



# Agilent 7697A Headspace Sampler

## *Leak Testing the Headspace Sampler*

Parts Supplied	1
Leak Check General Procedure	2
Flow Paths	4
Checking for External Leaks	6
To Run the Restriction and Pressure Decay Test	8
To Run the Cross Port Leak Test	18
To Check for Leaks in the Transfer Line	22

This document discusses leak testing in the headspace sampler (HS). Chromatographic symptoms such as loss of sensitivity can result from leaks and restrictions. Use the following set of tests for verifying leaks and restrictions within the HS. Verify that the GC is leak free before checking for leaks within the HS.

## Parts Supplied

**Table 1** G4556-67010 Leak Test Kit

Description	Part Number
<b>Leak Test Kit</b>	<b>G4556-67010</b>
• Leak Test Instruction Sheet/Quick Reference Guide	G4556-90001
• Blue leak test vial	G4556-20600
• six port valve cap	G6600-80039
• No hole ferrule	5181-7458
• 11-mm low bleed septa, 5 PK	5182-3413
• Plug, 1/8-in tube fitting, nylon	0100-2414



## Leak Check General Procedure

When checking for leaks, consider the system in three parts: external leak points, GC leak points, and HS leak points.

- **External leak points** include the gas cylinder (or gas purifier), the regulator and its fittings, the supply shutoff valves, and the connections to the HS and GC supply fittings.
- **For GC leak points**, refer to the GC user documentation.
- **HS leak points** include the connections at the six port valve (sample loop and transfer line), the transfer line connection to the GC inlet, and the connections for the sampling probe.

### WARNING

**Hydrogen (H<sub>2</sub>) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, a flow meter). Purge flow meters with inert gas as needed. Always measure gases individually.**

---

### WARNING

**Hazardous sample may be present.**

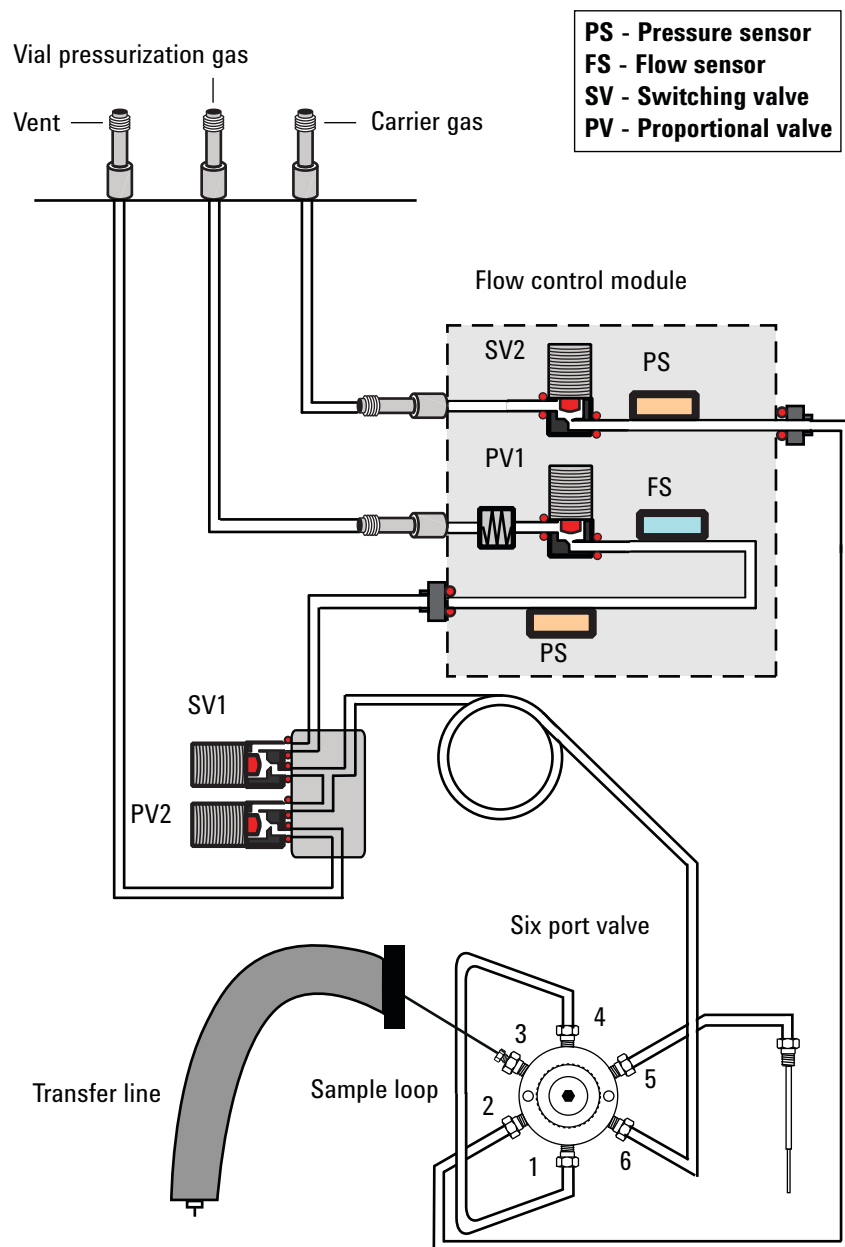
---

- 1 Gather the following:
  - Electronic leak detector capable of detecting the gas type (Agilent part number G3388A).
  - 7/16-inch, 9/16-inch, and 1/4-inch wrenches for tightening Swagelok and column fittings.
  - Leak test kit, Agilent part number G4556-67010. Includes: no hole ferrule, 11 mm low bleed septa, headspace (blue) leak test vial, 1/8-in. nylon tube fitting plug, 1/16-in. stainless steel ZDV plug (six port valve cap).
- 2 Check any potential leak points associated with any recent maintenance.
- 3 Check for external leaks. See ["Checking for External Leaks"](#).
- 4 Check the GC for leaks. See the GC user documentation.
- 5 Check HS fittings and connections that undergo thermal cycling, since thermal cycling tends to loosen some fitting types. Use the electronic leak detector to determine if a fitting is leaking.
  - Start by checking any newly made connections first.
  - Remember to check connections in the gas supply lines after changing traps or supply cylinders.

