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The Split/Splitless Inlet

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 allow 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

For the standard septum retainer nut, an internal spring in the septum retainer 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.

If using a Merlin MicrosealTM septum, finger tighten the septum nut, until snug (not loose). The pressure capacity depends on the septum used.

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 Agilent catalog for consumables and supplies.

Procedure: Changing the liner

Parts list:

- Liner, part no. 5183-4647 (split) or 5062-3587 (splitless)
- Tweezers
- Septum wrench (part no. 19251-00100)
- Viton O-ring (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 42 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.

<u>Figure 43</u> shows the pneumatics for this inlet in split mode operation.



Figure 43 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. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 2. Press [Front Inlet] or [Back Inlet]
 - a. Scroll to Mode: and press [Mode/Type]. Select Split.
 - b. Set the inlet temperature.
 - 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 <u>285</u>) before manually injecting the sample.

	() ()	Press [Mode/Type]
Mode: Temp 250 Pressure 10.0 Split ratio Split flow	Split 250 < 10.0 76.6	FRONT INLET MODE Split < *Splitless Pulsed split Pulsed splitless
Gas saver Saver flow Saver flow	80.3 0n 20.0 2.00	If using gas saver, set time after injection time.

Split ratio = <u>Split flow</u> Column flow

Procedure: Using the split mode with the column not defined

- 1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 2. Press [Front Inlet] or [Back Inlet]

FRONT I	NLET	(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 <u>286</u> for nominal septum purge flows by carrier gas type) from Total flow to get column flow.
- d. Calculate the split ratio. Adjust as needed.



Front of instrument

Split ratio = <u>Split flow</u> Column flow

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 44 Splitless flow diagram, pre-run to purge time

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.

	FRON	T INLET (S/SL)		
	Mode:	Spl	itless		
	Temp	250	250	<	
	Pressu	<u>ire 10.0</u>	10.0		
	Purge	time	0.75		
	Purge	flow	15.0		
	Total	flow	77.6		
	Gas sa	iver	0 n		
	Saver	flow	20.0		It using gas saver, set saver time atter
	Saver	time	2.00		purge now time.
122.00					

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. <u>Table 33</u> provides starting values for the critical 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	O 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	O to 999.9 minutes	After purge time
Gas saver flow	15 to 1000 mL/min	15 mL/min greater than maximum column flow

 Table 33
 Splitless Mode Inlet Parameters

Procedure: Using splitless mode with the column defined

- 1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 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 <u>285</u>) before manually injecting a sample.

Procedure: Using splitless mode with the column not defined

- 1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 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 286—to guarantee adequate column flow.



3. Use the [Prep Run] key (see page <u>285</u>) 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 <u>285</u> 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 45 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 2 control table. Appears only if the column is defined.

Split flow Flow, in mL/min from the split/purge vent. Appears only if the column is defined.

Total flow The 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.

-					
Г 	FRONT IN	ILET	(S/SL)		
2	Mode: Pul	sed	split		
1	Temp	250	250	<	
l L	Pressure	10.0	10.0	 	
	Pulsed pr	e s	30.0		Prossura nulsa satnointe
	Pulse time	e	1.0		
	Split rat	io	100		
	Split flo	W	67.0		
	Tot flow		70.9		
	Gas saver		0 n		
	Saver flo	W	20.0		
	Saver time	e	3.00		
				and the second second second	

Procedure: Using the pulsed split mode

- 1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 2. Press [Front Inlet] or [Back Inlet]
 - a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Split.
 - b. Set the inlet temperature.
 - c. Entervalues for Pulsed Pres and Pulse time.
 - 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 <u>285</u>) before injecting a sample manually.

Split ratio = Split flow Column flow

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.

100			1	
	FRONT INLET (S/SL)		
	Mode:Pulsed spl	itless		
	Temp 250	250 <		
	Pressure 10.0	10.0		
	Pulsed pres	30.0 -		Proseuro nulso sotnointe
	Pulse time	1.6 —		i lessure puise serpoints
	Purge time	1.5	<u> </u>	Inlat nurga astrointa
	Purge flow	15.0		mier purge serpoints
	Total flow	77.6		
	Gas saver	0 n		
	Saver flow	0.0		
	Saver time	3.00		
			1	

Procedure: Using the pulsed splitless mode

- 1. Verify that the column, carrier gas, and flow or pressure program (if used) are configured correctly. See <u>"Flow and Pressure Control"</u>.
- 2. Press [Front Inlet] or [Back Inlet]
 - a. Scroll to Mode: and press [Mode/Type]. Select Pulsed Splitless.
 - b. Set the inlet temperature.
 - c. Entervalues 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. If 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 <u>285</u>) before injecting a sample manually.

Maintaining a split/splitless inlet



Figure 46 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. Another available option is the Merlin Microseal[™] septum, a duckbill septum providing low bleed and longer life when used with the 7683 automatic Sampler and recommended syringes. You can order septa directly from Agilent Technologies; refer to the Agilent catalog for consumables and supplies for ordering information.

