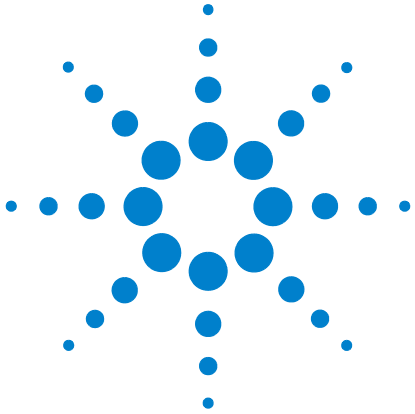


Agilent 7697A Headspace Sampler

Installation and First Startup

Agilent Technologies

Notices

Manual Part Number

G4556-90013

Edition

Fourth edition, May 2012
Third edition, February 2012
Second edition, November 2011
First edition, January 2011

Printed in USA

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

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.

Safety Notices

CAUTION

A CAUTION notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Contents

1 Installation

This guide describes how to install the Agilent 7697A Headspace Sampler (HS). This guide assumes that the GC is installed, configured, and operating properly, along with any associated data system.

This chapter describes the installation steps to unpack and install the instrument. The next chapter describes how to start up the HS for the first time and verify it is operating properly.

Before You Begin

Before installing the headspace sampler (HS), review the information below.

Tools required for installation

The following tools and parts are needed for installation.

Table 1 Tools and parts for installation

Tool	Agilent part number	Use
Required		
Torx T-20 driver	5182-3465	Transfer line installation
Open-end wrenches: • 7/16-inch (2) • 5/16-inch	 8710-0972 8710-0510	Swagelok fittings for gas connections, sample loop, and transfer line
Tubing cutter	8710-1709	Cut 1/8-inch gas supply plumbing
Precision tubing cutter	5190-1442	Cut 1/16-inch stainless steel tubing
Included		
1/4-in. x 3/16-in. wrench	8710-2618	Transfer line installation
Column cutting wafer	5181-7487	Transfer line installation

The other necessary parts and tools are supplied with the new instrument.

Personnel needed for installation

The instrument is heavy. When lifting or moving the instrument, two persons are required.

Parts supplied with the instrument

The headspace sampler ships with consumables and hardware sufficient for typical installation and checkout. See Table 2 below.

Table 2 Parts supplied with the headspace sampler

Description	Part number	Quantity	Use
Wrench, open end 3/16-in. x 1/4-in.	8710-2618	1	Transfer line connection.
LAN cable	8121-0940	1	Communications.
Cable, APG remote, 9M/9M	G1530-60930	1	Communications.
Flat bottom headspace vials, 20 mL, 100/pk	5182-0837	1	Checkout.
Crimp cap, headspace, Al, PTFE/Si septum, 20 mm, 100/pk	5183-4477		Checkout.
Ergonomic manual crimper for 20 mm caps	5040-4669		Checkout.
Leak Test Kit	G4556-67010	1	
Vial Pressurization Connection Kit			
Tubing, post-drawn, 1650 mm	1530-2239	1	Connect HS **Vial Pressure** fitting to the gas supply.
Nut, stainless steel, for 1/16-in. tubing	0100-0053	1	Connect the 1/16-in. end of the post-drawn tubing to the HS **Vial Pressure** fitting.
Ferrule set, stainless steel, for 1/16-in. tubing	0100-1490	1	
Nut and ferrule set, 1/8-in. Swagelok, stainless steel	5181-7482	1	Connect the 1/8-in. end of the tubing to the gas supply.
Transfer Line Connection Kit			
Fused silica, deactivated, .530 mm id x 5 m long	160-2535-5	1	Transfer line.
Fitting, internal reducer, 1/16-in. to 1/32-in.	0100-2594	1	Transfer line connection.
Ferrule, polyamide graphite 1/32-in.	0100-2595	1	Transfer line connection.
Transfer line support assembly	G3504-60620	1	Support transfer line at GC inlet. Consists of bracket, clamp, and knurled thumbscrew.
Tubing, post-drawn, 1650 mm	1530-2239	1	Connect HS **Carrier** fitting to the gas supply from the GC inlet flow module.
Union SS 1/16-inch tubing	0100-0124	1	Connect the 1/16-in. end of the post-drawn tubing to the open end of the GC inlet carrier line.
Column cutting wafer	5181-7487	1	Trim fused silica transfer line.