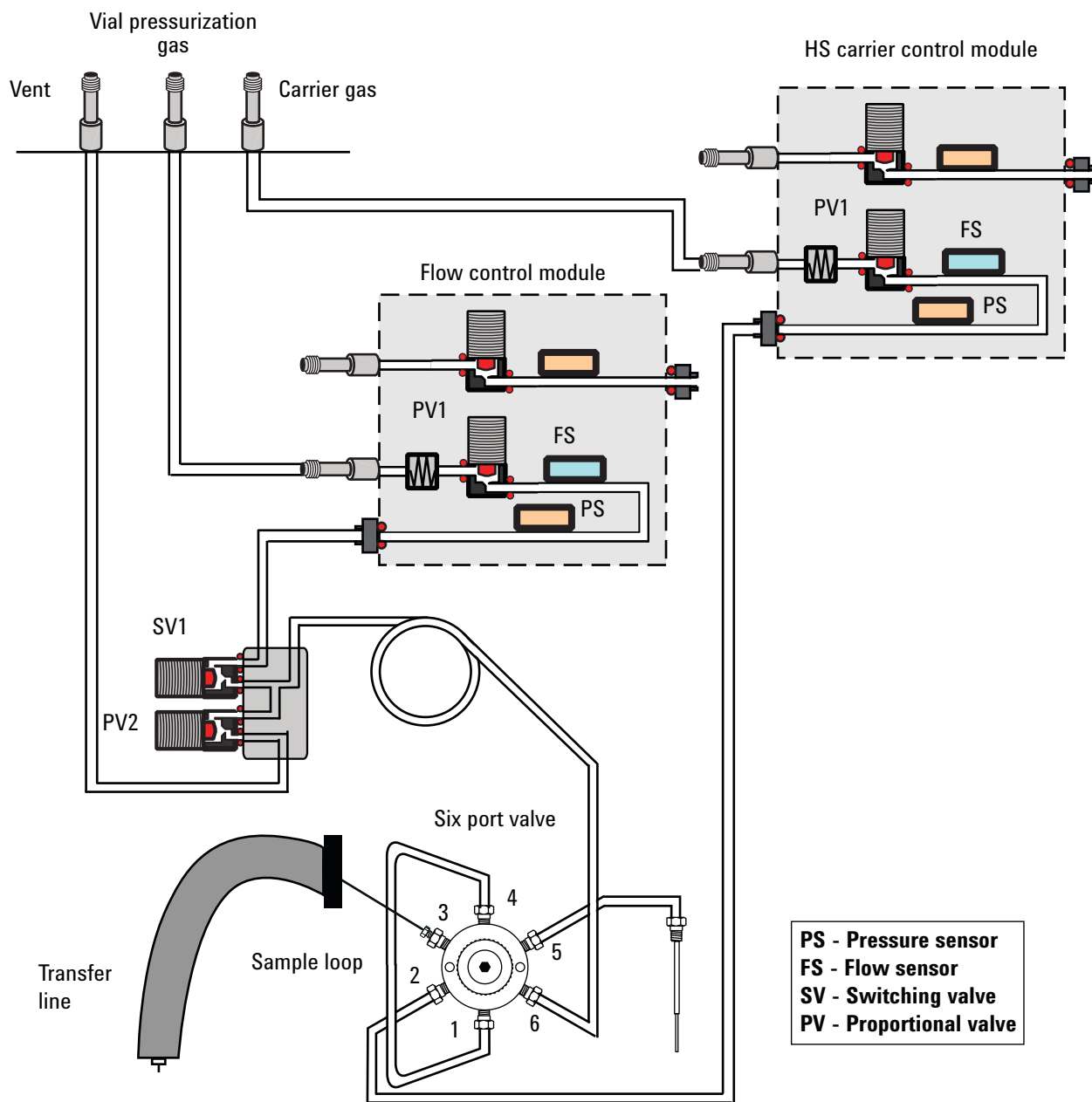
- 6 Run the HS **Restriction & pressure decay** test. See [To Run the Restriction and Pressure Decay Test](#).
- 7 Run the HS **Cross port leak test**. See [To Run the Cross Port Leak Test](#).

## Flow Paths

Figure 1 and Figure 2 below show the flow paths within the headspace sampler (HS).



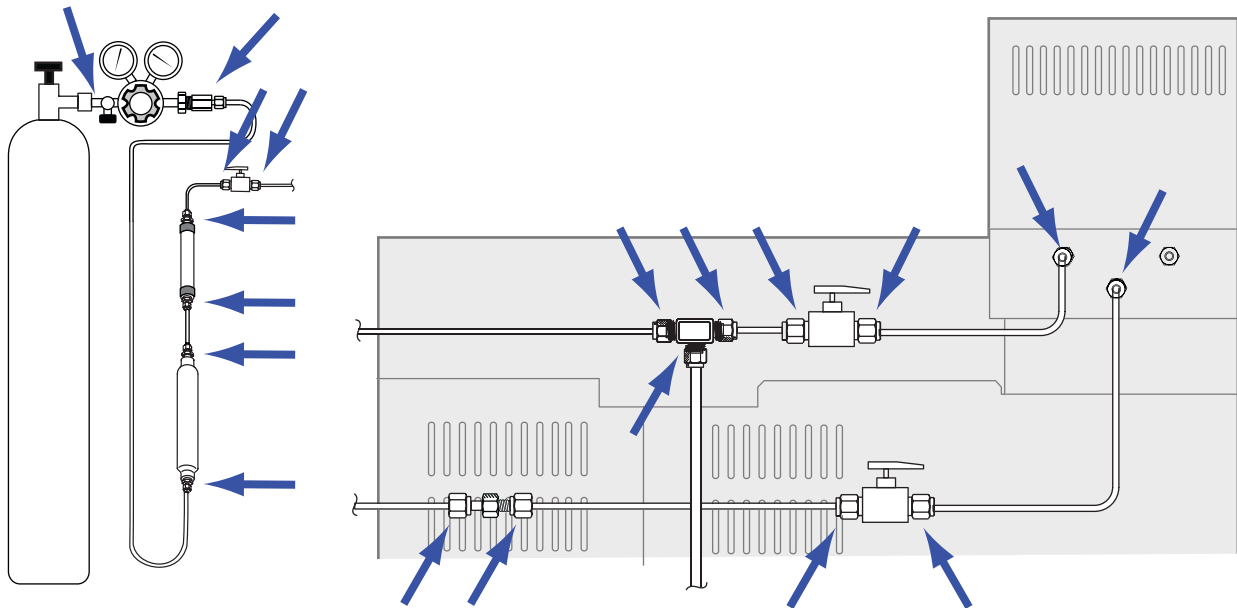
**Figure 1** HS flow paths (standard installation)



**Figure 2** HS flow paths with optional G4562A 7697A Carrier Gas EPC Module Accessory installed

## Checking for External Leaks

Figure 3 below shows typical external leak points to check. Check all fittings and correct as needed.