 Table 34
 Recommended Septa for the Split/Splitless Inlet

Description	Part no.	
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 (30 psi)	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—see <u>Table 34</u> on page <u>307</u> for part numbers
- Septum nut wrench (part no. 19251-00100)
- A 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.
 - Turn the oven and detector off.
 - Cool the oven and inlet to room temperature.
 - Turn the inlet pressure off.
- 2. Remove the septum retainer nut or Merlin cap, using the wrench if the nut is hot or sticks. Remove the old septum or Merlin Microseal. If the septum sticks, use a sharp tool to remove it. Be sure to get all of it. Take care to avoid gouging or scratching the interior of the septum head.

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, use a small piece of rolled-up steel wool and forceps or tweezers to scrub the residue from the retainer nut and septum

holder. Use compressed air or nitrogen to blow away the pieces of steel wool and septum.

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

If installing a Merlin Microseal, install it so that the side with the metal parts faces down (toward the oven).



5. Replace the septum retainer nut or Merlin cap, tightening it finger-tight. If using the standard septum retainer nut, the C-ring is about 1 mm above the nut. Avoid overtightening.



6. Restore normal operating conditions.

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.



Figure 47 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 35. O-Rings for the Split/Splitless Inlet

Description	Part no.
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 <u>Table 35</u> on page <u>310</u>
- Septum nut wrench (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.
 - Turn the oven and detector off.
 - Cool the oven and inlet to room temperature.
 - Turn the inlet pressure off.
- 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. Use the forceps or tweezers to grasp the liner and pull it out.



- 4. Remove the old O-ring and slide a new one onto the liner.
- 5. Use the forceps to 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.

Three types of inlet base seals are available:

- Gold-plated seal, part no. 18740-20885
- Gold-plated seal, cross, part no. 5182-9652
- Stainless steel seal, part no. 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 <u>"Columns and Traps"</u>.

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 list of part numbers)
- A new washer (part no. 5061-5869)
- 1/4-inch wrench (for column)
- 1/2-inch wrench
- 1. Complete the following preliminary steps:
 - If you have entered parameters that you do not want to lose, store them as a method.
 - Turn the oven and detector off.
 - Cool the oven and 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-inch 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 gloves 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.



5. Replace the reducing nut. Use the 1/2-inch wrench to tighten the nut. Replace the column and the insulation cup. After the column is installed, you can restore normal operating conditions.

Replacing the split vent trap filter cartridge

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

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

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



- 5. Remove the old filter cartridge and O-rings and replace them.
- 6. Reassemble the trap.
- 7. Check for leaks.

Procedure: Leak testing the gas plumbing

Leaks in the gas plumbing can affect chromatographic results dramatically. The following procedure checks the flow system up to but not including the 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-inch wrenches ٠
- 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-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch 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.
 - Turn the oven off.
 - Cool the oven and inlet to room temperature.
 - Turn the inlet pressure off.
 - 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 <u>"Changing septa"</u>.
 - Inspect the O-ring and replace it if it is hard and brittle or cracked. See <u>"Changing the O-ring"</u> for instructions.

2. Cap the septum purge fitting with a 1/8-inch SWAGELOK cap.



- 3. Set the oven to its normal operating temperature.
- 4. Configure the column as 0 length.
- 5. Press [Front Inlet] or [Back Inlet] to open the inlet's control table.
 - Set the inlet to its normal operating temperature.
 - Enter a pressure setpoint of 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.
 - Set the total flow to 60 mL/min.
 - Set the inlet to Split Mode.

Wait a few moments for the pressure and flow to equilibrate. If pressure cannot be achieved, there is either a large leak or the supply pressure is to low.

- 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.
- 7. Monitor the pressure for 10 minutes. A pressure drop of less than 0.5 psig (0.05 psi/min or less) is acceptable.

If the pressure drops much faster than the acceptable rate, see <u>"Procedure: Correcting leaks"</u>.

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-inch wrench
- Gloves (if the inlet is hot)
- Septum nut wrench (part no. 19251-00100)
- 9/16-inch wrench
- 1/8-inch 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 <u>307</u>.
 - Inspect the O-ring and replace it if it is hard and brittle or cracked. See page <u>310</u> for instructions.
- 2. Cap the purge vent with a 1/8-inch 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 to 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. If the system cannot reach the pressure setting, their either is a large leak or the supply pressure is too low.
- 6. Verify that the split flow 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 clock-wise an additional 1/2 turn.

Observe the column pressure for approximately 10 minutes. If the pressure drops less than 0.5 psig (0.5 psi/min or less), you can consider the inlet leak-free.

If the pressure drops much 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, and 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 Agilent 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 (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.
 - Turn the heated zones off—wait for them to cool.
 - Turn off all flows to the inlet at the initial gas supply.
 - Turn off the GC and unplug it.
 - Remove the inlet liner.
 - Remove the column adapter. See <u>"Columns and Traps"</u>.
 - Remove the inlet base seal. See page <u>313</u> for instructions.
- 2. Illuminate the inside of the inlet from below 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.
- 3. Blow out loose particles and dry thoroughly with compressed air or nitrogen before reassembling.
- 4. Reassemble the inlet. Use a new inlet base seal. Restore to normal operating conditions.