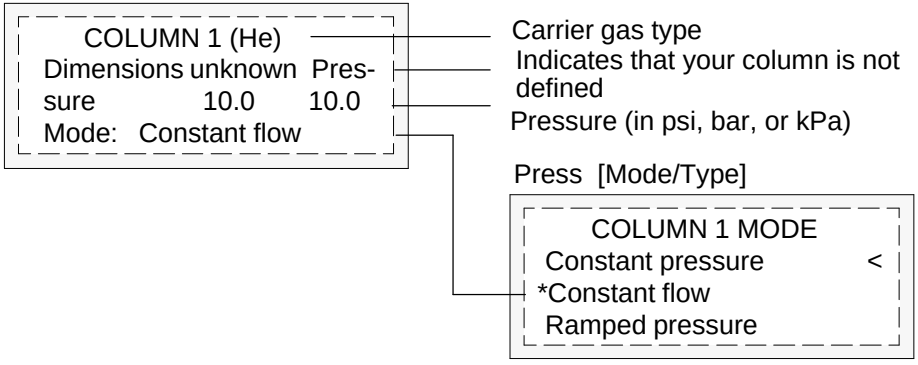
The purged packed inlet is flow controlled. The actual pressure is displayed, but is not controllable by the user.

Mode: You have a choice of three modes if using a split/splitless or cool on-column inlet—constant pressure, constant flow, and ramped flow. The packed inlet gives you only the two flow modes—constant and ramped.

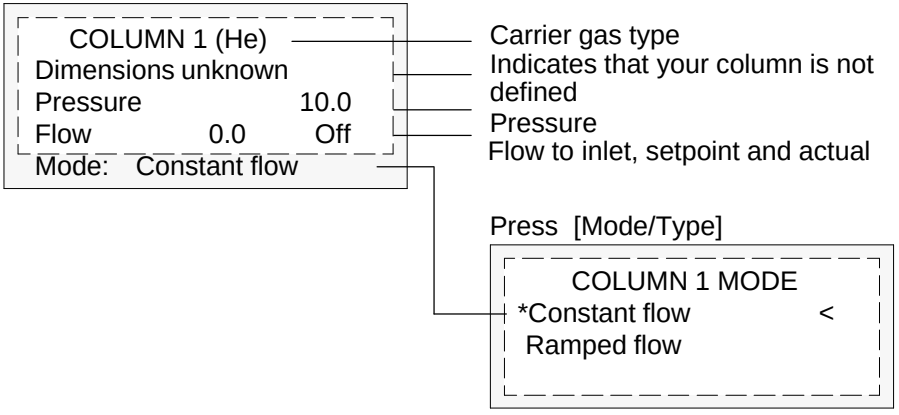
The “Flow and Pressure Control” chapter of the General Information volume explains how to set pressure and flow programs.

Figure 2. Column Display — Packed or Undefined Capillary Columns

Split/splitless or cool on-column inlets



Purged packed inlet

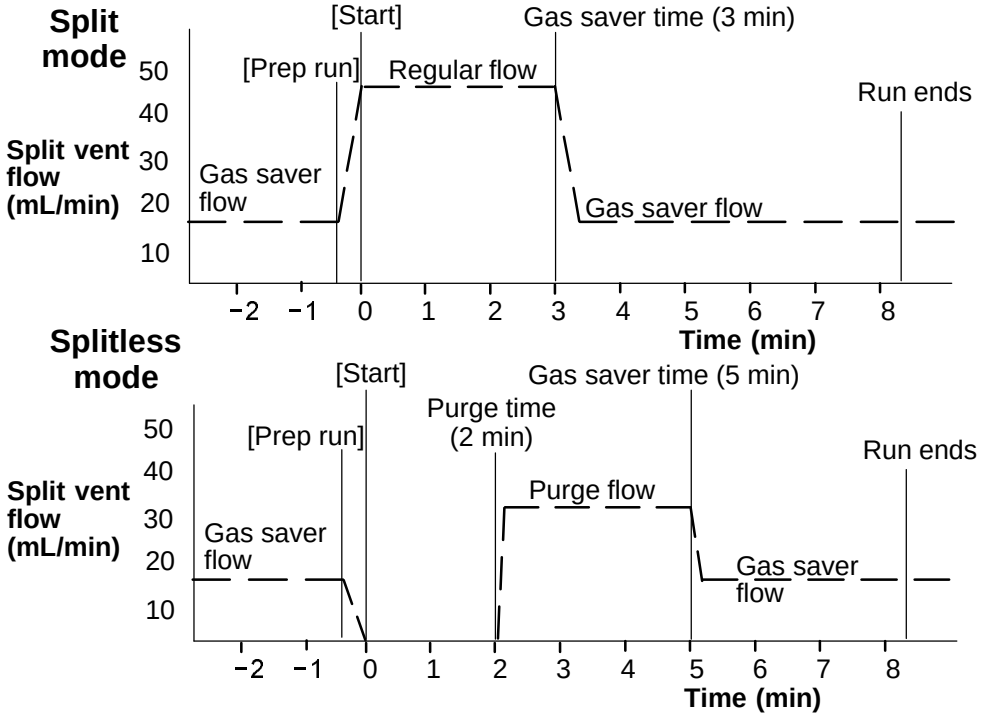


What is gas saver?

Gas saver reduces carrier gas flow from the split vent after the sample is on the column. Column head pressure and flow rate are maintained, while purge and split vent flows decrease. Flows—except column flow—remain at the reduced level until you press [Prep Run].

You can use gas saver in all modes of operation of the Split/Splitless and PTV inlets and in the split and splitless modes of the Volatiles Interface.

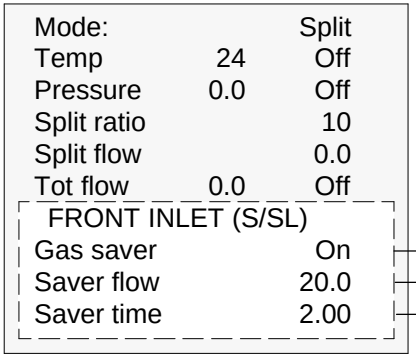
Figure 3. Gas Saver Operation



The pulsed modes of the split/splitless and PTV inlets are similar except for the pressure pulse starting at [Prep Run] and ending at Pulse time. The solvent vent mode of the PTV is more complex; see chapter 5 for details.

Procedure: Using gas saver

Press [Front Inlet] or [Back Inlet].



1. Turn on gas saver.

2. Set a flow. Must be at least 15 mL/min greater than the column flow.

3. If in split mode, set after injection time. In all other modes, set after purge time.

Pre Run and Prep Run

With some inlets and operating modes, certain instrument setpoints are different between runs than during an analysis. To restore the setpoints for injection, you must place the GC into the Pre Run state.

You must use the Pre Run state when:

- Using gas saver with any inlet.
- Using splitless mode with any inlet.
- Using a pressure pulse mode with any inlet.
- Using the solvent vent mode of the PTV inlet.
- Using the direct or splitless mode of the Volatiles Interface.

There are two ways to begin Pre Run—manually push the [Prep Run] key before each run or configure the GC to enter the Pre Run state automatically. The two methods are discussed below and on the next page.

During the Pre Run state:

- The Pre Run light blinks and Not Ready is on.
- Setpoints change to the correct values for injection.
- Inlet, detector, and oven equilibration times begin.

When all equilibration times expire, the Pre Run light is on steadily. When all criteria for a run are met, the Not Ready light turns off. The GC is now ready for sample injection.

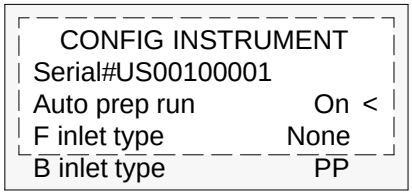
The [Prep Run] key

Press the [Prep Run] key before you inject a sample manually. The GC enters the Pre Run state. When the Pre Run light is steady and the Not Ready light goes off, begin the analysis.

Procedure: Auto Prep Run

With most automatic injection systems, you do not need to use the [Prep Run] key. If your sampler or automation controller (for example, an integrator or workstation) does not support the [Prep Run] function, you must set the GC to Auto Prep Run. To do this:

1. Press the [Config] key to view a list of configurable parameters.
2. Scroll to the Instrument parameter and press [Enter].
3. Scroll to Auto prep run and press [On].



Septum purge

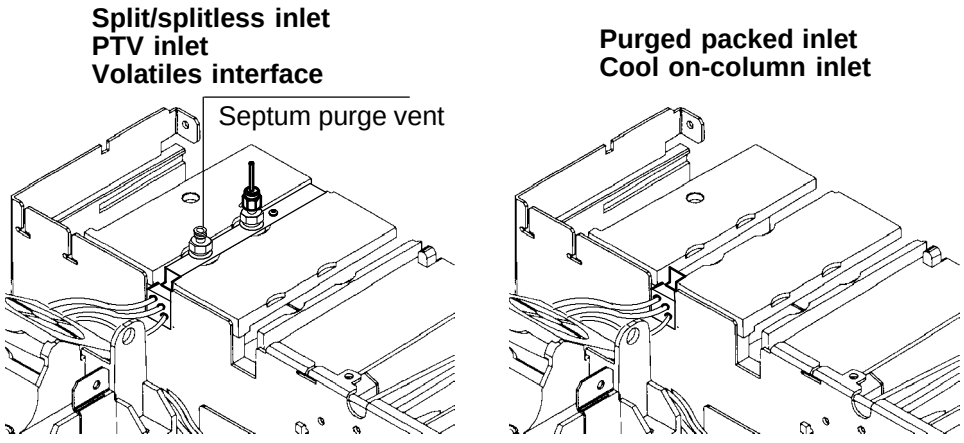
The septum purge line is near the septum where the sample is injected. A small amount of carrier gas exits through this line to sweep out any bleed.

Each inlet has a different septum purge flow. The GC automatically sets the purge flow for EPC inlets, but you can measure it from the septum purge vent at the flow manifold if you like.

Table 5. Septum Purge Flows

Inlet	Carrier	Septum Purge
Split/splitless, all modes	He, N_2, Ar/5%Me	3 mL/min
	H_2	6 mL/min
Purged packed	All	1 to 3 mL/min
Cool on-column	He, N_2, Ar/5%Me	15 mL/min
	H_2	30 mL/min
PTV	He, N_2, Ar/5% Me	3 mL/min
	H_2	6 mL/min
Volatiles interface	He, N2, Ar/5%Me	3 mL/min
	H_2	6 mL/min

Figure 4. Septum Purge Vents



2

The Split/Splitless Inlet

Chapter 2.
The Split/Splitless Inlet

Part 1. Using a Split/Splitless Inlet

This inlet is used for split, splitless, pulsed splitless, or pulsed split analyses. You can choose the operating mode from the inlet control table. The split mode is generally used for major component analyses, while the splitless mode is used for trace analyses. The pulsed splitless and pulsed split modes are used for the same type of analyses as split or splitless, but allows you to inject larger samples.

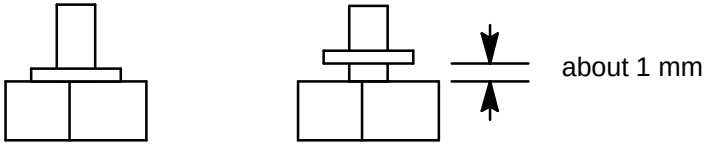
Standard and high-pressure versions

The standard split/splitless inlet is rated to 120 psi pressure at the gas supply fitting. It is appropriate for most columns. The high-pressure inlet is rated to 170 psi pressure—it is useful with very small diameter capillary columns that offer considerable resistance to gas flow.

To determine the version that you have, press [Front Inlet] or [Back Inlet], scroll to the Pressure line, and press the [Info] key. The display will show the pressure range for the inlet—either 1 to 100 psi (for the standard version) or 1 to 150 psi (for the high-pressure version).

Septum tightening

The septum retainer has an internal spring that applies pressure to the septum. For inlet pressures up to 100 psi, tighten the retainer until the C-ring lifts about 1 mm above the top surface. This is adequate for most situations.



With higher inlet pressures, tighten the septum retainer until the C-ring stops turning, indicating that the retainer is in firm contact with the septum. Then tighten one additional full turn.

Liners

Choose liners according to the type of injection you are doing—split or splitless. Many liners are available and can be ordered from the Hewlett-Packard Analytical Columns and Supplies Catalog.

Procedure: Changing the liner

Parts list:

- Liner, HP part no. 19251-60540 (split) or 5062-3587 (splitless)
- Tweezers
- Septum wrench (HP part no. 19251-00100)
- Viton O-ring (HP part no. 5180-4182)

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.

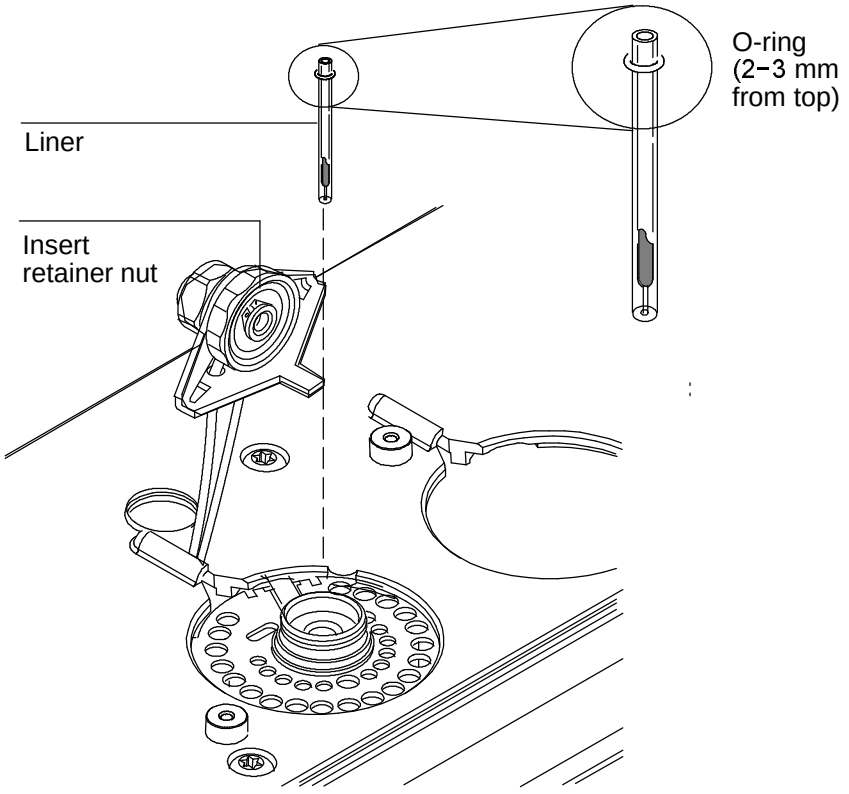
WARNING Be careful! The inlet fittings may be hot enough to cause burns.

2. Remove the insert retainer nut. Use a septum wrench, if needed.
3. If a liner is present, remove it with tweezers or a similar tool. Be careful not to chip the liner.
4. Hold the new liner with tweezers, and inspect it. Make sure it is the correct type for the injection mode you are using—split or splitless.
5. Place a Viton O-ring on the liner about 2 to 3 mm from its top end.
6. Press the liner straight down into the inlet.

Caution Do not add an O-ring or other seal either at the bottom of the inlet or at the bottom of the liner; this will damage the inlet and shatter the liner.

7. Replace the insert retainer nut, tightening it to firm finger tightness. Do not overtighten.

Figure 5. Installing a liner

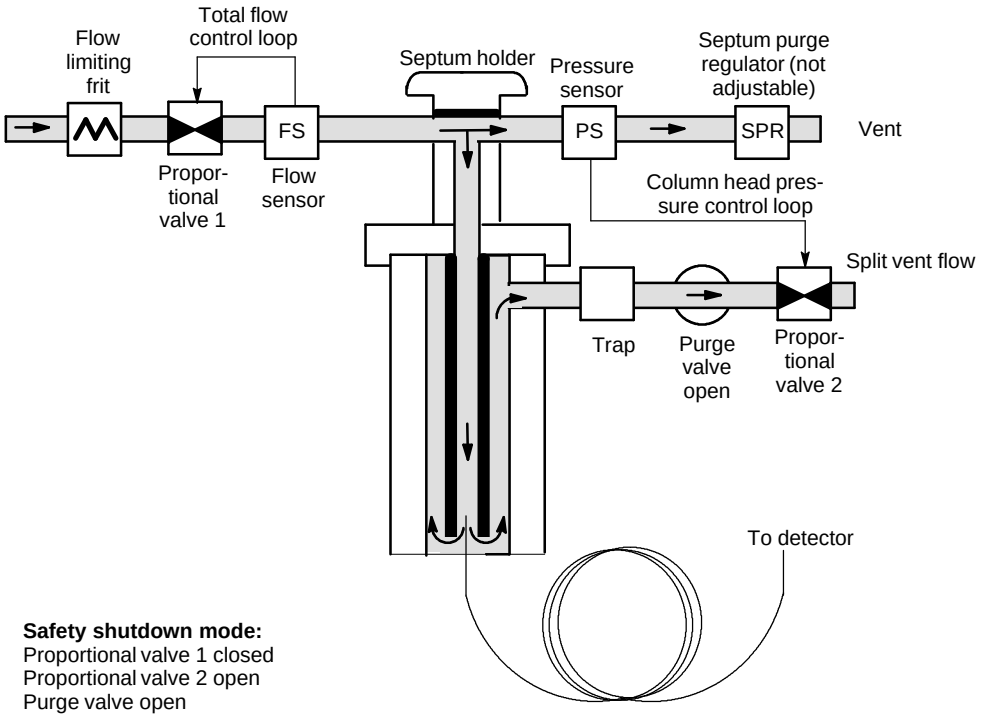


Split mode pneumatics

During a split injection, a liquid sample is introduced into a hot inlet where it vaporizes rapidly. A small amount of the vapor enters the column while the major portion exits from the split/purge vent. The ratio of column flow to split flow is controlled by the user. Split injections are primarily used for high concentration samples when you can afford to lose most of the sample out the split/purge vent. It is also used for samples that cannot be diluted.

Figure 6 shows the pneumatics for this inlet in split mode operation.

Figure 6. Split flow pneumatics



The control table—split operation

Mode: The current operating mode—split

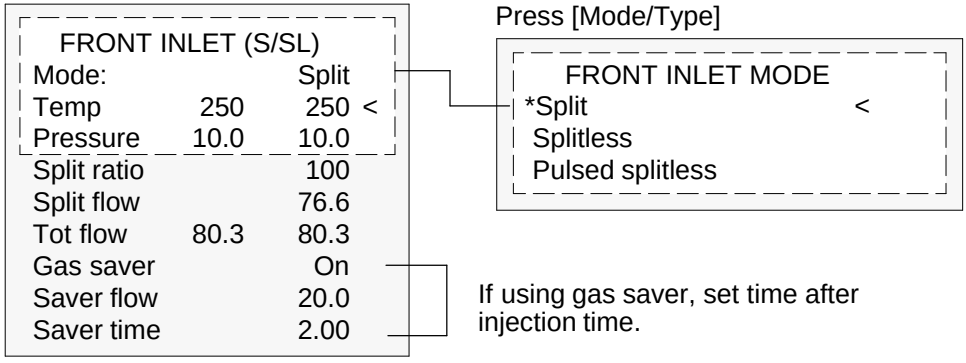
Temp Actual and setpoint inlet temperatures

Pressure Actual and setpoint inlet 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 line does not appear if your column is not defined.

Split flow Flow, in mL/min, from the split/purge vent. This line does not appear if your column is not defined.

Total flow This is the total flow into the inlet, which is the sum of the split flow, column flow, and septum purge flow. When you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same.

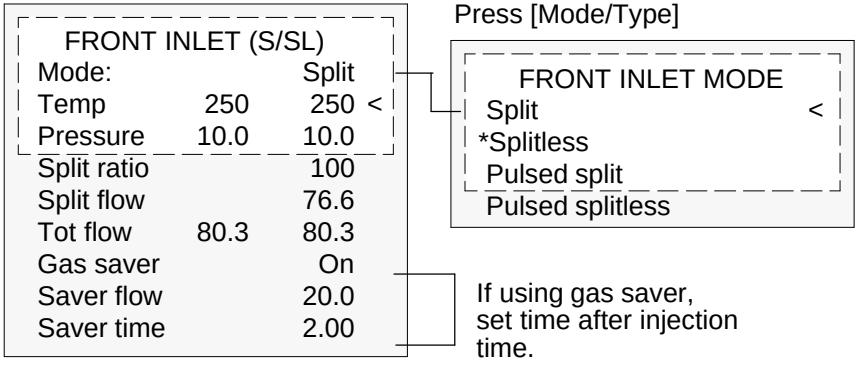


Procedure: Using the split mode with the column defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Split.
   b. Set the inlet temperature.

Split ratio = $\frac{\text{Split flow}}{\text{Column flow}}$

   c. If you want a specific split ratio, scroll to Split ratio and enter that number. The split flow will be calculated for you.
   d. If you want a specific split flow, scroll to Split flow and enter that number. The split ratio will be calculated for you.
   e. If desired, turn on Gas saver. Set the Saver time after the injection time. Use the [Prep Run] key (see page 13) before manually injecting the sample.



Procedure: Using the split mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]

FRONT INLET (S/SL)			
Mode:		Split	
Temp	250	250	<
Pressure	10.0	10.0	
Tot flow	79.1	79.1	

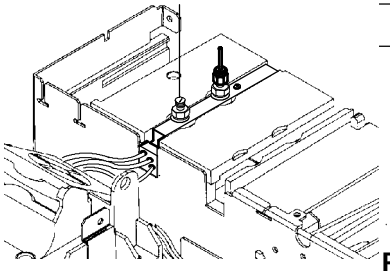
   a. Set temperature.

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

   c. Subtract split vent flow and septum purge flow (see page 15 for nominal septum purge flows by carrier gas type) from Total flow to get column flow.

$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$

   d. Calculate the split ratio. Adjust as needed.



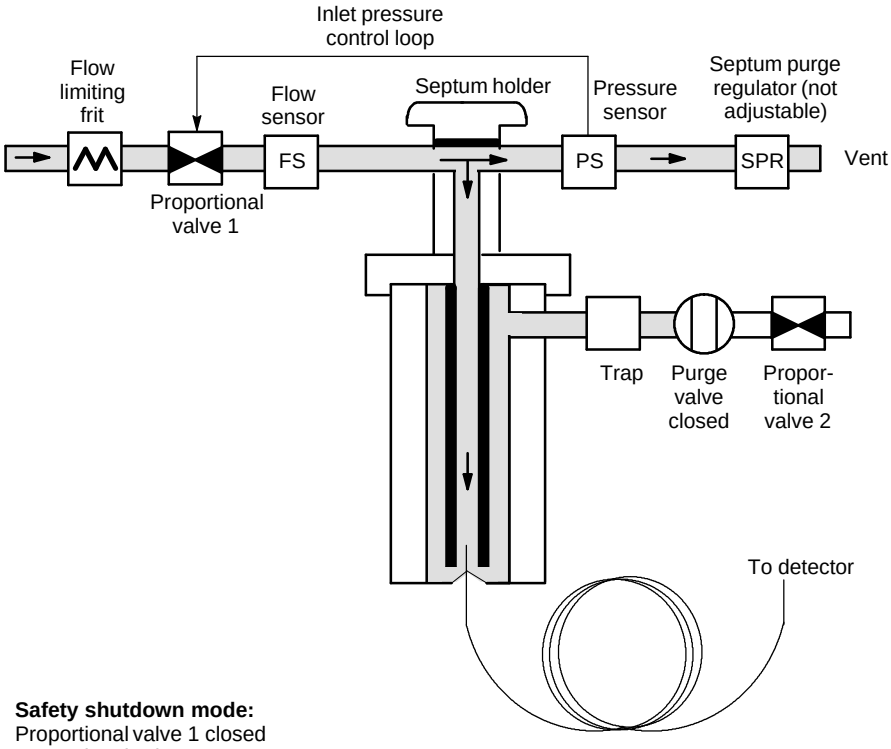
Front of instrument

Splitless mode pneumatics

In this mode, the purge valve is closed during the injection and remains so while the sample is vaporized in the liner and transferred to the column. At a specified time after injection, the purge valve opens to sweep any vapors remaining in the liner out the split vent. This avoids solvent tailing due to the large inlet volume and small column flow rate. Specify the purge time and purge flow rate in the inlet control table.

If you are using gas saver, the gas saver time should be after the purge time.

Figure 7. Splitless Flow Diagram, Pre-Run to Purge Time



Safety shutdown mode:
Proportional valve 1 closed
Proportional valve 2 open
Purge valve open

The control table—splitless operation

Mode: The current operating mode—splitless

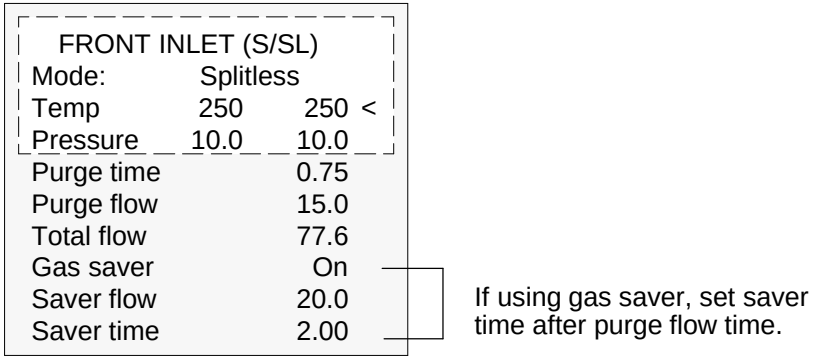
Temp Actual and setpoint inlet temperatures

Pressure Actual and setpoint inlet pressure in psi, bar, or kPa

Purge time The time, after the beginning of the run, when you want the purge valve to open.

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

Total flow The Total flow line displays the actual flow to the inlet during a Pre-run (Pre-run light is on and not blinking) and during a run before purge time. You cannot enter a setpoint at these times. At all other times, Total flow will have both setpoint and actual values.



Operating parameters

A successful splitless injection consists of these steps:

1. Vaporize the sample and solvent in a heated inlet.
2. Use a low flow and low oven temperature to create a solvent-saturated zone at the head of the column.
3. Use this zone to trap and reconcentrate the sample at the head of the column.
4. Wait until all, or at least most, of the sample has transferred to the column. Then discard the remaining vapor in the inlet—which is mostly solvent—by opening a purge valve. This eliminates the long solvent tail that this vapor would otherwise cause.
5. Raise the oven temperature to release the solvent and then the sample from the head of the column.

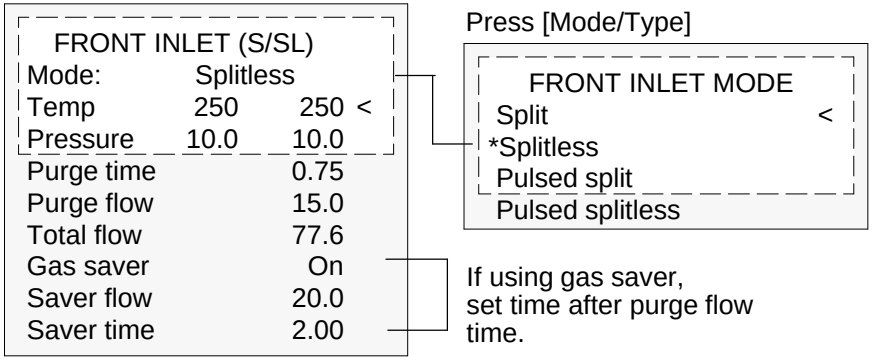
Some experimentation is needed to refine the operating conditions. Table 5 provides starting values for the critical parameters.

Table 5. Splitless Mode Inlet Parameters

Parameter	Allowed Setpoint Range	Suggested Starting Value
Oven temperature	No cryo, 24°C to 450°C CO_2 cryo, −60°C to 450°C N_2 cryo, −80°C to 450°C	10°C below solvent boiling point
Oven initial time	0 to 999.9 minutes	$\geq$ Inlet purge time
Inlet purge time	0 to 999.9 minutes	$\frac{\text{Liner volume}}{\text{Column flow}} \times 2$
Gas saver time	0 to 999.9 minutes	After purge time
Gas saver flow	15 to 1000 mL/min	15 mL/min greater than maximum column flow

Procedure: Using splitless mode with the column defined

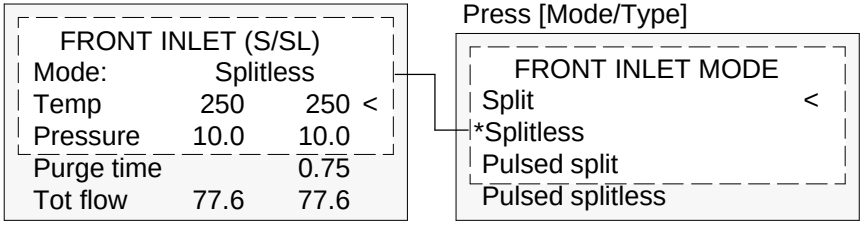
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Splitless.
   b. Set the inlet temperature.
   c. Enter a purge time and a purge flow.
   d. If desired, turn Gas saver on. Make certain the time is set after the purge flow time.



3. Use the [Prep Run] key (see page 13)before manually injecting a sample.

Procedure: Using splitless mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Splitless.
   b. Set the inlet temperature.
   c. Enter a purge time.
   d. Set your total flow greater than the column flow plus the septum purge flow—see page 15—to guarantee adequate column flow.



3. Use the [Prep Run] key (see page 13) before manually injecting a sample.

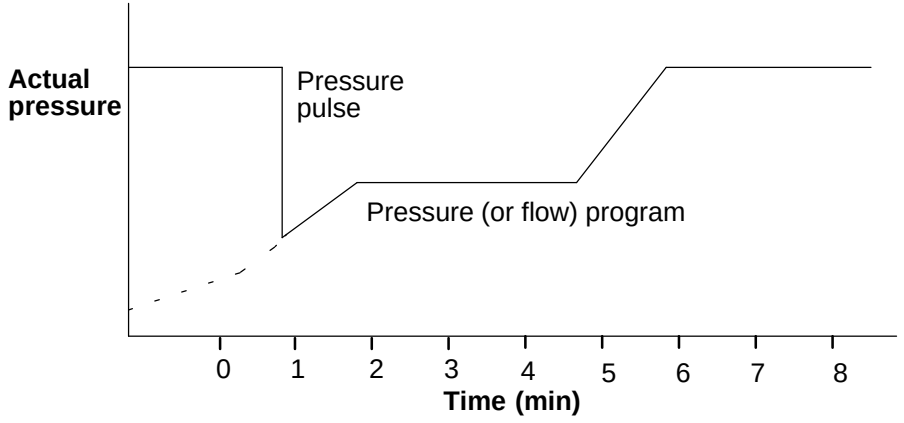
Pulsed split and splitless modes

The pressure pulse modes increase inlet pressure just before the beginning of a run and returns it to the normal value after a specified amount of time. The pressure pulse sweeps the sample out of the inlet and into the column faster, reducing the chance for sample decomposition in the inlet. If your chromatography is degraded by the pressure pulse, a retention gap may help restore peak shape.

You must press the [Prep Run] key before doing manual injections in the pressure pulse mode. See page 13 for details.

You can do column pressure and flow programming when in the pressure pulse mode. However, the pressure pulse will take precedence over the column pressure or flow ramp.

Figure 8. Pressure Pulse and Column Flow or Pressure



The control table—pulsed split mode

Mode: The current operating mode—pulsed split

Temp Actual and setpoint inlet temperatures

Pressure Actual and setpoint inlet pressure at the beginning of a run, ignoring the effect of a pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

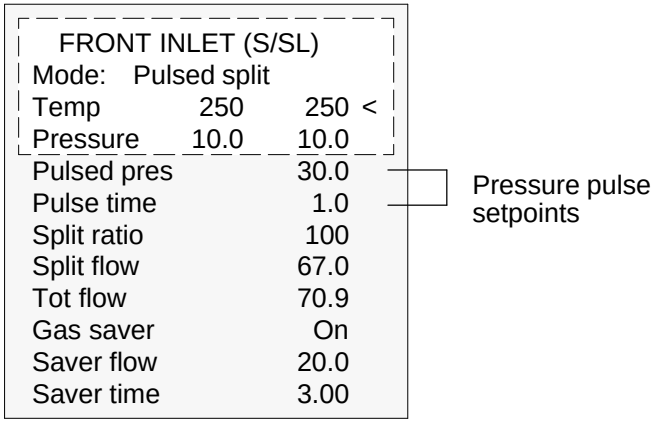
Pulsed pres The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until Pulse time elapses, when it returns to Pressure.

Pulse time Pressure returns to its normal setpoint at this time.

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

Split flow Flow, in mL/min from the split/purge vent. This line does not appear if your column is not defined.

Total flow The total flow into the inlet, a sum of the split flow, column flow, and septum purge flow. If you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same. When a pressure pulse is used, total flow increases to keep the split ratio constant.

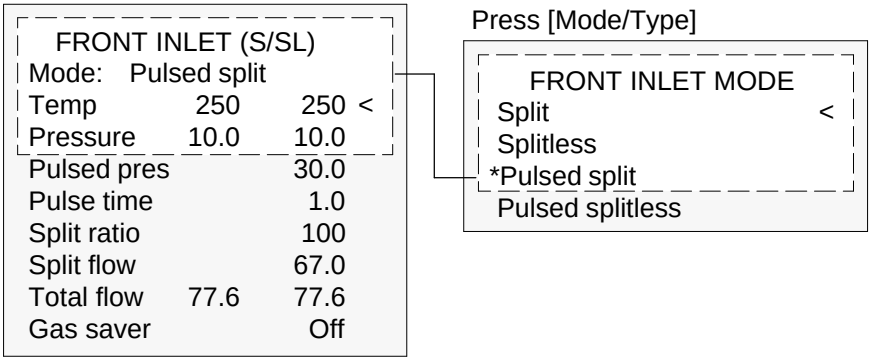


Procedure: Using the pulsed split mode

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Split.
   b. Set the inlet temperature.
   c. Enter values for Pulsed Pres and Pulse time.

Split ratio = $\frac{\text{Split flow}}{\text{Column flow}}$

   d. If you want a specific split ratio, scroll to Split ratio and enter that number. The split flow is calculated for you if the column is defined.
   e. If you want a specific split flow, scroll to Split flow and enter that number. The split ratio is calculated for you if the column is defined.
   f. Turn Gas saver on, if desired. Make certain the time is set after Pulse time.



3. Press the [Prep Run] key (see page 13) before injecting a sample manually.

The control table—pulsed splitless operation

Mode: The current operating mode—pulsed splitless

Temp Actual and setpoint inlet temperatures

Pressure Actual and setpoint inlet pressure at the beginning of a run, ignoring the effect of a pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

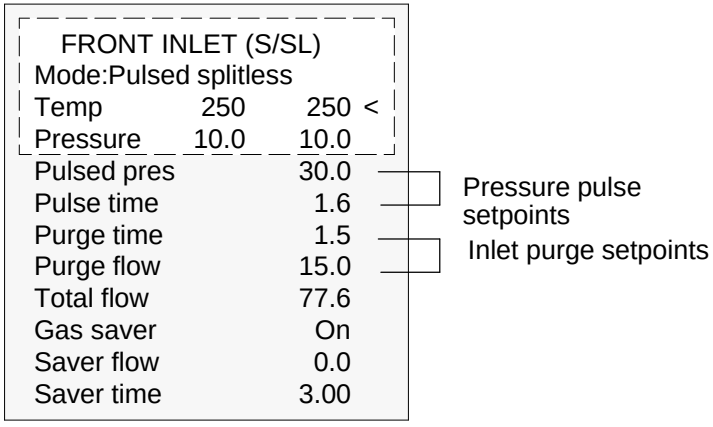
Pulsed pres The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until Pulse time elapses, when it returns to Pressure.

Pulse time Pressure returns to its normal setpoint at this time.

Purge time The time, after the beginning of the run, that you wish the purge valve to open. Set purge time 0.1 to 0.5 minutes before pulse time.

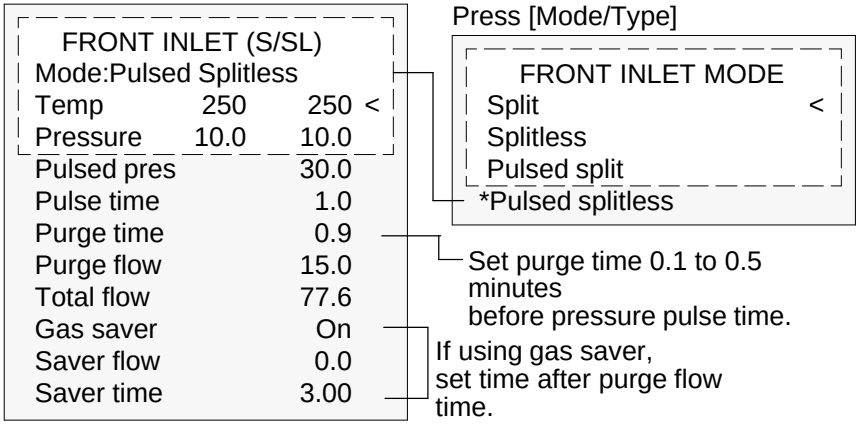
Purge flow The flow, in mL/min, from the purge vent, at Purge time. The column must be defined.