**Figure 3** External leak points (HC configured with GC carrier gas control, with optional valves and tee fittings shown)

- Gas supply bulkhead fittings
- Gas cylinder fittings
- Regulator fittings
- Traps
- Shut-off valves (if installed, not included in ship kit)
- Unions and tee fittings (if installed, not included in ship kit)
- Transfer line connection to the GC

Perform a pressure drop test.

### CAUTION

A pressure decay test cuts off carrier flow through the HS and GC. To prevent damage to the flow path components (column, inlet liner, and so forth), cool the HS and GC before beginning.

- 1 Cool the HS sample loop, transfer line, and GC inlet and column oven to prevent damage during the test.
- 2 Turn off the HS.
- 3 Set the regulator pressure to 415 kPa (60 psi).
- 4 Fully turn the regulator pressure adjustment knob counterclockwise to shut the valve.
- 5 Wait 5 min. If there is a measurable drop in pressure, there is a leak in the external connections. No drop in pressure indicates that the external connections are not leaking.

## To Run the Restriction and Pressure Decay Test

The built-in restriction and pressure decay test sequentially checks for restrictions and leaks in the sampling system. The test checks for:

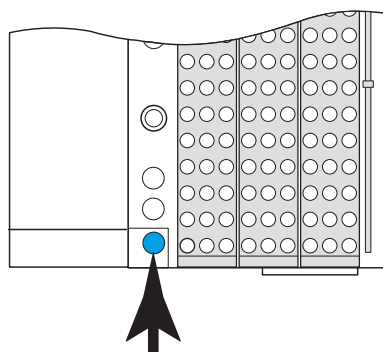
- Restrictions in the sample probe
- Restrictions in the vent line
- Leaks around the sample probe
- Leaks around the sample loop
- Leaks in valves and in the six port valve

The test consists of several parts. If one part fails, the test reports the failure and stops before attempting any subsequent parts. Correct the problem, then re-run the test. Continue correcting problems and retesting until the test completes successfully.

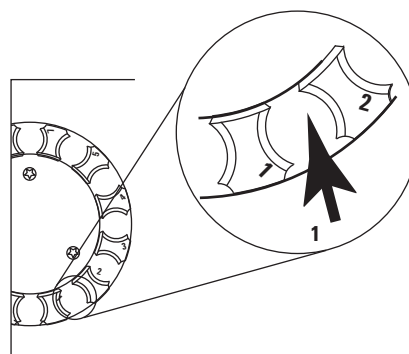
Before running the built-in leak check, check the GC for leaks. See "[Leak Check General Procedure](#)" and the GC user documentation.

- 1 Gather the following:
  - Leak test kit (G4556-67010). Includes: six port valve cap (G6600-80039), no hole ferrule (5181-7458), blue leak test vial (G1290-20600), 1/8-inch Nylon Swagelok plug (0100-2414)
  - GC liner, O-ring, and septum as needed
  - 3/16-inch wrench
  - 7/16-inch wrench
  - 1/4-inch wrench
  - 5/16-inch wrench
- 2 If unsure of the quality of the GC inlet septum, liner, and liner O-ring, change them now. Refer to the GC user documentation for details.
- 3 Set the active method vial size to 20 mL. Press [**Vial**], scroll to **Vial size**, press [**Mode/Type**] to select the vial size from a list, then press [**Enter**].
- 4 Install a new septum into the blue leak test vial.
- 5 Place the blue leak test vial into priority position 1 (111 vial model) or into tray position 1 (12 vial model).





G4556A



G4557A

- 6 Set the carrier pressure to a low value.

If using HS Control or GC Control:

- a Cool the GC inlet and column oven.
- b When cool, press [**Carrier**].
- c Set the carrier pressure setpoint to 14 kPa (2 psi). (If in a flow mode, reduce the flow setpoint until the carrier pressure is below 14 kPa (2 psi).)

If using GC + HS Control:

- Press [**Carrier**], then reduce the flow setpoint until the carrier pressure is below 14 kPa (2 psi).
- Leave the GC carrier pressure or flow unchanged.

- 7 Start the test. Press [**Service Mode**], select **Restriction & pressure decay**, then press [**Enter**].

The test begins.

The test displays information for the current action (for example, "Purging," "Zeroing Pressure Sensor," or "Moving Test Vial," and the pressure and flow readings).

See "If the test passes" or "If the test fails".

- 8 After the test passes, restore the instrument to operating condition.

## If the test passes

If the test passes all stages, the sampling system is leak free. If still experiencing leak-like symptoms:

- Check the transfer line.
- Check the interface to the GC.
- If not already done, check the GC. The HS leak test cannot test for leaks in the GC.

## If the test fails

If the test fails, the display provides:

- The reading for the stage that failed (for example, leak rate or flow rate).
- Command lines to toggle related valves (for example, a switching valve or the six port valve).

**SV1, SV2, PV1, or PV2:** Scroll to this line and press [**On/Yes**] to turn on (energize) the valve, or [**Off/No**] to turn it off.

**Six-port valve:** Scroll to this line and press [**On/Yes**] to switch the valve to the load position, or [**Off/No**] to switch it to the inject position.

- A selection to exit the test.