Table 2 Parts supplied with the headspace sampler (continued)

Description	Part number	Quantity	Use
Cap, 1/16-in., stainless steel	0100-0050	1	Cap the cut end of the SSL or MMI inlet carrier tubing.
Nut and ferrule set, 1/8-in. Swagelok, stainless steel	5181-7482	1	Connect the 1/8-in. end of the post-drawn tubing to the HS.
Nut, stainless steel, for 1/16-in. tubing	0100-0053	1	Cap the cut end of the SSL or MMI inlet carrier tubing.
Ferrule set, stainless steel, for 1/16-in. tubing	0100-1490	1	
Septum retainer nut for transfer line	G3452-60835	1	Support transfer line on GC SSL or MMI inlet
Liner, Direct Connect, 2 mm ID, deactivated	5181-8818	1	GC SSL or MM inlet liner for checkout.
O-Ring, 2-010, Fluoroelastomer, 10/pk	5188-5365	1	GC SSL or MM inlet liner O-ring.
Septa, 11 mm low bleed, 5/pk	5182-3413	1	GC SSL or MM inlet septum (for checkout).

Verify site preparation

Verify that the site has been prepared according to the site preparation manual. Sufficient bench space, power supply receptacles, supplies, gases, traps, required accessories, and so forth must be available.

Prepare the GC and data system

If not already installed, install the gas chromatograph and verify its performance before beginning HS installation.

- Install the GC.
- Install the data system.
- Verify GC and data system performance.
- Cool all heated zones in the GC to safe handling temperatures, especially the column oven and the inlet. HS installation requires carrier gas flow interruption, which can damage a hot column. HS installation requires contact with the GC inlet, which can be hot enough to cause burns.
- If you purchased the add-on HS control software, install it after installing the data system.

Determine how you will connect the headspace sampler to the GC

How you connect the headspace sampler to the GC depends on:

- The GC model
- The GC inlet type
- Optional HS accessories
- Whether or not you need to also use this same GC inlet for other injection types (ALS or manual, for example)

See Table 3. Determine the connection best suited to your hardware and needs.

Table 3 Supported headspace sampler–GC connections and control modes

Inlet type	GC models	Connection type	Supports HS carrier mode(s)	Comments
Recommended configurations				
Split/ splitless, Multimode	7890, 7820, 6890, 6850, 5975T	Transfer line through septum head	• GC controls carrier flow • GC + HS (additive)*	• Standard configuration. • Can be disconnected to support autosampler or manual injection. • With GC control of carrier flow, HS must be On for non-HS injections. • Supports frequent column changes. Optional hardware: • G4562A Carrier Gas EPC Module Accessory. • G3521A 7890A S/SL MMI Weldment for Headspace (7890A, 5975T only).

* Requires G4562A Carrier Gas EPC Module Accessory

Table 3 Supported headspace sampler–GC connections and control modes (continued)

Inlet type	GC models	Connection type	Supports HS carrier mode(s)	Comments
Split/ splitless, Multimode	7890	G3520A Transfer Line Interface Accessory	GC controls carrier flow	• Allows alternating HS and ALS usage, without hardware changes. • Transfer line bypasses septum, reducing cross-contamination between runs. • Design reduces cold spots, improves performance for samples with analytes such as high weight alkenes (C40). • Best performance for widest range of analyses. • Supports frequent column changes. • Reduces potential for leaks. • With GC control of carrier flow, HS must be On for non-HS injections. Required hardware: • G3520A Transfer Line Interface Accessory. • 1/16-inch stainless steel tubing. Optional hardware: • G4562A Carrier Gas EPC Module Accessory.
Volatiles interface	7890, 6890	Transfer line direct to volatiles interface	GC controls carrier flow	• No support for additive flow. • High sensitivity. • Allows small volume injections. • Most inert choice. • Not recommended for frequent column changes. • Does not support ALS or manual injections. Required hardware: • Strain relief adapter.
Optional configurations				
Split/ splitless, Multimode	7890, 7820, 6890, 6850, 5975T	Transfer line through septum head	• HS controls carrier flow* • GC + HS (additive)*	• Can be disconnected to support autosampler or manual injection. • Carrier flow control not as robust as control available using GC. Required hardware: • G4562A Carrier Gas EPC Module Accessory.