Total flow This is the total flow into the inlet, representing a total of the column flow and the septum purge flow.



Procedure: Using the pulsed splitless mode

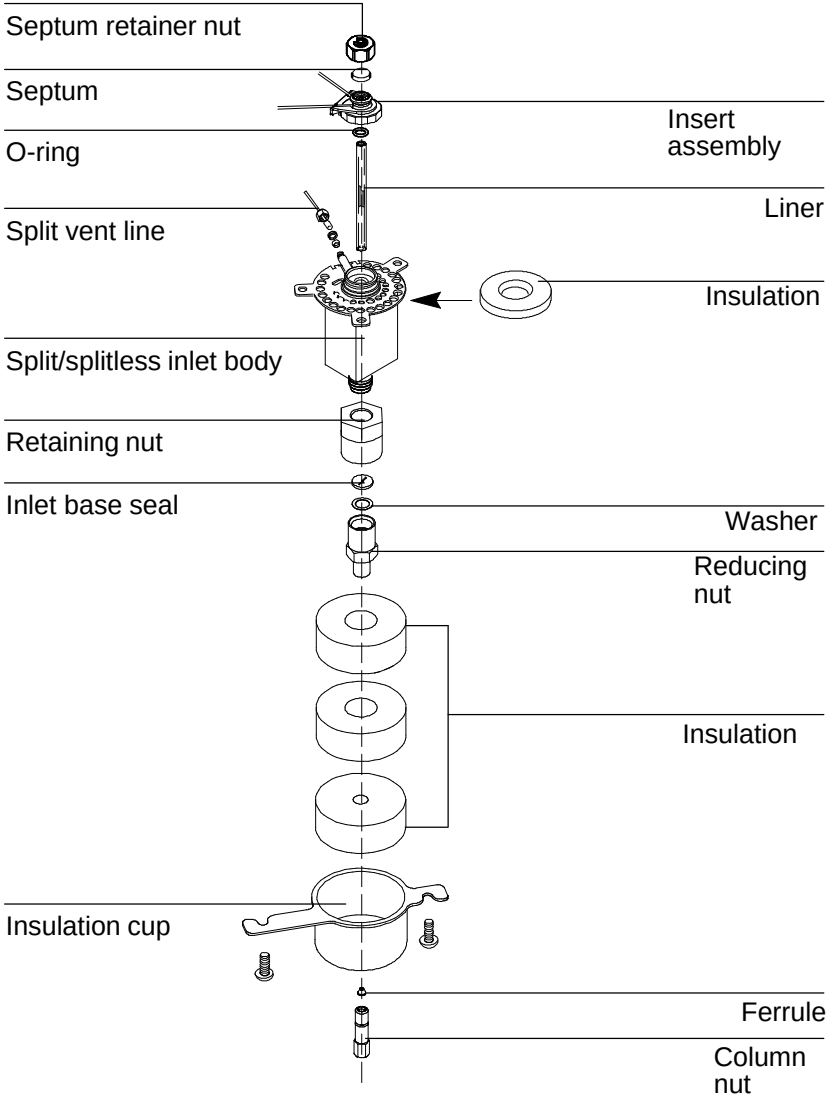
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Splitless.
   b. Set the inlet temperature.
   c. Enter values for Pulsed pres and Pulse time.
   d. Enter the Purge time when you wish the purge valve to open. Set 0.1 to 0.5 minutes before Pulse time.
   e. If your column is defined, enter a Purge flow.
   f. f your column is defined, turn Gas saver on, if desired. Make certain the time is set after the purge flow time.



3. Press the [Prep Run] key (see page 13) before injecting a sample manually.

Part 2. Maintaining a Split/Splitless Inlet

Figure 9. The Split/Splitless Capillary Inlet



Changing septa

If a septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, signal noise will increase.

The useful lifetime of septa depends upon injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is in steady use, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Hewlett-Packard; refer to the Analytical Columns and Supplies Catalog for ordering information.

Table 6. Recommended Septa for the Split/Splitless Inlet

Description	HP part number
11-mm septum, low-bleed red	5181-1263
11-mm septum with partial through-hole, low-bleed red	5181-3383
11-mm septum, low-bleed gray	5080-8896
Merlin microseal septum (see page 170)	5181-8815
11-mm high-temperature silicon septum (350°C and higher)	5182-0739

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

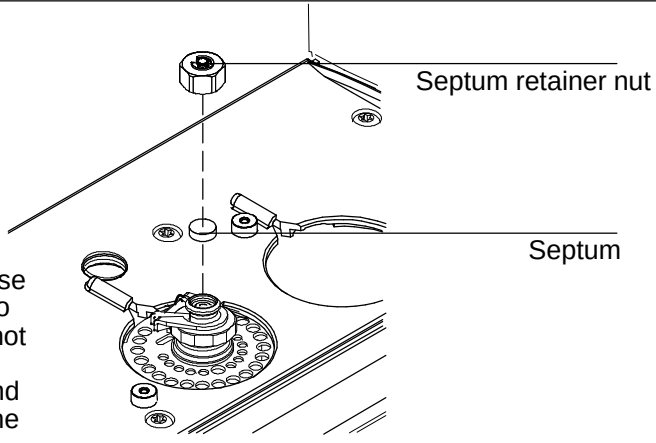
Procedure: Changing the septum

Materials needed:

- Gloves (if inlet is hot)
- New septum—refer to Table 6 on page 36 for part numbers
- Septum nut wrench (HP part no. 19251-00100)
- A nonmetallic (plastic or wood) tool with a sharp tip to remove septum from inlet
- 0- or 00-grade steel wool (optional)
- Forceps or tweezers
- Compressed, filtered, dry air or nitrogen (optional)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.
   - If the inlet is hot, wear gloves to protect your hands from burns.

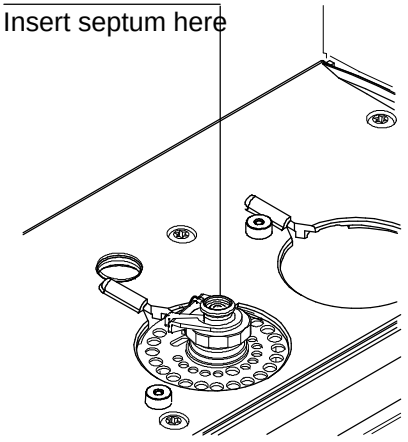
2. Remove the septum retainer nut, using the wrench if the nut is hot or sticks. Remove the old septum.



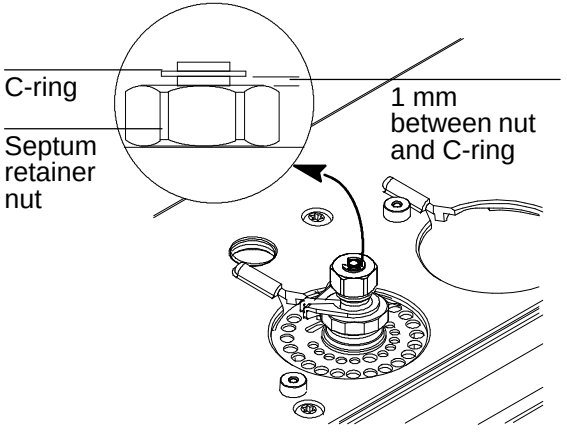
If the septum sticks, use the sharp-tipped tool to remove it. Take care not to gouge the metal around the septum, and remove all pieces of the old septum.

3. If pieces of the septum are sticking, grasp a small piece of rolled-up 0- or 00-grade steel wool with the forceps (or tweezers) and scrub the residue from the retainer nut and septum holder. Use the compressed air or nitrogen to blow away the pieces of steel wool and septum.

4. Use the forceps to insert a new septum. Press it into the fitting firmly.



5. Replace the septum retainer nut, tightening it finger-tight until the C-ring is approximately 1 mm above the nut. When the nut is replaced, you can restore normal operating conditions.



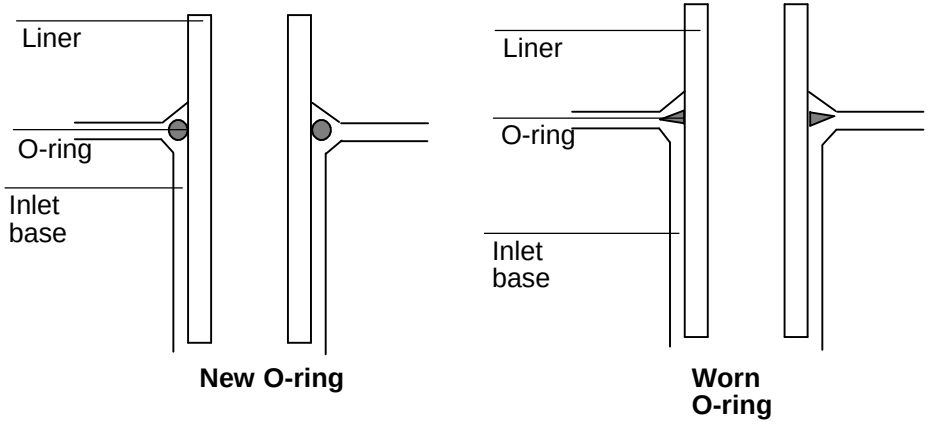
Do not overtighten the nut! Over-tightening over-compresses the septum, which causes premature failure and pieces of septum to fall in the inlet.

Changing the O-ring

You will need to change the O-ring each time you change the liner, or if it wears out and becomes a source of leaks in the inlet. To determine if the O-ring leaks, run the leak test for the split/splitless inlet.

O-rings contain plasticizers that give them elasticity. The O-ring seals the top of the inlet, the inlet base, and the liner. However, at high temperatures the plasticizers bake out, and the O-rings become hard and are no longer able to create a seal (this is referred to as “taking a set”).

Figure 10. Cross Section of Inlet, Liner, and O-ring.



If you regularly operate the inlet at high temperatures, you may want to use graphite O-rings. Although they have a longer life-time, they too will eventually take a set. Refer to the table below to make sure you are using the correct O-ring for your inlet.

Table 7. O-Rings for the Split/Splitless Inlet

Description	HP Part Number
Viton O-ring for temperatures up to 350°C	5181-4182
Graphite O-ring for split liner (temperatures above 350°C)	5180-4168
Graphite O-ring for splitless liner (temperatures above 350°C)	5180-4173

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, wear gloves to protect your hands.

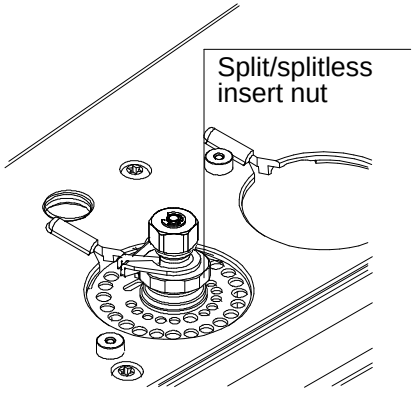
Procedure: Changing the O-ring

Materials needed:

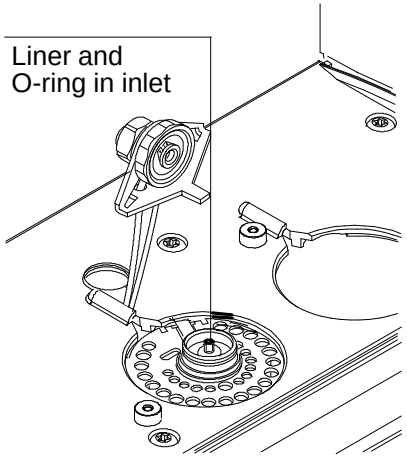
- Gloves (if inlet is hot)
- A new O-ring—refer to Table 7 on page 40
- Septum nut wrench (HP part no. 19251-00100)
- Forceps or tweezers

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.
   - Wear gloves if the inlet is hot to protect your hands from burns.

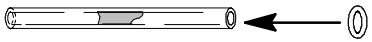
2. Locate the split/splitless insert nut, and loosen it using the wrench if necessary. Lift it straight up to avoid chipping or breaking the liner.



3. You should see the top of the liner with the O-ring around it. Using the forceps (or tweezers), grasp the liner and pull it out.

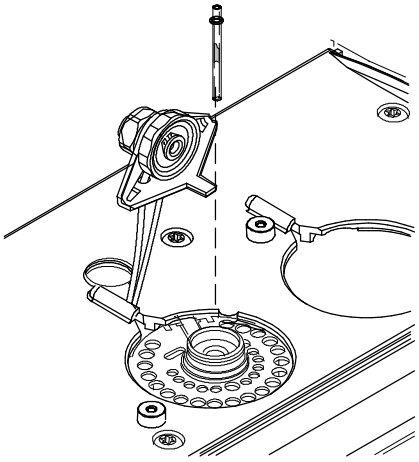


4. Replace the old O-ring with a new one.



Slide new O-ring onto the liner

5. Using the forceps, return the liner to the inlet, replace the insert assembly nut, and use the wrench to tighten the nut just to snugness.



6. Restore the GC to normal operating conditions.

Replacing the inlet base seal

You must replace the inlet base seal whenever you loosen or remove the reducing nut. In addition, chromatographic symptoms such as ghost peaks indicate that the inlet base seal is dirty and should be replaced.

Two types of inlet base seals are available:

- Gold-plated seal, HP part number 18740-20885
- Stainless steel seal, HP part number 18740-20880

You change the inlet base seal from inside the oven, so you must remove the column. If you are unfamiliar with column installation and removal, see the “Columns and Traps” chapter in the General Information volume.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

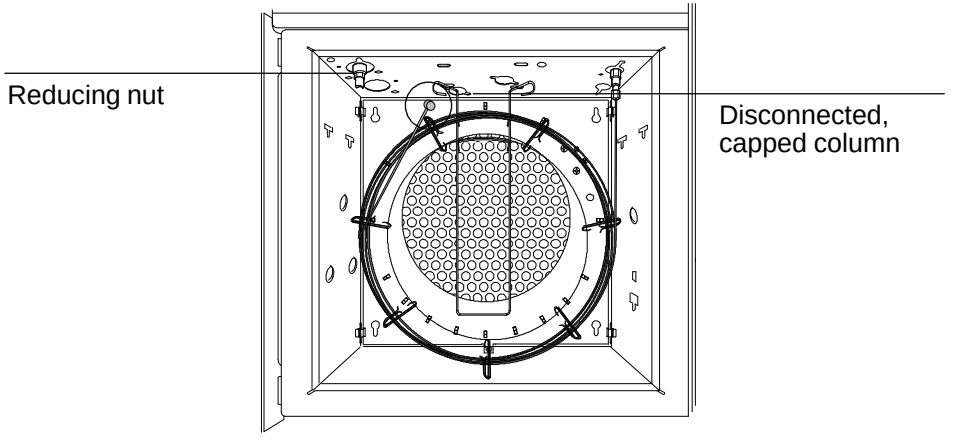
Procedure: Replacing the inlet base seal

Materials needed:

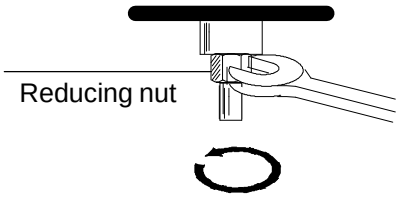
- Clean, lint-free, non-nylon gloves (must wear when handling seal)
- A new seal (see page 43 for part numbers)
- A new washer (HP part no. 5061-5869)
- 1/4-in. wrench (for column)
- 1/2-in. wrench

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.

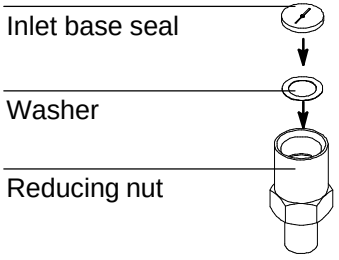
2. Remove the column from the inlet. Cap the open end of the column to prevent contamination. If an insulation cup is installed around the base of the inlet, remove it.



3. Use the 1/2-in. wrench to loosen the reducing nut, and then remove it. The washer and seal are inside the reducing nut. Remove them. You will probably want to replace the washer when you replace the inlet seal.



4. Put on the golves to protect the inlet base seal and washer from contamination. Place the washer in the reducing nut. Place the new inlet base seal on top of it.



Side view of inlet base seal:

Make sure this raised portion faces down.

5. Replace the reducing nut. Use the 1/2-in. wrench to tighten the nut. Replace the column and the insulation cup. If you are not sure how to do so, see the General Information volume. After the column is installed, you can restore normal operating conditions.

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 inlet flow manifold. If this portion of the system proves to be leak-free, refer to the next procedure to check the inlet and inlet manifold.

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, turn the GC off and disconnect the main power cord. 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, remove excess fluid when you have completed the test.
- Two 7/16-in. wrenches

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

Procedure: Leak testing an EPC split/splitless inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

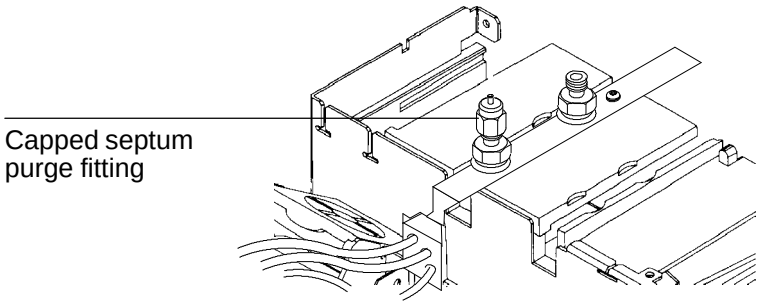
WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-in. wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (HP part no. 19251-00100)
- 9/16-in. wrench
- 1/4-in. SWAGELOK cap
- Bubble flow meter

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
   - Remove the old septum and replace it with a new one. For instructions, see “Changing Septa” on page 36.
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page 40 for instructions.

2. Cap the septum purge fitting with a 1/4-in. SWAGELOK cap.



3. Set the oven to its normal operating temperature. Set the inlet to its normal operating temperature. 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 gas supply is at least 10 psi higher than the inlet pressure.

Press [Front Inlet] or [Back Inlet]

FRONT INLET (S/SL)

Mode		Split
Temp	150	150
Pressure	0.0	24.0<
Split ratio		25.0
Split flow		0.0
Total flow	0.0	Off
Gas saver		Off

4. Set the total flow to 60 ml/min. Wait a few moments for the pressure and flow to equilibrate.

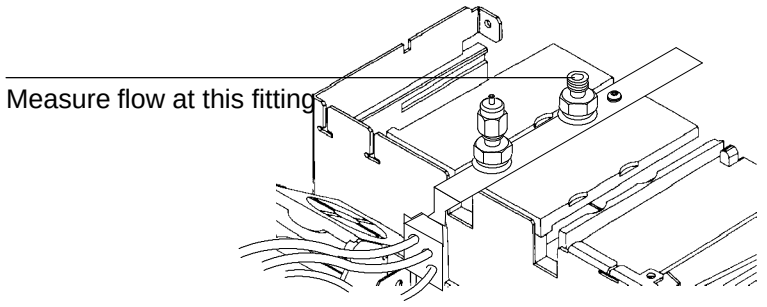
Press [Front Inlet] or [Back Inlet]

The GC may exceed the pressure setpoint slightly while equilibrating.

FRONT INLET (S/SL)

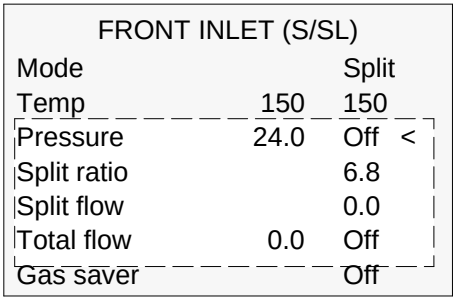
Mode		Split
Temp	150	150
Pressure	24.2	24.0<
Split ratio		25.0
Split flow		0.0
Total flow	60.0	60.0
Gas saver		Off

5. Verify that the flow is actually 60 ml/min by measuring the flow rate at the split purge vent on the manifold. Use a bubble flow meter to measure the flow.



6. Turn either the pressure or the flow off. Because the septum purge and the column fittings are capped, gas should be trapped in the system and the pressure should remain fairly constant.

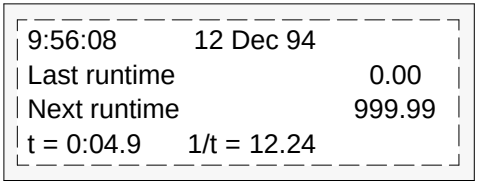
Press [Front Inlet] or [Back Inlet]



Because the pneumatics have been turned off, the alarm does not sound even though there is no flow through the column.

7. Continue to monitor pressure for 10 to 15 minutes. You can use the GC's Stopwatch function. A pressure drop of 0.03 psi/min or less is acceptable.

Press [Time]



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

If the pressure drops faster than the acceptable rate, see "Correcting Leaks" on page 53.

Procedure: Leak testing a nonEPC split/splitless inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Materials needed:

- No-hole ferrule
- 7/16-in. wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (HP part no. 19251-00100)
- 9/16-in. wrench
- 1/8-in. SWAGELOK cap
- Bubble flow meter

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and plug the column fitting with the column nut and a no-hole ferrule.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see page 36.
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page 40 for instructions.
2. Cap the purge vent with a 1/8-in. SWAGELOK cap.
3. Set the oven to its normal operating temperature.
4. Set the inlet to its normal operating temperature. Make sure that the pressure at the initial gas supply is at least 35 psi.

5. Set the inlet pressure between 20 and 25 psi, or to your normal operating pressure, if it is higher. Set the split flow to 60 ml/min. Wait a few moments for the pressure and flow to equilibrate.
6. Verify that the septum purge is off by using a bubble flow meter.
7. Turn off flow to the inlet by turning off the carrier gas at the flow controller. Then, adjust the back pressure regulator clockwise and additional 1/4 turn.

   Observe the column pressure for approximately 15 minutes. If the pressure remains between 19 and 20 psi, or if the pressure drop is 0.03 psi/min or less, you can consider the inlet leak-free.

   If the pressure drops faster than the acceptable rate, go to the next section, “Correcting Leaks.”

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- Tools to tighten connections

1. Use the electronic leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:

   - The capped purge vent
   - The plugged column connection
   - The septum and/or septum nut
   - The area where the gas lines are plumbed to the inlet
   - The O-ring
   - The O-ring nut
   - The inlet base seal

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 inlet 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 inlet system is still losing too much pressure, you may need to replace the inlet manifold. Contact your Hewlett-Packard service representative.

Procedure: Cleaning the inlet

It is unlikely that the inlet will frequently require the thorough cleaning that this procedure presents; however, deposits from injected samples occasionally build up inside the split/splitless inlet. Before cleaning the inlet, replace dirty column liners and inserts with clean ones. If changing them does not correct the problems, then clean the inlet.

Materials needed:

- Cleaning brushes—The FID cleaning kit contains appropriate brushes
  (HP part no. 9301-0985)
- Solvent that will clean the type of deposits in your inlet
- Compressed, filtered, dry air or nitrogen

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the heated zones to room temperature. Turn them off when they cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - Remove the liner.
   - Remove the column and the column liner. See the “Columns and Traps” chapter in the General Information volume.
   - Remove the inlet base seal. See page 43 for instructions.

2. Using a suitable light source, illuminate the inside of the inlet from inside the oven and look for signs of contamination or deposits. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits.

4. Dry thoroughly with the compressed air or nitrogen before reassembling. You can also use compressed air to blow out any loose particles.

Caution
To avoid eye injury, wear eye protection when using compressed gas.

5. Reassemble the inlet. You must replace the inlet base seal at this time. You should replace the septum with a new one. Plug in the GC's power cord, turn it on, and restore it to normal operating conditions.

3

The Purged Packed Inlet

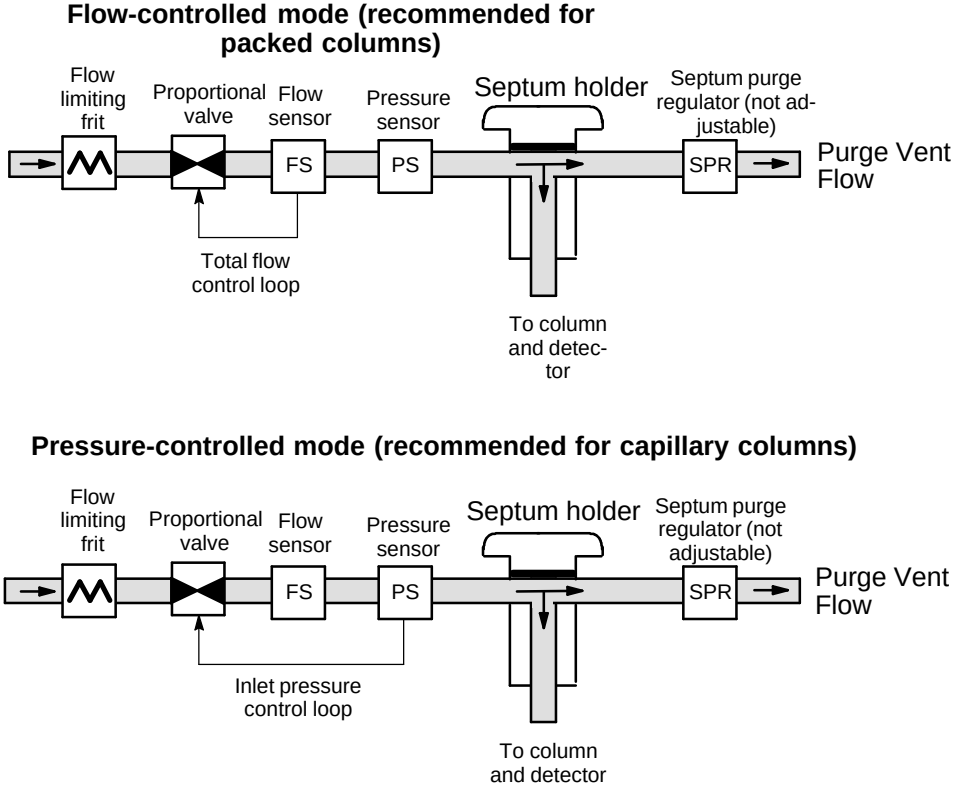
Chapter 3.
The Purged Packed Inlet

Part 1. Using a Purged Packed Inlet

This inlet is used with packed columns when high-efficiency separations are not required. It can also be used with wide-bore capillary columns, provided that flows greater than 10 mL/min are acceptable.

If a capillary column is used and the column is defined, the inlet is pressure-controlled. If the column is not defined (packed columns and undefined capillary columns), the inlet is flow-controlled.

Figure 11. Packed Column Inlet with Electronic Pneumatics Control



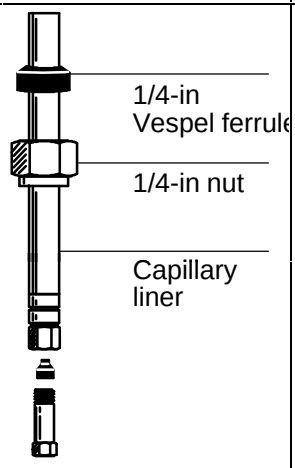
Liners and inserts

Liners. Your choice of liner depends on the type of column you are using. Liners are available for use with wide-bore capillary, 1/4-in. packed, or 1/8-in. packed columns. The liner functions as an adapter so that columns can be connected to the inlet. Installation instructions are on page 61.

Inserts. Glass inserts are often used with metal liners to reduce reactivity and trap nonvolatile residues. They are always used with capillary columns. Inserts are installed from the top of the inlet and should be installed before the column. Installation instructions are on page 63.

The purged packed inlet is shipped with a liner and insert for use with capillary columns (see Table 8.) If you are using packed columns, consult Table 9.

Table 8. Liner and Insert for Wide-Bore Capillary Columns
(Narrow-bore capillary columns are not recommended for use with this inlet.)

Column type	Liner	Insert
530 μm or 320 μm	19244-80540 	5080-8732 or 5181-3382* 

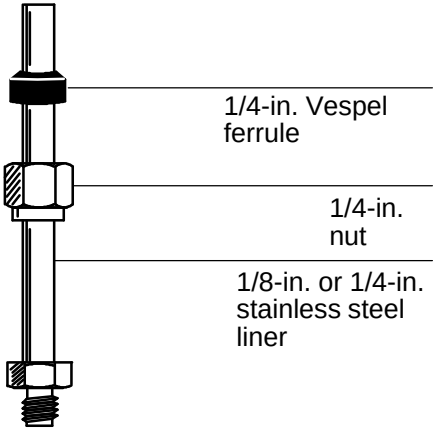
*Deactivated

Table 9. Liner and Insert for Packed Columns

Column Type	Liner	Insert
1/8-in. metal	1/8-in. stainless steel 19243-80510	None
	19243-80530	5080-8732 or 5181-3382*
1/4-in. metal	1/4-in. stainless steel 19243-80520	none
	19243-80540	5080-8732 or 5181-3382*
1/4-in. glass	No liner required. Column end functions as liner. Can also use 1/4-in. metal liner.	Not applicable

*Deactivated

1/4- or 1/8-inch liner (stainless steel)



Insert

Procedure: Installing liners

Use these instructions for installing all liner types. Graphitized Vespel ferrules are recommended because metal ferrules tend to lock permanently onto the liner. If a leak develops when using metal ferrules, you must replace the entire liner.

Materials needed:

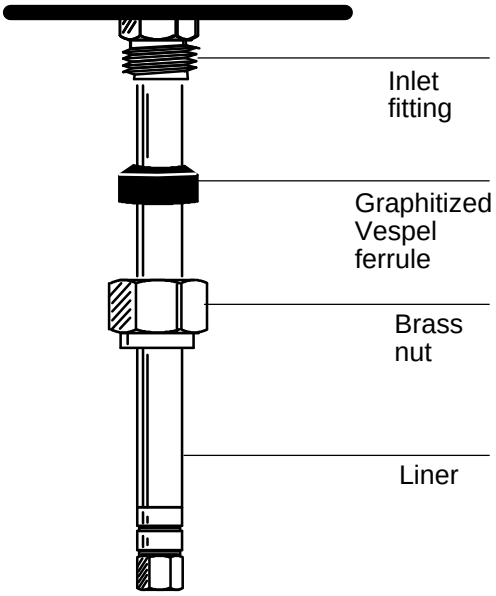
- Liner, brass nut, and ferrule (see Table 8 or Table 9)
- Lint-free cloth
- Methanol
- 9/16-in. wrench

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure or flow.

WARNING Be careful. The oven and inlet fittings may be hot enough to cause burns.

2. Clean the end of the liner with a lint-free cloth to remove contamination such as fingerprints. Use methanol as a solvent.
3. Place a brass nut and graphitized Vespel ferrule on the liner.
4. Open the oven door and locate the inlet base. Insert the liner straight into the inlet base as far as possible.
5. Hold the liner in this position and tighten the nut finger tight.
6. Use a wrench to tighten the nut an additional 1/4 turn.
7. Install the column.
8. Establish a flow of carrier gas through the inlet, and heat the oven and inlet to operating temperatures. Allow these to cool, and then retighten the fittings.

Figure 12. Installing a Liner



Procedure: Installing glass inserts

Materials needed:

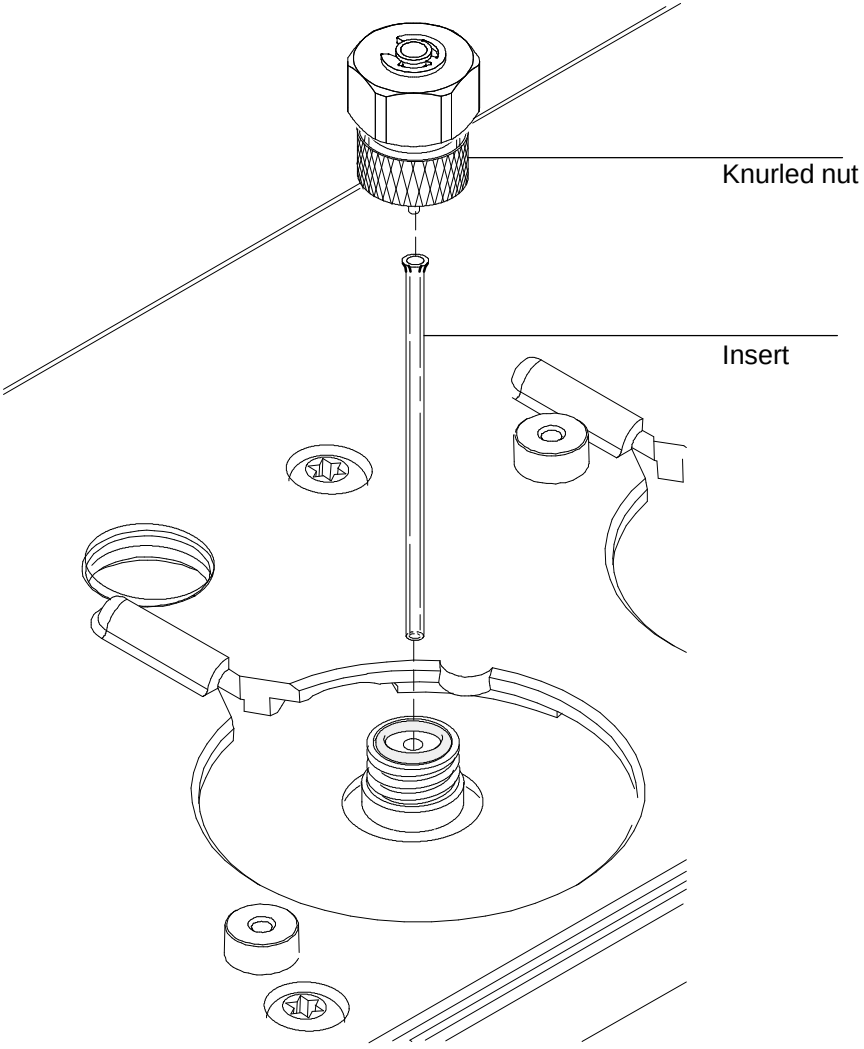
- Insert (see Table 8 or Table 9)
- Tweezers or hemostats
- Wire

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure or flow.

WARNING Be careful. The inlet fittings may be hot enough to cause burns.

2. Remove the knurled nut at the top of the inlet.
3. Carefully remove the old insert. A thin wire (such as a paper clip) may be helpful when lifting the insert from the inlet.
4. Using tweezers or similar tool, grasp the top of the insert and install in the inlet with the flared end up.
5. If a capillary column is installed and the insert does not seat properly, you must remove the capillary column, install the insert, and replace the column.
6. Reinstall the knurled nut and tighten finger tight.

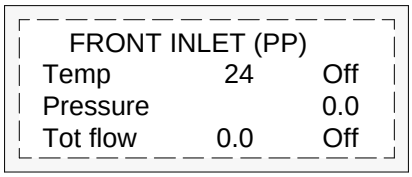
Figure 13. Installing a Glass Insert in a Purged Packed Inlet



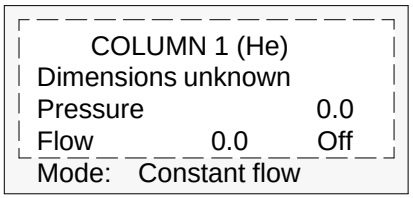
The control table

Packed columns or column not defined

(The inlet)



(The column)



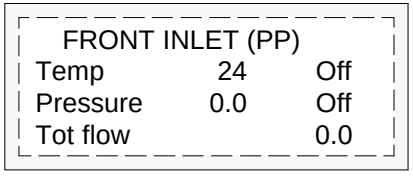
Temp The setpoint and actual temperature values.

Pressure The actual pressure (in psi, bar, or kPa) supplied to the inlet. You cannot enter a setpoint here.

Tot flow Enter your setpoint here, actual value is displayed. Inlet is mass flow controlled.

Defined capillary columns

(column defined)



Temp The setpoint and actual temperature values

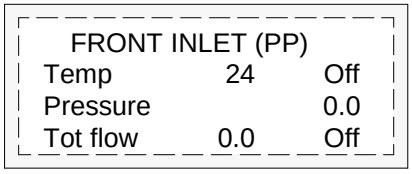
Pressure Inlet is pressure controlled. Enter your setpoint here (in psi, bar, or kPa) and actual value is displayed.

Tot flow The actual total flow to the inlet. This is a reported value, not a setpoint.

Procedure: Using packed and undefined capillary columns

If the column is not defined, only the flow-controlled modes are available.

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet] and enter a temperature. (The flow was set at the column in step 4.)

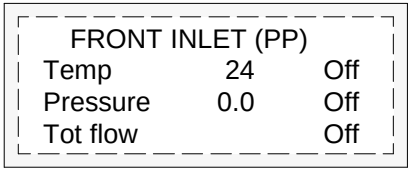


3. Inject a sample.

Set column flow from the Column table, as described in Appendix A. Total flow in the inlet table is the sum of column flow and septum purge flow.