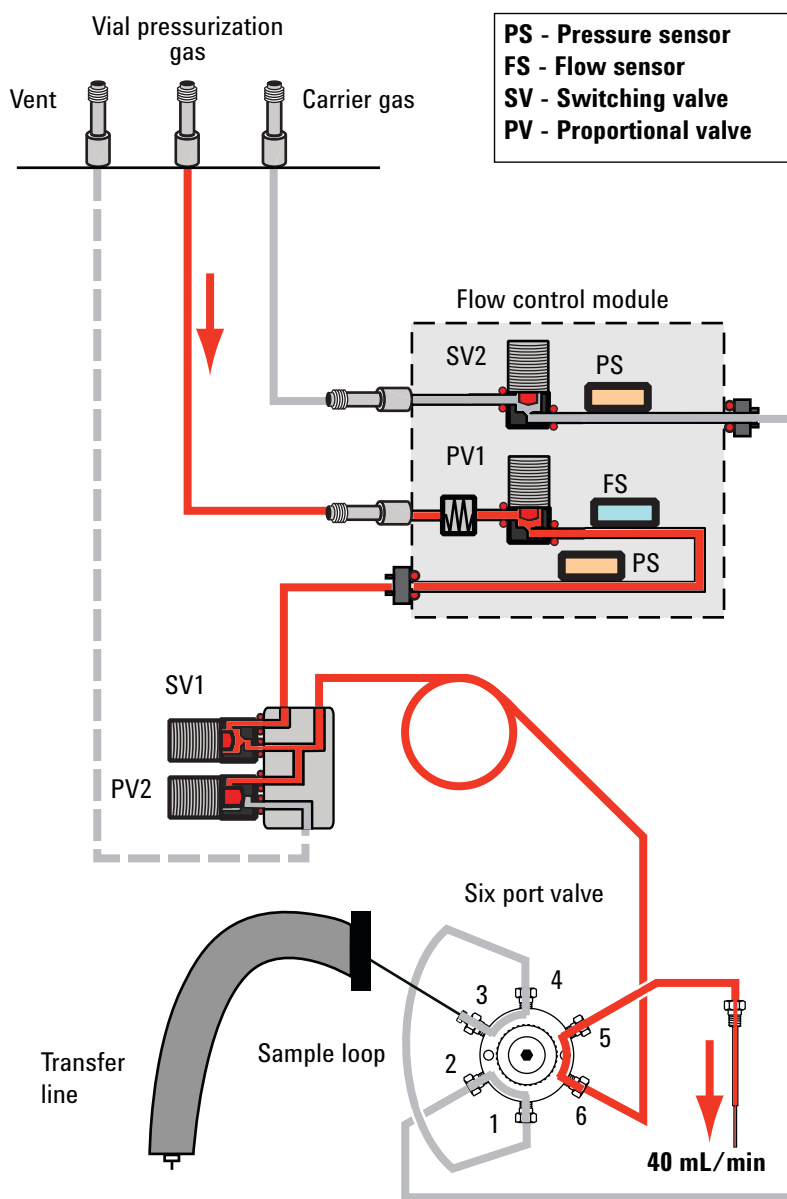
**Exit test?:** Select this line then press [**On/Yes**] to abort the test.

- A failure code.

See the sections below for troubleshooting information for each stage of the test.

**Needle (Sample Probe) restriction test, stage 1: Vent CLOSED (INJECT)**

During this stage, the HS flow paths are as shown in [Figure 4](#):



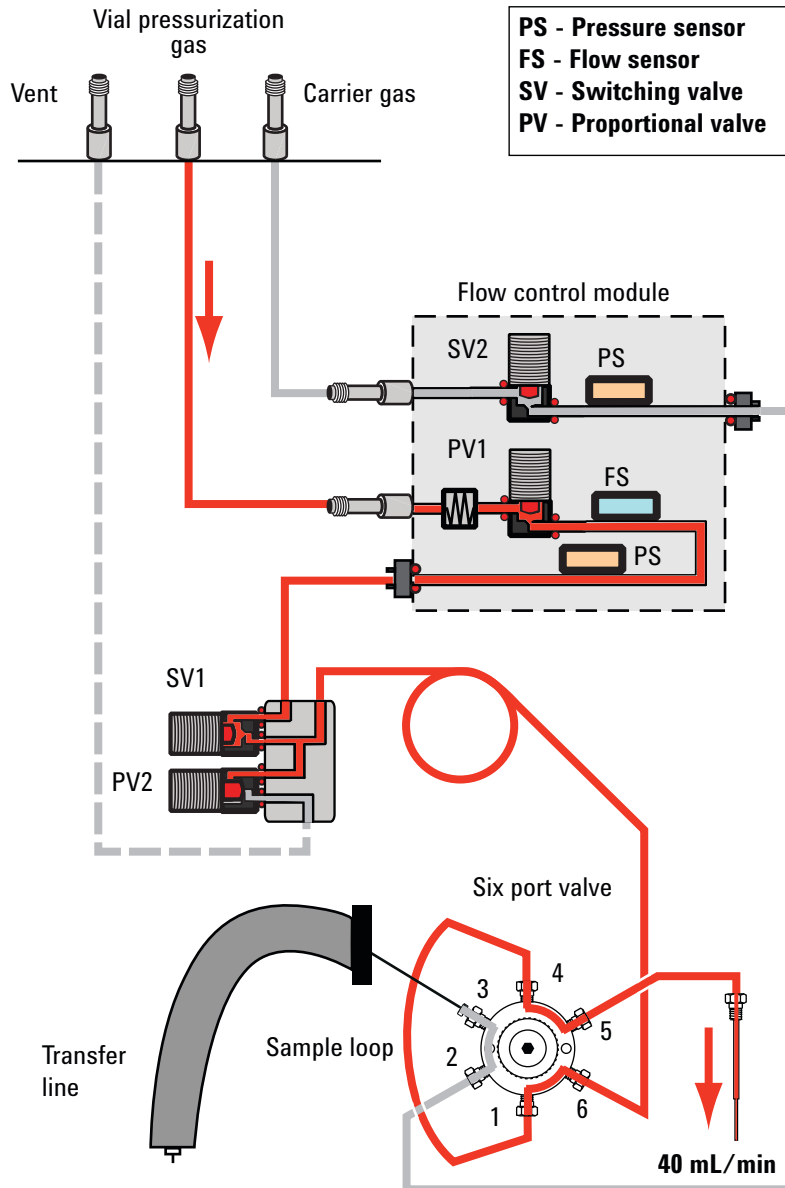
**Figure 4** Flow paths: Needle restriction test, stage 1: Vent CLOSED (INJECT)

In this stage, the displayed pressure reading should stabilize at some value. This is your baseline value. If the test fails this stage, check:

- The sample probe
- A leak causing no restriction value

**Needle (Sample Probe) restriction test, stage 2: Vent CLOSED (STANDBY)**

During this stage, the HS flow paths are as shown in [Figure 5](#):