* Requires G4562A Carrier Gas EPC Module Accessory

Table 3 Supported headspace sampler–GC connections and control modes (continued)

Inlet type	GC models	Connection type	Supports HS carrier mode(s)	Comments
Purged packed	7890, 7820, 6890, 6850	Transfer line through septum head	GC + HS (additive)*	**Purged packed inlet is not recommended for HS use.** The flow rate through the transfer line needs to be high, which broadens peaks. If used, set only a small flow from the PP inlet (to sweep the inlet septum). Provide the main column flow using the HS additive flow. • Supports frequent column changes. • Can be disconnected to support autosampler or manual injection. • Users will need to experiment with flows to optimize the analysis. Required hardware: • G4562A Carrier Gas EPC Module Accessory.
Cool on-column	7890, 6890, 6850	Transfer line through septum head	GC controls carrier flow	**Cool on-column inlet is not recommended for HS use.** For most analytes, instead use a SSL or MMI and run the column up and through the transfer line to the HS 6 port valve. (Use of columns > 530 µm is not recommended in the transfer line. Larger id columns are not supported for this use.) If connecting the column directly to the HS sampling valve, for most analytes provide a small split flow so that the inlet provides a small amount to sweep septum, and let the HS provide most flow, for example 0.1:1. • The flow rate through the transfer line needs to be high, which broadens peaks. • Supports frequent column changes. • Can be disconnected to support autosampler or manual injection, but realignment of ALS or change of inlet hardware may be required. • Users will need to experiment with flows to optimize the analysis. • With the cool on-column inlet, the transfer line butts against column. This is not preferred. Required hardware: • G4562A Carrier Gas EPC Module Accessory. • Cool on-column inlet hardware for transfer line size.

* Requires G4562A Carrier Gas EPC Module Accessory

Table 3 Supported headspace sampler–GC connections and control modes (continued)

Inlet type	GC models	Connection type	Supports HS carrier mode(s)	Comments
Any non-Agilent	any non-Agilent	User must determine connection method	• GC + HS (additive)* • HS controls carrier flow	• Determine best modes and use cases based on similar Agilent hardware. Required hardware: • G4562A Carrier Gas EPC Module Accessory.

* Requires G4562A Carrier Gas EPC Module Accessory

Install the GC consumables needed for HS checkout

HS checkout requires the GC consumables listed in Table 4. Install them before installing the HS. See your GC user documentation for details.

Table 4 GC consumables needed for HS performance verification

Description	Part number
GC checkout column:	
7890, 7820, 6890:	19091J-413
6850:	19091J-413E
If using MS or MSD:	
7890, 7820, 6890:	19091S-433
6850:	19091S-433E
5975T:	G3900-63001
Helium carrier gas	
For split/splitless or multimode inlet	
Liner	5181-8818*
Liner O-ring	5188-6405*
If using FID or NPD	
Makeup gas	Nitrogen or helium
If using µECD	
Makeup gas	Nitrogen or argon/methane

*Supplied with the HS.

Configure the column and any other items, as needed.

Inspecting the Headspace Sampler

WARNING **The instrument is heavy. Use extreme caution when handling the instrument. A two person lift is recommended. Failure to perform a two person lift may result in personal injury.**

Inspect the headspace sampler (HS) shipping container(s).

1 Inspect the shipping containers for damage. If a container is damaged or shows signs of stress, notify both the carrier and your local Agilent office.

   Keep all shipping materials for inspection by the carrier.

2 Check the items received against the packing lists. If there are discrepancies, notify your local Agilent office immediately.

   Keep the shipping containers until you have checked their contents for completeness and verified instrument performance.

Place the Headspace Sampler on the Bench

The HS requires a benchtop that can support its weight plus that of other equipment you will use with it. The area must be free of overhanging obstructions that might interfere with cooling and limit access to the top of the instrument.

WARNING **The instrument is heavy. Use extreme caution when handling the instrument. A two person lift is recommended. Failure to perform a two person lift may result in personal injury.**

1 Remove the HS from its shipping box.

- Be careful when removing the packing foam from around the instrument. The foam also secures the transfer line.

2 Place the HS on the benchtop. Make sure gas and power supplies are accessible. Place other required equipment near the HS. **Install only on the right side of the GC.**

3 Remove caps and packing.

Protective caps cover gas input and vent fittings on the back of the unit.

Remove any packing tape that secures the transfer line against the instrument.

For the 12 vial model, lift the tray cover and remove any packing material from the tray.