Procedure: Using defined capillary columns

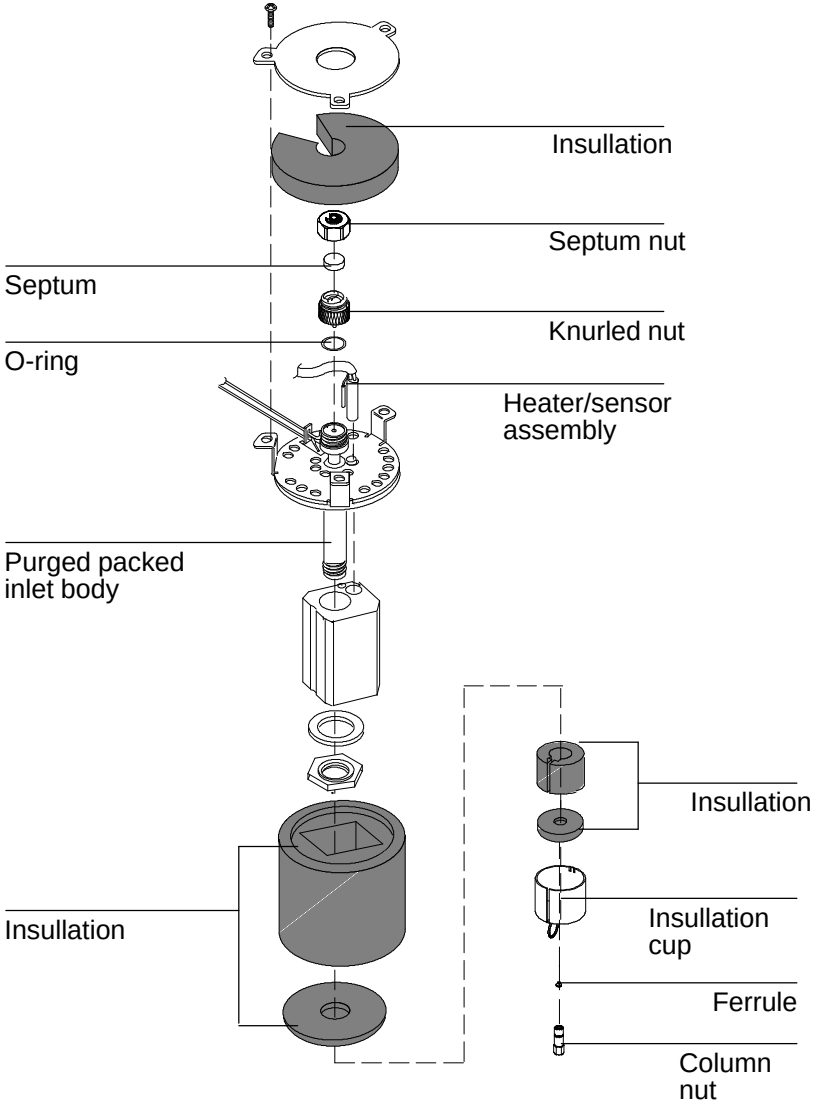
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet] and enter a temperature.



3. Inject the sample.

Part 2. Maintaining a Purged Packed Inlet

Figure 14. The Purged Packed Inlet



Procedure: Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Hewlett-Packard; see the Analytical Columns and Supplies Catalog for ordering information.

Table 10. Recommended Septa for the Purged Packed Inlet

Description	HP Part Number
11-mm septum, low-bleed red	5181-1263
11-mm septum with partial through-hole, low-bleed red	5181-3383
11-mm septum, low-bleed gray	5080-8896
Merlin microseal septum	5181-8815
11-mm high-temperature silicon septum (350°C and higher)	5182-0739

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Caution Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

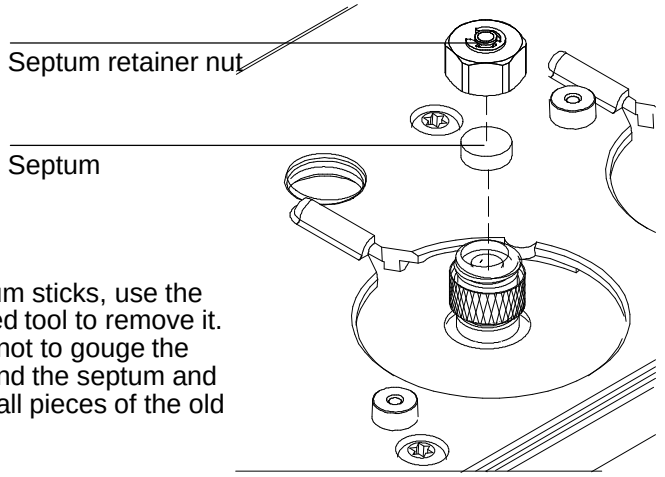
Materials needed:

- Gloves (if the inlet is hot)
- New septum—see Table 10 on page 68 for part numbers
- Septum nut wrench (HP part no. 19251-00100)
- A nonmetallic (plastic or wood) tool with a sharp tip to remove septum from inlet
- 0- or 00-grade steel wool (optional)
- Forceps or tweezers
- Compressed, filtered, dry air or nitrogen (optional)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.

Caution
If you are using packed columns, decrease head pressure to prevent a sudden release of inlet pressure from blowing the packing out of the column.

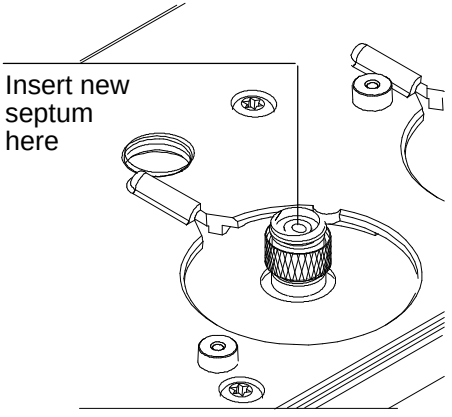
2. If the inlet is hot, wear the gloves to protect your hands from burns. Remove the septum retainer nut, using the wrench to loosen or remove the nut if it is hot or sticks. Remove the old septum.



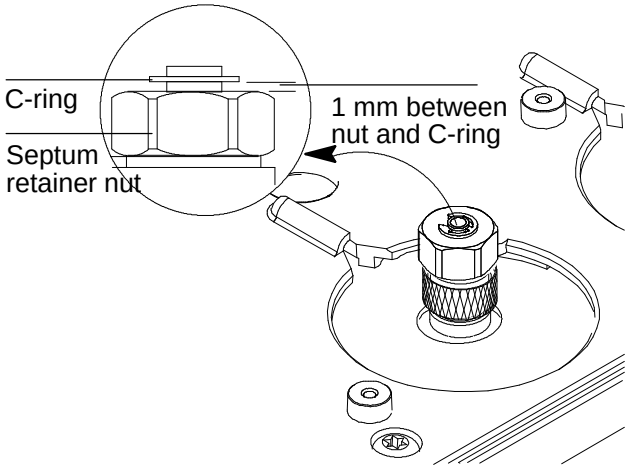
If the septum sticks, use the sharp-tipped tool to remove it. Take care not to gouge the metal around the septum and to remove all pieces of the old septum.

3. If pieces of the septum are sticking, grasp a small piece of rolled-up 0- or 00-grade steel wool with the forceps (or tweezers), and scrub the residue from the retainer nut and septum holder. Use the compressed air or nitrogen to blow away the pieces of steel wool and septum.

4. Use the forceps to insert a new septum. Press it into the fitting firmly.



5. Replace the septum retainer nut, tightening it finger-tight until the C-ring is approximately 1 mm above the nut. When the nut is tightened, you can restore normal operating conditions.



Do not overtighten the nut! Over-tightening over-compresses the septum, which could cause premature failure and pieces of septum to contaminate the inlet.

Procedure: Changing the O-ring

You will need to change the O-ring periodically because it wears out and becomes a source of leaks in the inlet. To determine if the O-ring leaks, perform the leak test presented later in this chapter.

O-rings contain plasticizers that give them elasticity. The O-ring seals the top of the inlet and the inlet base. However, at high temperatures the plasticizers bake out, and the O-rings become hard and are unable to create a seal (this is referred to as "taking a set"). If you operate the inlet at high temperatures, you will probably need to replace the O-ring frequently.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, be sure to wear gloves to protect your hands.

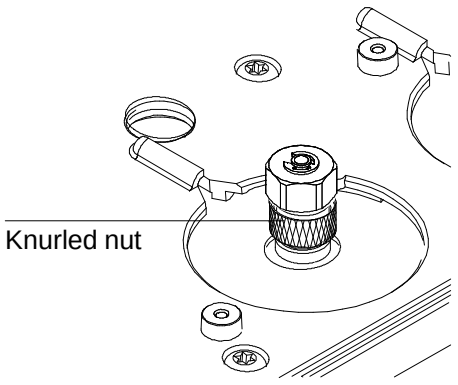
Materials needed:

- Gloves (if the inlet is hot)
- A new Viton O-ring (HP part no. 5080-8898)
- Septum nut wrench (HP part no. 19251-00100)
- Forceps or tweezers (optional)

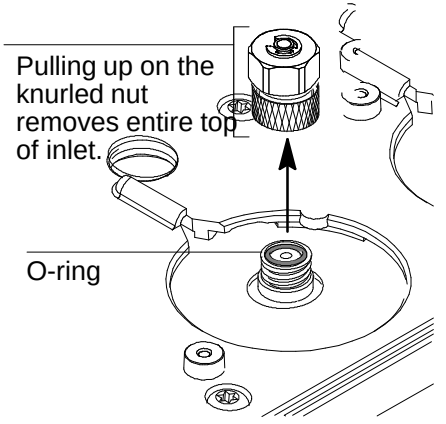
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Turn off the detector.
   - Cool the inlet to room temperature.
   - Turn the inlet pressure off.

Caution
If you are using packed columns, decrease head pressure to prevent a sudden release of inlet pressure from blowing the packing out of the column.

2. If the inlet is hot, use the septum nut wrench. Loosen the knurled nut completely. Pull up on the nut to remove the top portion of the inlet.



3. The O-ring will be visible. Remove the old O-ring. You may need to use forceps to grab it. Using the tweezers, insert the new O-ring.



4. Replace the top portion of the inlet and tighten the knurled nut until you cannot tighten it further. Restore the GC to normal operating conditions.

Procedure: Leak testing the gas plumbing

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

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, turn the GC off and disconnect the main power cord. Be careful not to spill leak solution on electrical leads, especially the detector heater leads.

Materials needed:

- Electronic leak detector or liquid leak detection fluid. If you use leak detection fluid, wipe off excess fluid when you have completed the test.
- Two 7/16-in. wrenches

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

Procedure: Leak testing an EPC purged packed inlet

This procedure allows you to determine if the inlet is leaking. It is recommended that you leak test the inlet at your normal operating temperature since the O-ring may leak if it is cooled to ambient.

Materials needed:

- Gloves (if the inlet is hot)
- Septum nut wrench (HP part no. 19251-00100)

If you are using capillary columns:

- No-hole ferrule
- 7/16-in. wrench

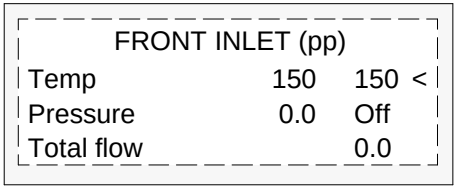
If you are using packed columns:

- Solid Vespel plug
- 9/16-in. wrench

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and cap the column fitting.
     If you are using capillary columns, insert a no-hole ferrule in the column nut to create a plug.
     If you are using packed columns, use the Vespel plug.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see “Changing Septa” on page 68.
   - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page 72 for instructions on changing the O-ring.
   - Make sure that the pressure at the initial gas source is at least 35 psi.

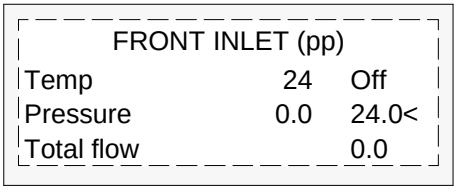
2. Set the inlet to its normal operating temperature.

Press [Front Inlet] or [Back Inlet]



3. Set the inlet pressure between 20 and 25 psi.

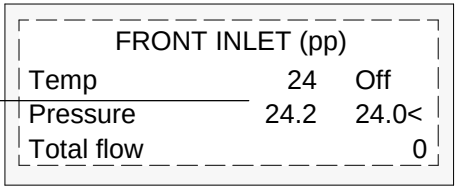
Press [Front Inlet] or [Back Inlet]



4. Wait a few minutes for the pressure to equilibrate.

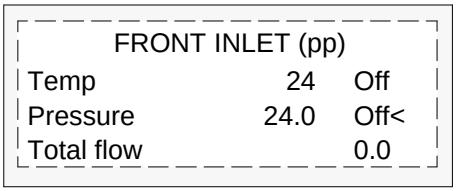
Press [Front Inlet] or [Back Inlet]

The GC may exceed the pressure setpoint slightly while equilibrating.



6. Turn the pressure off. Because the column is capped, the pressure should remain fairly constant.

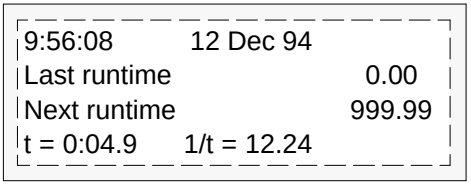
Press [Front Inlet] or [Back Inlet]



Because the pneumatics have been turned off, the alarm does not sound even though there is no flow through the column.

7. Continue to monitor pressure for 10 to 15 minutes. You can use the GC's Stopwatch function. A pressure drop of 0.03 psi/min or less is acceptable.

Press [Time]



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

If the pressure drops faster, go to "Correcting Leaks" on page 80.

Procedure: Leak testing a nonEPC purged packed inlet

This procedure allows you to determine if the inlet leaks. It is recommended that you leak test the inlet at your normal operating temperature since the O-ring is likely to leak if it is cooled to ambient.

Materials needed:

- Gloves (if the inlet is hot)
- Septum nut wrench (HP part no. 19251-00100)
- 1/8 in. SWAGELOK cap (HP part no. 5180-4120)

If you are using capillary columns:

- No-hole ferrule
- 7/16-in. wrench

If you are using packed columns:

- Solid Vespel plug
- 9/16-in. wrench

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and cap the column fitting.
     If you are using capillary columns, insert a no-hole ferrule in the column nut and then attach the nut to the fitting.
     If you are using packed columns, attach the Vespel plug to the fitting.
   - Remove the old septum and replace it with a new one. For instructions on changing septa, see “Changing Septa” on page 68.
   - Inspect the O-ring, and replace it if it is hard and brittle or cracked. See page 72 for instructions on changing the O-ring.

2. Set the inlet to its normal operating temperatures.
3. Cap the septum purge vent with a 1/8-in. SWAGELOK cap.
4. Turn on the gas to the inlet at its source and adjust the supply pressure to 50 psi. Completely open the mass flow controller by turning the knob counterclockwise as far as it can go. Wait 2 minutes to insure equilibrium.
5. Turn off the gas to the inlet at its source. Monitor the pressure for 10 to 15 minutes. You can use the GC's Stopwatch function. A pressure drop of 0.03/min psi or less is acceptable.

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

   If the pressure drops faster than this, go to "Correcting Leaks" on page 80.

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector suitable for the gas type
- Tools to tighten parts of the inlet that leak (if leaks are detected)

1. Use the leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:

   - The septum and/or septum nut
   - The 1/4-in. ferrule (if a liner is being used)
   - The O-ring
   - The capped purge vent
   - The plugged column connection
   - The knurled nut
   - The area where the gas line is plumbed to the inlet

   If no liner is used, then column must be plugged with 1/4-in. SWAGELOK cap or equivalent

2. Correct leaks using a wrench to tighten loose connections, if necessary. You may need to repeat the leak test.

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

   If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet is still losing too much pressure, you may need to replace the inlet manifold. Contact your Hewlett-Packard service representative.

Procedure: Cleaning the inlet

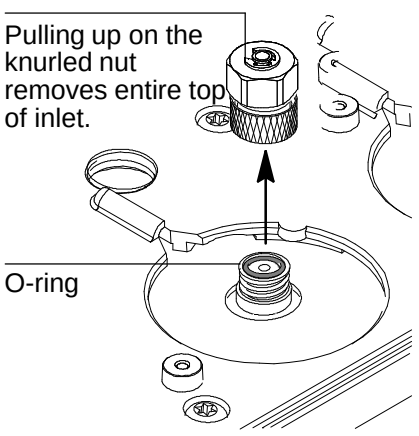
It is unlikely that the inlet will frequently require cleaning as thoroughly as this procedure presents; however, deposits from injected samples occasionally build up inside the purged packed inlet. Before cleaning the inlet, replace dirty column liners and inserts with clean ones. See the Inlets volume for instructions. If changing them does not correct the problems, then clean the inlet.

Materials needed:

- Cleaning brushes—The FID cleaning kit contains appropriate brushes
  (HP part no. 9301-0985)
- Solvent that will clean the type of deposits in your inlet
- Compressed, filtered, dry air or nitrogen

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Allow the heated zones to cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - If the septum is worn out or dirty, replace it. See page 68 for instructions.
   - Remove the column and the column liner and insert. See the “Columns and Traps” chapter in the General Information volume.

2. Loosen the knurled nut and then pull it upward. The O-ring will be visible. Replace it if it is hard and brittle or cracked. See page 72 for the procedure.

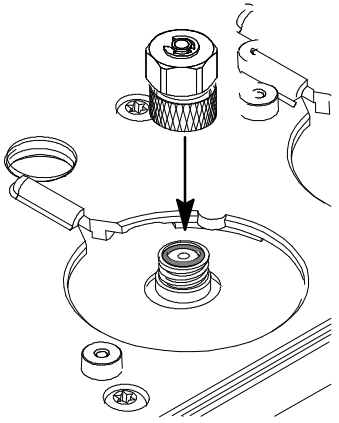


3. Using a suitable light source, illuminate the inside of the inlet from inside the oven while looking through the inlet from the top. If deposits are present, they should be visible.

4. Insert the brush into the inlet. Scrub the interior walls of the inlet vigorously to remove all deposits. You may need to wet the brush with solvent. Use a burst of the compressed air or nitrogen to dry the inlet and remove loose contaminants.

WARNING
To avoid eye injury, wear eye protection when using compressed gas.

5. Replace the top of the inlet and tighten the knurled nut. Replace the column (the procedure is in the General Information volume).



6. Plug in the GC's power cord, turn it on, and restore it to normal operating conditions.

4

The Cool On-Column Inlet

Chapter 4.
The Cool On-Column Inlet

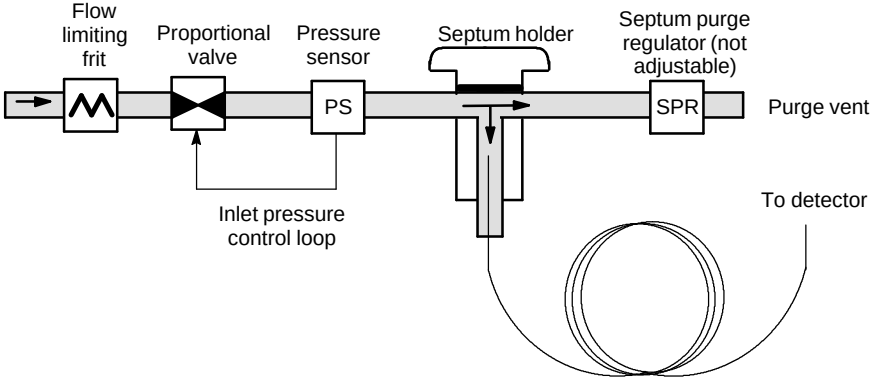
Part 1. Using a Cool On-Column Inlet

This inlet introduces liquid sample directly onto a capillary column. To do this, both the inlet and the oven must be cool at injection, at or below the boiling point of the solvent. Because the sample does not vaporize immediately in the inlet, problems with sample discrimination and sample alteration are minimized. If done properly, cool-on column injection also provides accurate and precise results.

You can operate the inlet in track oven mode, where the inlet temperature follows the column oven, or you can program up to three temperature ramps. There is also a cryogenic cooling option that uses liquid CO_2 or N_2 to reach subambient temperatures.

This inlet is only available with electronic pneumatics control. Figure 15 shows the inlet pneumatics.

Figure 15. Cool On-Column Capillary Inlet with EPC



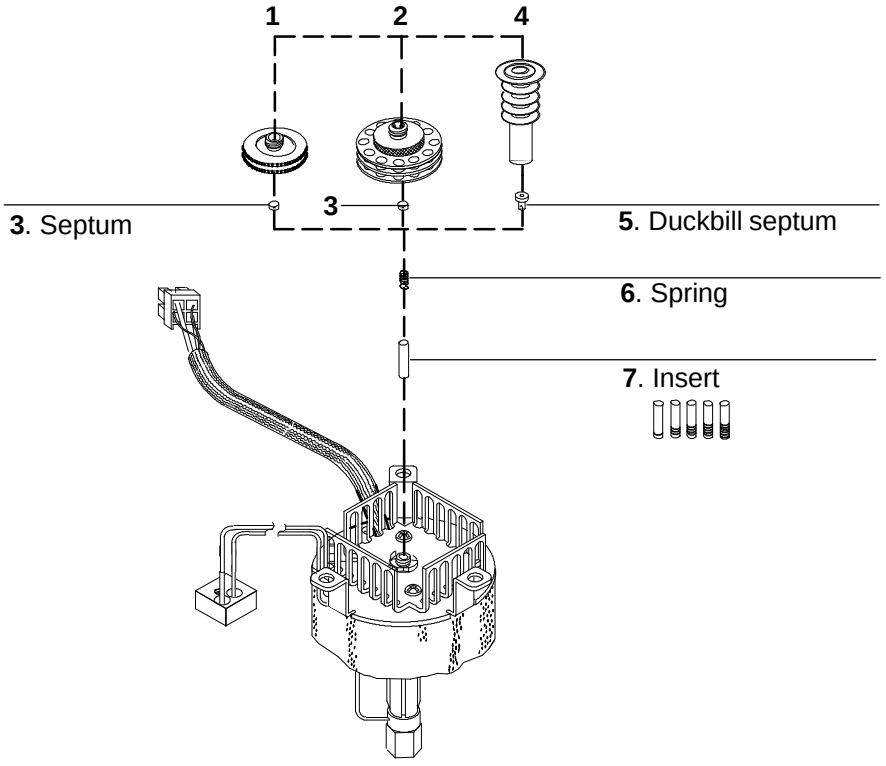
Hardware

Because you are injecting sample directly into the column, most of the hardware required is determined by your column inside diameter. Injection technique, manual or automatic, must also be considered. Table 11 is a checklist for choosing hardware and shows where to find instructions for installing the hardware and injecting the sample.

Table 11. Hardware and Procedures Checklist

Automatic Injection	Manual Injection with Septum Nut	Manual Injection with Cooling Tower
Hardware		
See Table 12 for part numbers	See Table 12 for part numbers	See Table 13 for part numbers
☐ Septum nut	☐ Septum nut	☐ Cooling tower
☐ Insert	☐ Solid septum	☐ Duckbill septum
☐ Stainless steel needle	☐ Insert	☐ Insert
	☐ Stainless steel needle	☐ Fused silica needle (columns $\geq$200 µ) or ☐ Stainless steel needle (columns $\geq$250 µ)
Where to find instructions		
☐ Installing an Insert, page 90	☐ Installing an Insert, page 90	☐ Installing an Insert, page 90
☐ Changing the septum nut or cooling tower assembly, page 89	☐ Changing the septum nut or cooling tower assembly, page 89	☐ Changing the septum nut or cooling tower assembly, page 89
☐ Checking the needle-to-column size, page 91	☐ Manual Injection technique with septum nut and stainless steel needle, page 92	☐ Manual injection technique with cooling tower, page 93 and Replacing the fused silica syringe needle, page 101
☐ Automatic Sampler Operating Manual, HP part no. G1513-90100		

Figure 16. Hardware for the Cool On-Column Inlet



Septum nut and septum, manual or automatic injection
1. Septum nut (HP part no. 19245-80521) for use with 250-µ and 320-µ columns.
See Sampler manual for needle support assembly requirements.
2. Septum nut (HP part no. 19245-20670) for use with 530-µ columns
3. Septum

Cooling tower and duckbill septum, manual injection
4. Cooling tower assembly (HP part no. 19320-80625)
5. Duckbill septum (HP part no. 19245-40050) for use with columns 200 µ and larger

For all applications:
6. Spring. Keeps insert in position.
7. Insert. Guides the needle smoothly into the column. Choose based on column and needle.

Automatic or manual injection with septum nut

Choose a needle, septum nut, and insert based on your column inside diameter. Use Table 12 to select hardware for your injection. See Table 13 if you are doing manual injections with a duckbill septum.

Table 12. Automatic or Manual Injection with a Stainless Steel Needle

Column Type and Inside Diameter	**Needle** HP part no.:*	**Septum nut** HP part no.:	**Insert** HP part no.:
Fused silica:			
530 µ id	5182-0832**	19245-20670 	19245-20580 
320 µ id	5182-0831	19245-80521 	19245-20525  (five rings)
250 µ id	5182-0833	19245-80521 	19245-20515  (six rings)
200 µ id	Use cooling tower and duckbill septum		
Aluminum-clad, 530 µ id	5182-0832	19245-20670 	19245-20780  (four rings)
Glass capillary			
320 µ id	5182-0831	19245-20670 	19245-20550  (three rings)
250 µ id	5182-0833		

* Order removable needle syringe, HP part no. 5181-0836. If doing a manual injection, you must also order a plunger button, HP part no. 5181-8866.

** Many other needles can be used to inject onto a 530-µ column. Consult the Hewlett-Packard Anal −ytical Columns and Supplies catalog for details.

Manual injection with a cooling tower and duckbill septum

If you are doing this type of manual injection, use either fused silica or metal removable stainless steel needles. Use Table 13 to choose the correct insert and syringe.

Table 13. Manual Injection Hardware—Cooling Tower & Duckbill Septum

Column Type and Inside Diameter	**Insert** (HP part no.)	
Fused silica		
530 µ	19245-20580	 (no rings)
320 µ	19245-20525	 (five rings)
250 µ	19245-20515	 (six rings)
200 µ (fused silica needle only)	19245-20510	 (one ring)
Aluminum-clad, 530 µ	19245-20780	 (four rings)
Glass capillary	19245-20550	 (three rings)

Syringe and needle

Fused silica needle syringe (HP part no. 9301-0658)
Replacement needles (HP part no. 19091-63000)

or

Metal removable needle syringe (HP part no. 9301-0562)
Replacement needles (HP part no. 9301-0561)

Procedure: Changing the septum nut or cooling tower and septum

If you need to change the insert, refer also to the next section, "Installing the Insert."

1. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.

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

2. Locate the septum nut or cooling tower assembly at the top of the inlet and remove (see Figure 16). If you are using a cooling tower, grasp the three rings and unscrew. If you are using a septum nut, grasp the knurling and unscrew.

   There should be a small spring at the inlet base. If the spring is stuck to the septum nut, place it back in the inlet base.

3. If you are using a septum nut, remove the old septum with tweezers, hemostat, or septum remover. Use tweezers to install a new septum. Push the septum into the septum nut until properly seated.

   If you are using a cooling tower assembly, locate the duckbill septum and install in the inlet base so that the duckbill is inserted inside the coil spring.

4. Install the septum nut or cooling tower assembly and tighten firmly.

5. Before making an injection, check the alignment of the entire assembly.

Procedure: Installing an insert

1. Choose an insert. See Table 12 or Table 13 for instructions on choosing an insert.
2. Press [Oven] and set the oven to 35°C. When the temperature reaches setpoint, turn the oven off. Press [Front Inlet] or [Back Inlet] and turn off the inlet temperature and pressure.
3. Remove the column, column nut, and ferrule.
4. Locate the septum nut or cooling tower assembly at the top of the inlet and remove it.

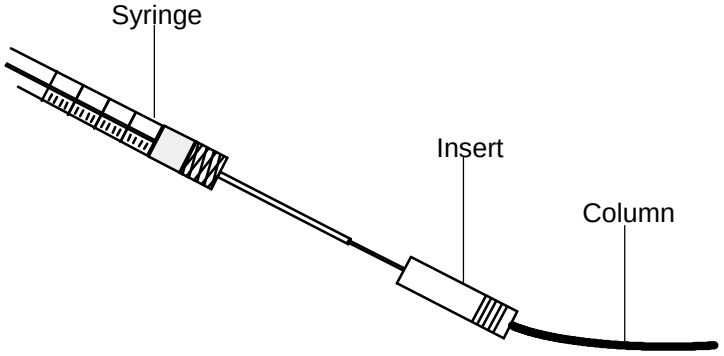
   If the septum remains in the septum nut, do not remove it unless you want to change it. If necessary, replace the existing septum or duckbill with a new one. See the Maintenance part of this chapter for detailed instructions. Set the inlet septum nut or cooling tower assembly aside.
5. Remove the spring from the inlet with an extraction wire, and set it aside. Be careful not to lose or damage it because you will use the spring to keep the new insert in position.
6. Remove the existing insert from the inlet by gently pushing it out from below with a wire or piece of column. Store the insert for possible later use.
7. Drop the new insert straight into the inlet from the top.
8. Replace the spring on top of the insert.
9. Reinstall the septum nut or duckbill septum and cooling tower assembly and tighten finger tight.
10. Reinstall the column, nut, and ferrule.

Procedure: Check the needle-to-column size

Caution This applies to 250 µ and 320 µ columns only.

After selecting an insert and before installing a column, you need to check the needle-to-column size to make certain your needle fits in the column. You could bend the needle if you try to inject it into a smaller column. Use the insert that is the same size as your syringe needle to verify that the column you plan to use is the correct size.

1. Identify the correct insert.
2. Insert the column into one end of the insert as shown below.



3. Insert the syringe needle through the other end of the insert and into the column. If the needle cannot pass easily into the column, reverse the insert to try the needle and column in the other end.

If the needle still cannot pass into the column, you may have a column with an incorrect id. Check the column to make sure it is labeled correctly, and try a new column.

Procedure: Manual injection with septum nut

Before making your injection, make sure the correct septum nut and septum are installed.

1. Immerse the syringe needle in sample; pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe.
3. Remove the needle from the sample and draw about 1 µL of air into the syringe.
4. Wipe the needle dry if it is wet.
5. Guide the needle straight into the septum nut, pierce the septum, and insert the needle fully into the inlet until it bottoms.
6. Start the run, depress the syringe plunger as quickly as possible, and withdraw the needle from the inlet.

These steps should be done smoothly, with minimal delay.

Procedure: Manual injection with cooling tower

When injecting with fused silica or metal removable stainless steel needles, be sure the cooling tower assembly and duckbill are installed on the inlet. Initial pressure must be set at less than 30 psi. Higher pressures will make needle insertion difficult.

1. Immerse the syringe needle in the sample and pump the syringe plunger to expel air from the barrel and needle.
2. Draw the sample into the syringe. Allow enough time for fluids to pass through the small bore of the needle.
3. Remove the needle from the sample and draw about 1 µl of air into the syringe. Wipe the needle with a tissue wetted with solvent.
4. Press down the top of the cooling tower with a pencil to open the duckbill.

WARNING The cooling tower may be hot!

5. Hold down the cooling tower and guide the needle until it is fully inserted in the inlet. You may observe a drop in the pressure reading on the control table.

   If the needle does not go in all the way, try rotating the syringe and slightly releasing pressure on the cooling tower.

   If you still cannot get the needle in, the duckbill opening may be stuck. Try removing the duckbill, opening it manually, and reinstalling it.
6. Once the needle has entered the column, release the cooling tower and continue to insert the needle. Allow 1 to 2 seconds for back pressure on the duckbill to seal it around the inserted needle.
7. Start the GC, depress the syringe plunger as quickly as possible, and withdraw the needle from the inlet.

Retention gaps

Because the sample is injected directly onto the column, it is strongly suggested that a retention gap—or guard column—be used to protect your column. A retention gap is a deactivated column that is connected between the inlet and the analytical column. If you choose to use one, it is suggested that at least 1 m of retention gap be installed per 1 μL of sample injected. Information on ordering retention gaps can be found in the Hewlett-Packard Analytical Columns and Supplies Catalog.

If you are using a retention gap and are operating with column defined, the length of the retention gap could affect the calculations for flow and velocity through your column. If your retention gap is the same inside diameter as your column, it is a good idea to add the retention gap and column length before entering the number on the Configure Column control table. If the retention gap inside diameter is larger than your column, this step may not be necessary.

Inlet temperature

CryoBlast (optional)

CryoBlast shortens the cycle time between runs. If you have a CO_2 or N_2 cryogenic valve and the cryoblast feature, you can cool the inlet to 37°C in either the track oven or temperature program modes.

Track oven mode

In the Track oven mode, the inlet temperature stays 3°C higher than the oven temperature throughout the oven program. You cannot enter a temperature setpoint—it is set automatically. If you have CryoBlast, the inlet will track oven temperatures to 40°C; without CryoBlast, the lower limit is set by room temperature.

Temperature programming mode

In this mode, you can enter up to three temperature ramps in the inlet control table so that the inlet and the oven operate independently. This is the recommended mode if operating below 20C.

At these very low oven temperatures, the inlet temperature should be at least 20C higher than the oven temperature. This will be more than adequate for solvent focusing.

At temperatures greater than ambient, the inlet should always be at least 3C warmer than the oven for proper control of the inlet temperature.

The oven temperature program controls the run. If it is longer than the inlet temperature program, the inlet will remain at its final temperature until the oven program (and the run) ends.

Cryogenic considerations

When using track oven mode with a cryogenic oven, all other inlets must be off or in track oven mode.

Setpoint ranges

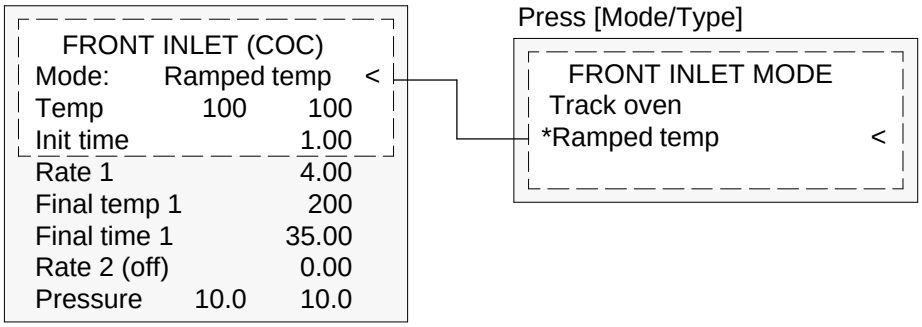
The table below lists setpoint ranges for the inlet parameters.

Temperature	Allowed setpoint range
Track oven	3°C higher than the oven temperature to a maximum of 450°C. If you have cryoblast, the inlet can maintain temperatures down to −40°C, although allowable oven setpoints are 60°C for CO_2 and −80°C for N_2
Ramped temp without cryoblast	24°C to 450°C
Ramped temp with cryoblast	40°C to 450°C

Procedure: Programming the temperature

1. Press [Front Inlet] or [Back Inlet].
2. Press [Mode/Type] and select Ramped temp.

Ramped temp mode



3. Enter a Temperature. This is the starting temperature.
4. Enter an Init time. This is the length of time the inlet will stay at the starting temperature after a run has begun.
5. Enter a Rate. This is the rate at which the inlet will be heated or cooled. A Rate of 0 halts further programming.
6. Enter the Final temp. This is the inlet temperature at the end of the first ramp.
7. Enter the Final time. This is the number of minutes the inlet holds the Final temp.
8. To enter a second (or third) ramp, scroll to the appropriate Rate line and repeat steps 5 through 7.

Procedure: Operating the cool on-column inlet

Verify that a column and suitable insert and septum nut or cooling tower are installed. Make certain you are using a needle that will fit the column.

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.

   Pressure can be set from either the column or inlet table. In constant or ramped flow mode, the pressure will be determined from the flow requirements. It is best to set flow only.

Track oven mode

FRONT INLET (COC)		
Mode:	Track oven	
Temp	24	Off
Pressure	10.0	10.0

Ramped temp mode

FRONT INLET (COC)		
Mode:	Ramped temp	
Temp	100	100
Init time		1.00
Rate 1		4.00
Final temp 1		200
Final time 1		35.00
Rate 2 (off)		0.00
Pressure	10.0	10.0

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

   a. Choose a temperature mode: Track oven or Ramped temp.

   b. For Ramped temp mode, enter your temperature ramps (page 96). There is no setpoint for Track oven mode.

3. Inject a sample.

Part 2. Maintaining a Cool On-Column Inlet

Maintaining the cool on-column inlet includes changing septa, cleaning inlet components, and checking and correcting leaks in the system.

The cool on-column inlet's hardware will vary depending on whether you will be making manual or automated injections, the type of needle you use, and the size of column you use.