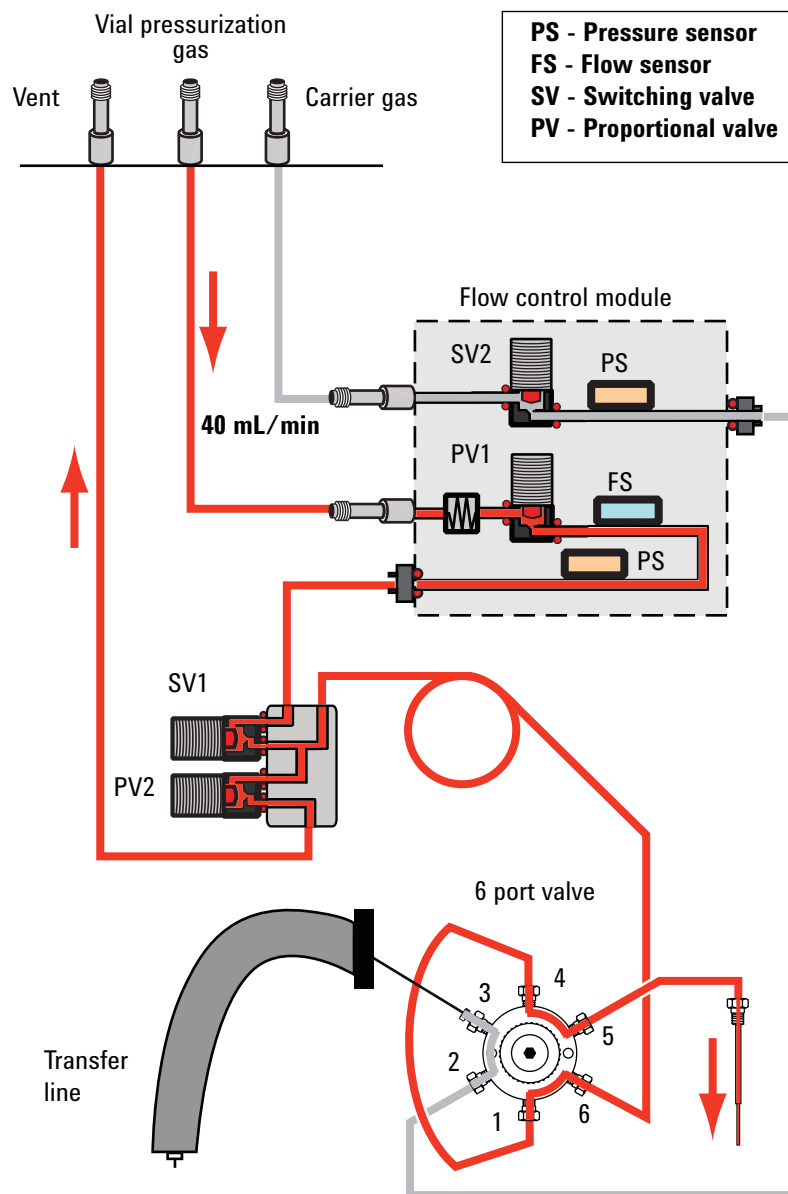
**Figure 5** Flow paths: Needle restriction test, stage 2: Vent CLOSED (STANDBY)

In this stage, the pressure should stabilize at a higher value than in stage 1, depending on your sample loop size. If the test fails this stage, check:

- The sample probe
- The sample loop or sample loop configuration

### Needle (Sample Probe) restriction test, stage 3: Vent OPEN

During this stage, the HS flow paths are as shown in [Figure 6](#):



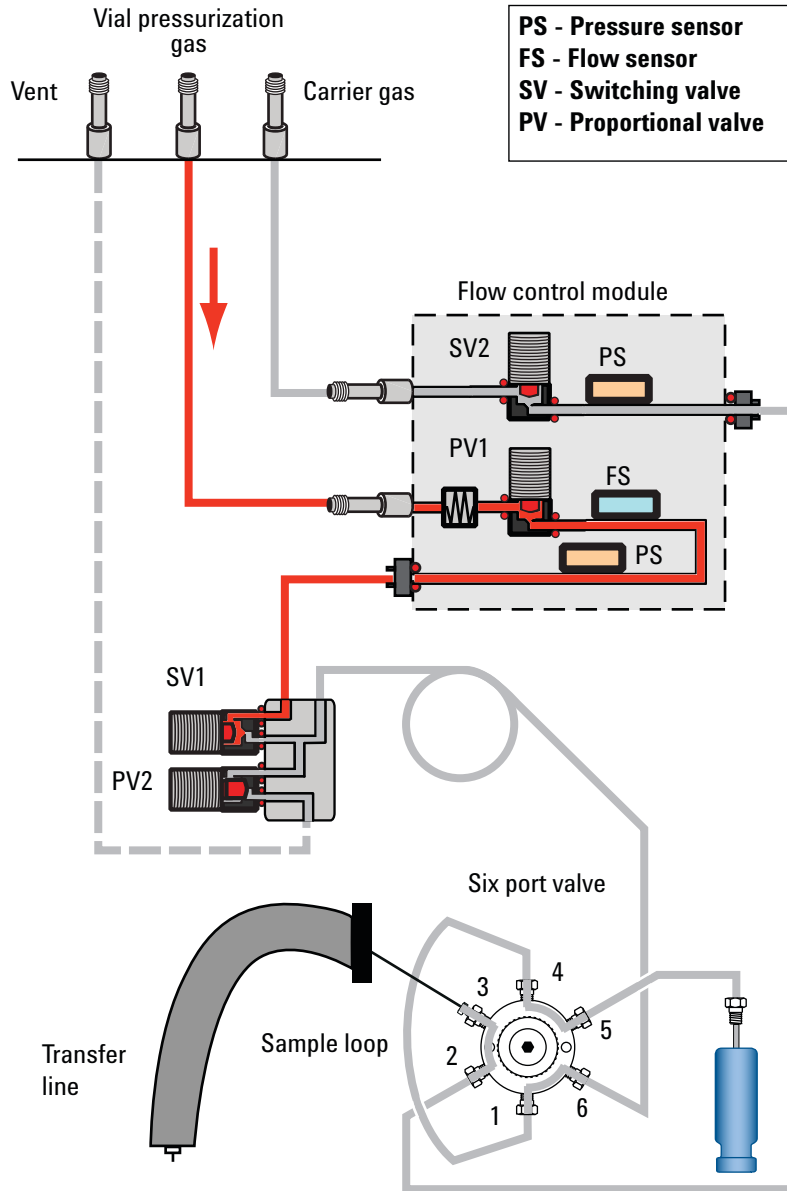
**Figure 6** Flow paths: Needle restriction test, stage 3: Vent OPEN