CAUTION The tray gantry in secured only by the foam packing material. Be careful when unpacking the tray. The gantry can move when you lift the tray.

4 Unpack the tray, if purchased.

Check the tray for shipping tape and packing screws. Remove them. Packing screws will be labeled with a flag or ribbon for identification. Remove any tape or packing screws found.

Install the Configuration Plug

Before use, set the HS power configuration.

Verify the available power configuration

1 Locate the configuration plug that shipped with the headspace sampler. Compare the voltage shown on the configuration plug label with the laboratory line voltage. (For customers in Japan using 100 VAC, the unit is 120 VAC with an external 100 V/200 V step up transformer.) See Table 5.

Table 5 Headspace sampler power configuration plugs

Part number	Voltage	Color
G4556-60390	120 VAC	Brown
G4556-60400	200 VAC	Red
G4556-60410	220 VAC	Orange
G4556-60420	230 VAC	Yellow
G4556-60430	240 VAC	Violet

2 Locate the power cord that shipped with the headspace sampler. Verify the power cord is correct for the locale and voltage.

WARNING **Measuring the mains voltage can expose you to hazardous electrical current. Only a trained technician should perform this step.**

Measure the power outlet voltage and verify it meets the tolerance requirements listed in Table 6.

Table 6 Power consumption

Product	Line voltage (VAC)	Frequency (Hz)	Maximum continuous power consumption (VA)	Current rating (amps)	Power outlet current rating
111 Vial model	100*/120 single phase (–10% / +10%)	50/60	850	6.2	15 Amp
111 Vial model	200/220/230/240 single/split phase (–10% / +10%)	50/60	850	3.8/3.4/3.3/3.1	10 Amp
12 Vial model	100*/120 single phase (–10% / +10%)	50/60	850	6.2	15 Amp
12 Vial model	200/220/230/240 single/split phase (–10% / +10%)	50/60	850	3.8/3.4/3.3/3.1	10 Amp

* Users of 100 V power (Japan) receive the 120 V US power option with a 100 V/120 V step up transformer.

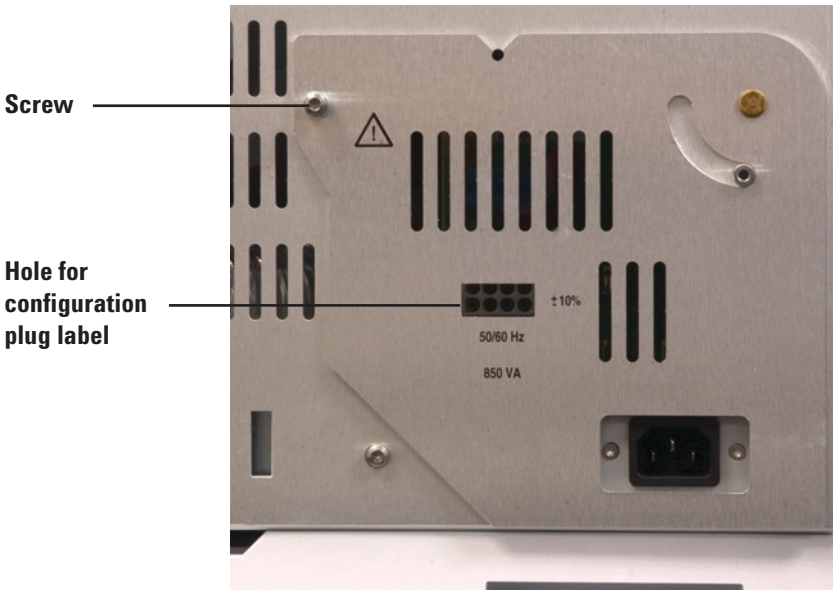
Install the configuration plug

Install the configuration plug as follows:

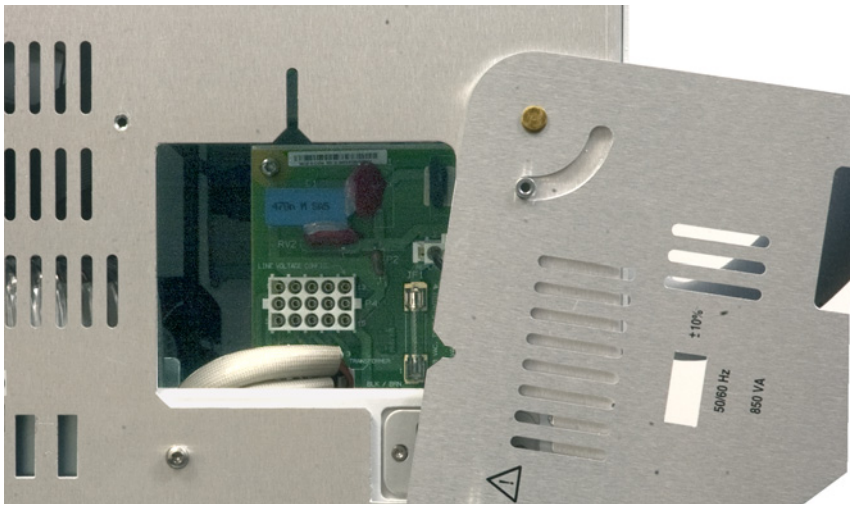
WARNING **Only an Agilent-trained technician should perform this procedure.**

CAUTION Components can be damaged by static electricity: be sure to wear an ESD strap grounded to the GC chassis while performing this procedure.

1 Locate the cover plate for the AC board on the lower back right corner of the HS.



2 Loosen the T-20 Torx screw in the cover plate and rotate the cover plate to expose the AC board.



3 Install the configuration plug into the AC board (connector P4). See Figure 1.

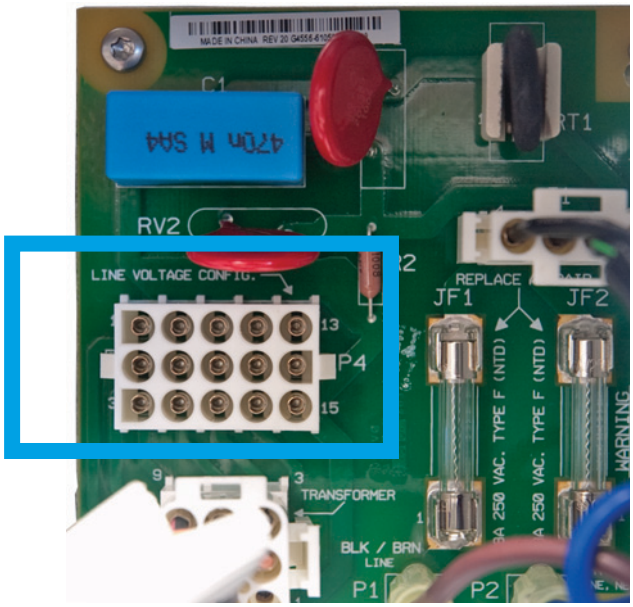


Figure 1 Close up of AC board

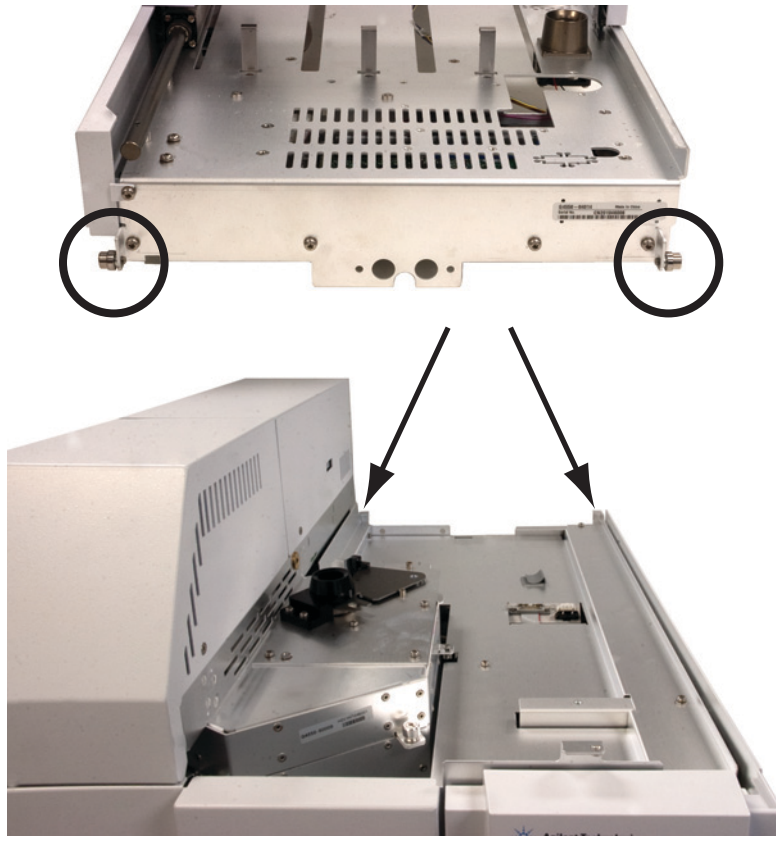
4 Rotate the cover plate until it covers the plug. Reinstall the screw to secure in place. The power rating label can be seen through the cover plate.