Figure 17. The Cool On-Column Inlet for Automatic Injection Systems

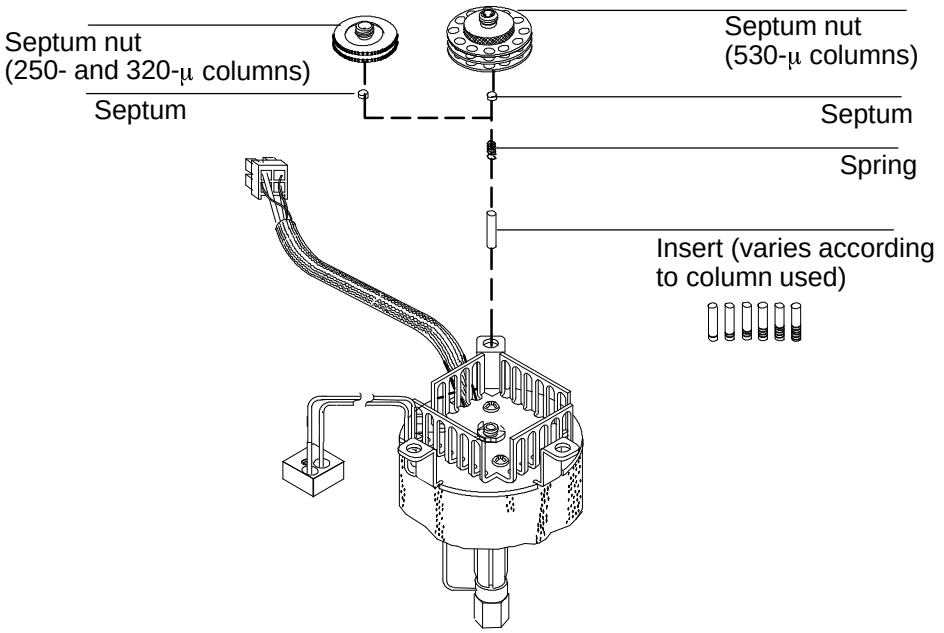
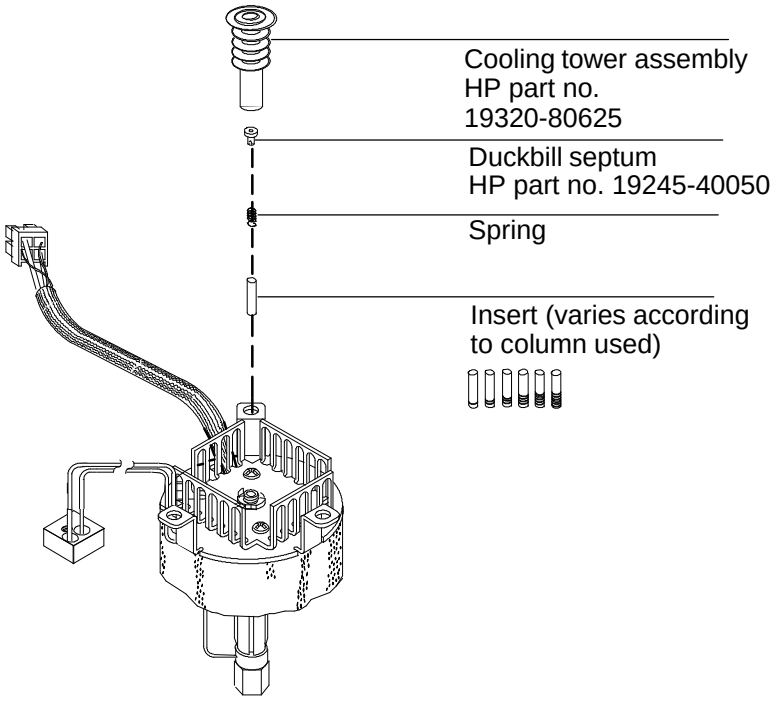


Figure 18. The Cool On-Column Inlet for Manual Injection Systems



Cool on-column inlet hardware problems

The inlet cools very slowly

- The inlet fan is not running or is blowing away from the inlet. Check the fan to make sure it is operating. If it is not, contact your Hewlett-Packard service representative.

The inlet is unable to reach a temperature setpoint

- Check the temperature equilibration time. If the equilibration time is too short, the inlet may oscillate. Increase the equilibration time.
- Check that the cryogenic cooling is turned off. If you do not turn off cryogenic cooling when it is not in use, both the inlet and the oven may be unable to reach a temperature setpoint, particularly temperatures near room temperature. If you turn the cryogenic cooling off and the inlet still fails to reach the setpoint temperature, contact your Hewlett-Packard service representative.

The syringe needle bends during injections

- The needle may have been defective before the injection was made. Check each syringe before injection to make sure the needle is straight.
- Check that the needle support assembly is installed correctly
- Check that the correct insert is installed and that it is installed correctly
- Check the alignment of the inlet septum and the septum nut.
- The inlet septum hole may have closed. Replace the septum.

If you are using the GC Automatic Liquid Sampler (GC ALS):

See the GC ALS manual for additional information.

- The sampler vials may be over-crimped.
- Check the needle guide for signs of wear or damage. Replace the needle guide if necessary.
- Check the alignment of the inlet and the automatic sampler.

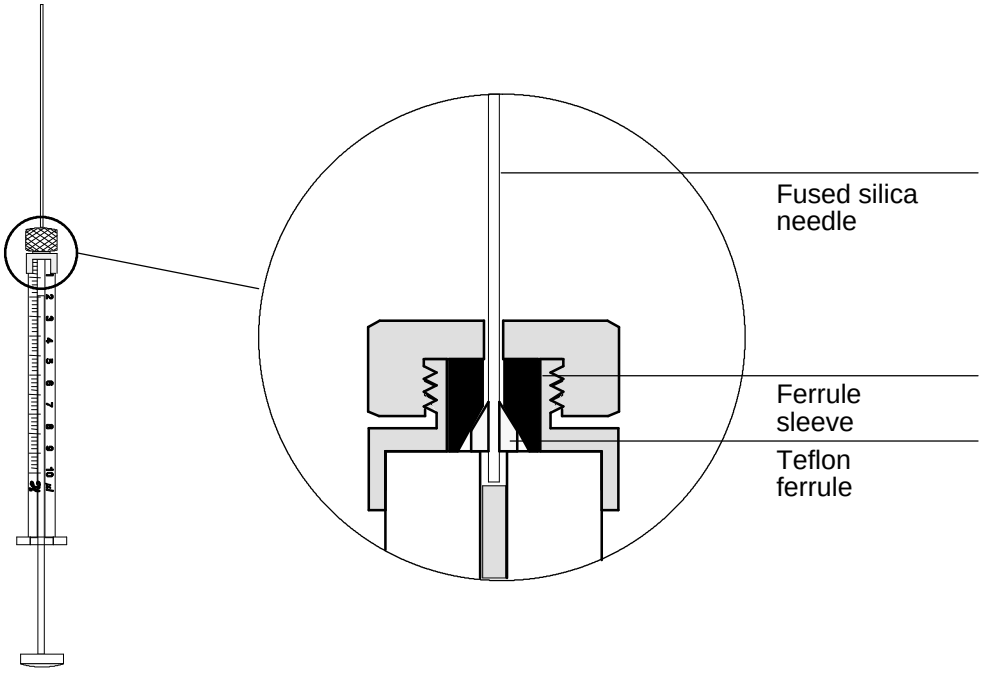
Procedure: Replacing the fused silica syringe needle

1. Hold the syringe vertically and insert the fused silica needle so it is visible inside the syringe barrel. If the fused silica needle cannot be inserted into the syringe barrel, the Teflon ferrule (HP part no. 0100-1389) may be blocked. You may need to replace the ferrule. Push the plunger down until it bottoms. The needle will now be flush with the end of the plunger.
2. When the needle is inserted, tighten the retaining nut to firm finger tightness. Pull the needle gently to be sure the Teflon ferrule has formed a tight seal with the needle. Tighten the retaining nut further, if necessary.
3. Loosen the retaining nut just enough so the needle is again free. Depress the syringe plunger slowly until it pushes the needle to the end of the barrel, then tighten the retaining nut to firm finger tightness.
4. Use a solvent to rinse the syringe and check for leaks or blocks.
5. Leaks (inability to eliminate air bubbles) may be fixed by further tightening the retaining nut. Blocks (or serious leaks) require repeating this procedure.

The Teflon ferrule may lose its seal in time. If so, first retighten the retaining nut and, if the seal still leaks, install a new Teflon ferrule and needle.

When not in use, loosen the retaining nut to avoid premature leaks.

Procedure: Installing a fused silica needle



If you are cutting replacement needles directly from fused silica column material:

1. Column material for making needles must have an outside diameter smaller than both the inside diameter of the on-column inlet (0.23 mm) and the inside diameter of the installed column.
2. Column material must be washed free of active stationary phase.
3. Score the column material about 1/4 inch from its end. Break off the end and discard. Then measure, score, and break off a 115 $\pm$ 5 mm length to use as the syringe needle.

Changing septa

If the septum leaks, you will see symptoms such as longer or shifting retention times, loss of response, and/or loss of column head pressure. Additionally, the detector signal will become increasingly noisy.

The useful lifetime of septa is determined by injection frequency and needle quality; burrs, sharp edges, rough surfaces, or a blunt end on the needle decrease septum lifetime. When the instrument is used regularly, daily septum replacement is recommended.

The type of septa you use will depend on your chromatography needs. You can order septa directly from Hewlett-Packard; see the Analytical Columns and Supplies Catalog for ordering information.

Caution The procedure for changing septa differs depending on whether you cool on-column inlet has a cooling tower assembly or a septum nut. Make sure to follow the correct procedure for your inlet!

Table 14. Recommended Septa for the Cool On-Column Inlet

Description	HP Part Number
Solid septum for manual and automatic injection	5181-1261
Duckbill septum for manual injection only (must use cooling tower with the duckbill)	19245-40050

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

Caution Column flow is interrupted while changing septa; since columns may be damaged at elevated temperatures without carrier flow, cool the oven to room temperature before proceeding.

Procedure: Changing septa

Materials needed:

- New septum—see Table 14 on page 103 for part numbers
- Forceps (or tweezers)
- A thin wire (0.2-in. diameter) for removing septum from inlet

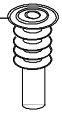
1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Cool the oven to room temperature and then turn the oven off.
   - Cool the inlet to room temperature and then turn the inlet off.

Depending on your analysis and injection technique, the inlet will have one of the following septum nuts or a cooling tower assembly.

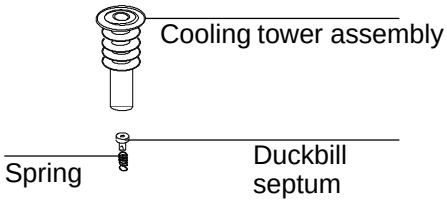
Septum nut for injections onto 250- and 320-μ columns

Septum nut for injections onto 530-μ columns

Cooling tower assembly (for manual injections only)

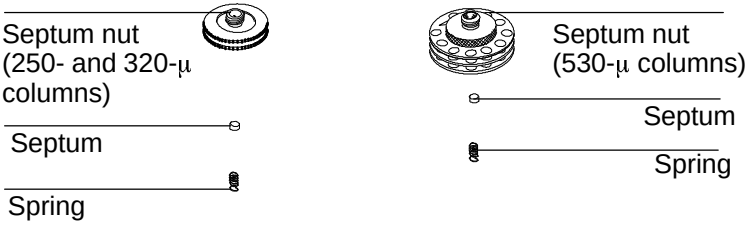


2. If you have a cooling tower assembly installed:
   Remove the assembly by grasping it and turning counterclockwise. The duckbill septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower. Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.



   Insert the duckbill septum into the spring and place them in the inlet. Reattach the cooling tower assembly. Tighten it finger-tight.

2. If you have a septum nut installed:
   Remove the septum nut by grasping the knurling and turning counter-clockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it. If the septum is not attached, you may need to use tweezers to grasp and remove it.



   Make sure the spring is in the inlet. Use the tweezers to place a new septum on the bottom of the septum nut, and then reattach the septum nut to the inlet. Tighten the nut firmly.

3. Restore normal GC operating conditions.

Procedure: Cleaning the inlet

Most laboratories have airborne lint and dust that accumulate on the cooling tower or septum nut and can enter the inlet or column on the syringe needle. Particulate matter in the inlet interferes with easy passage of the syringe needle. If dirt enters the column, it can alter the chromatography.

You can clean the needle guides, springs and inserts according to the following procedure.

WARNING Be careful! The oven and/or inlet may be hot enough to cause burns.

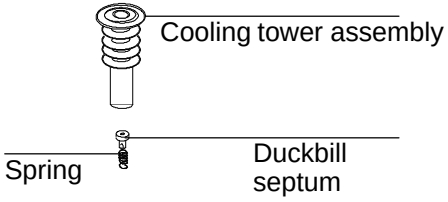
Materials needed:

- 9/16-in. wrench
- Narrow wire (0.02-in. diameter) or piece of capillary column (250-µ diameter) for removing spring and insert
- Small ultrasonic cleaning bath with aqueous detergent
- Distilled water
- Methanol
- Compressed, filtered, dry air or nitrogen

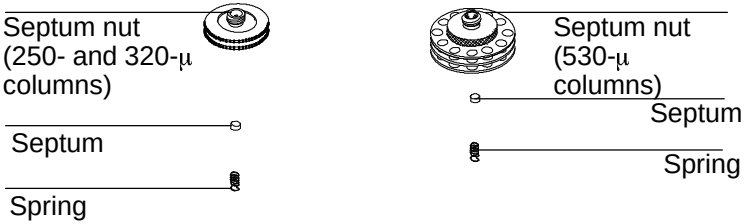
Cool on-column maintenance

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Allow the oven and inlet to cool.
   - Turn off all flows to the inlet at the initial gas supply.
   - Turn off the GC and unplug it.
   - Remove the column. See the "Columns and Traps" chapter in the General Information volume.

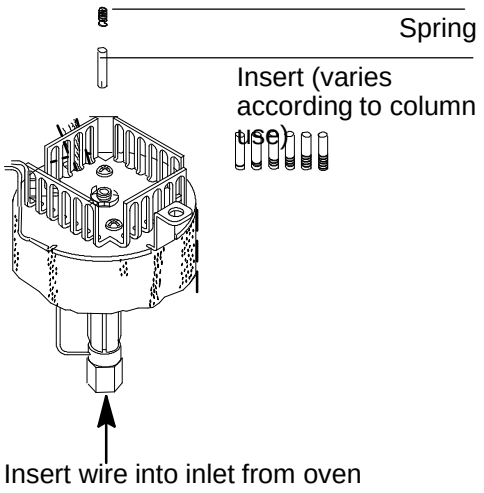
2. If you have a cooling tower assembly installed:
   Remove the assembly by grasping it and turning counterclockwise. The septum is underneath the cooling tower inside the spring. The spring and septum may pop out of the inlet when you remove the cooling tower. Be careful not to lose them. If they do not pop out, use a thin wire to remove them from the inlet.



2. If you have a septum nut installed:
   Remove the septum nut by grasping the knurling and turning counter-
   clockwise. The septum is probably attached to the septum nut. The spring may also pop out when you remove the septum nut. Be careful not to lose it.



3. Insert the narrow wire (or a piece of capillary column) into the inlet through the oven, and push the insert and spring (if they did not come out previously) out through the top of the inlet.



4. Cleaning procedure:
   a. Fill the ultrasonic cleaning bath with aqueous detergent, and place the spring and the insert into it. Sonicate for one minute.
   b. Drain the aqueous detergent, and fill the bath with distilled water. Sonicate for one minute.
   c. Remove the parts from the bath, and rinse them thoroughly with water and methanol.
   d. Dry the parts with a burst of compressed air or nitrogen.

5. Reinstall the insert. If you are using a septum nut, insert the spring and insert, with the spring on top.

6. Attach a new septum to the bottom of the septum nut. If you are using the cooling tower assembly, insert a new duckbill septum into the spring, and place them in the inlet.

7. Attach the septum nut or the cooling tower and tighten finger-tight. Reinstall the column, and then restore normal operation conditions.

Procedure: Leak testing the gas plumbing

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

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, turn the GC off and disconnect the main power cord. 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, remove excess fluid when you have completed the test.
- Two 7/16-in. wrenches

1. Using the leak detector, check each connection you have made for leaks.

2. Correct leaks by tightening the connections. Retest the connections; continue tightening until all connections are leak-free.

Procedure: Leak testing a cool on-column inlet

There are numerous places in the inlet that can leak. This procedure lets you determine, in general, if there is an unacceptable leak in the inlet. If the inlet is leaking, you should use an electronic leak detector to pinpoint the component that is leaking.

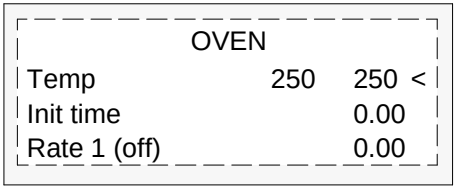
Materials needed:

- No-hole ferrule
- 1/4-in. wrench
- Gloves (if the inlet is hot)

1. Complete the following preliminary steps:
   - If you have entered parameters that you do not want to lose, store them as a method.
   - Allow the oven to cool to room temperature and then turn it off.
   - When the oven is cool, turn off the inlet pressure.
   - Remove the column, if one is installed, and plug the column fitting with the column nut with a no-hole ferrule installed.
   - Remove the old septum and replace it with a new one. For instructions, see page 104.

2. Set the oven temperature to its normal operating temperature.

Press [Oven]



3. Set inlet to normal operating temperature. Enter a pressure between 20 and 25 psi, or enter your normal operating pressure, if it is higher. Make sure that the pressure at the initial gas supply is at least 10 psi higher than the inlet pressure.

Press [Front Inlet] or [Back Inlet]

FRONT INLET (coc)		
Mode:	Track oven	
Temp	150	150 <
Pressure	24.0	24.0
Total flow		0.0

OR

FRONT INLET (coc)		
Mode:	Ramped temp	
Temp	150	150 <
Init time		0.00
Rate 1 (Off)		0.00
Pressure	24.0	On

4. Wait a few minutes for the GC to equilibrate after the system has reached the pressure. (The rest of the examples of the Front Inlet control table will show the table that appears if the inlet is in track mode.)

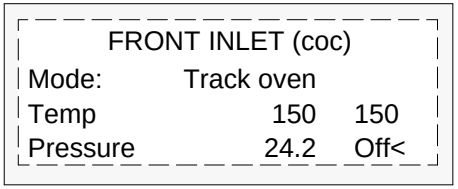
Press [Front Inlet] or [Back Inlet]

FRONT INLET (coc)		
Mode:	Track oven	
Temp	150	150
Pressure	24.2	24.0<

The GC may exceed the pressure setpoint slightly while equilibrating.

5. Turn the pressure off. Because the column is capped, the pressure should remain fairly constant.

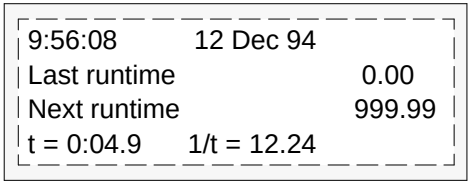
Press [Front Inlet] or [Back Inlet]



Because the pneumatics have been turned off, the alarm does not sound even though there is no flow through the column.

6. Continue to monitor pressure for 10 to 15 minutes. You can use the GC's Stopwatch function. A pressure drop of 0.03 psi/min or less is acceptable.

Press [Time]



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

If the pressure drops faster than the acceptable rate, go to the next section, "Correcting Leaks."

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- 1/4-in. wrench

1. Use the electronic leak detector to check all areas of the inlet that are potential sources of a leak. Potential leak areas are:
   - The plugged column connection
   - The septum nut, if present
   - The cooling tower assembly, if present
2. Correct leaks, using the wrench if necessary, 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 inlet system leak-free.

If the pressure drops faster than the acceptable rate, continue to search for leaks and repeat the pressure test. If all fittings appear to be leak free, but the inlet system is still losing too much pressure, you may need to replace the inlet manifold. Contact your Hewlett-Packard service representative.

5

The Programmable Temperature Vaporization Inlet

Chapter 5.
The Programmable Temperature Vaporization Inlet

Part 1. Introducing the HP PTV

Operating modes

The Hewlett-Packard Programmed Temperature Vaporization (PTV) Inlet System has five operating modes:

- The split mode is generally used for major component analyses.
- The pulsed split mode is like the split mode, but with a pressure pulse applied to the inlet during sample introduction to speed the transfer of material to the column.
- The splitless mode is used for trace analyses.
- The pulsed splitless mode allows for a pressure pulse during sample introduction.
- The solvent vent mode is used for large volume injection. Either single or multiple injections can be made for each run.

System requirements

The PTV inlet can be used with both manual and automatic injection.

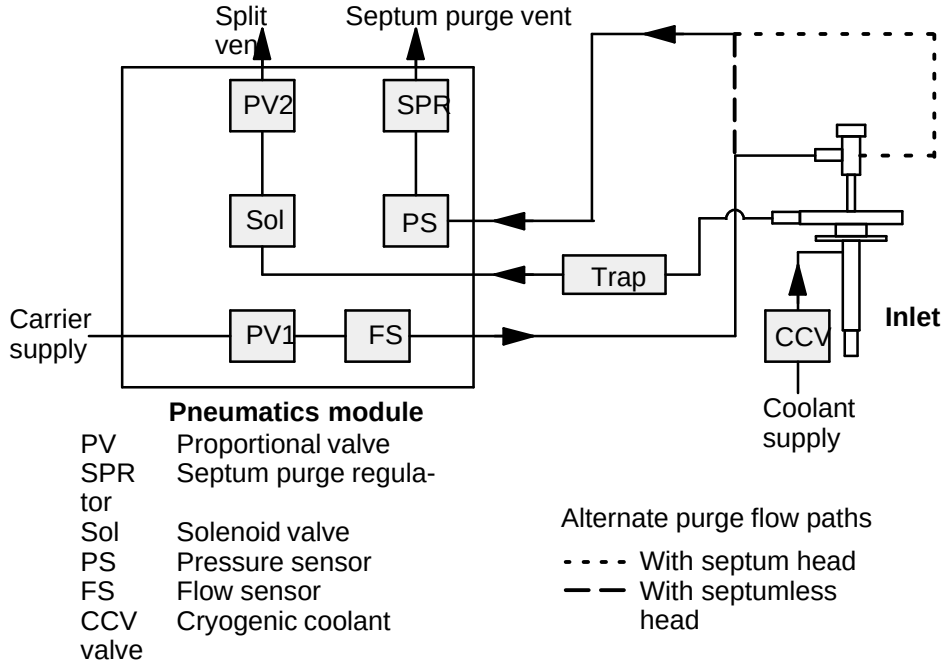
If an HP automatic sampler is used, it must be a model G1513A autosampler (firmware G1513A.09.14 or later) with a G1512A controller (firmware G1512A.01.08 or later)

For automatic multiple injections (large volume injections), an HP GC or MSD ChemStation is required. This function is not available under HP 6890 control alone. See part 4 of this chapter.

System components

1. The pneumatics module, located at the top rear of the GC.
2. The inlet body, always mounted in the front inlet position.
3. The trap, which is in the split line and placed to the left of the pneumatics carrier at the top rear of the chromatograph.
4. The coolant control valve. For liquid nitrogen, this valve is on the left outside wall of the oven. For liquid carbon dioxide, it is in the pneumatics carrier. These valves are not interchangeable—if you change coolants, you must also change all of the coolant plumbing including the valve and inlet jacket.
5. The thermocouple conversion board. It converts thermocouple readings from the inlet for use by the GC and is near the trap.

Figure 19. PTV system components



Sampling heads

Two heads are available for the PTV inlet.

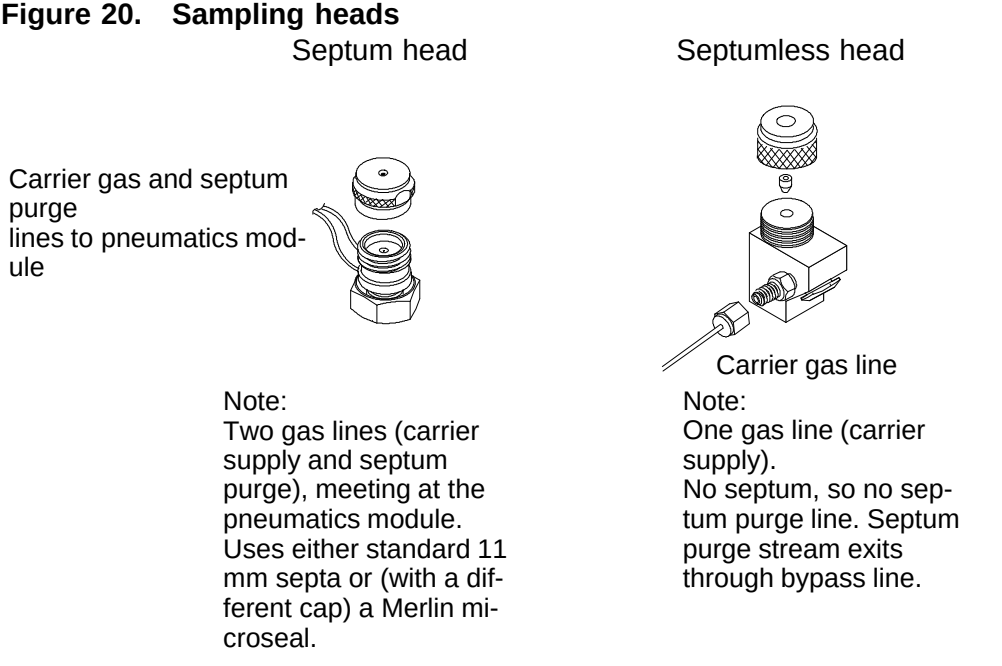
- The septum head uses either a regular septum or a Merlin microseal to seal the syringe passage. A stream of gas sweeps the inner side of the septum and exits through the septum purge vent on the pneumatics module. It may be used with either automatic or manual injection.

Caution

At inlet temperatures below 40C, the Merlin microseal may not seal effectively—use a regular septum instead.

- The septumless head uses a check valve instead of a septum to seal the syringe entrance passage. It may be used with either automatic or manual injection.

Figure 20. Sampling heads



The flow diagrams in the rest of this book show the septum head in place with a separate drawing for the septumless head plumbing.

Heating the inlet

The control parameters for PTV temperature programming are the same as for the column oven, but are reached by pressing [Front Inlet]. Temperature can be programmed with an initial temperature and up to 3 rates and plateaus. Rates between 0.1 and 720C/min can be selected. See chapter 4 of the General Information volume for details.

Caution

If the initial inlet temperature and the oven initial temperature are too close, the inlet may be unable to maintain its setpoint. We recommend a difference of at least 6C, either higher or lower.

Additional temperature ramps

For most purposes, the PTV is designed to hold the sample in the inlet liner until the entire sample—there could be several injections—has been injected. Then the PTV is heated rapidly to transfer the sample to the column. This can be accomplished with an initial hold, a single ramp, and a hold at the end to complete sample transfer.

Two additional ramps are available and have several possible uses:

- The inlet can be heated to a high temperature to thermally clean the liner for the next run.
- The inlet can be programmed downward—just set the Final temp below the previous temperature—to reduce thermal stress on the inlet.
- Downward programming can be used to prepare the inlet for the next run. This can reduce cycle time for greater sample throughput.

Cooling the inlet

The sample may be injected into either a cooled or heated chamber. The initial chamber temperature can be reduced to 60C (with CO_2 cooling) or to 160C (with liquid N_2 cooling).

Caution

If the initial inlet temperature and the oven initial temperature are too close, the inlet may be unable to maintain its setpoint. We recommend a difference of at least 6C, either higher or lower.

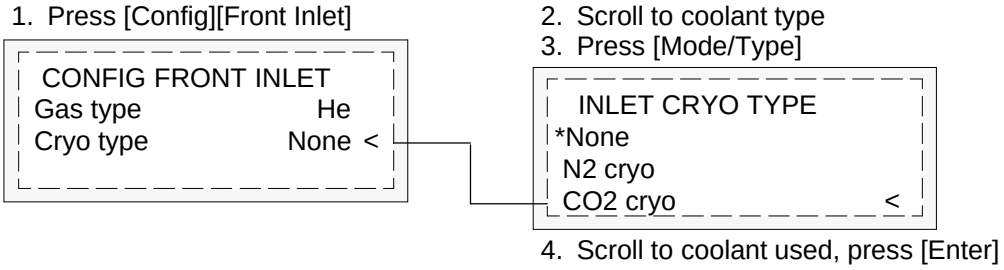
The HP 6890 GC supports only one type of coolant at a time.

Once a coolant is selected for any cryogenic device, that same coolant must be used for all such devices, including the column oven.

Since the GC can sense which coolant is used by the oven, if oven cooling is installed that coolant becomes the one that must be used by all other cooling devices.

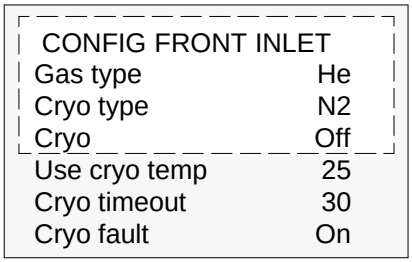
Configuring the PTV

To configure the PTV, press [Config] [Front Inlet]. If the inlet has not been configured previously, this screen is displayed.



If oven cooling is installed, your choices are restricted to the coolant used by the oven or None. If oven cooling is not installed, you must specify the coolant using the procedure in the figure.

If the Cryo type selection is anything other than None, several other parameters appear.



Cryo [ON] enables cryogenic cooling of the inlet as soon as the column oven reaches its initial temperature. [OFF] disables cooling.

Use cryo temp If Cryo is ON, this is the upper limit of temperatures at which cryo cooling is used to hold the inlet at its setpoint. If the setpoint is higher than this limit, cryogenic cooling is used to bring the inlet down to its setpoint but is not used to hold it at the setpoint.

Cryo timeout Cryo timeout occurs, and the inlet temperature shuts down, when a run does not start within a specified time (range 5 to 120 minutes, default 30 minutes) after the oven equilibrates. Turning cryo timeout off disables this feature. We recommend that it be turned on because cryo timeout conserves coolant at the end of a sequence or if automation fails. A Post Sequence method could also be used.

Cryo fault Shuts down the inlet temperature if it does not reach setpoint in 16 minutes of continuous cryo operation. Note that this is the time to reach the setpoint, not the time to stabilize and become ready at the setpoint.

Shutdown behavior

Both Cryo timeout and Cryo fault can cause cryo shutdown. If this happens, the inlet heater is turned off and the cryo valve closes. The GC beeps and displays this message:

```
SHUTDOWN (#18):
Front inlet cryo
shutdown
```

The inlet heater is monitored to avoid overheating. If the heater remains on at full power for more than 2 minutes, the heater is shut down. The GC beeps and displays this message:

```
SHUTDOWN (#22):
Front inlet heating
too slowly;
temperature shut off
```

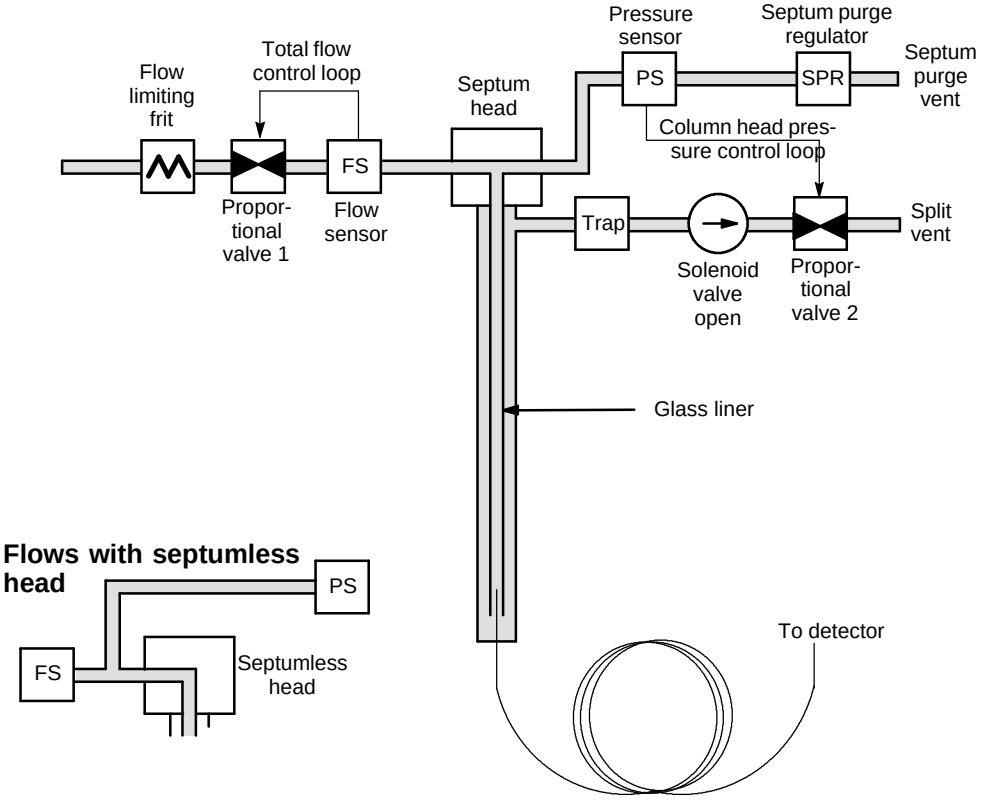
To recover from either condition, turn the GC off, then on, or enter a new setpoint.

Part 2. Using the Split Modes

Flow pattern

The two split modes—with or without a pressure pulse—divide the gas stream entering the inlet between the column flow, the split vent flow through the solenoid valve, and the septum purge flow. The ratio of the split vent flow to the column flow is called the split ratio.

The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).



Temperature considerations

Cold split introduction

For cold split sample introduction, use an initial inlet temperature below the normal boiling point of the solvent. If the liner volume is enough to hold all the vaporized solvent, start the first inlet temperature ramp at 0.1 minutes with a high heating rate (500C/min or higher). The final temperature should be high enough to volatilize the heaviest analytes from the liner and should be held for at least 5 minutes. A final temperature of 350C for 5 minutes has proven sufficient to quantitatively transfer C_{44}.

For larger injection volumes or to eliminate the solvent, hold the initial temperature long enough to vent the solvent through the Split vent and then begin the first ramp. Use a fast rate for thermally stable analytes. Slower rates may help minimize thermal degradation in the inlet.

A single temperature ramp is enough for the injection process. The remaining ramps may be used to clean the liner or to reduce the inlet temperature in preparation for the next injection.

Hot split introduction

For hot split introduction, set an initial temperature high enough to volatilize the analytes. No additional thermal parameters are required as the inlet will maintain the setpoint throughout the run.

Because of the small liner volume (about 120 microliters), the PTV has a limited injection capacity with hot split introduction. Injection volumes exceeding 1 µL in the hot split mode may overflow the inlet causing analytical problems. Cold split introduction avoids this potential problem.

Control table parameters—split mode operation

Mode: The current operating mode—split

Temp Actual and setpoint inlet initial temperatures.

Init time Hold time at the inlet initial temperature.

Rate # Temperature program rate for inlet thermal ramps 1, 2, and 3.

Final temp # Final inlet temperature for ramps 1, 2, and 3.

Final time # Hold time at Final temp 1, 2, and 3.

Pressure Actual and setpoint inlet 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 line does not appear if your column is not defined.

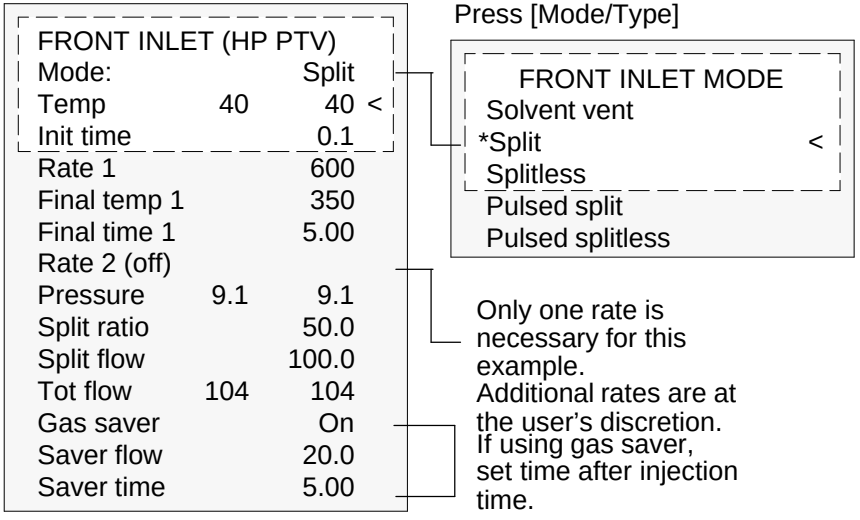
Split flow Flow, in mL/min, from the split/purge vent. This line does not appear if your column is not defined.

Total flow These are the actual and setpoint values of the total flow into the inlet, which is the sum of the split flow, column flow, and septum purge flow. When you change the total flow, the split ratio and split flow change while the column flow and pressure remain the same.

Procedure: Using split mode with the column defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet]
   a. Scroll to Mode: and press [Mode/Type]. Select Split.
   b. Set the inlet temperature and any desired ramps.
   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 displayed for you.
   e. If desired, turn on Gas saver. Set the Saver time after the injection time.