In this stage, the pressure should drop to a very low value since the vent is open. If the test fails this stage, check:

- The vent line
- The vent valve (contact Agilent for service)

**Vial leak test, stage 1: Test up to SV1**

During this stage, the HS flow paths are as shown in [Figure 7](#):



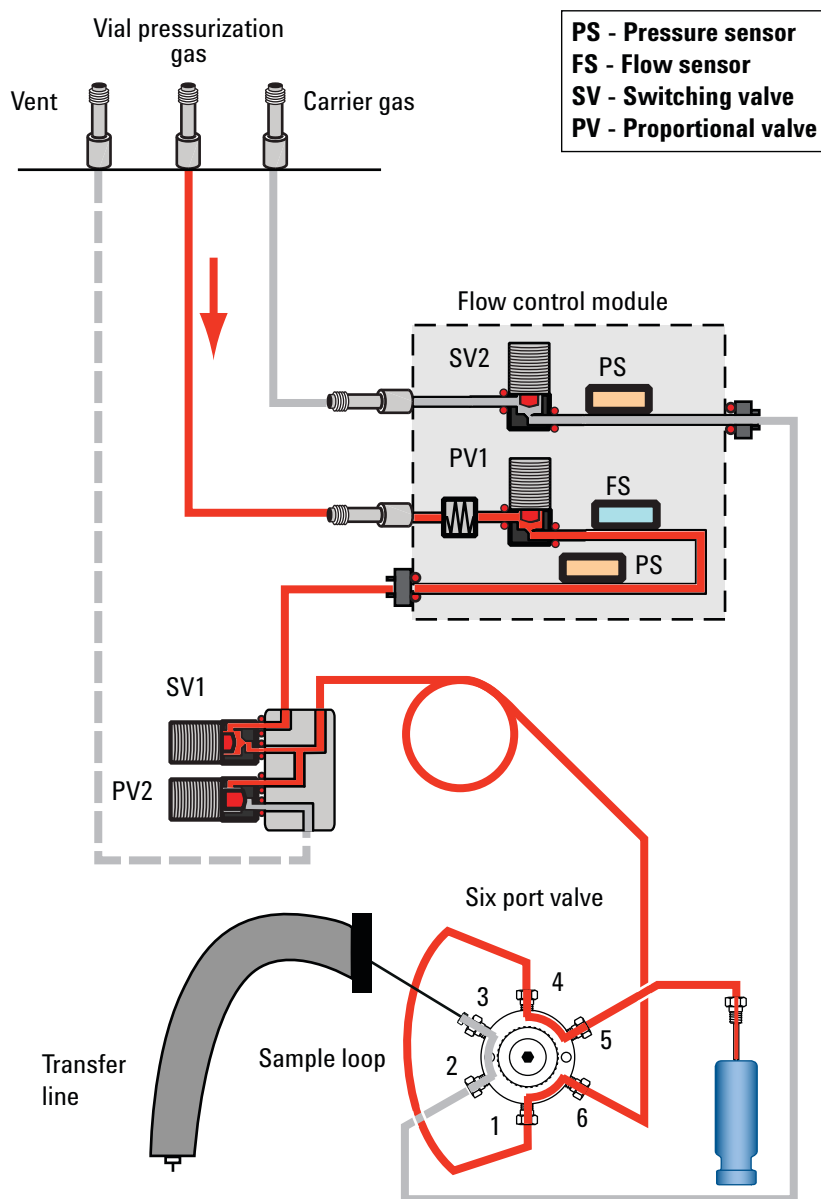
**Figure 7** Flow paths: Vial leak test, stage 1: Test up to SV1

If the test fails this stage, check:

- SV1, the pressurization valve (contact Agilent for service)
- The O-ring seats on the PCM module connection (contact Agilent for service)

**Vial leak test, stage 2: Test 6PORT (STANDBY)**

During this stage, the HS flow paths are as shown in [Figure 8](#):



**Figure 8** Flow paths: Vial leak test, stage 2: Test 6PORT (STANDBY)

If the test fails this stage, check:

- The vent valve. Cap the vent line and retest. If the test passes, contact Agilent for service. If the test fails again, the vent valve is operating properly.
- The sample probe connections to the six port valve