If the plate does not close, check the configuration plug.

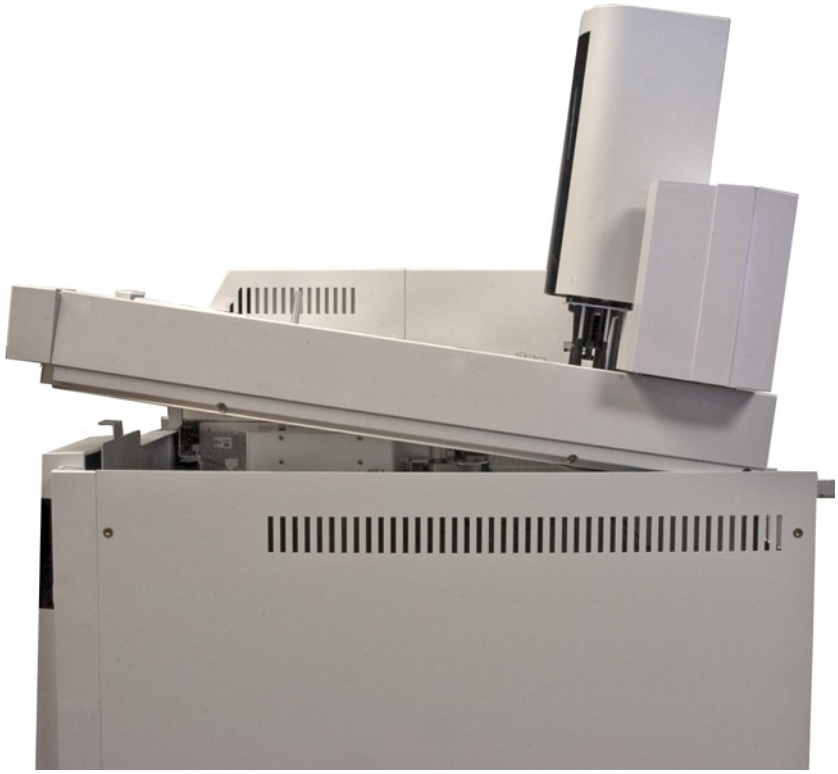
Install the Vial Tray (111 Vial Model Only)

If you did not purchase the 111 vial 7697A Headspace Sampler model, skip to the next section.

The headspace sampler tray fits on the top right side of the instrument. Two rollers at the back of the tray slide into mounting clips on top of the HS chassis.



1 Lift the tray and place onto the headspace sampler chassis so that the two rollers at the back of the tray rest on the HS chassis and the front of the tray is a few centimeters above the chassis.



2 Roll the tray back until the rollers engage the clips in the chassis. See Figure 2.

Figure 2 Placing the tray

3 Connect the tray control cables to the connectors in the chassis. See Figure 3. Route the control cables through the plastic clip on the chassis.

- Carefully remove the packing tape from the cables.
- When finished, check that the cables route through the clip.