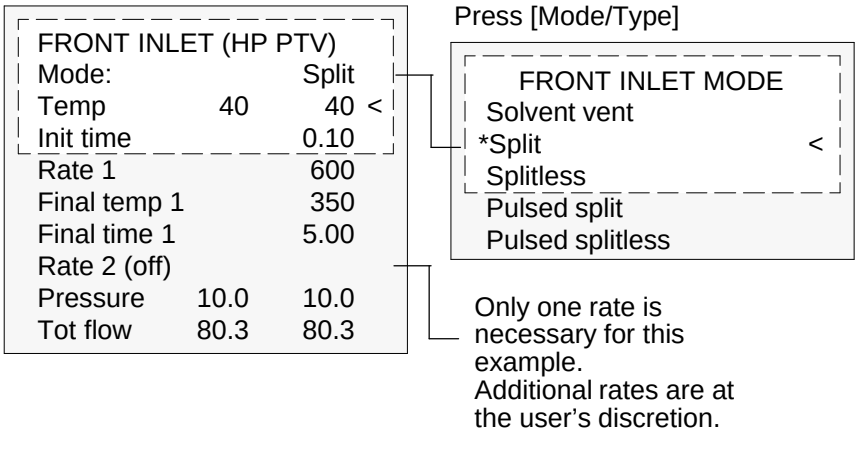
Split ratio = Split flow / Column flow

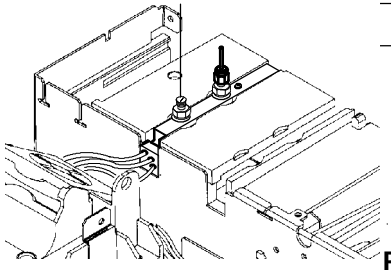


3. Press [Prep Run] before manually injecting the sample if the Gas Saver is on (see page 13).

Procedure: Using split mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Set temperature.
   b. Set total flow into the inlet. Measure flows out of the split vent and septum purge vent using a flow meter.
   c. Subtract the septum purge flow from Total flow to get split flow.
   d. Calculate the split ratio. Adjust as needed.





Front of instrument

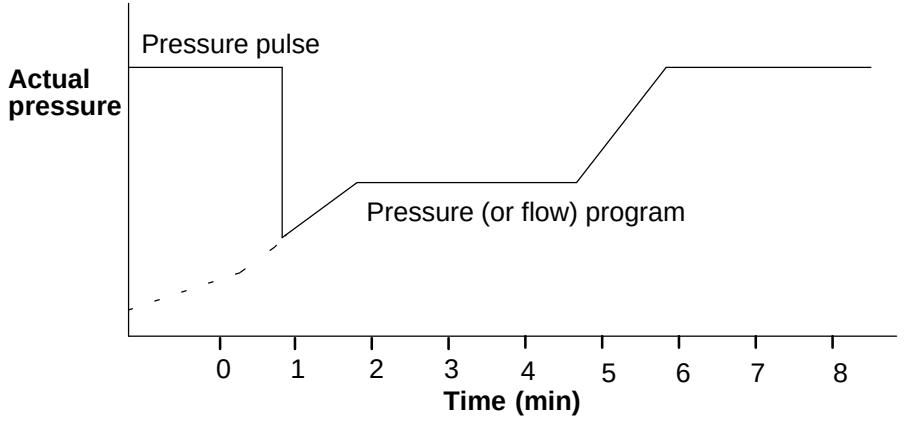
Pulsed modes

The pressure pulse modes (split and splitless) increase inlet pressure just before the beginning of a run and return it to the normal value after a specified amount of time. The pressure pulse sweeps the sample out of the inlet and into the column faster, reducing the chance for sample decomposition in the inlet. If your chromatography is degraded by the pressure pulse, a retention gap may help restore peak shape.

You must press the [Prep Run] key before doing manual injections in the pressure pulse mode.

You can do column pressure and flow programming when in the pressure pulse mode. However, the pressure pulse will take precedence over the column pressure or flow ramp.

Figure 21. Pressure Pulse and Column Flow or Pressure



Control table parameters—pulsed split mode

Mode: The current operating mode—pulsed split

Temp Actual and setpoint inlet temperatures

Init time Hold time at the initial inlet temperature.

Rate # Temperature program rate for inlet thermal ramps 1, 2, and 3.

Final temp # Final inlet temperature for ramps 1, 2, and 3.

Final time # Hold time at Final temp 1, 2, and 3.

Pressure Actual and setpoint inlet pressure before and after the pressure pulse. This is the starting point of a pressure program or the fixed pressure if a program is not used.

Pulsed pres The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until Pulse time elapses, when it returns to Pressure.

Pulse time Inlet pressure returns to its normal setpoint at this time after Start Run.

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

Split flow Flow, in mL/min from the split/purge vent. This line does not appear if your column is not defined.

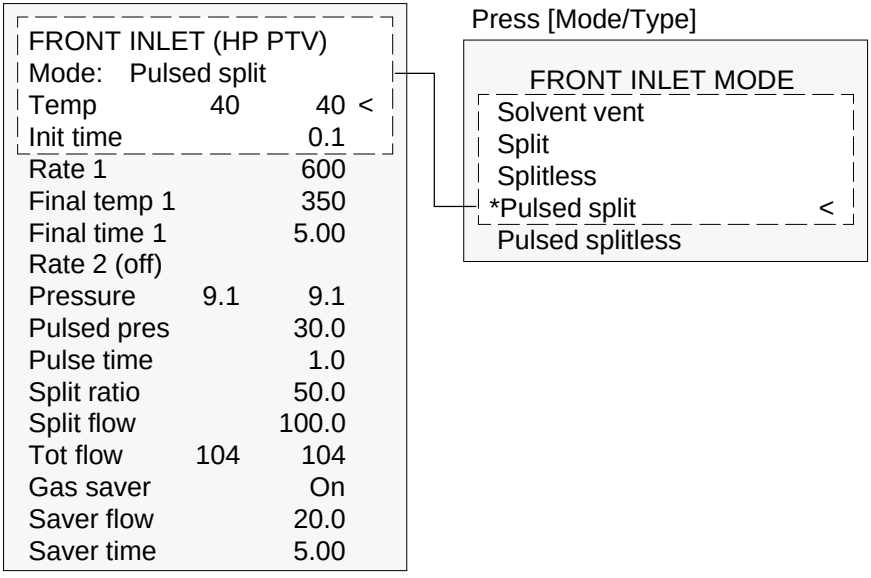
Total flow The total flow into the inlet, the sum of the split flow, column flow, and septum purge flow. When you change total flow, the split ratio and split flow change while column flow and pressure remain the same. When a pressure pulse is used, total flow increases to keep the split ratio constant.

Procedure: Using pulsed split mode with the column defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Split.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for Pulsed Pres and Pulse time.

Split ratio = Split flow / Column flow

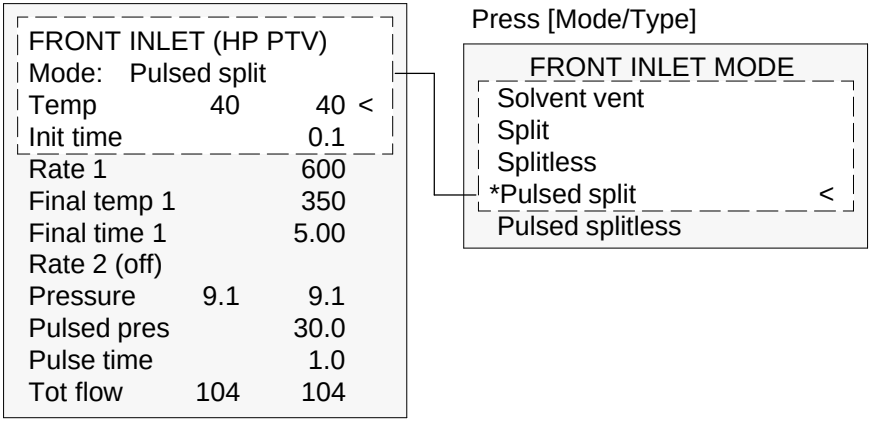
   d. If you want a specific split ratio, scroll to Split ratio and enter that number. The split flow is calculated and set for you.
   e. If you want a specific split flow, scroll to Split flow and enter that number. The split ratio is calculated and displayed for you.
   f. Turn Gas saver on, if desired. Set the time greater than Pulse time.



3. Press [Prep Run] (see page 13) before injecting a sample manually.

Procedure: Using pulsed split mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Split.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for Pulsed Pres and Pulse time.
   d. Set total flow into the inlet. Measure flows out of the split vent and septum purge vent using a flow meter.
   e. Subtract the septum purge flow from Total flow.
   f. Calculate the split ratio. Adjust as needed.



Part 3. Using the Splitless Modes

Flow patterns

In these modes—with or without a pressure pulse—the solenoid valve is closed during injection and vaporization of the sample and stays so while the sample transfers to the column. At a specified time after injection, the valve opens to sweep vapors left in the liner out the split vent. This avoids solvent tailing due to the large inlet volume and small column flow rate.

The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).

Figure 22. Stage 1. Sample injection

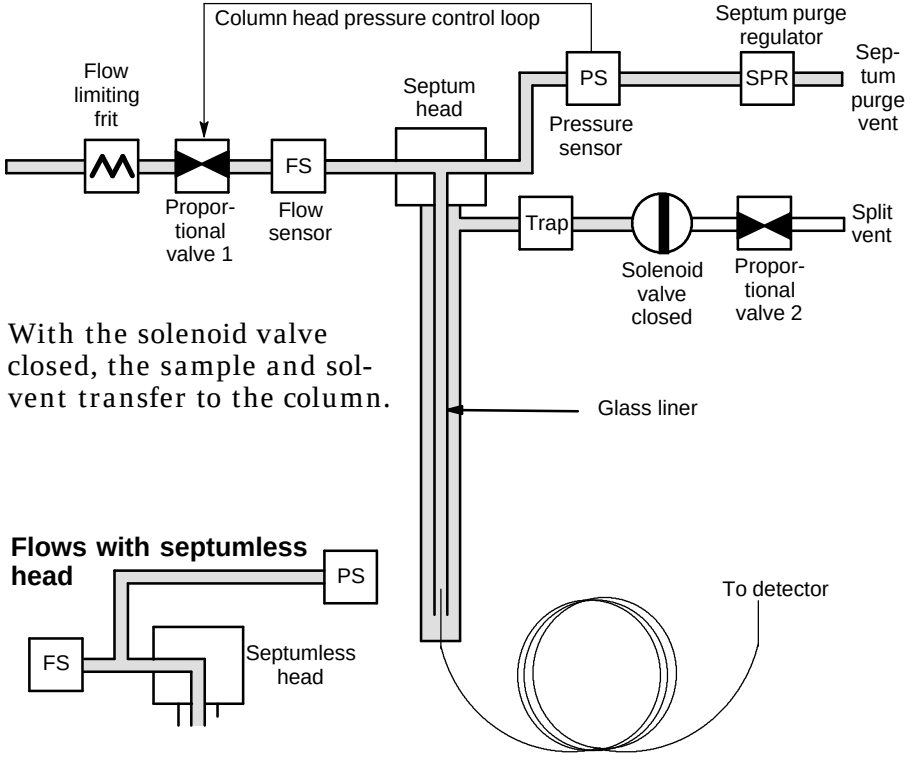


Figure 23. Stage 2. Purging

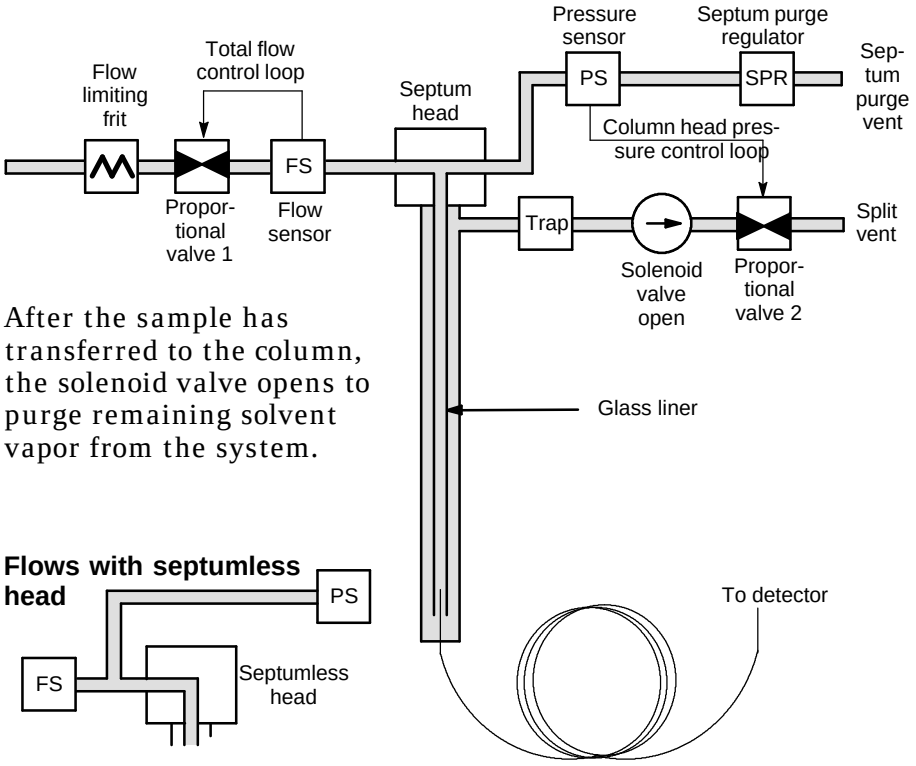
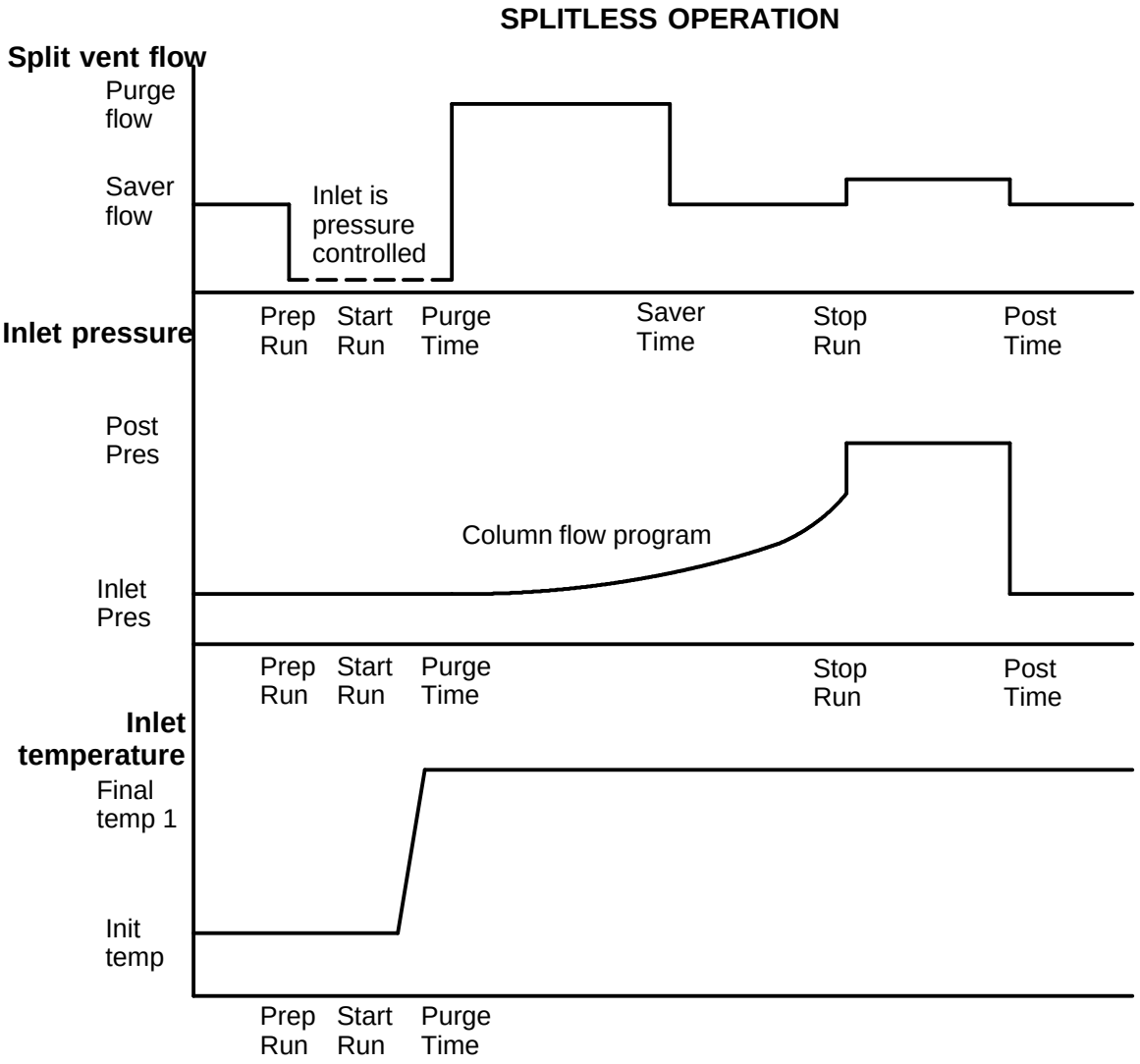


Figure 24. Flows, pressures, and temperatures



Temperature considerations

Cold splitless introduction

For cold splitless introduction, use an initial inlet temperature below the normal boiling point of the solvent. For most solvents, starting the first inlet temperature ramp at 0.1 minutes provides good transfer and reproducibility. A program rate of 500C/min or higher is appropriate for thermally stable analytes. A final temperature of 350C, held for 5 minutes, has quantitatively transferred up to C_{44} alkane.

A main advantage of temperature programmability is that the inlet can be heated gently to transfer delicate analytes. If the oven temperature is initially low enough to refocus the analytes on the column, the inlet heating rate can be made slower (eg, 120C/min). This reduces thermal degradation from the inlet and can improve peak shape and quantitation.

For most applications of cold splitless, a single temperature ramp is enough. The remaining ramps can be used to clean the liner or to decrease the inlet temperature to prepare for the next injection.

Hot splitless introduction

For hot splitless introduction, select an initial temperature high enough to volatilize the analytes. No additional temperature parameters are required as the inlet will maintain the setpoint throughout the run.

Because of the small liner volume (about 120 µL), the PTV cannot contain vapor resulting from large liquid injection volumes. Injection volumes greater than 1 µL may overflow vapor from the inlet, causing analysis variations. Cold splitless introduction avoids this problem.

Control table parameters—splitless operation

Mode: The current operating mode—splitless

Temp Actual and setpoint inlet temperatures

Init time Hold time at the initial inlet temperature.

Rate # Temperature program rate for inlet thermal ramps 1, 2, and 3.

Final temp # Final inlet temperature for ramps 1, 2, and 3.

Final time # Hold time at Final temp 1, 2, and 3.

Pressure Actual and setpoint inlet pressure in psi, bar, or kPa

Purge time The time, after the beginning of the run, when you want the purge valve to open.

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

Total flow The Total flow line displays the actual flow to the inlet during a Pre-run (Pre-run light is on and not blinking) and during a run before purge time. You cannot enter a setpoint at these times. At all other times, Total flow will have both setpoint and actual values.

Starting values

A successful splitless injection consists of these steps:

1. Inject the sample and temperature program the inlet to vaporize it.
2. Use a low column flow and low oven temperature to create a solvent-saturated zone at the head of the column.
3. Use this zone to trap and reconcentrate the sample at the head of the column.
4. Wait until all, or at least most, of the sample has transferred to the column. Then discard the remaining vapor in the inlet—which is mostly solvent—by opening a purge valve. This eliminates the long solvent tail that this vapor would otherwise cause.
5. Raise the oven temperature to analyze the sample.

Some experimentation is needed to refine the operating conditions. Table 16 provides starting values for the critical parameters.

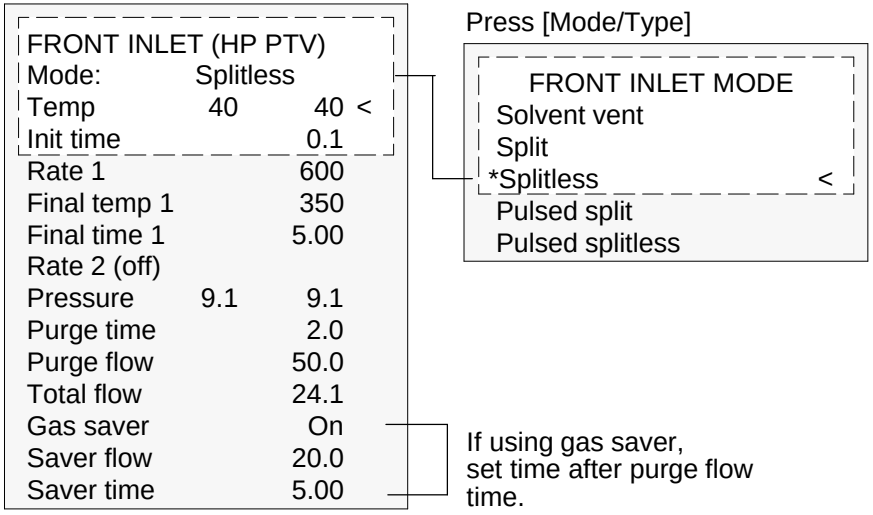
Table 16. Splitless Mode Inlet Parameters

Parameter	Allowed Setpoint Range	Suggested Starting Value
Oven temperature	No cryo, ambient+10°C to 450°C CO_2 cryo, −60°C to 450°C N_2 cryo, −80°C to 450°C	10°C below solvent boiling point
Oven initial time	0 to 999.9 minutes	≥ Inlet purge time
Inlet purge time	0 to 999.9 minutes	$\frac{\text{Liner volume}^*}{\text{Column flow}} \times 5$
Gas saver time	0 to 999.9 minutes	After purge time
Gas saver flow	15 to 1000 mL/min	15 mL/min greater than maximum column flow
Inlet temperature	No cryo, oven temp + 10°C CO_2 cryo, −50°C to 450°C N_2 cryo, −160°C to 450°C	10°C below solvent boiling point for 0.1 min, then ramp up

* Liner volume is about 120 µL

Procedure: Using splitless mode with the column defined

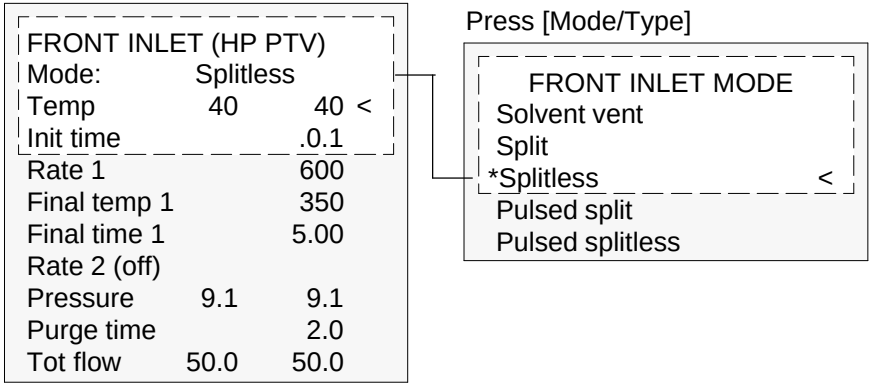
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Splitless.
   b. Set the inlet temperature and any desired ramps.
   c. Enter a purge time and a purge flow.
   d. If desired, turn Gas saver on. Make certain the time is set after the purge flow time.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

Procedure: Using splitless mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Splitless.
   b. Set the inlet temperature and any desired ramps.
   c. Enter a purge time.
   d. Set your total flow greater than the column flow plus the septum purge flow (about 3 to 6 mL/min) to guarantee adequate column flow.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

Pulsed splitless mode operation

See page 128 for a discussion of the pulsed pressure modes.

Control table parameters—pulsed splitless operation

Mode: The current operating mode—pulsed splitless

Temp Actual and setpoint inlet temperatures

Init time Hold time at the initial inlet temperature.

Rate # Temperature program rate for inlet thermal ramps 1, 2, and 3.

Final temp # Final inlet temperature for ramps 1, 2, and 3.

Final time # Hold time at Final temp 1, 2, and 3.

Pressure Actual and setpoint inlet pressure before and after the pressure pulse. It sets the starting point of a pressure program or the fixed pressure if a program is not used.

Pulsed pres The inlet pressure you desire at the beginning of a run. The pressure rises to this setpoint after [Prep Run] is pressed and remains constant until Pulse time elapses, when it returns to Pressure.

Pulse time Pressure returns to its normal setpoint at this time.

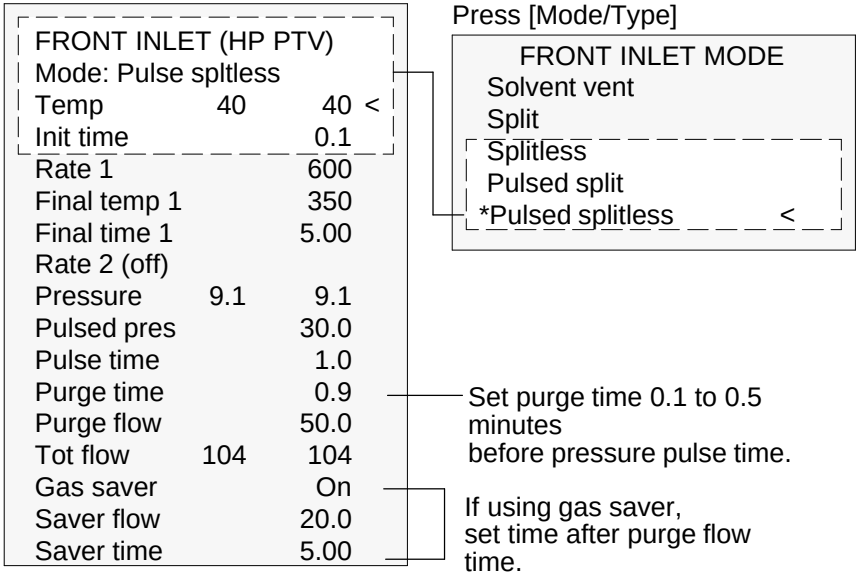
Purge time The time, after the beginning of the run, that you wish the purge valve to open. Set purge time 0.1 to 0.5 minutes before pulse time.

Purge flow The flow, in mL/min, from the purge vent, at Purge time. The column must be defined.

Total flow This is the total flow into the inlet, representing a total of the column flow and the septum purge flow.

Procedure: Using pulsed splitless mode with the column defined

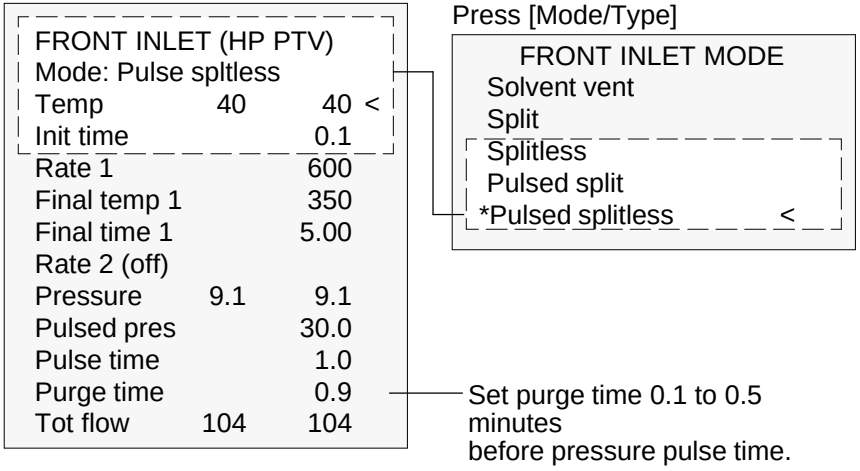
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Splitless.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for Pulsed pres and Pulse time.
   d. Enter the Purge time when you wish the purge valve to open.
   e. Enter a Purge flow.
   f. Turn Gas saver on, if desired. Set the time after the purge flow time.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

Procedure: Using pulsed splitless mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Splitless.
   b. Set the inlet temperature and any desired ramps.
   c. Enter values for Pulsed Pres and Pulse time.
   d. Enter the Purge time when you wish the purge valve to open.
   e. Enter a Purge flow.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

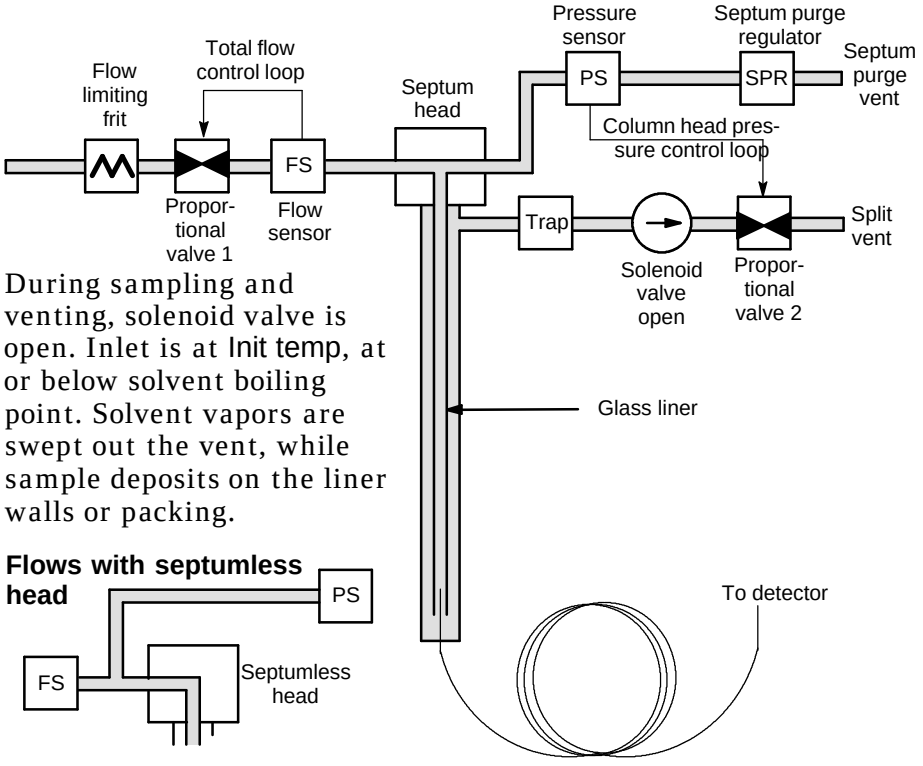
Part 4. Using the Solvent Vent Mode

Flow patterns

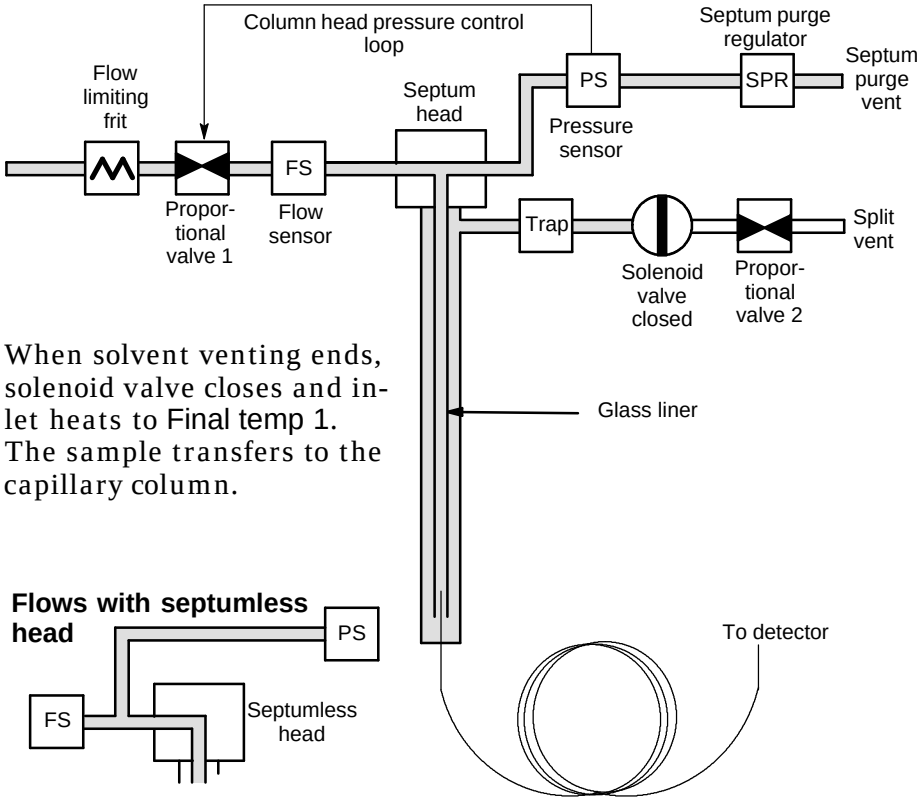
The sample is injected into a cold inlet. If conditions are properly chosen and the sample is suitable, analytes deposit in the inlet liner while the solvent evaporates and is swept out. Large or multiple injections can be used to concentrate sample in the inlet before transferring to the column for analysis.

The main figure shows the flows with the septum head. Flows with the septumless head are the same except that the septum purge flow bypasses the head (lower left).

Figure 25. Stage 1. Sample and vent



Stage 2. Sample transfer



Stage 3. Purge and cleanup

The solenoid valve opens again and the system returns to the Stage 1 configuration but with different setpoints. The PTV inlet is flushed. Additional ramp rates are available to thermally clean the inlet or to reduce inlet temperature after sample transfer. This can extend the life of the liner.

Temperature, pressure, and flow considerations

The solvent vent mode goes through three distinct pneumatic states; venting, sample transfer, and purging. The vent portion allows the inlet pressure and the vent flow to to be adjusted to optimize solvent elimination. The transfer state mimics traditional splitless operation and transports the analytes from the liner to the column. The purging mode allows the user to prepare the inlet for the next run.

A fundamental difficulty with solvent vent mode is the potential loss of volatile analytes with the solvent. Several solutions are possible for this situation:

- The inlet liner can be packed with a more retentive material, such as Tenax. This greatly improves volatile analyte recovery but may impact recovery of higher boiling materials.
- Some of the solvent can be left in the liner when sample transfer begins. The residual solvent acts like a stationary phase and retains volatile material, but at the expense of a larger solvent peak.
- The inlet temperature can be reduced. This reduces the vapor pressure of the volatile analytes and permits higher recoveries.

Solvent removal can be speeded up by:

- Reducing pressure in the inlet during sample introduction—the Vent pressure parameter
- Increasing flow through the inlet—the Vent flow parameter

While all these possibilities do complicate use of the PTV, they provide increased flexibility and new potential to solve difficult problems.

Sequence of operations

These are the steps in a typical analysis using the solvent vent mode.

Step		Parameter	Value
1.	**Before injection**	Flow at split vent	— Either Purge flow or Saver flow
		Inlet pressure	— Derived from column setpoint
	The system is resting, with Purge flow (or Saver flow, if on) through the inlet.		
2.	**Prep Run begins**	Flow at split vent	— Vent flow setpoint
		Inlet pressure	— Vent pressure setpoint
	Setpoints change to prepare for injection. When GC is ready, the sample is injected. Inlet and oven temperature program Init times begin. Solvent venting and analyte trapping begin.		
3.	**At Vent end time**	Flow at split vent	— None, solenoid valve closed
		Inlet pressure	— Column pressure setpoint
	Solvent venting ends, analyte transfer begins as inlet heats up.		
4.	**At Purge time**	Flow at split vent	— Purge flow setpoint
		Inlet pressure	— Column pressure setpoint
	Analyte transfer ends, inlet is purged of residual vapor. Analysis begins.		
5.	**At Saver time**	Flow at split vent	— Saver flow setpoint
		Inlet pressure	— Column pressure setpoint
	Analysis ends, gas flow reduced to save gas (if Saver is on).		

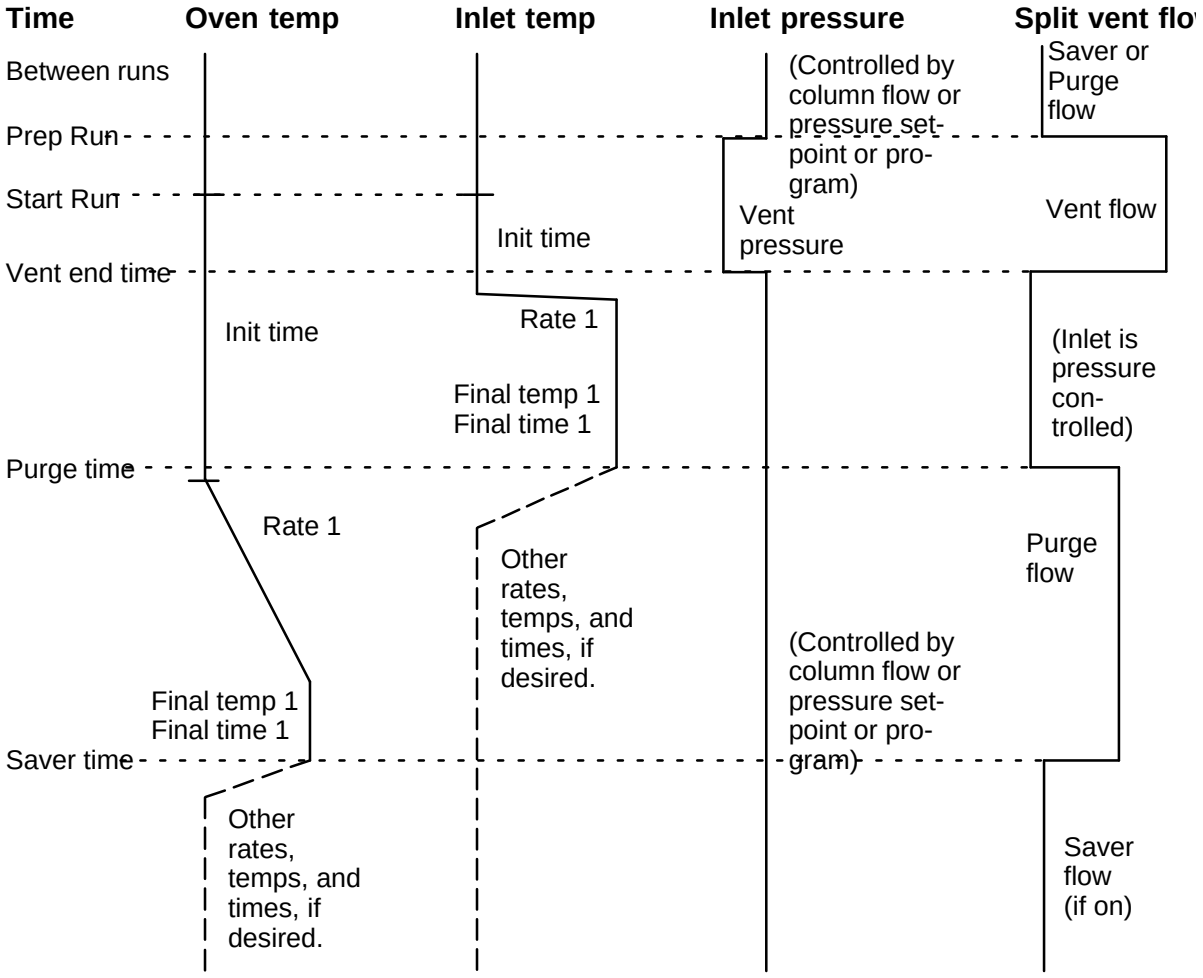
Some important points

- The flow through the column is governed by the pressure in the inlet. This is controlled, during the analysis part of the process, by the flow or pressure setpoint or program entered for the column.
- The controlling times must be in the order shown; Vent end time before Purge time before Saver time.
- Vent end time must occur before the inlet starts to heat and release analytes.
- Purge time must occur before the oven begins to heat and move sample through the column.