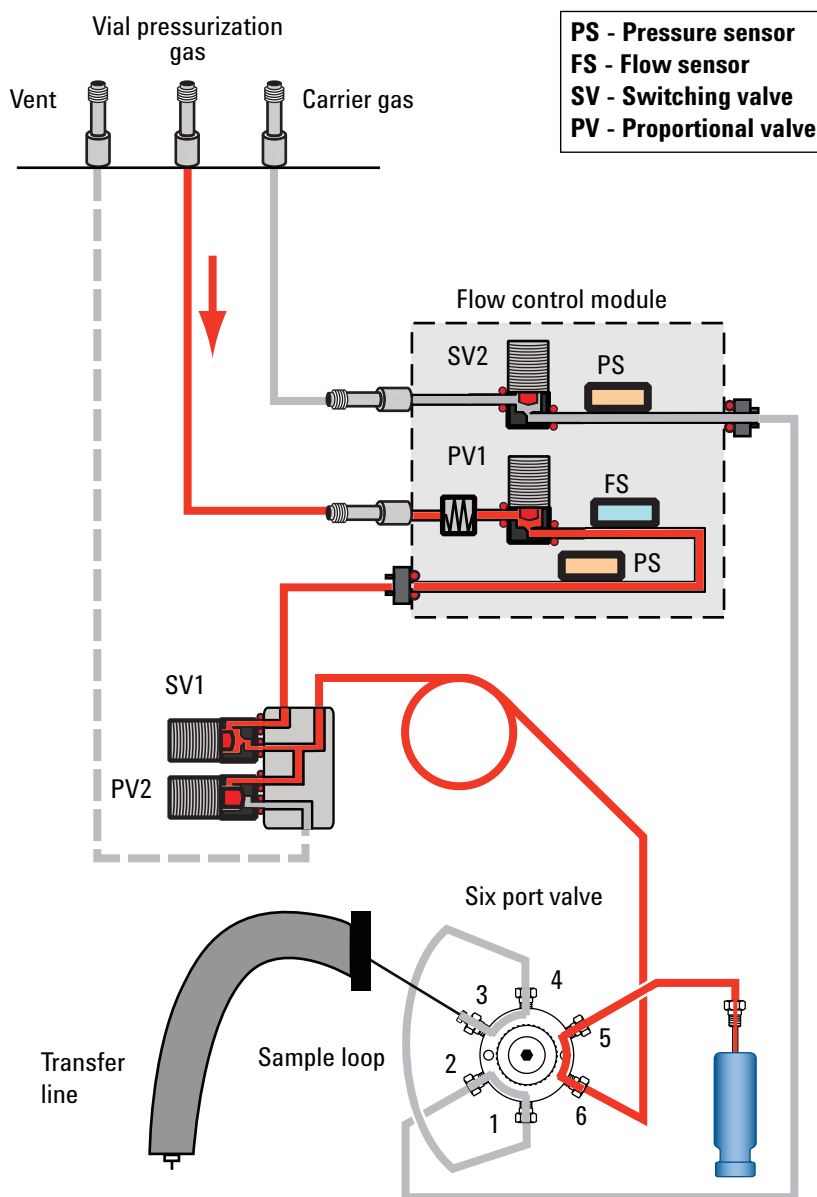
## Agilent 7697A Headspace Sampler

- The sample loop connections to the six port valve
- Port 6 on the six port valve



### Vial leak test, stage 3: Test 6PORT (INJECT)

During this stage, the HS flow paths are as shown in [Figure 9](#):



**Figure 9** Flow paths: Vial leak test, stage 3: Test 6PORT (INJECT)

If the test fails this stage, check:

- The sample probe connections to the six port valve
- Port 6 on the six port valve

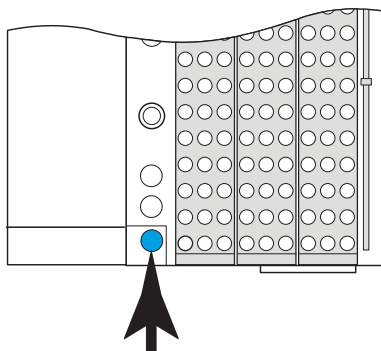
## To Run the Cross Port Leak Test

Run the cross port leak test immediately after the HS passes the restriction and pressure decay test. See ["To Run the Restriction and Pressure Decay Test"](#). These instructions assume that the HS is already prepared and that the GC is leak-free.

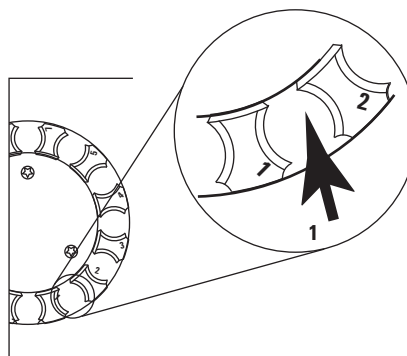
The built-in leak test sequentially checks for leaks across the six port valve, from the carrier side to the vial pressurization side.

The test consists of two parts. If one part fails, the test reports the failure and stops before attempting any subsequent parts. Correct the problem, then re-run the test. Continue correcting problems and retesting until the test completes successfully.

- 1 Install a new septum into the blue leak test vial.
- 2 Place the blue leak test vial into priority position 1 (111 vial model) or into tray position 1 (12 vial model).



G4556A



G4557A

- 3 Set the carrier pressure to 172 kPa (25 psi).

If using GC + HS Control:

- Press [**Carrier**], then increase the flow setpoint until the carrier pressure is 172 kPa (25 psi).
- Leave the GC carrier pressure or flow unchanged.

- 4 Start the test. Press [**Service Mode**], select **Cross port leak test**, then press [**Enter**].

The test begins.

The test displays information for the current activity (for example, venting or moving the test vial, and the pressure and calculated leak rates).

To stop the test, press [**Clear**] during a purge step (monitor the HS display). Otherwise, the test continues until it passes or fails.

See "If the test passes" or "If the test fails".

- 5 After the test passes, restore the instrument to operating condition.

### If the test passes

If the test passes all stages, the sampling system is leak free. If still experiencing leak-like symptoms:

- Check the transfer line.
- Check the interface to the GC.
- If not already done, check the GC. The HS leak test cannot test for leaks in the GC.

### If the test fails

If the test fails, the display provides:

- The reading for the stage that failed (for example, leak rate or flow rate).
- Command lines to toggle related valves (for example, a switching valve or the six port valve).

**SV1, SV2, PV1, or PV2:** Scroll to this line and press [**On/Yes**] to turn on (energize) the valve, or [**Off/No**] to turn it off.

**Six-port valve:** Scroll to this line and press [**On/Yes**] to switch the valve to the load position, or [**Off/No**] to switch it to the inject position.

- A selection to exit the test.

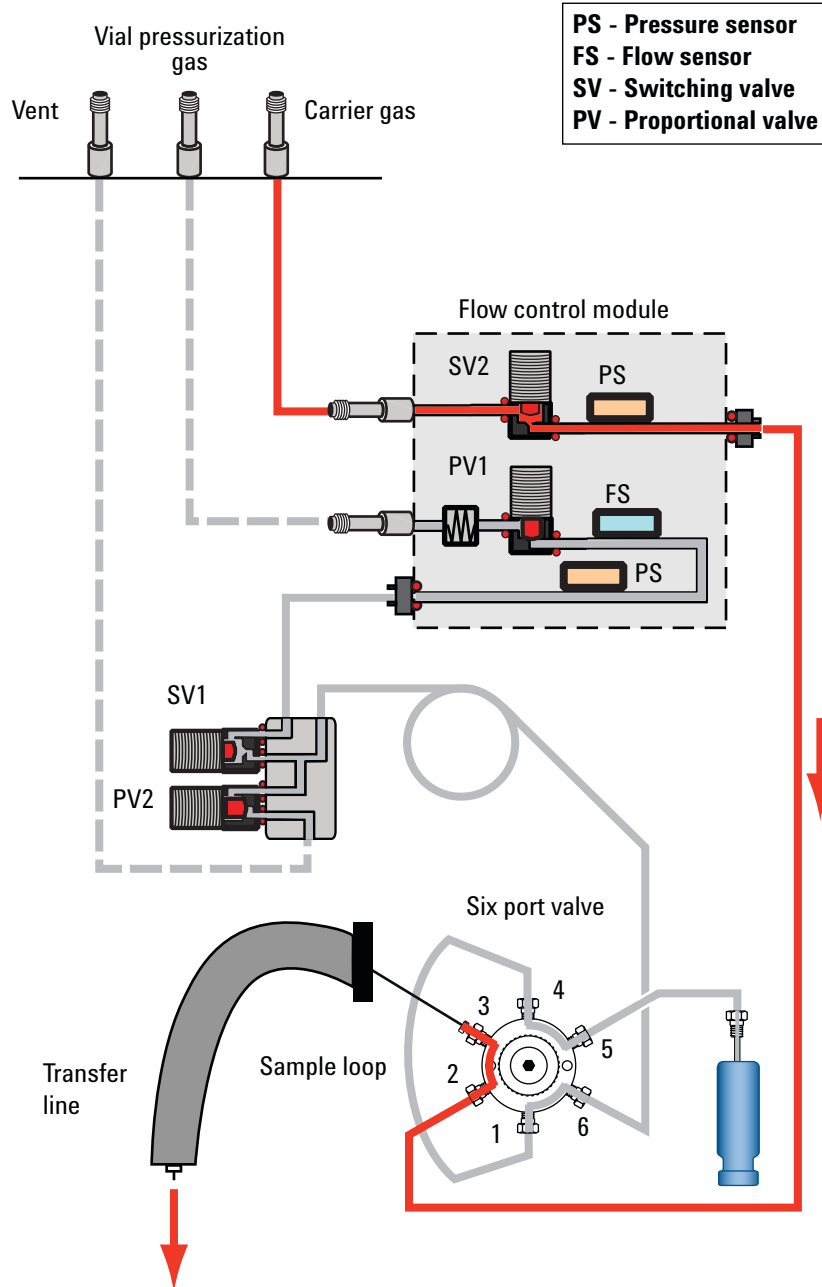
**Exit test?:** Select this line then press [**On/Yes**] to abort the test.