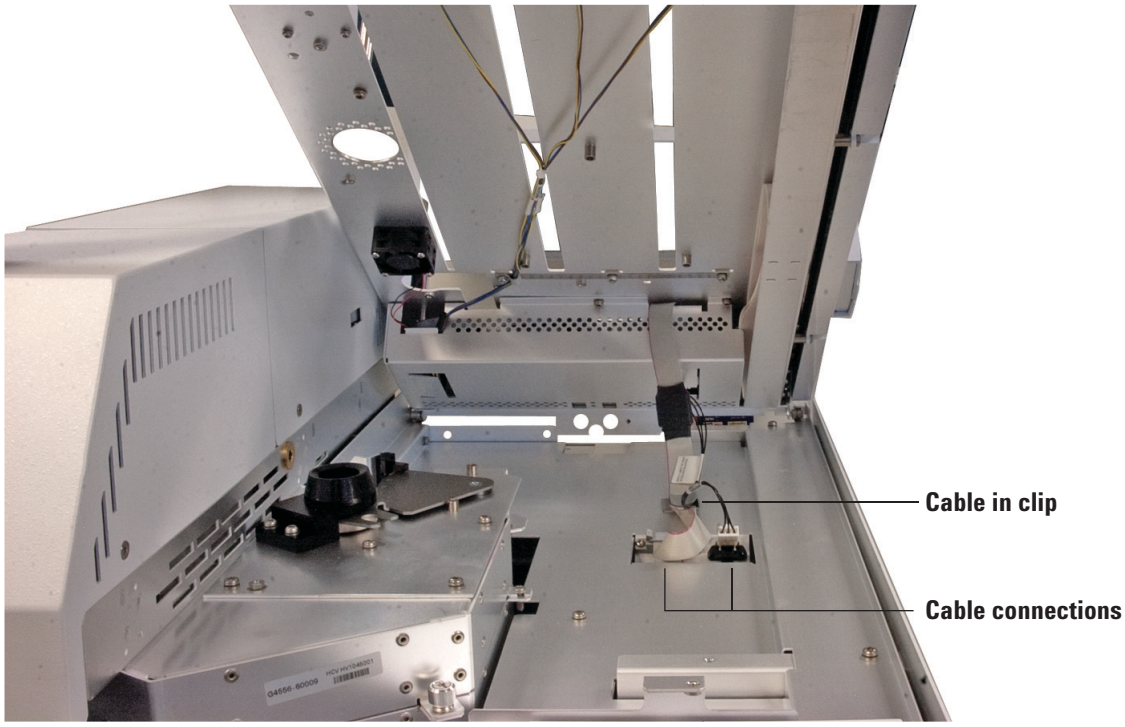


Figure 3 Connecting and routing the cables

4 Slowly lower the front of the tray. Align the vial oven access through-hole over the raised rim of the vial oven access port. The tray front aligns with the front of the instrument. The tray should be level and flat against the instrument chassis.

5 If needed, gently slide the gantry all the way to the back of the tray.

6 Install the 3 vial racks into the tray.

Connect Cabling

1 Connect the power cable. See Figure 4.

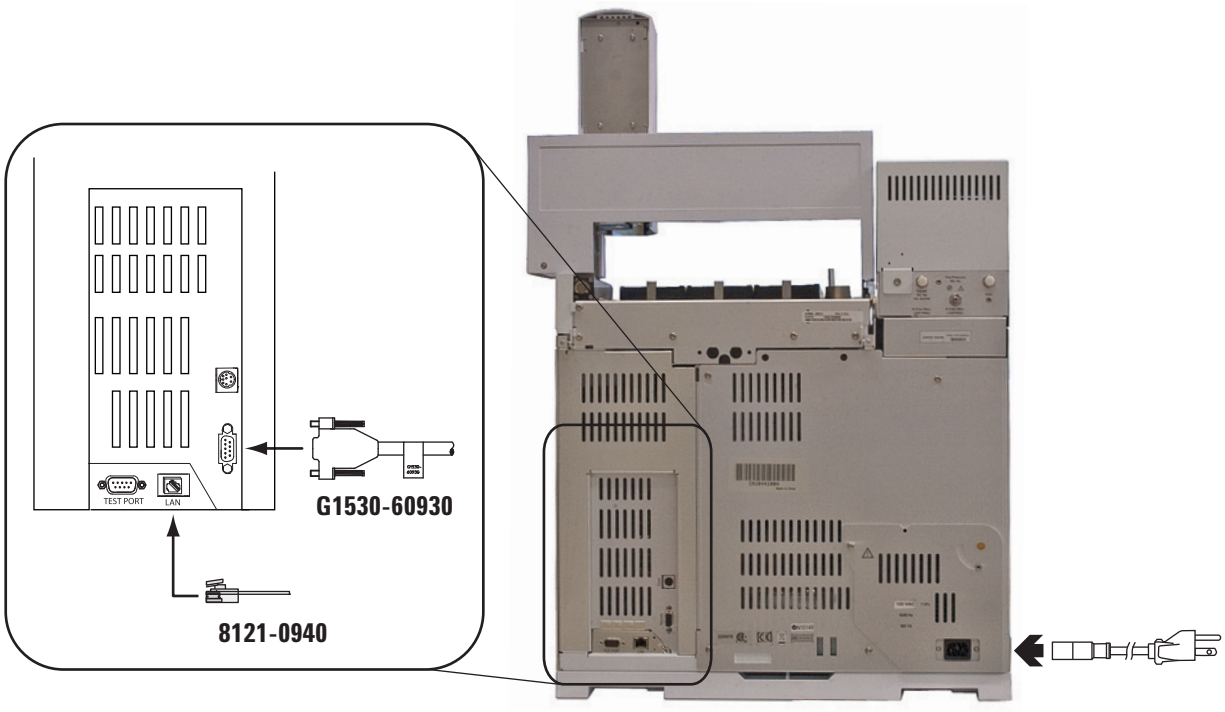
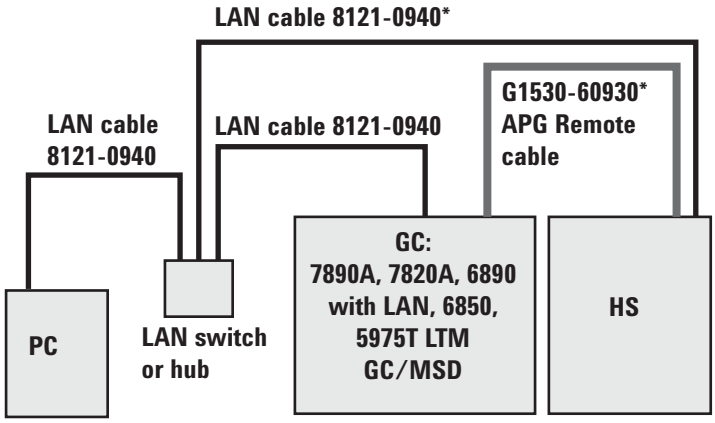