Timelines

Time increases downward; all other quantities increase to the right.

Figure 26. Time Relationships



When is Start Run?

Both the inlet and oven temperature programs begin at Start Run. All times—such as Purge time—are measured from Start Run. When does Start Run occur?

- If the sample is injected manually, Start Run occurs when the user presses the Start Run key.
- If a single injection per run is made using an autosampler, Start Run occurs when the syringe carrier moves down to make the injection.
- If multiple injections per run are made using an autosampler, Start Run occurs when the syringe carrier moves down to make the first injection of the set. There are no Start Run signals for the rest of the injections in the set.

  These additional injections take time. The inlet and oven temperature programs, mainly the Init time values, must be adjusted to allow for this. So must the various time values that control the inlet operation. This is discussed in more detail under Large Volume injections, later in this chapter.

Control table parameters—solvent vent operation

Mode: The current operating mode—solvent vent

Temp Actual and setpoint initial inlet temperatures

Init time The time, measured from Start Run, when the initial inlet temperature hold ends. Usually greater than Vent end time.

Rate # Temperature program rate for inlet thermal ramps 1, 2, and 3.

Final temp # Final inlet temperature for ramps 1, 2, and 3.

Final time # Hold time at Final temp 1, 2, and 3. This time is a duration; it is not measured from Start Run.

Pressure Actual and setpoint inlet pressure before and after the vent period. It sets the starting point of column head pressure.

Vent pressure The inlet pressure during the vent period. By decreasing the inlet pressure while venting, solvent elimination proceeds faster. Also, the pressure reduction decreases the amount of carrier gas—and solvent vapor—that enters the column during this time.

Users select from 0 to 100 psig. If 0 is chosen, the inlet uses the lowest pressure possible at the given vent flow. Table 17 shows approximate values for this minimum at various vent flows of helium. Pressures less than those in the table are not possible unless the flow is reduced.

Table 17. Minimum attainable pressures

Vent flow (mL/min)	Actual vent pressure at “0“ psig setpoint	Actual vent pressure at “0” kPa setpoint
50	0.7	5
100	1.3	10
200	2.6	18
500	6.4	44
1000	12.7	88

Vent flow The flow of carrier gas out the split vent during the vent period. Higher flows sweep the liner more quickly and reduce the time for solvent elimination. For most columns, 100 mL/min vent flow eliminates solvent at an acceptable rate but puts minimal material on the column.

Vent end time The time, measured from Start Run, when solvent venting ends. For large volume injections, this time is normally greater than the time for the injection to complete.

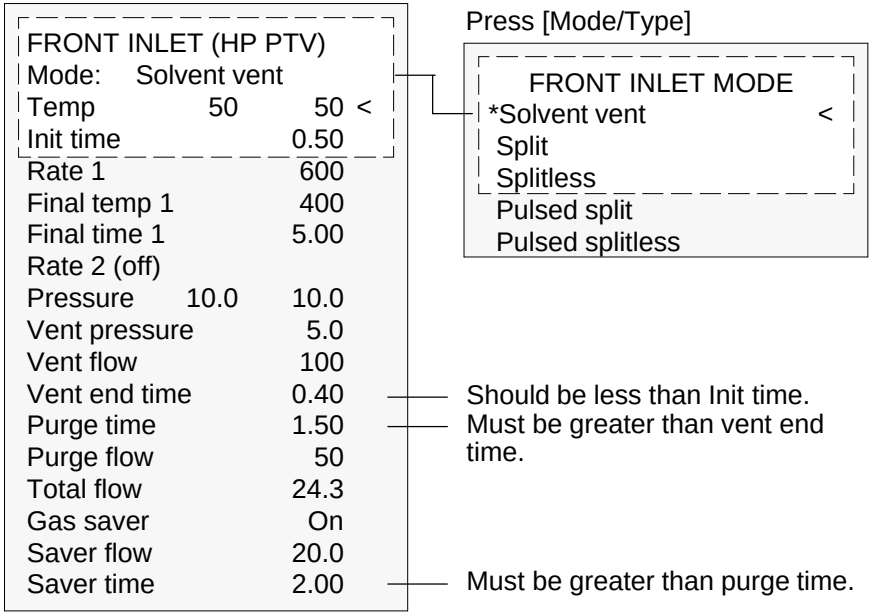
Purge time The time, measured from Start Run, when sample transfer ends. It began at Vent end time.

Purge flow The flow of carrier gas to the inlet beginning at Purge time.

Total flow The Total flow displays the actual flow to the inlet.

Procedure: Using solvent vent mode with the column defined

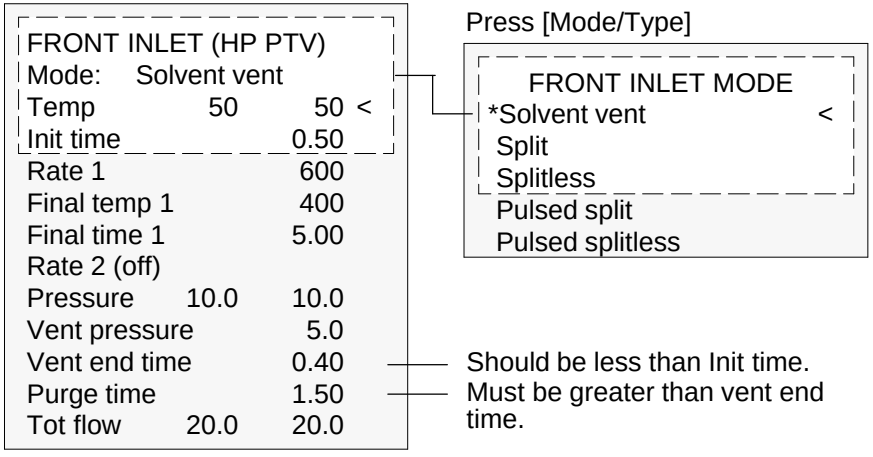
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Solvent vent.
   b. Enter a vent pressure, a vent flow, and a vent end time.
   c. Set the inlet temperature and ramps, as desired.
   d. Enter a purge time and a purge flow.
   e. If desired, turn Gas saver on. Make certain the time is set after the purge time.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

Procedure: Using solvent vent mode with the column not defined

1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet].
   a. Scroll to Mode: and press [Mode/Type]. Select Solvent vent.
   b. Enter a vent end time and a vent pressure.
   c. Set the inlet temperature and ramps, as desired.
   d. Enter a purge time. It must be greater than the vent end time.
   e. Set total flow greater than the column flow plus the septum purge flow (about 6 mL/min) to guarantee adequate column flow.



3. Press [Prep Run] (see page 13) before manually injecting a sample.

Large volume injection

Most vaporizing inlets are designed for liquid injections in the 1 to 5 µL range. With larger injections, the vapor cloud created when the sample vaporizes may overflow the inlet and degrade the chromatography. For the PTV, the nominal liner liquid capacities are:

Table 18. Liner capacities

Liner	Nominal liquid capacity	Inertness
Open baffle	5 µL	High
Glass wool packed	25 µL	Lower, because of greater surface area

In the solvent vent mode, analytes are thermally trapped in the liner while the solvent is removed. With the solvent gone, the liner volume can be used for another injection. Injection can be repeated several times to concentrate the analytes from a large sample volume. After injection and solvent removal, the analytes are transferred to the column. This can replace the need for offline concentrating and minimize loss of sample.

Multiple injections by an automatic sampler can be used with the PTV to achieve large volume injection. A ChemStation controls the process.

Gas chromatograph requirements

- Model 6890 GC with A.2.00 or later firmware
- HP PTV

Automatic sampler requirements

- Model G1512A controller with G1512A.01.08 or later firmware.
- Model G1513A injector with G1513A.09.14 or later firmware. A second injector can be mounted for synchronous injections to the rear inlet, but only one PTV can be mounted and it must be in the front position.

Caution Use of the 18593A or 18593B injection tower may damage the system.

- Operates with or without sample tray, and with either standard or 8-sample turret.

ChemStation requirements

A GC or MSD ChemStation is necessary for multiple injection because the needed parameters are not available through the HP 6890 GC keyboard.

- GC ChemStation Software revision A.04.02 or later
  or Software revision A.04.01 plus the software provided with the PTV.
- MSD ChemStation Software revision A.03.00 or later

Control parameters—Injector configuration subscreen

Parameter	Range	Default
Syringe size	0.1 to 100 µL	10 µL
Nanoliter Adapter	Present or not present	Not present
Multiple Injections	Single or Multiple	Single

- Syringe size Full volume of the syringe.
- Nanoliter Adapter Controlled by a checkbox. If checked, indicates that a nanoliter adapter is present on the injector. If not checked, means that a nanoliter adapter is not present on the injector.
- Multiple Injections Controlled by a checkbox.If checked, the sampler makes multiple injections into the inlet for each run according to the other parameters. It issues a Start Run command at the first injection only.

  If not checked, the sampler makes one injection—and issues a Start Run command—for each run. This is the default mode of operation.

Control parameters—Injector screen

Parameter	Range	Default
Inject **X** µL **Y** times	**X**: 0.1 to 0.5 x syringe volume **Y**: 1 to 100	**X**: 0.1 x syringe volume **Y**: 1
Delay between injections	0 to 100 seconds	0
Preinjection washes	0 to 15	0
Postinjection washes	0 to 15	0
Pumps	0 to 15	0

- Inject **X** µL **Y** times X is the amount to be injected; Y is the number of injections to make. If the nanoliter adapter is checked on the Injector Configuration screen, the range becomes 0.02 to 0.4 x syringe volume.
- Delay A pause time, in seconds, between injections. This is added to the minimum hardware cycle time.
- Preinjection washes Number of times to wash the syringe with solvent and/or sample before the first injection. No washes are performed before the rest of the injections in a multiple injection set.
- Postinjection washes Number of times to wash the syringe with solvent after the last injection. No washes are performed after the rest of the injections in a multiple injection set.
- Preinjection pumps Number of times to pump the syringe plunger before drawing up the measured sample. Pump are performed only before the first injection of a multiple injection set.

Calculated values

The software calculates and displays:

- On the Injector screen: Total Product of X (Volume per injection) and Y (Injections per run).
- On the Inlets screen: Estimated total injection time The approximate total time, in minutes, to make a set of multiple injections based on the parameters entered and the mechanical cycle time of the sampler. Includes Delay between injections, pre- and post-injection dwell times, and viscosity delays.

An example

These values were used for a sample with a broad range of boiling points.

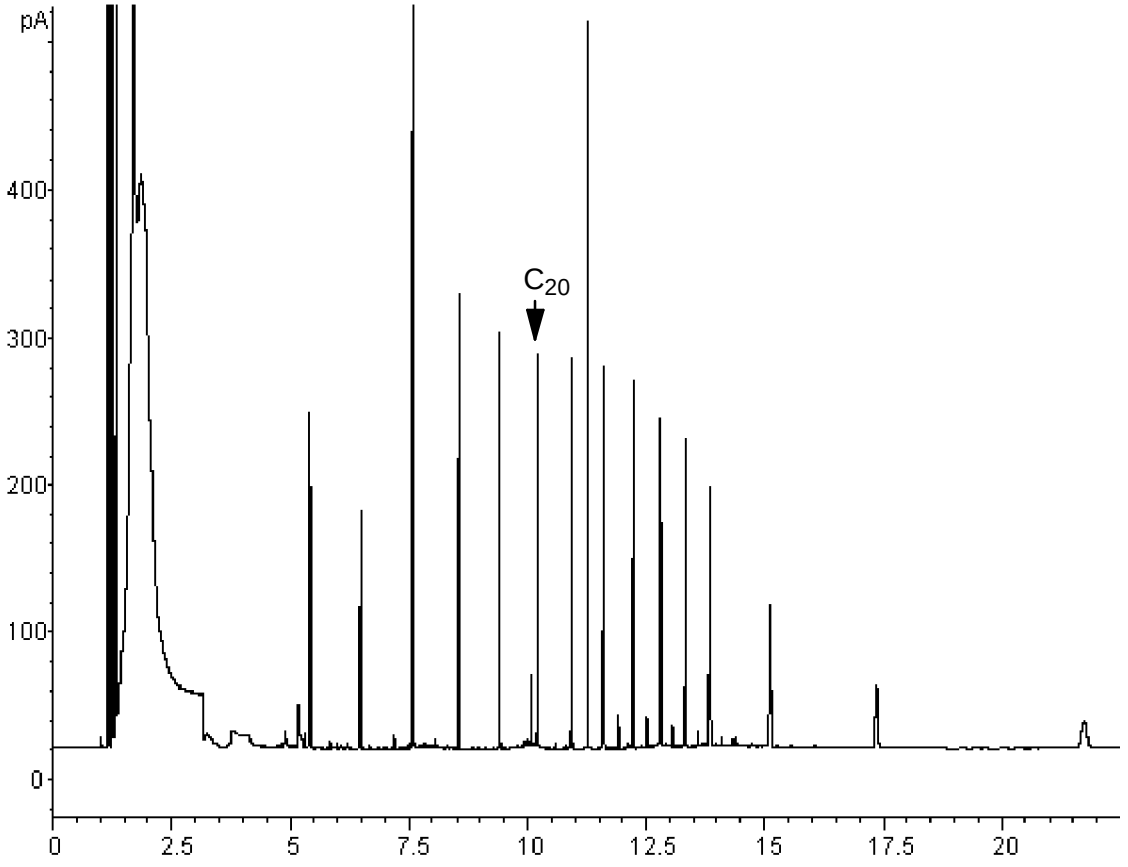
General parameters	
Name	**Value**
Sample	C_{10} to C_{44} hydrocarbons in hexane
Mode	Solvent vent
PTV liner	Glass wool packed
Injection volume	One 10.0 µL injection (25 µL syringe)
Injection speed	Fast
Column	30 m x 320 µm x 0.25 µm HP-5, HP p/n 19091J-413
Column flow	4 mL/min constant flow

Inlet parameters			
Name	**Value**	**Name**	**Value**
Init temp	40°C	Rate 2 (off)	
Init time	0.3 min	Pressure	15.6 psig
Rate 1	720°C/min	Vent pressure	0.0 psig
Final temp 1	450°C	Vent flow	100 mL/min
Final time 1	5 min	Vent end time	0.2 min
Rate 2	100°C/min	Purge time	2.0 min
Final temp 2	250°C	Purge flow	50 mL/min
Final time 2	0 min		

Oven parameters	
Name	**Value**
Init temp	40°C
Init time	2.5 min
Rate 1	25°C/min
Final temp 1	320°C
Final time 1	10.0 min
Rate 2 (off)	

Detector parameters	
Name	**Value**
Detector	FID
Detector temp	400°C
Hydrogen flow	40 mL/min
Air flow	450 mL/min
Makeup (N_2)	45 mL/min

Figure 27. Chromatogram From One 10 µL Injection



These results were compared with a splitless analysis of the same sample, which should produce 100% recovery of all analytes. The data showed that, under these conditions, compounds above C_{20} were completely recovered and that the recovery was independent of injection size; Compounds lower than C_{20} were partially vented with the solvent.

Possible adjustments

Depending on what you are trying to accomplish, you have a number of possible adjustments available.

- To eliminate more solvent
  - Increase the vent end time, inlet initial time, and purge time. This will not affect analytes that are quantitatively trapped but will eliminate more of the solvent peak.
  - Increase the vent flow to sweep the liner more rapidly with the same inlet timing. Increasing vent flow raises vent pressure if it is set to 0. This puts more solvent onto the column.
  - Raise the inlet initial temperature to vaporize more solvent and allow more to be eliminated. This also increases the loss of volatile analytes since their vapor pressures also increase.
- To improve recovery of low boiling analytes
  - Reduce inlet temperature to lower the vapor pressure of the analytes and trap them more effectively. This also reduces solvent vapor pressure and more time will be needed to eliminate it.
  - Use a retentive packing in the liner. Materials such as Tenax permit higher recovery of volatile analytes but may not release higher boiling compounds. This must be considered if quantitation on these high boiling peaks is desired.
  - Leave more solvent in the liner. The solvent acts as a pseudo stationary phase and helps retain volatile analytes. This must be balanced against the detector's tolerance for solvent.

An example—continued

The single injection example shown on the last few pages makes it clear that a 10 µL injection does not overload the glass wool packed liner. This means that multiple 10 µL injections are possible.

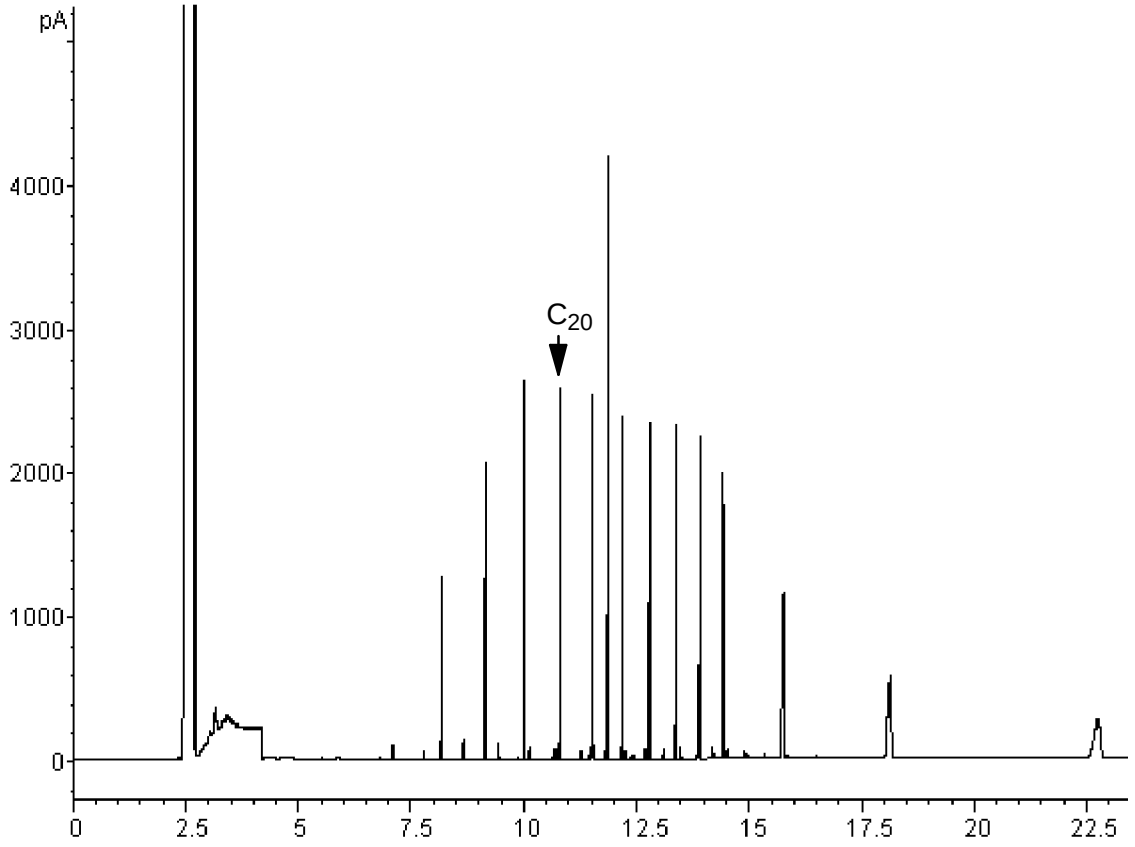
It was decided to make 10 injections per run, each of 10 µL size. This would increase analytical sensitivity substantially. No adjustments were made to improve recovery of the low boilers since the purpose of this analysis was to detect and measure the high boiling components.

The ChemStation estimated that 10 injections would require a total of 1.3 minutes. The following timing changes were made:

Parameter	increased from	to
Inlet Init time	0.3 minutes	1.6 minutes
Vent end time	0.2 minutes	1.5 minutes
Purge time	2.0 minutes	3.0 minutes
Oven Init time	2.5 minutes	3.0 minutes

The result is shown on the next page.

Figure 28. Chromatogram From Ten 10 µL Injections



Part 5. Maintaining a PTV

Inlet adapters

The Graphpack-2M connector (the inlet adapter) at the bottom of the inlet is sized to the column diameter. When a different diameter column is to be installed, the adapter must be changed.

The adapter number is stamped on the side of the adapters. Select the smallest hole diameter that will accept the column.

Table 19. Inlet adapters

Column ID	Inlet adapter number	Quanti-ty	HP part num-ber
200 µm	31	1	5182-9754
250 µm	45	1	5182-9761
320 µm	45	1	5182-9761
530 µm	70	1	5182-9762

Procedure: Replacing inlet adapters

1. Unscrew the column nut from the adapter. Remove the nut and the column from the inlet.
2. With a 6 mm wrench, remove the inlet adapter, being careful not to lose the silver seal inside. Save the adapter for later use.
3. Select the appropriate inlet adapter for the column to be installed. Insert a new silver seal (part number 51829763, pkg of 5) into the adapter and screw the adapter onto the inlet fingertight. Use the 6 mm wrench to tighten the adapter an additional $^{1}/_{16}$ to $^{1}/_{8}$ turn.

   Do not overtighten the adapter. The inlet can be damaged if the adapter is forced. If the adapter leaks, check the silver seal and replace it if necessary.

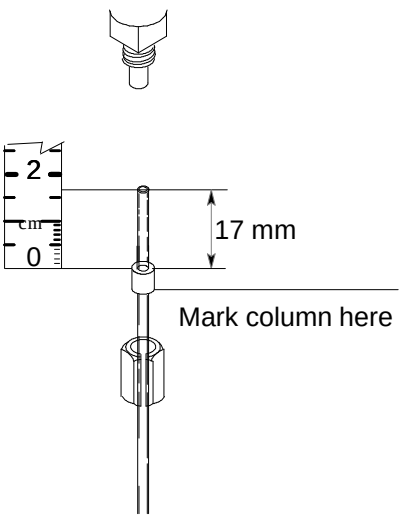
Procedure: Installing columns

Graphpack-2M ferrules are sized to the column outer diameter.

Table 20. Columns and ferrules

Column ID	Graphpack ferrule hole ID	Quantity	HP part number
200 µm	0.31 mm	10	5182-9756
250 µm	0.40 mm	10	5182-9768
320 µm	0.45 mm	10	5182-9769
530 µm	0.70 mm	10	5182-9770

1. Place the appropriate Graphpack ferrule onto the column inlet end and pull it at least 30 mm from the end.
2. With a glass knife or other fused silica cutter, remove approximately 10 mm from the column end to eliminate graphite contamination.
3. Position the ferrule so that it is 17 mm from the column end. Place a small mark (typewriter correction fluid is useful) at the back of the ferrule and, making sure that the column is correctly positioned, insert the column end into the adapter.



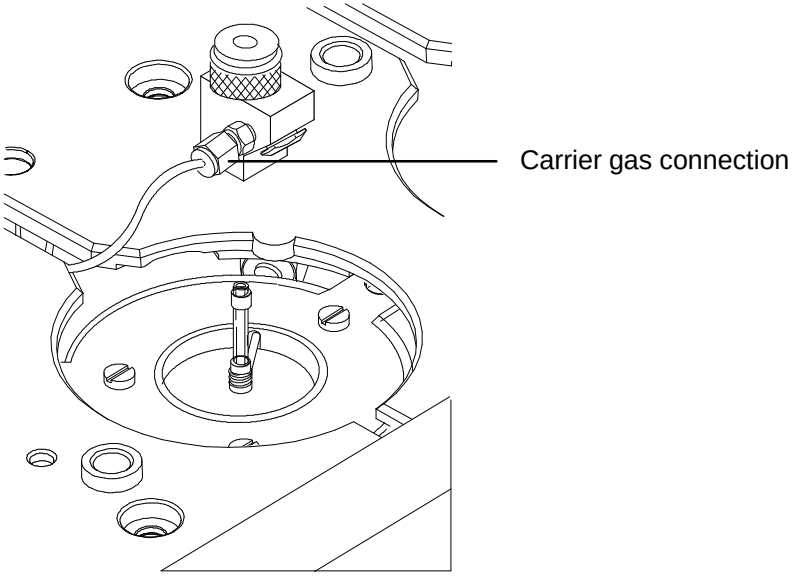
4. Screw the column nut on fingertight. Using a 5 mm wrench, tighten the column nut $^{1}/_{8}$ to $^{1}/_{4}$ turn. Be careful not to overtighten.
5. Check the connections for leaks. If there are any leaks at the column adapter, tighten it slightly more with the open end wrench provided.

The septumless head

This sampling head uses a check valve instead of a septum to seal the syringe entrance passage. It may be used with either automatic or manual injection. Syringes must have 23 gauge needles (see the last page of this chapter).

Procedure: Removing the septumless head

1. Cool the inlet to room temperature.
2. Disconnect the carrier gas line.
3. Unscrew the septumless head counterclockwise from the inlet.
4. Screw the new head onto the inlet. Tighten it $^{1}/_{8}$ turn past finger tight.

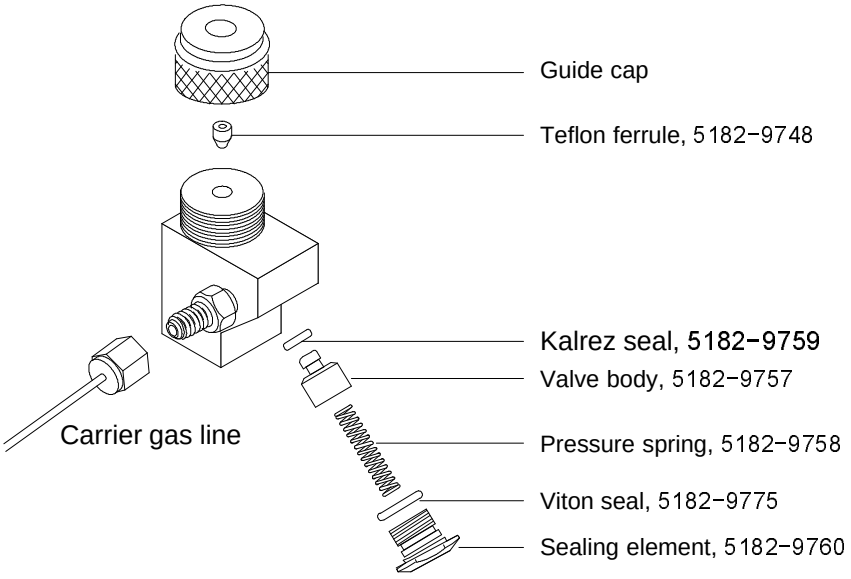


5. Reconnect the carrier gas line.
6. Check all connections on the sampling head for leaks. If necessary, tighten them again by hand.

Procedure: Cleaning the septumless head

Minor deposits from sample mixtures can collect in the head. Dust and abraded material particles can enter together with the syringe needle, eventually causing leaks. We recommend periodic cleaning.

1. Cool the inlet to room temperature.
2. Disconnect the carrier gas line and unscrew the head from the inlet.
3. Unscrew the sealing element from the head. Carefully remove the Viton seal and the pressure spring. Do not use a sharp object to extract the valve body—this can leave scratches that cause leaks.



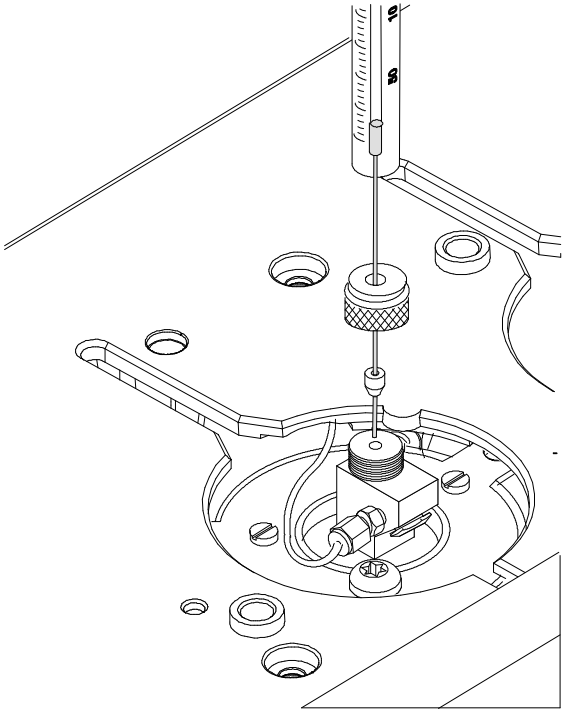
4. Unscrew the guide cap from the head and remove the Teflon ferrule.
5. Insert a syringe with a 23 gauge needle carefully into the head to press the valve body with the Kalrez seal slightly out of the head. Carefully tap the head on a soft smooth surface so that the valve body falls out completely or slips so far out that you can grasp it with your fingers.

6. Remove the seal from the valve body.
7. Carefully clean all components in hexane.
8. Assemble the head in reverse order. Make sure that you work absolutely lint-free and that the seals and the pressure spring are not damaged.
9. Use this opportunity to check the Teflon ferrule. If it must be replaced, see page 167 for instructions.
10. Check the entire system again for leaks; if necessary, carefully retighten the guide cap slightly more with the syringe needle inserted and/or replace the Kalrez seal.

    If the head leaks when a syringe is inserted, the Teflon ferrule is the problem. If the head leaks without a syringe inserted, the seals may need to be replaced.

Procedure: Replacing the Teflon ferrule

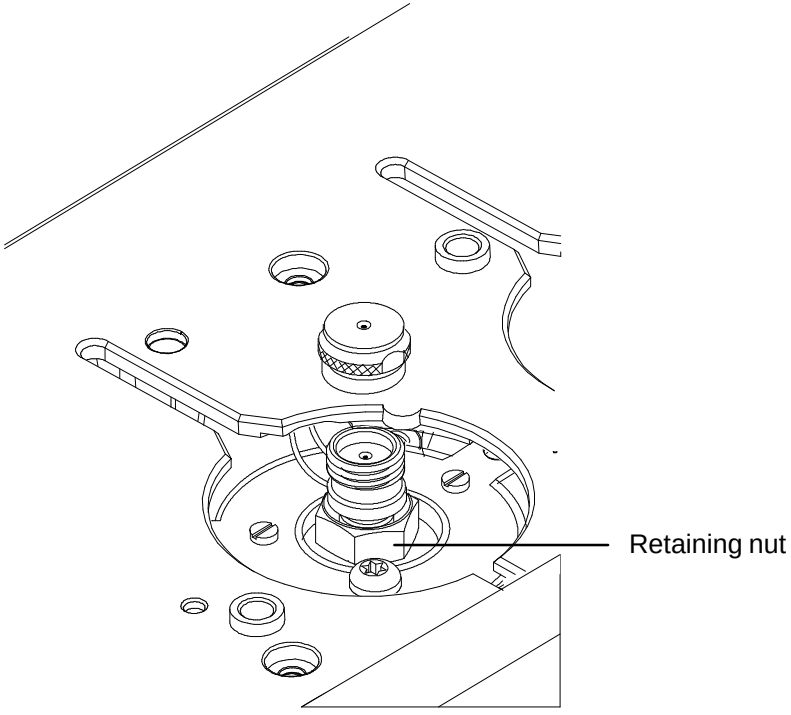
1. Unscrew the guide cap from the septumless head and remove the Teflon ferrule.



2. Push the guide cap and the new Teflon ferrule over the syringe needle so that at least 10 mm of the needle tip is exposed.
3. Guide the end of the syringe needle into the septumless head until the ferrule meets the septumless head.
4. Tighten the guide cap until resistance is first felt.
5. Check for leaks when the syringe needle has been fully introduced.
6. If necessary, carefully tighten the guide cap until the inlet stops leaking.

The septum head

The septum head uses either a regular septum or a Merlin microseal to seal the syringe passage. A stream of gas sweeps the inner side of the septum and exits through the septum purge vent on the pneumatics module.



Procedure: Removing the septum head

The septum head connects to the inlet via a free-spinning retaining nut.

1. Cool the inlet to room temperature.
2. Use a $^{5}/_{8}$ in. wrench to loosen the retaining nut on the septum head.
3. Gently remove the septum head assembly from the inlet. Be careful not to overly bend the $^{1}/_{16}$ inch lines. For best results, lift the head to clear the inlet and then push it to either side to allow access.
4. To reinstall the septum head, gently align the head with the inlet and manually engage the free-spinning nut to the inlet.

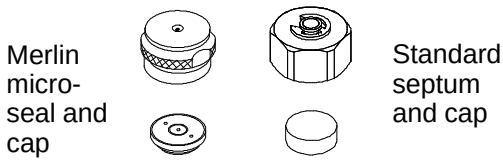
   The nut should easily turn on to the inlet. If resistance is felt, unscrew the nut and retry. Excessive force can irreparably damage the inlet.
5. Tighten the retaining nut ½ turn past finger tight.
6. Check all connections for leaks. If necessary, the retaining nut can be tightened an additional ¼ of a turn to eliminate leaks.

Procedure: Changing the septum

Either a regular septum or a Merlin microseal can be used with the septum head.

If the inlet temperature is set below 40C, the Merlin microseal may not seal effectively. For inlet temperatures below 40C, use a regular septum for the inlet seal.

1. To replace the septum, cool the inlet to ambient temperature.
2. Using the inlet tool or manually, unscrew the septum cap or Merlin cap counterclockwise. If the septum head begins to turn, support it manually while removing the cap.
3. Remove the septum or Merlin microseal, taking care not to scratch the interior of the septum head.
4. Install a new septum or Merlin microseal and the correct cap. When installing a Merlin microseal, note that the side where the metal parts are visible goes down.



5. Check for leaks out of the cap and tighten the cap if necessary.

Glass inlet liners

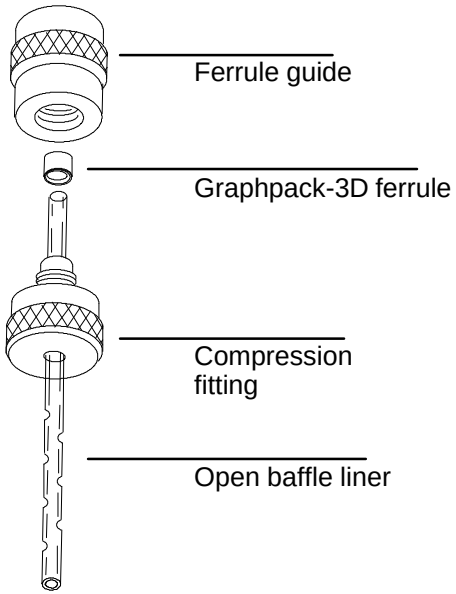
The liner is the chamber for sample deposition. Three kinds are available:

Table 21. Inlet liners

Type	Injection capacity	Inertness	Quantity	HP part number
Open baffled liner	lowest capacity	most inert	10	5182-9751
Liner packed with silanized glass wool	higher capacity	less inert	10	5182-9752
Unpacked liner, to be packed by the user	depends on the packing		10	5182-9753

Procedure: Replacing liners

1. Remove the head from the inlet See page 164 (septumless head) or 169 (septum head).
2. Grasp the liner by the Graphpack ferrule. Remove the liner and ferrule.
3. Unscrew the assembly tool (HP part number G261780540) into two pieces, the ferrule guide and the compression fitting.



4. Slide the compression fitting onto the longer straight end of the new liner with the threads pointing toward the end of the liner.
5. Place a Graphpack-3D ferrule on the same end of the the liner with the recessed graphite end towards the compression fitting. Slide the ferrule on so that about 2 mm of liner is exposed beyond the ferrule.
6. Slide the compression fitting up to meet the ferrule. Screw the ferrule guide gently onto the compression fitting until it is fingertight.

7. Unscrew and remove the ferrule guide. Slide the compression fitting off the other end of the liner. The ferrule should now be set with 1 mm of liner exposed. Check that the graphite within the ferrule is flush with the top of the metal collar.
8. Insert the glass liner into the inlet from above until the unpacked side of the ferrule rests on the top of the inlet.
9. Replace the sampling head and reconnect the lines, if necessary.
10. Check all connections for leaks. If necessary, tighten them again by hand.