- A failure code

See the sections below for troubleshooting information for each stage of the test.

**Cross port leak test, stage 1: Test 6PORT (STANDBY)**

During this stage, the HS flow paths are as shown in **Figure 10**:



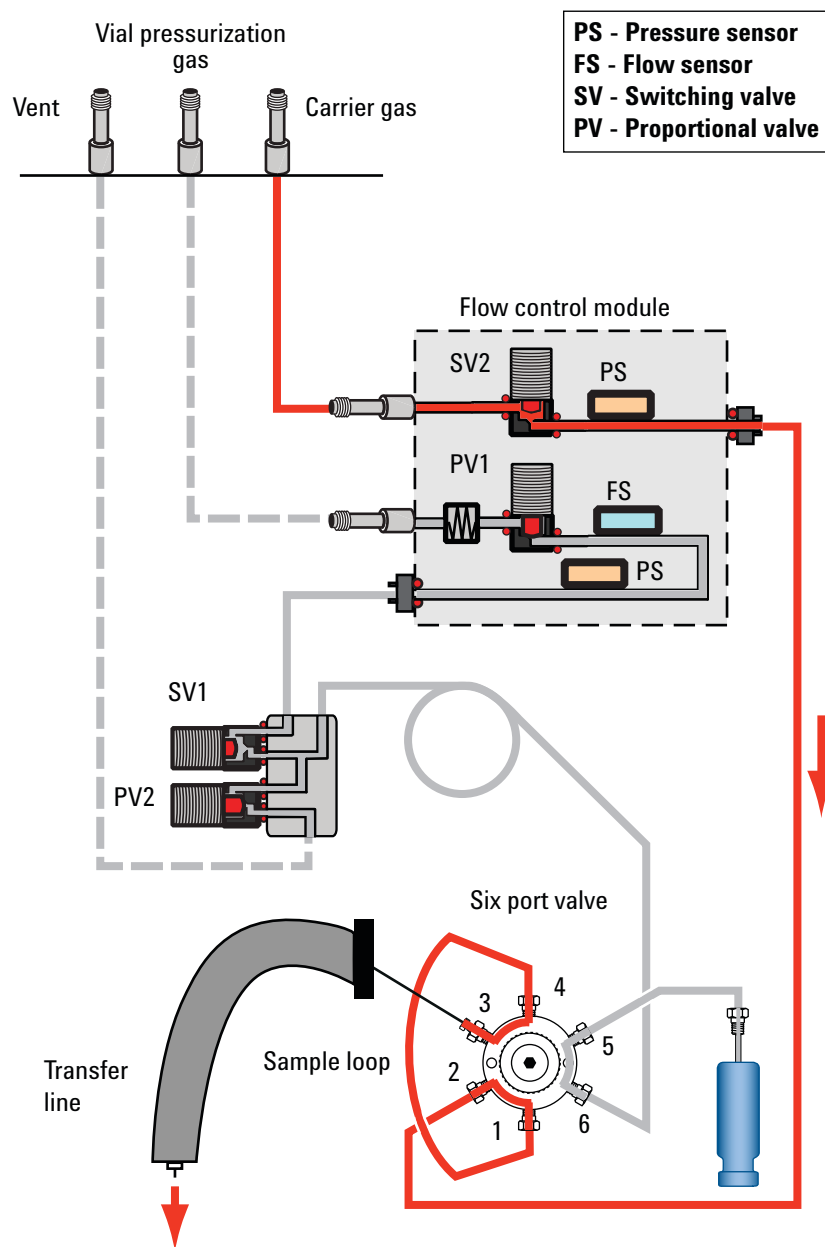
**Figure 10** Flow paths: Cross port leak test, stage 1: Test 6PORT (STANDBY)

If the test fails this stage, check:

- A leak across six port valve ports 3 and 4, or 1 and 2. Replace the valve rotor.

**Cross port leak test, stage 2: Test 6PORT (INJECT)**

During this stage, the HS flow paths are as shown in [Figure 11](#):



**Figure 11** Flow paths: Cross port leak test, stage 2: Test 6PORT (INJECT)

If the test fails this stage, check:

- A leak across six port valve ports 4 and 5, or 1 and 6. Replace the valve rotor.

## To Check for Leaks in the Transfer Line

- Check for leaks at the six port valve and interface connection to the GC.
- Check for flow out of the transfer line fused silica. No flow indicates broken fused silica.
- Also consider other leaks in the GC inlet (liner, O-ring, septum and so forth).

### Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

© Agilent Technologies, Inc. 2012  
Printed in USA and China

Second edition, January 2012  
Agilent Technologies, Inc.  
2850 Centerville Road  
Wilmington, DE 19808-1610 USA

安捷伦科技（上海）有限公司  
上海市浦东新区外高桥保税区  
英伦路 412 号  
联系电话：（800）820 3278



G4556-90001