Consumables and replaceable parts

Description	Quantity	HP part number
Septumless head assembly	1	G2617-60507
Service kit	1	5182-9747
Valve body	1	5182-9757
Pressure spring	1	5182-9758
Kalrez seal	1	5182-9759
Teflon guide	1	5182-9748
Sealing element	1	5182-9760
Graphpack-3D ferrule for liners	5	5182-9749
Assembly tool for Graphpack-3D ferrules	1	G2617-80540
Liner packed with silanized glass wool	10	5182-9752
Liner, open baffled	10	5182-9751
Liner, empty, for user packing	10	5182-9753
Graphpack-2M inlet adapter, 0.2 mm column id	1	5182-9754
Graphpack-2M inlet adapter, 0.32/0.25 mm column id	1	5182-9761
Graphpack-2M inlet adapter, 0.53 mm column id	1	5182-9762
Silver seal for Graphpack-2M inlet adapter	5	5182-9763
Nut for Graphpack inlet adapters	5	5062-3525
Ferrules for Graphpack-2M inlet adapter, 0.2 mm column id	10	5182-9756
Ferrules for Graphpack-2M inlet adapter, 0.25 mm column id	10	5182-9768
Ferrules for Graphpack-2M inlet adapter, 0.32 mm column id	10	5182-9769
Ferrules for Graphpack-2M inlet adapter, 0.53 mm column id	10	5182-9770

more>

Description	Quanti-ty	HP part num-ber
Syringes		
5 µL, 23 gauge fixed needle	1	9301-0892
10 µL, 23 gauge fixed needle	1	9301-0713
10 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5181-8809
10 µL, Teflon-tipped plunger, 23 gauge removable needle	1	5181-8813
25 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5183-0316
25 µL, Teflon-tipped plunger, 23 gauge removable needle	1	5183-0317
50 µL, Teflon-tipped plunger, 23 gauge fixed needle	1	5183-0318
50 µL, Reflon-tipped plunger, 23 gauge removable needle	1	5183-0319
Septa and seals		
Merlin microseal starter kit (cap + 1 microseal)	1	5182-3442
Merlin microseal replacement	1	5182-3444
11 mm HP septa, red	25	5181-1263

6

The Volatiles Interface

Chapter 6.
The Volatiles Interface

Part 1. 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 later in this chapter. Table 22 summarizes some issues to consider when choosing an operating mode. Specifications for the interface are also listed.

Table 22. 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

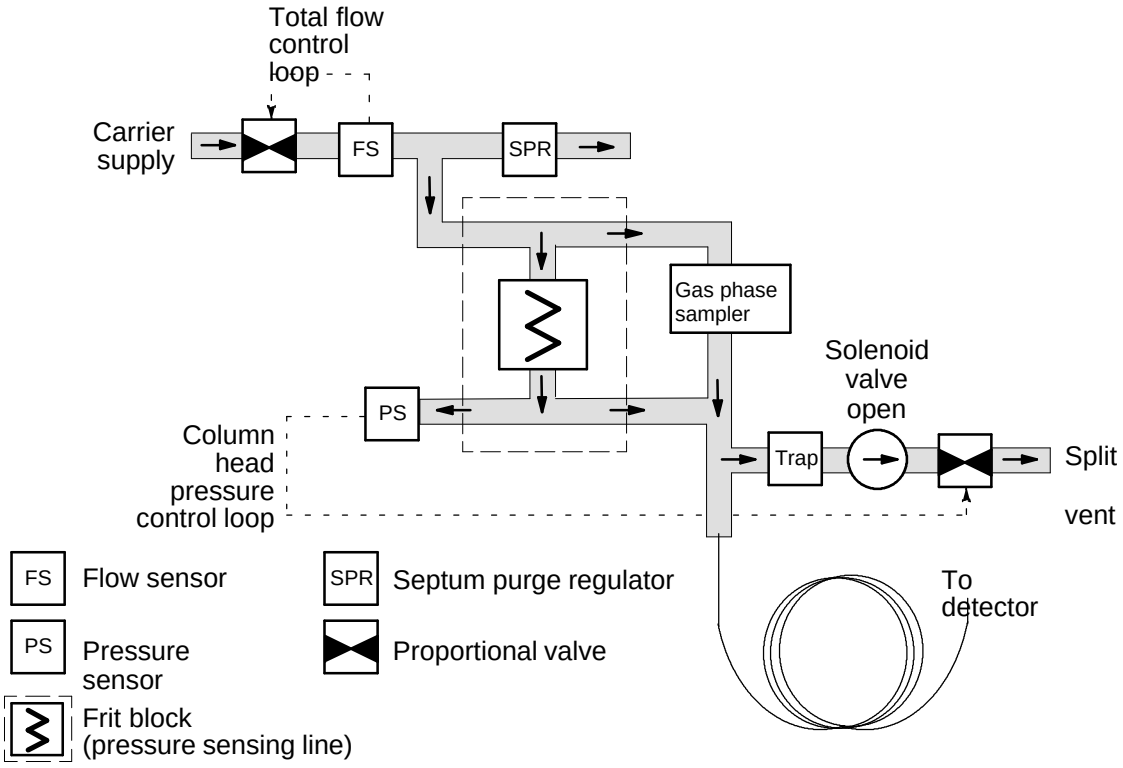
Split mode

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 29. 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

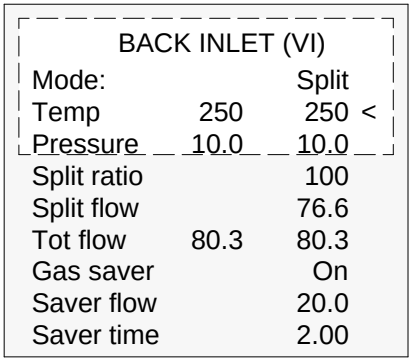
Pressure Actual and setpoint interface pressure

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.

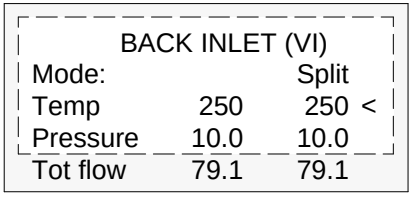
Split flow Flow, in mL/min, from the split vent. This parameter is not available if your column is not defined.

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 23. 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

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.

*This setpoint appears in the column control table.

Operating parameters

Use the information in Table 24 to help you set up the operating conditions for your interface.

Table 24. 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

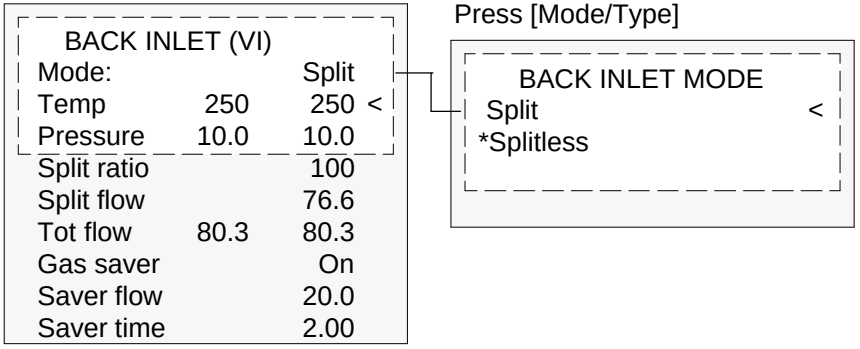
Split ratio

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:

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

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. See Appendix A to 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 Split.
   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 13) or use the [Prep Run] key before introducing the sample.

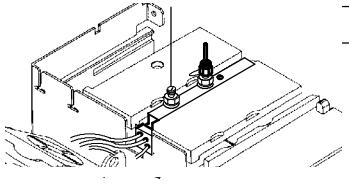
$$\text{Split ratio} = \frac{\text{Split flow}}{\text{Column flow}}$$

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. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
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” on page 15 for nominal septum purge flows).
   d. Calculate the split ratio. Adjust as needed.



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

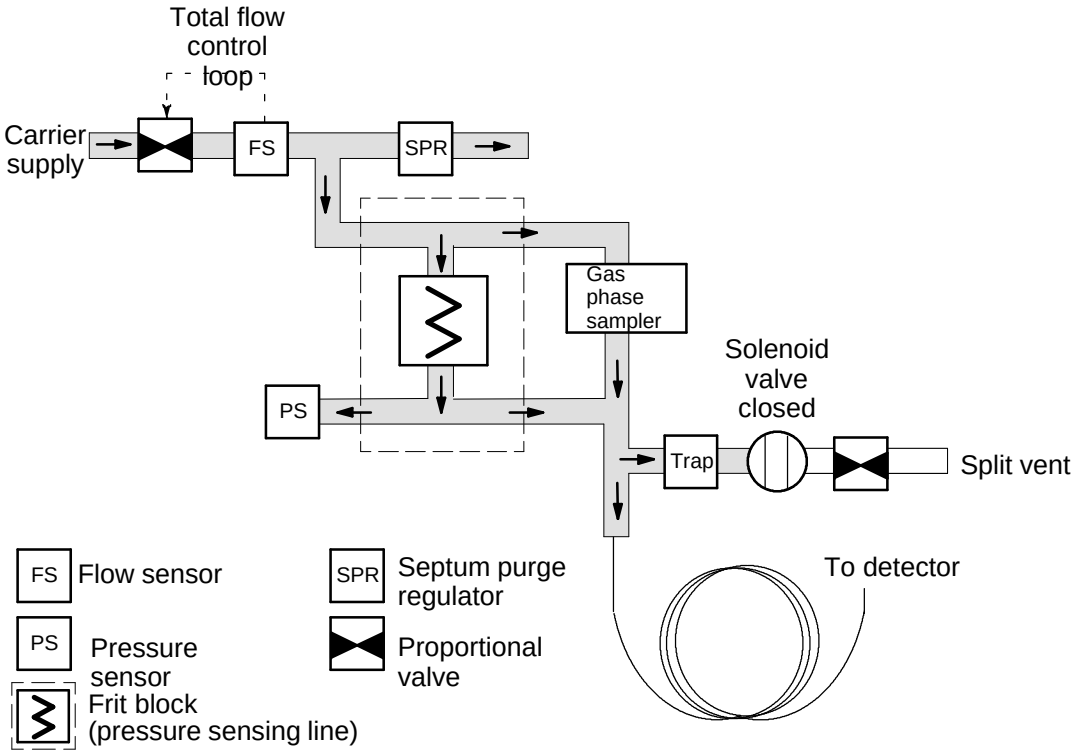
Before Pre Run, when the GC is preparing for sample introduction, total flow to the interface is measured by a flow sensor and controlled by a proportional valve. Column flow is controlled via back-pressure regulation. See Figure 29.

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 30.

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 29.

Figure 30. 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 HP 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 HP 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.

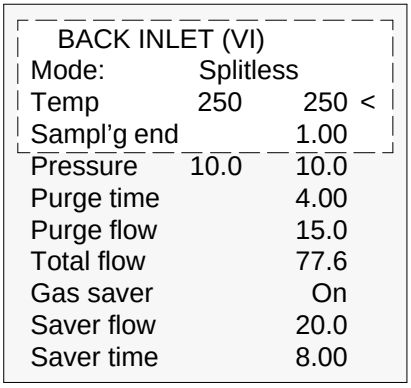
Pressure Actual and setpoint interface pressure in psi, bar, or kPa.

Purge time The time, after the beginning of the run, when purging resumes. Purge time must be greater than Sampling end.

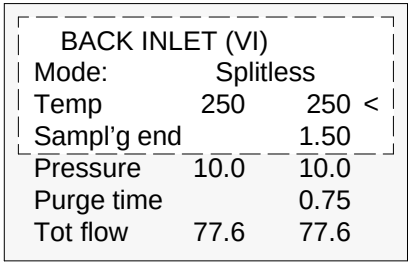
Purge flow The flow, in mL/min, from the split vent at Purge time. You will not be able access or to specify this value if operating with your column not defined.

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 25. Splitless mode pneumatic setpoints

Column defined	
When you change:	**These setpoints 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.	

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.

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

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 26. 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	Must be after Samplingend
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. See Appendix A to 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	

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

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	

   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 13) 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 later in this chapter.

Understanding the pneumatics

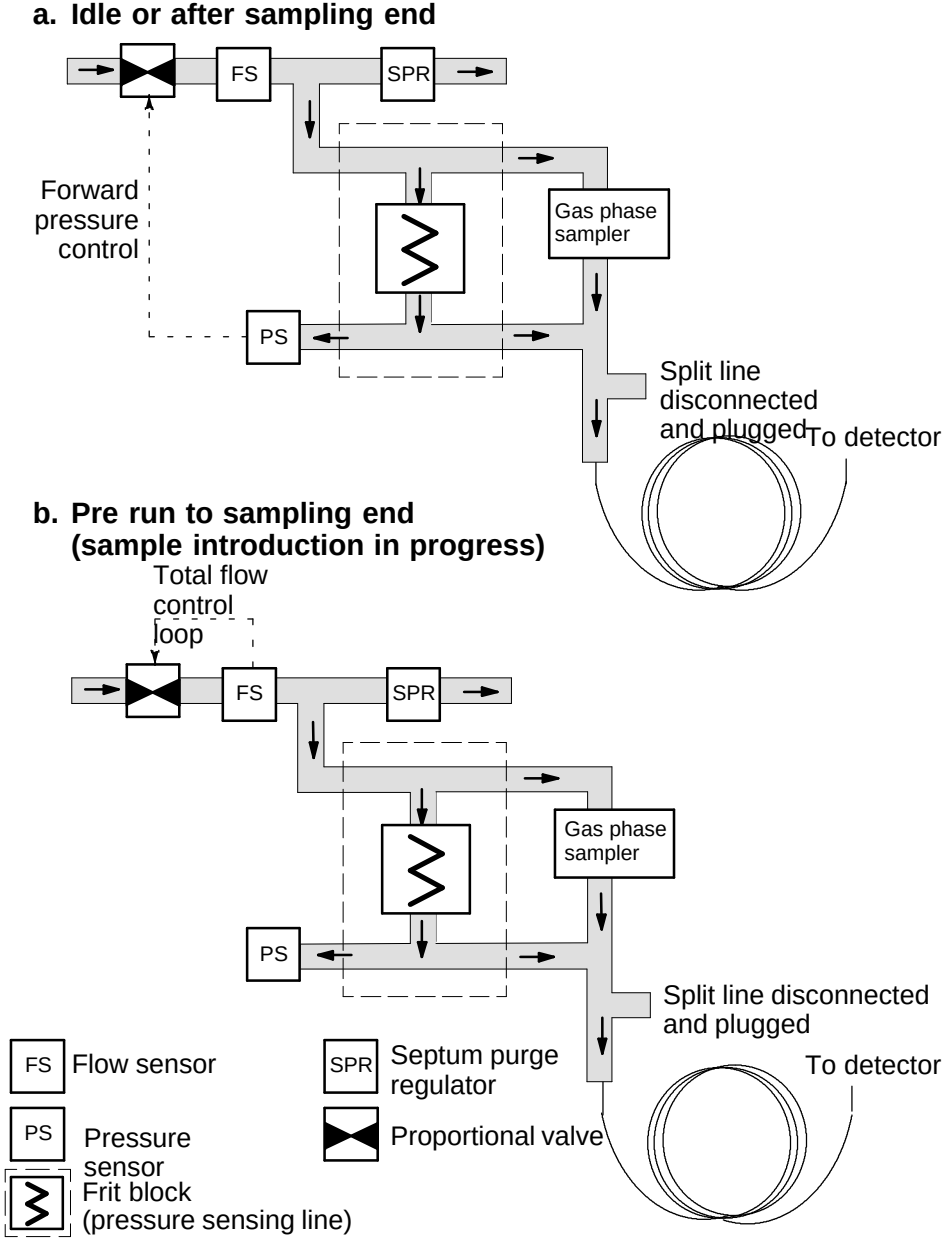
Before Pre Run, the interface is forward pressure controlled; pressure is sensed downstream from the flow proportional valve. See Figure 31a.

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 31b.

After sampling end, the interface is forward pressure controlled; pressure is sensed downstream from the proportional valve. See Figure 31a.

Figure 31. 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

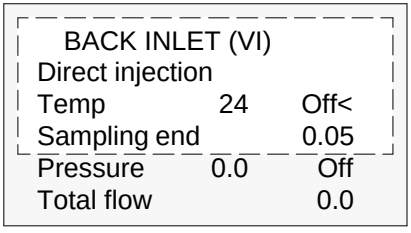
Procedure: Disconnecting the split vent line

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

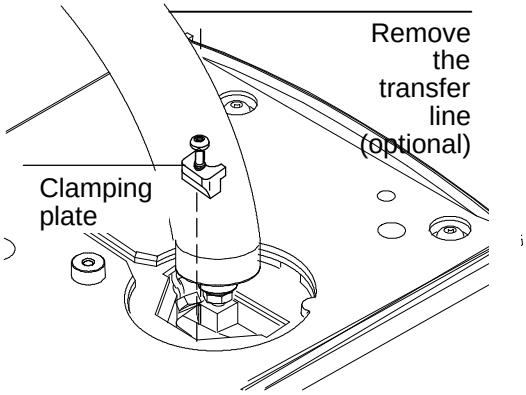
Materials needed:

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

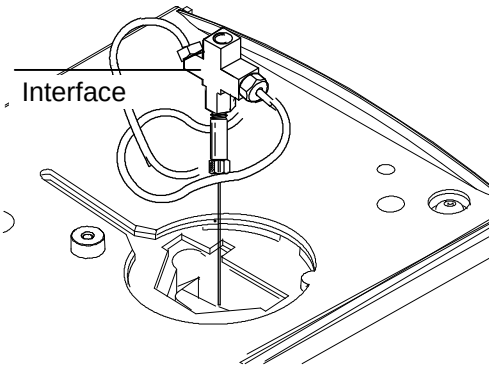
1. Press [Front Inlet] or [Back Inlet] and turn off the interface temperature and pressure. Allow the interface to cool.



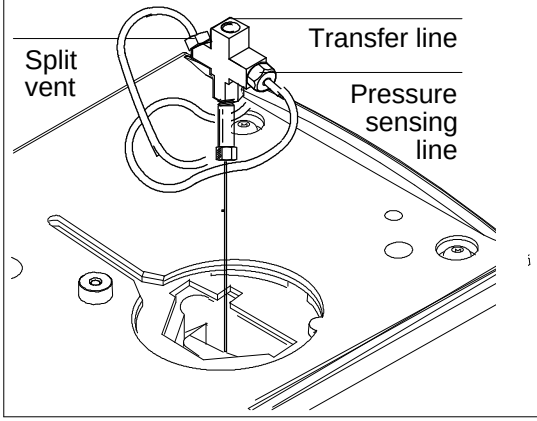
2. If desired, remove the transfer line by loosening the hex nut with a 1/4-in. wrench. Remove the clamping plate from the interface by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



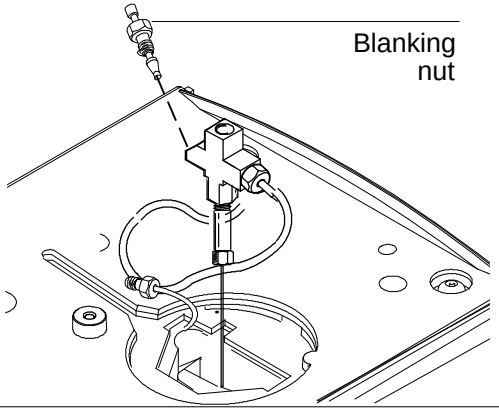
3. Carefully lift the interface out of the heater block.



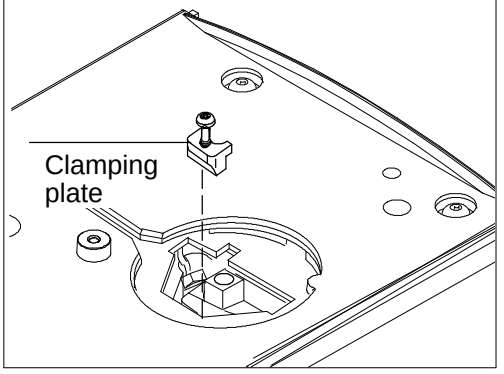
4. Loosen the the hex nut connecting the split vent line to the interface until you can remove the line. Put the line aside. You do not need to plug it.



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-in 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.



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:

```
    BACK INLET (VI)
Direct injection
Temp          250      250 <
Sampling end           0.05
Pressure      10.0     10.0
Total flow             0.0
```

If your interface is configured correctly, you will see this display

Using the control table

Direct injection If your GC is configured correctly, you will see this display. See “To configure your GC for a direct injection” for instructions.

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.

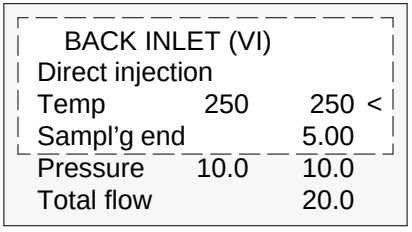
Set the samplng end setpoint 0.2 minutes longer than the time the sampler needs to introduce the sample. For example, the HP 7694 headspace sampler has an Inject time parameter which controls how long the valve remains in the inject position. If Inject time is 1 minute, the sampling end setpoint should be set to 1.2 minutes. If you’re using an HP 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 27. Direct mode pneumatic setpoints

When you change:	These setpoints change:
Column defined	
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 28 to help you set up the operating conditions for your interface.

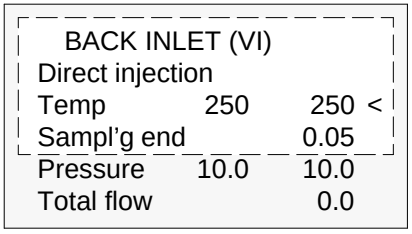
Table 28. Direct mode operating parameters

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.

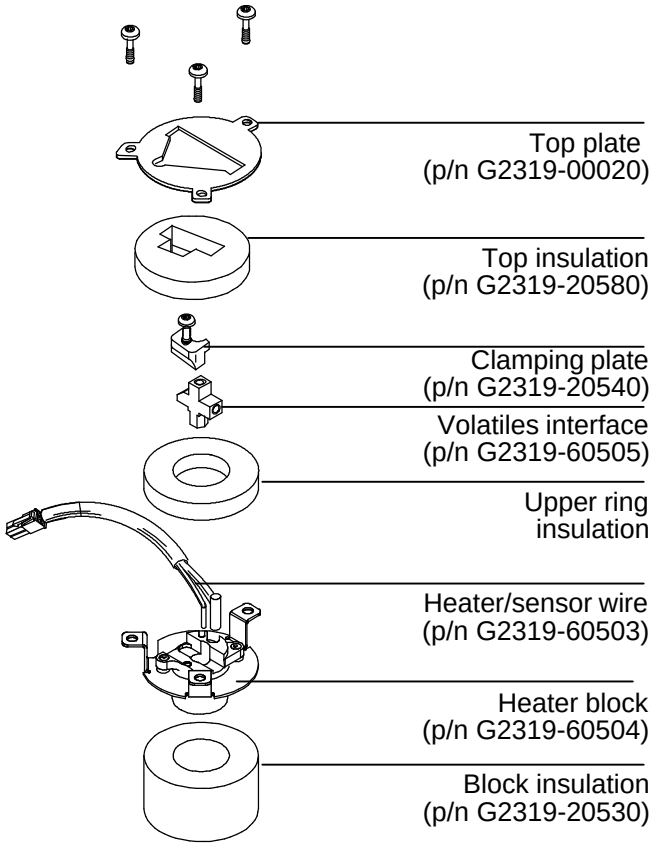
1. See Appendix A to verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly.
2. Press [Front Inlet] or [Back Inlet].
   a. Verify that your GC is configured for a direct injection.
   b. Set the interface temperature.
   c. Set sampling end. Set 0.2 minutes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



3. Make certain Auto Prep Run is On (see page 13) or use the [Prep Run] key before introducing a sample.

Part 2. Maintaining a Volatiles Interface

Figure 32. The volatiles interface parts breakdown



Not shown: Flow module, p/n G2319-60500
Pneumatic gang fitting assembly, p/n G2319-60501

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.

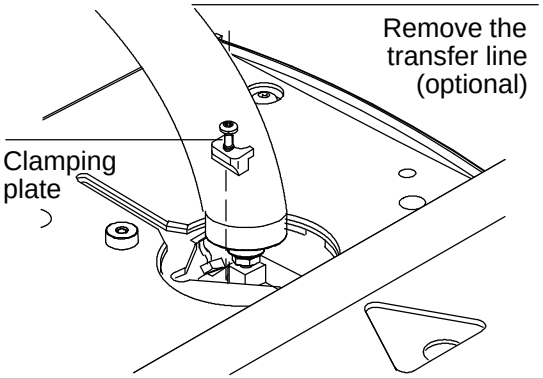
WARNING Be careful! The interface may be hot enough to cause burns.

Materials needed:

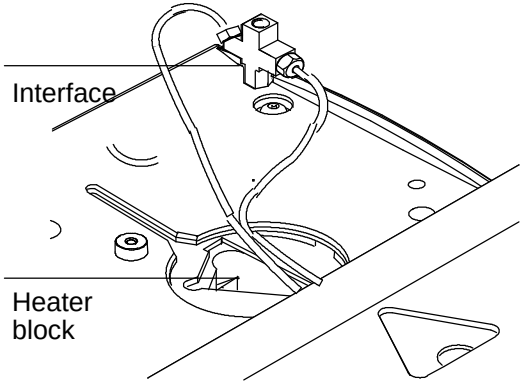
- Column nut
- Ferrule
- Column cutter
- Magnifying loop
- Isopropanol
- Tissue
- Typewriter correction fluid
- 1/4-in. wrench
- 5/16-in. or adjustable wrench
- Metric ruler
- T-20 Torx screwdriver

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.

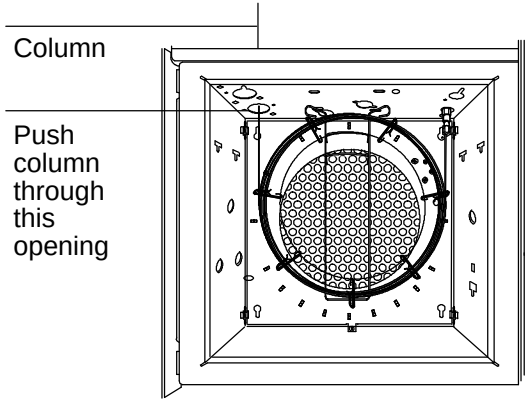
2. Disconnect the transfer line, if desired. Loosen the nut with a 1/4-in. wrench and remove the line. Remove the clamping plate from the interface by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



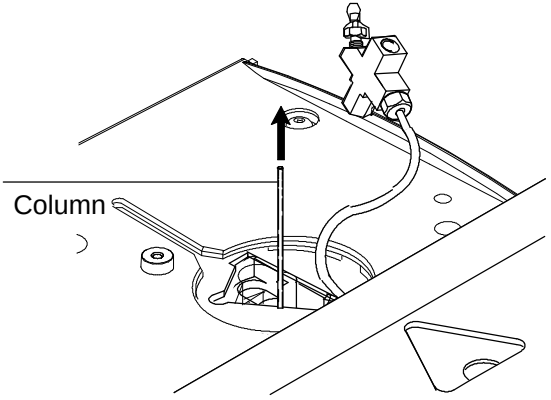
3. Lift the interface out of the heater block.



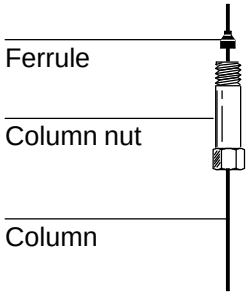
4. From inside the oven, push the column through the opening in the oven top.



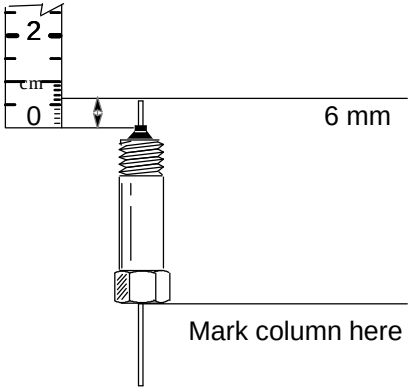
5. Grab the column from the oven top.



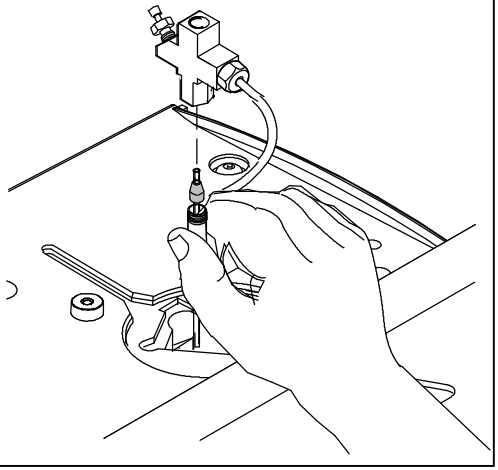
6. Place a capillary column nut and ferrule on the column and prepare the column end. If you need help with this step, see Columns and Traps in the General Information volume.



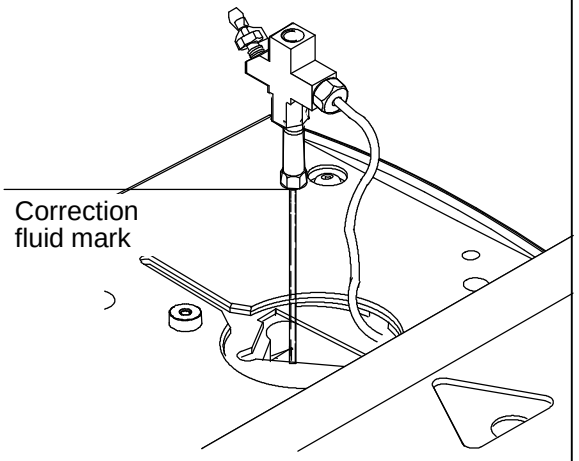
7. Position the column so it extends 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



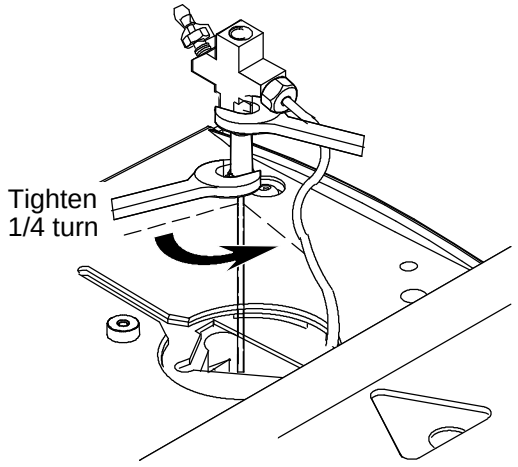
8. Insert the prepared column in the interface and finger tighten the column nut.



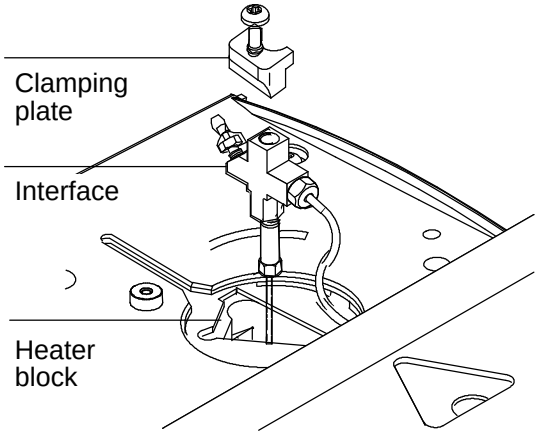
9. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.



10. Tighten the column nut an additional 1/4 to 1/2 turn using two wrenches in opposition. Use the adjustable wrench to hold the interface while you tighten the column nut with the 1/4-in. wrench until the column cannot be pulled from the fitting with gentle pressure.



11. 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.



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

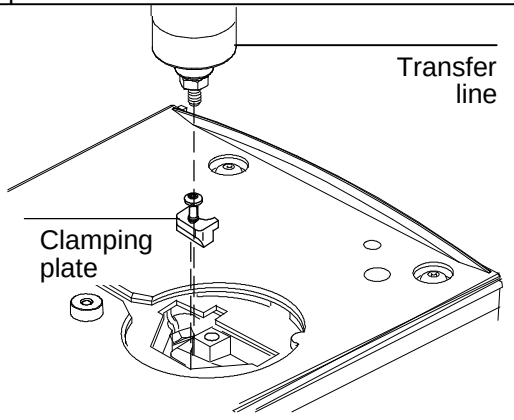
Materials needed:

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

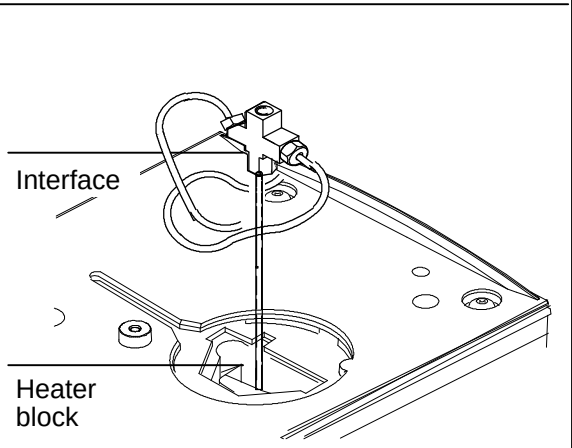
Preliminary Steps:

- 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.

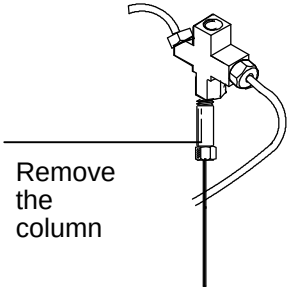
1. Disconnect the transfer line. Loosen the nut with a 1/4-in. wrench and remove the line. Remove the clamping plate from the interface by loosening the captive screw with a T-20 Torx screwdriver. Put the plate in a safe place.



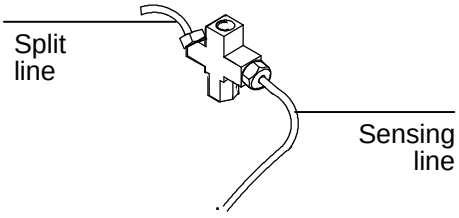
2. Lift the interface out of the heater block.



3. If a column is installed, removed it. See Columns and Traps in the General Information volume if you need help with this step.

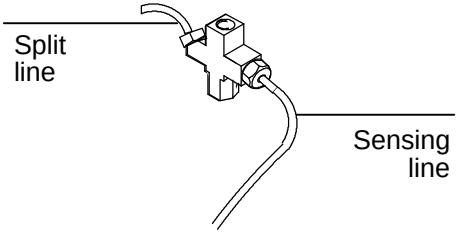


4. Remove the split and pressure sensing lines by loosening the hex nuts with the wrench.

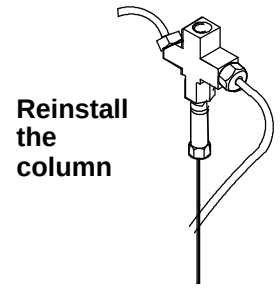


5. Clean or replace the interface. If you are cleaning the interface, sonicate it twice and then rinse.

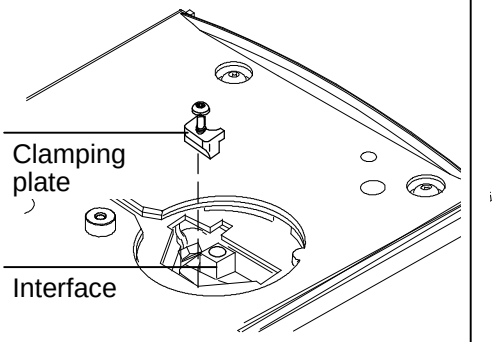
6. Reinstall the split line and pressure sensing lines and finger tighten the hex nuts. Tighten the hex nuts an additional 1/4 turn with the wrench.



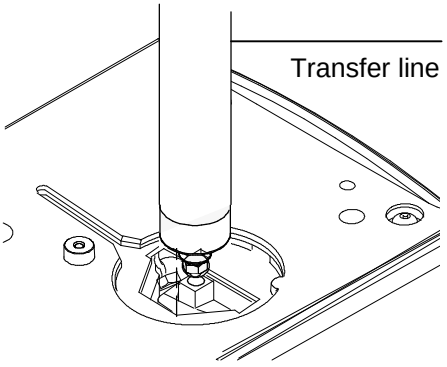
7. Reinstall the column in the interface. See Columns and Traps in the General Information volume for instructions.



8. Place the interface in the heater block. Replace the clamping plate you removed in step no. 1 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.



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.

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-in. 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.

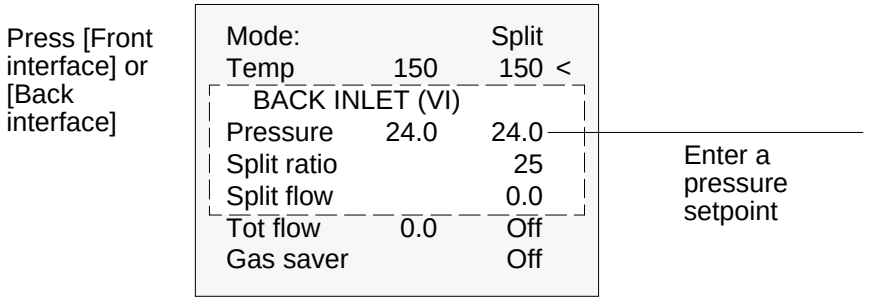
WARNING Be careful! The oven and interface may be hot enough to cause burns.

Materials needed:

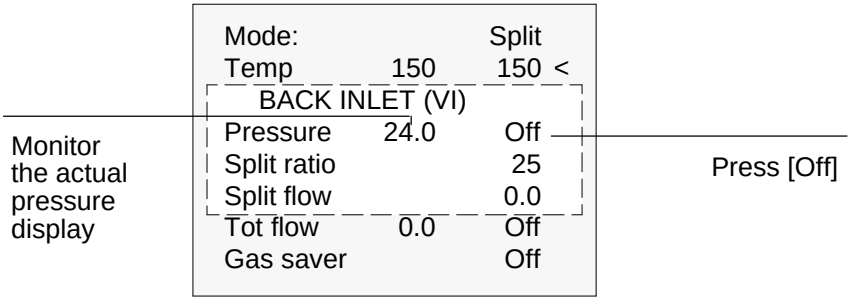
- No-hole ferrule
- 7/16-in. wrench
- 2, 1/8-in. SWAGELOK caps
- Gloves (if the interface is hot)
- 1/4-in. or 7 mm wrench

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 (see p. 33).
2. Cap the septum purge and split vent fittings located on the flow module with 1/8-in. Swagelock caps.

3. 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.



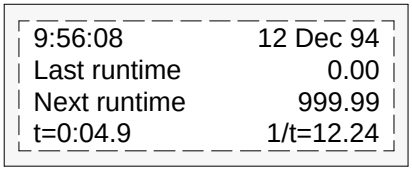
4. Turn the pressure off from the inlet control table. Because the septum purge, split vent, and column fittings are capped, gas should be trapped in the system and the pressure should remain fairly constant. Turn the pressure off at the source if you want to isolate the pneumatic system completely.



Because the pneumatics have been turned off, the alarm does not sound even though there is no flow through the column.

5. Continue to monitor pressure for 10 to 15 minutes. You can use the GC's Stopwatch function. 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.

   To access stopwatch, press [Time]

   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 "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 "Correcting Leaks" in this chapter.

   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-in. male GC nut and
- Graphite/vespel ferrule

1. Disconnect the transfer line from the interface (see p. 37, step 1).
2. Disconnect the carrier line from the sampler (see page 218 if you have a Headspace sampler or page 221 if you have a Purge and Trap Concentrator.)
3. Prepare the end of the carrier line using the 1/16-in. 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-in. wrench.
5. Return to “Leak testing the system” in this chapter and repeat steps 3 to 5.

Procedure: Correcting leaks

Materials needed:

- Electronic leak detector
- Tool that will tighten leaking fittings — 1/4-in., 5/16-in., 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.

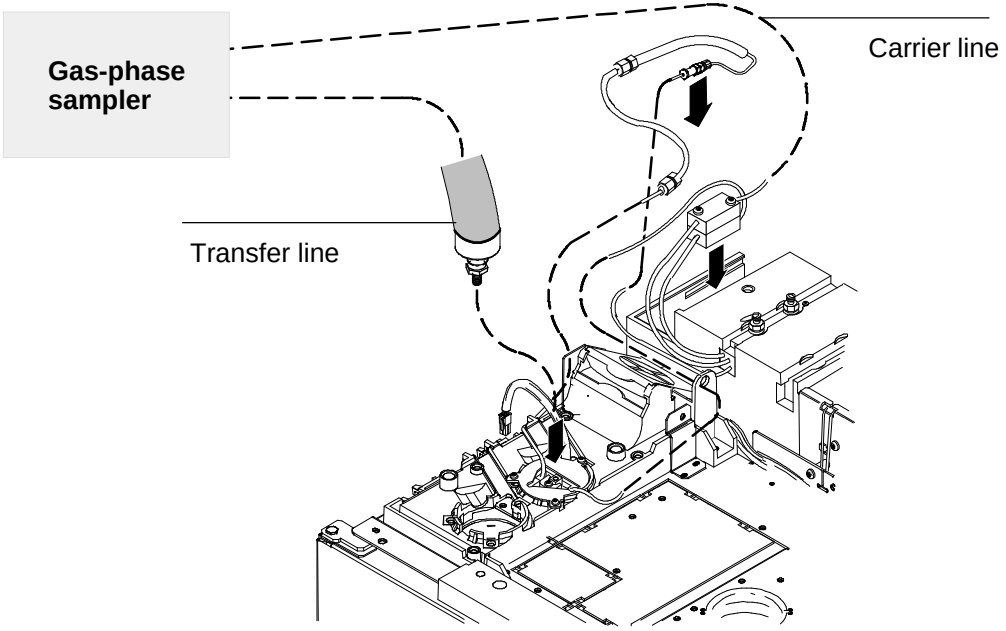
   If the pressure drop is now 0.03 psi/min or less, you can consider the interface system leak-free.

   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 Hewlett-Packard service representative.

Part 3. Connecting to an External Gas Sampler

Figure 33 illustrates a gas sampling device connected to the volatiles interface.

Figure 33. Flow diagram of an external sampling device

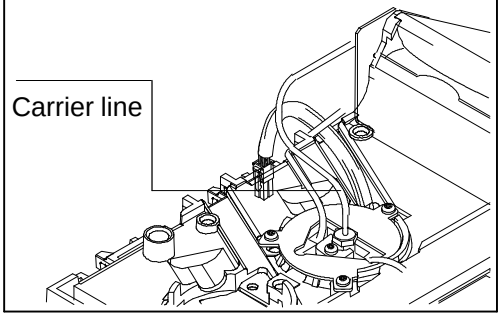


Procedure: Connecting the HP 7694 headspace sampler

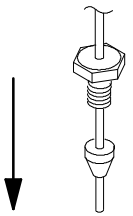
Materials needed:

- 1/16-in. to 1/8-in. reducer
- 1/8-in. female Swagelok nut
- 1/8-in. back ferrule
- 1/8-in. front ferrule
- Wrenches:
  - — One 7/16-in.
  - — Two 5/16-in.
  - — One 1/4-in.
  - — One 7-mm

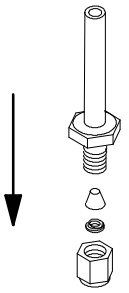
1. Locate the GC's carrier line tubing labeled "supply" attached to the volatiles interface, and remove the carrier line from the interface using a 1/4-in. wrench.



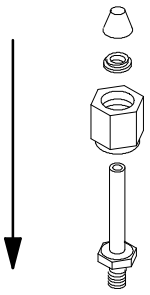
2. Remove the male fitting and Vespel/graphite ferrule from the carrier line. Keep the ferrule in a safe place.



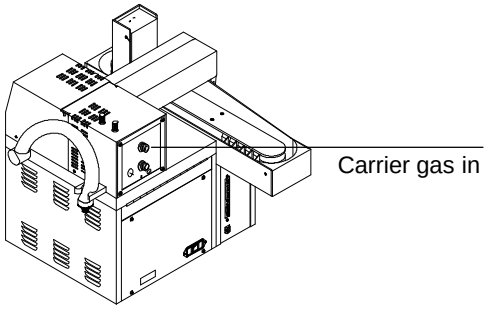
3. Get a 1/16-in. to 1/8-in. reducer, and remove the female nut and the metal front and back ferrules. Keep the female nut in a safe place.



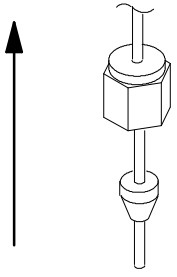
4. Slide a 1/8-in. female Swagelok nut, a 1/8-in. back ferrule, and a 1/8-in. front ferrule onto the unthreaded end of the reducer.



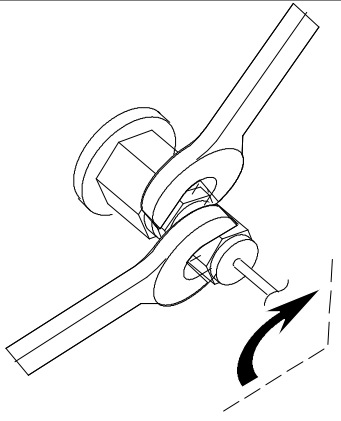
5. Connect the reducer to the gas supply port labeled “Carrier” on the back of the headspace sampler by tightening the 1/8-in. female Swagelok nut using a 7/16-in. wrench. Tighten the nut 1/4 turn past finger tight.



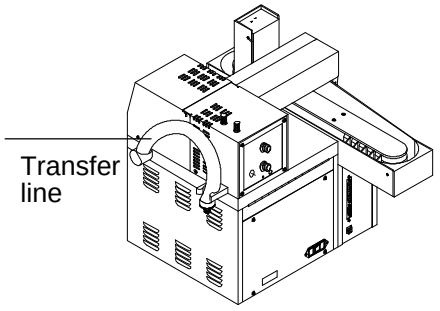
6. Slide the 1/16-in. female nut from step 3 and then the 1/16-in. Vespel/graphite ferrule from step 2 onto the end of the carrier line.



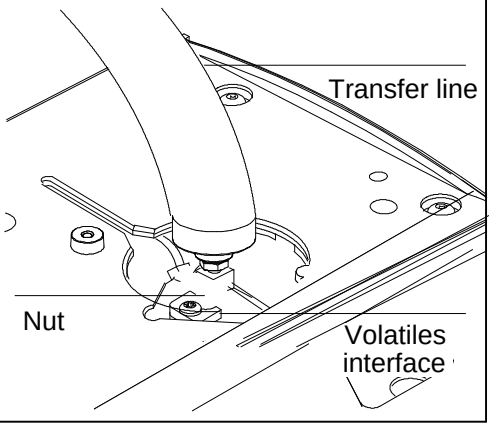
7. Connect the carrier line to the gas supply port by holding the reducer with one 5/16-in. wrench while tightening the 1/16-in. female Swagelok nut with another 5/16-in. wrench. Tighten the fitting 1/4 turn past finger tight. Do not overtighten. If the fitting leaks, tighten an additional 1/8 turn until it seals.



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



9. Connect the transfer line (with the preattached M5 nut and steel ferrule) to the interface using a 7-mm wrench. Tighten the M5 nut 1/4 turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8 turn until it seals.

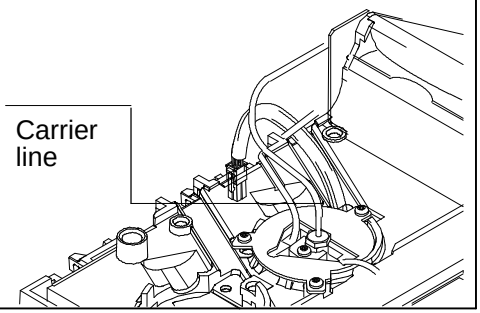


Procedure: Connecting the HP 7695 purge and trap concentrator

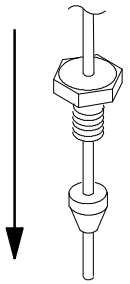
Materials needed:

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

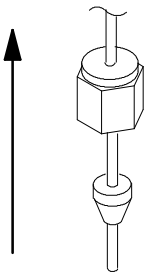
1. Locate the GC's carrier line tubing labeled "supply" attached to the volatiles interface, and remove the carrier line from the interface using a 1/4-in. wrench.



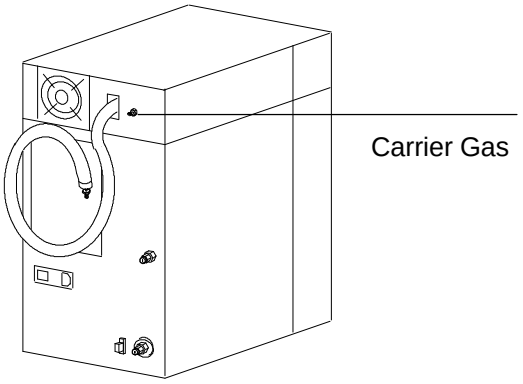
2. Remove the male nut and Vespel/graphite ferrule from the carrier line. Keep the male nut and ferrule in a safe place.



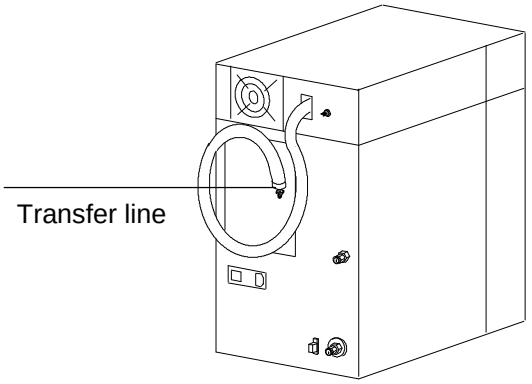
3. Slide a 1/16-in. female Swagelok nut and then the Vespel/graphite ferrule from step 2 onto the end of the carrier line.



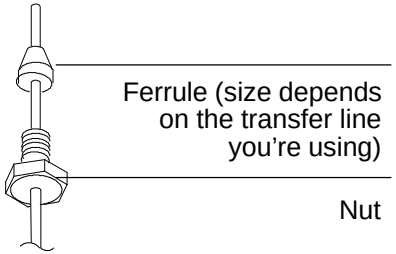
4. Connect the carrier line to the gas supply port labeled “Carrier Gas” on the back of the P&T concentrator using a 5/16-in. wrench. Tighten the nut 1/4 turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8 turn until it seals.



5. Locate the P&T concentrator’s transfer line tubing.

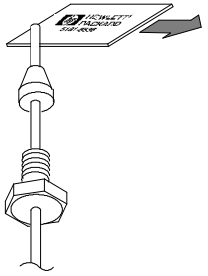


6. Slide the 1/16-in. male nut from step 2 and then an appropriate Vespel/graphite ferrule onto the end of the transfer line.

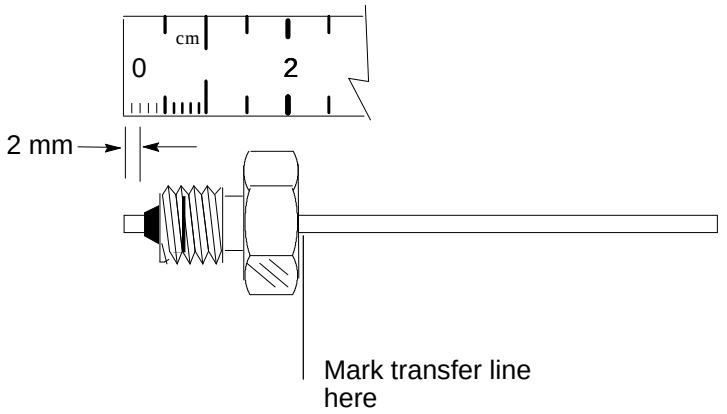


7. If you are using a nickel-plated transfer line, proceed to step 8.

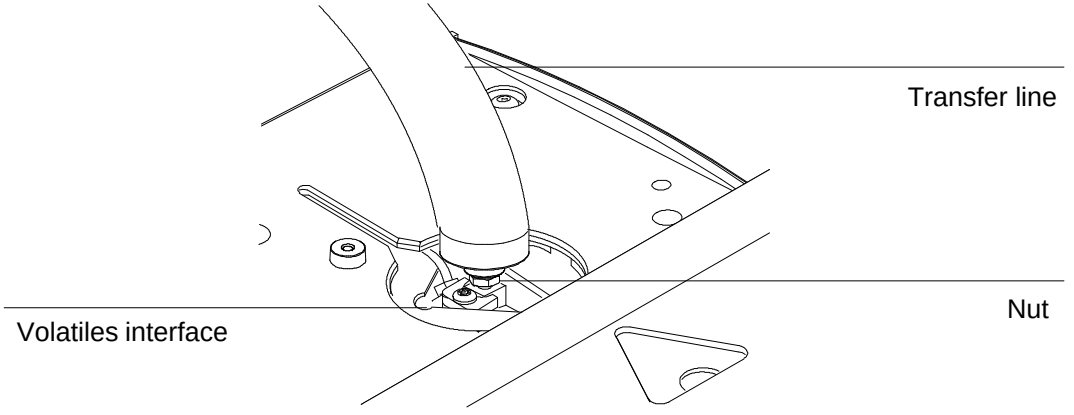
   If you are using a fused-silica transfer line, prepare the end of the fused silica line. See “Columns and Traps” in the General Information volume if you need help with this step.



8. 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.



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



7

NonEPC Inlets

Chapter 7.
NonEPC Inlets

Controls for these inlets are located on a pneumatics module attached to the left side of the GC.

Purged packed inlet

The only adjustment for this inlet is the carrier gas flow through the column. Septum purge flow is set automatically based on the source gas pressure. It can be measured at a vent on the front panel.

Split/splitless inlet—split mode

The carrier gas divides between the column and the split vent depending on their relative flow resistances. A small amount of carrier gas sweeps the lower side of the septum and exits through the septum purge control and vent.

Split/splitless inlet—splitless mode

In a splitless injection, a valve is actuated by [Prep Run] that prevents carrier gas from exiting the bottom of the inlet liner. Total flow does not change, but most of it exits through the septum purge line. All carrier gas that passes through the liner goes to the column—the sample is not split.

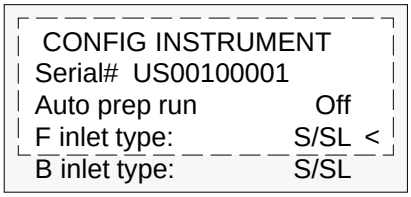
At purge time, the valve switches to sweep out residual vapor in the inlet. The system is now in the split configuration, with the purge flow and residual vapor—mostly solvent—exiting through the split vent.

Configuration

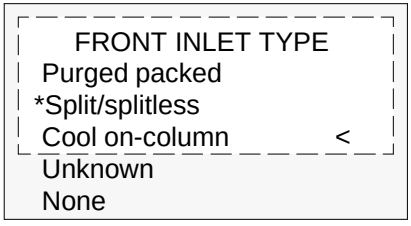
The GC is aware that a nonEPC inlet is present—it looks for the heater/sensor connections—but does not know what kind. You must supply this information through configuration.

Procedure: Configuring a nonEPC inlet

1. Press [Config], select Instrument., and [Enter].



2. Select the inlet and press [Mode/Type].



3. Select a type and [Enter].

4. Press [Config][Front Inlet] (or [Back Inlet]).

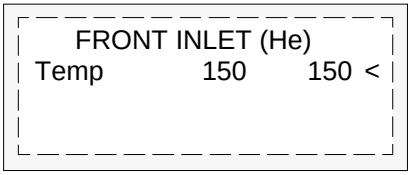
5. Press [Mode/Type], select a gas, and [Enter].

Inlet control tables

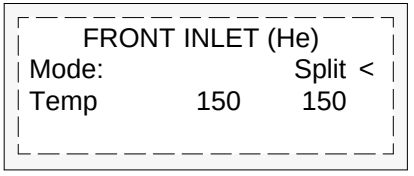
The inlet control tables for nonEPC inlets are similar to those for the EPC versions except that flow and pressure settings are absent. See Figure 34.

Figure 34. NonEPC inlet control tables

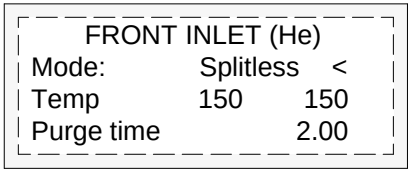
Purged packed inlet



Split/splitless inlet in split mode

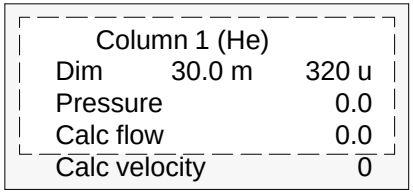


Split/splitless inlet in splitless mode



Column control tables

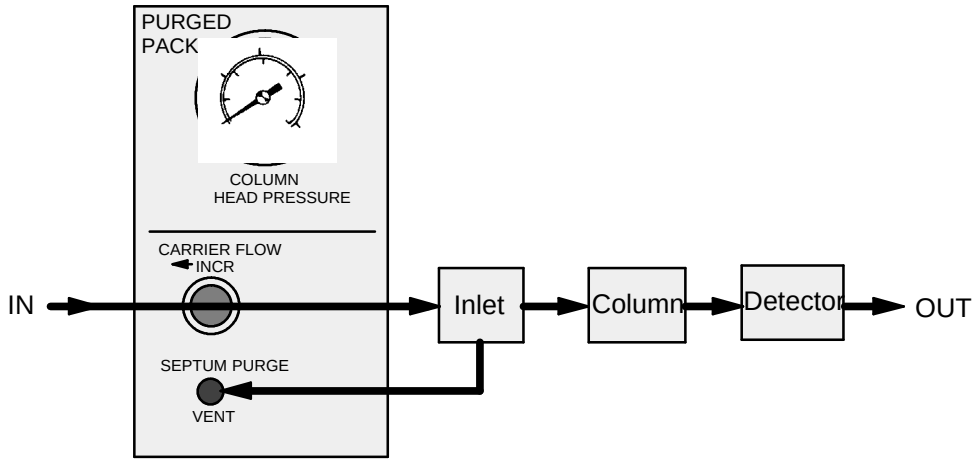
When a nonEPC split/splitless inlet is used with a defined column, the column control table becomes a calculator. Although you cannot control flows from the keyboard, you can determine the flows to be set manually.



Enter a pressure. Flow and average linear velocity are calculated and displayed.

Procedure: Setting carrier flow for the purged packed inlet

The internal flow path in the instrument is:

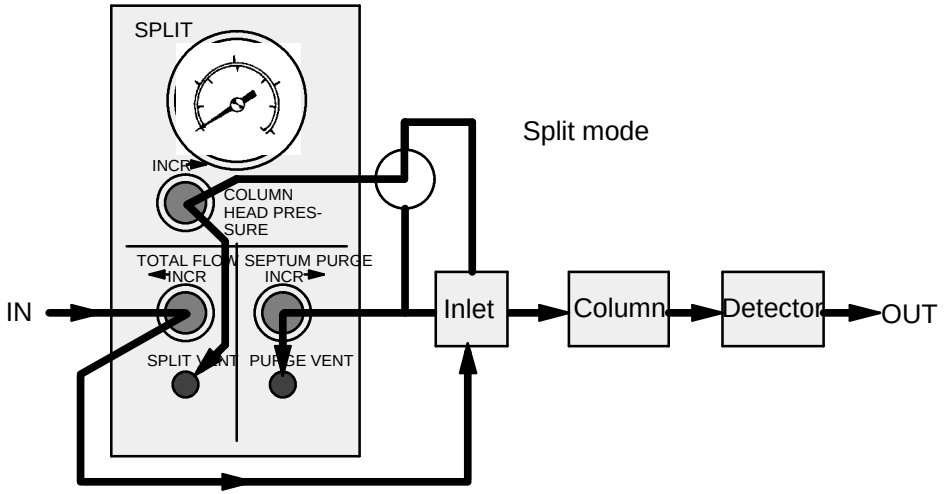


1. Locate the knob labeled CARRIER FLOW. Turn it clockwise as far as it will go. Do not force the knob; when it closes it comes to a slightly "soft" stop.
2. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi).
3. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas flows off from the keyboard.
4. Turn the CARRIER FLOW knob in the ◄INCR direction to turn the carrier gas on. Adjust and measure to achieve the desired flow. If necessary, increase the source pressure.

The septum purge is set automatically.

Procedure: Setting flows for the split mode inlet

The internal flow path in the instrument is:

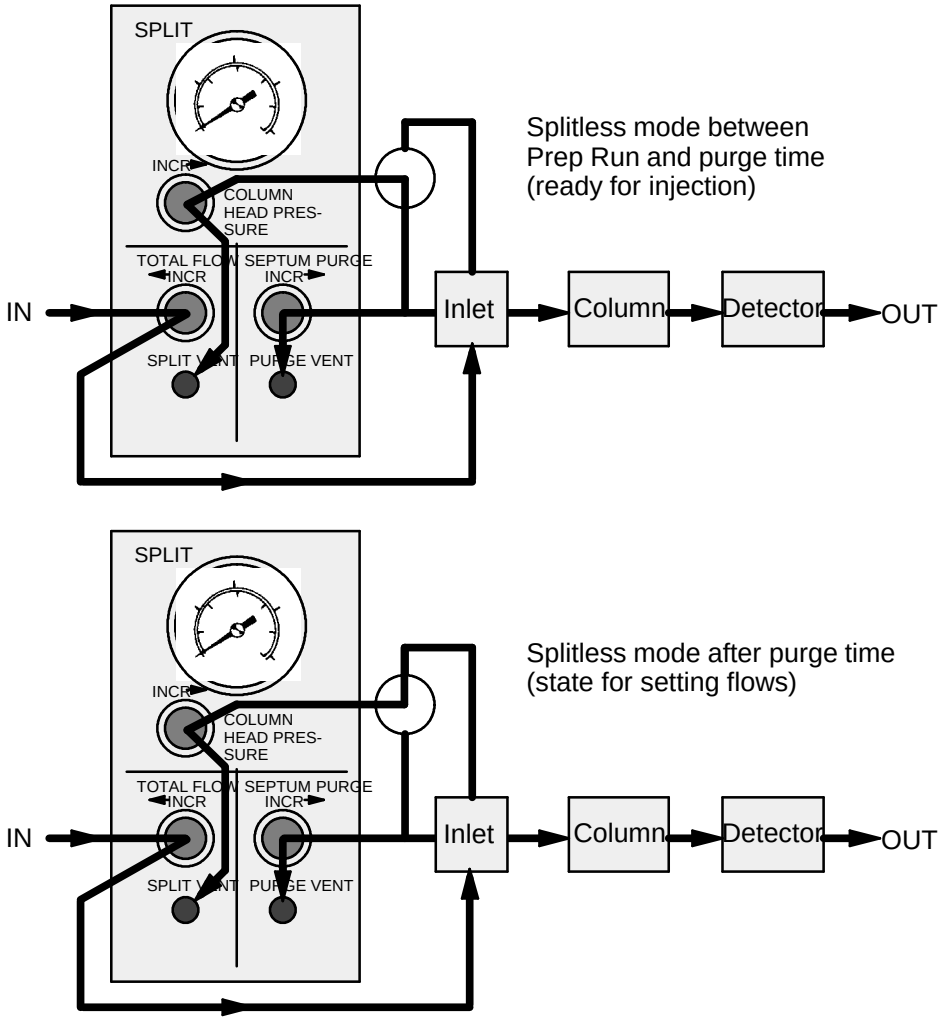


1. Locate the knob labeled TOTAL FLOW. Turn it clockwise as far as it will go. Do not force the knob; when it closes it comes to a slightly "soft" stop.
2. Locate the knob marked SEPTUM PURGE. Turn it counterclockwise to turn the flow off. There is no definite stop position; when the knob turns freely (does not seem to be touching anything inside), it is off.
3. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi). If you are using small-bore capillary columns, you may have to use higher pressures.
4. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas controls off from the keyboard.

5. Turn the TOTAL FLOW knob in the ◄INCR direction to turn the carrier gas flow on.
6. Turn the COLUMN HEAD PRESSURE knob in the INCR► direction. Adjust and measure to achieve the desired column flow. If you cannot, increase TOTAL FLOW until you can. Use TOTAL FLOW for coarse and COLUMN HEAD PRESSURE for fine adjustment.
7. Move the flow meter to the SPLIT VENT. Measure and adjust TOTAL FLOW to achieve the desired split flow. If necessary, increase the source pressure.
8. Move the flow meter to the PURGE VENT. Turn the SEPTUM PURGE knob in the INCR► direction to achieve the desired septum purge flow.
9. Repeat steps 6, 7, and 8 until all flows are correct.

Procedure: Setting flows for the splitless mode

The internal flow paths in the instrument are:



1. Locate the knob labeled TOTAL FLOW. Turn it clockwise as far as it will go. Do not force the knob; when it closes it comes to a slightly "soft" stop.
2. Locate the knob marked SEPTUM PURGE. Turn it counterclockwise to turn the flow off. There is no definite stop position; when the knob turns freely (does not seem to be touching anything inside), it is off.
3. Open the carrier gas cylinder valve and set the delivery pressure of the two-stage regulator to 410 kPa (60 psi). If there is a local regulator in the carrier gas line, set the cylinder regulator to 550 kPa (80 psi) and the local regulator to 410 kPa (60 psi). If you are using small-bore capillary columns, you may have to use higher pressures.
4. Attach a flow meter to the detector outlet. There should be no flow at this time. If there is, turn the detector gas controls off from the keyboard.
5. Turn the TOTAL FLOW knob in the ◄INCR direction to turn the carrier gas flow on.
6. Turn the COLUMN HEAD PRESSURE knob in the INCR► direction. Adjust and measure to achieve the desired column flow. If you cannot, increase TOTAL FLOW until you can. Use TOTAL FLOW for coarse and COLUMN HEAD PRESSURE for fine adjustment.
7. Move the flow meter to the SPLIT/SPLITLESS INLET VENT. Measure and adjust TOTAL FLOW to achieve the desired split flow. If necessary, increase the source pressure.
8. Move the flow meter to the SEPTUM PURGE VENT. Turn the SEPTUM PURGE knob in the INCR► direction to achieve the desired septum purge flow.
9. Repeat steps 6, 7, and 8 until all flows are correct.

Appendix A

Configuration Information

Appendix A:
Configuration Information

Preparing for analysis

All operating procedures begin with the four steps below. Note that there are two variations of step 2, one for column defined and one for column not defined.

The rest of the material in this appendix provides the details of these steps. It is copied from the General Information volume and printed here as a convenience to eliminate jumping back and forth between the two books.

1.. Verify that a column is installed and the correct liner is in the inlet.

2.. Configure the column. Press [Config][Col 1] or [Config][Col 2].

   a. To define the column, enter the dimensions requested.

   or

   b. To leave the column not defined, enter 0 for either column length or column diameter.

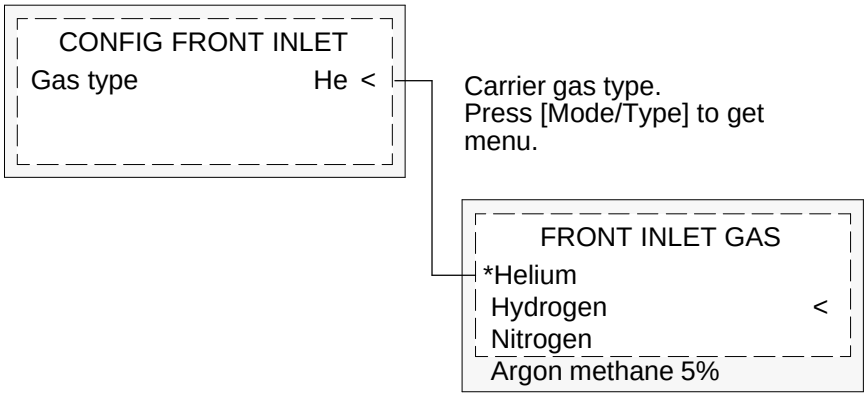
3.. Press [Col 1] or [Col 2]. Verify that the gas type in the title line is correct. Change if necessary.

4.. Specify a column flow or pressure mode and a starting flow or pressure. Enter a flow or pressure program, if desired.

To configure the carrier gas

1.. Press [Config] [Front Inlet] or [Config] [Back Inlet].

2.. Press [Mode/Type] to see the carrier gas menu.



3.. Scroll to the gas you will use. Press [Enter].

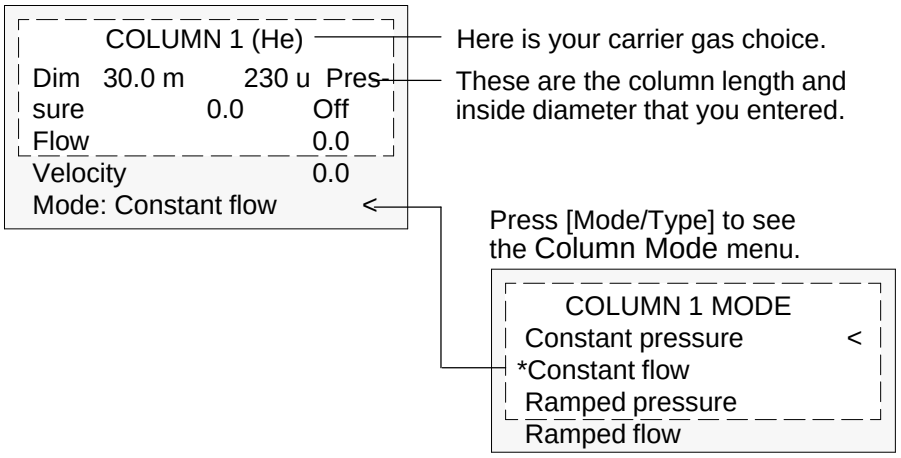
This completes carrier gas configuration. See the Inlets volume for more detail.

To select a column mode

1.. Press [Col 1] or [Col 2].

2.. Scroll to the Mode line.

3.. Press [Mode/Type] to see the column mode menu.

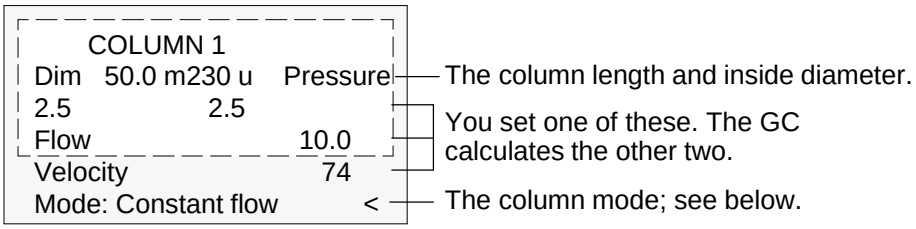


4.. Scroll to the column mode you want. Press [Enter].

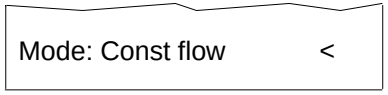
This completes the column mode selection. Next you must specify the inlet conditions either during the entire run (if you selected either of the constant modes) or at the beginning of the run (if you selected either of the ramped modes).

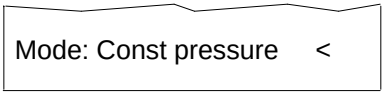
To set the initial flow or pressure or average linear velocity

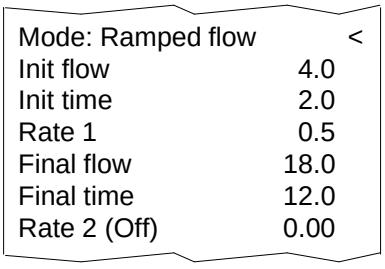
1.. Press [Col 1] or [Col 2].

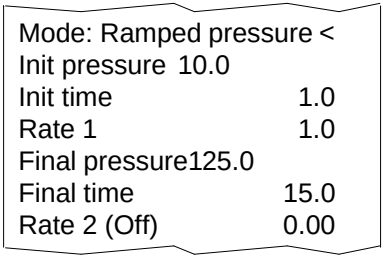


The control table will have one of these, depending on the column mode selected:









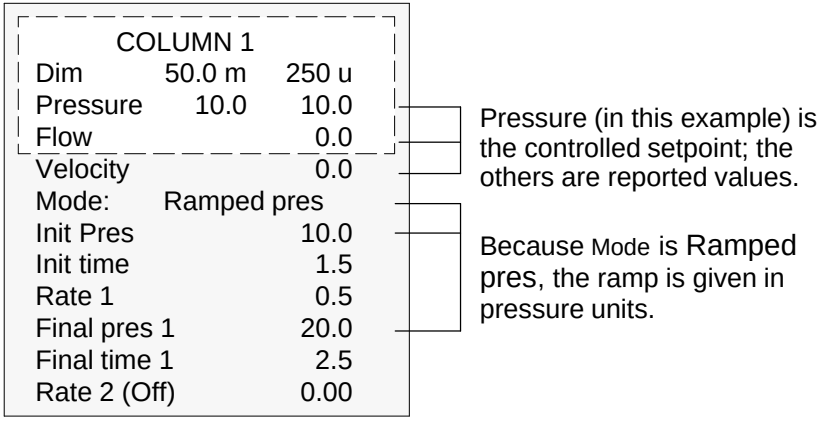
2.. Scroll to the Pressure or Flow or Velocity line.

3.. Type the desired initial value, followed by [Enter]. The GC computes and displays the other two values. Adjust them, if you choose to, by repeating steps 2 and 3. Note that changing any one changes all three.

This completes setting the initial carrier gas condition.

To enter a pressure or flow program

1.. Press [Col 1] or [Col 2].



2.. Scroll to Init Pres (or Init flow). Type the desired value and press [Enter].

3.. Similarly, enter a value for Init time. This completes the initial (constant pressure) part of the program.

4.. To begin a ramp, enter a positive value for Rate 1. It does not matter whether you are programming up or down—the rate is always positive.

5.. If Rate 1 is zero, the program ends here. If you enter any other value, the Final value lines for the first ramp appear and the cursor moves to the line.

6.. Enter values for Final pres 1 (or Final flow 1) and Final time 1. This completes the first ramp.

7.. To enter a second (or third) ramp, scroll to the appropriate Rate line and repeat steps 5 and 6.

A

C

D

F

G

R

S

T

U

V

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Manual Part No. G1530-90450

H l P k d C