Figure 4 Back panel connections

2 Connect the LAN cable. See Figure 4.

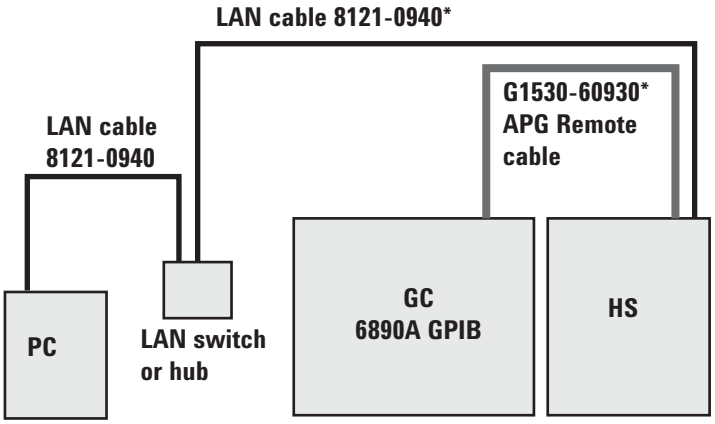
3 Connect the APG Remote cable. See Figure 4 through Figure 7.

Cabling diagrams for Agilent GCs



*Included in ship kit.

Figure 5 HS–GC (LAN)–PC



*Included in ship kit.

Figure 6 HS–GC (GPIB)–PC

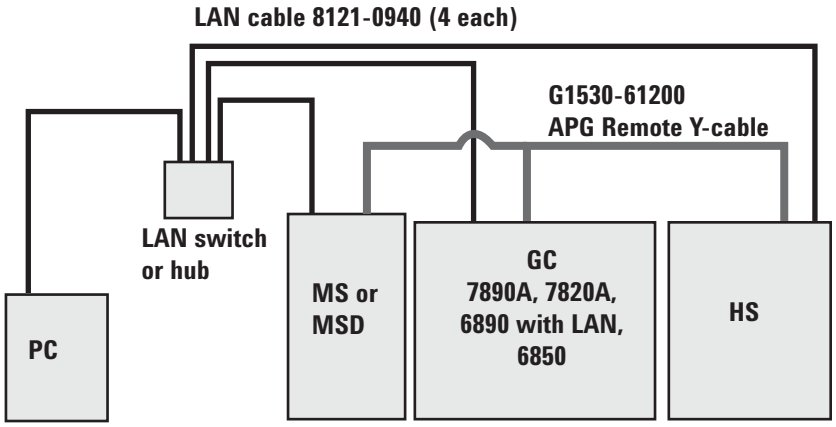


Figure 7 HS–GC (LAN)–PC–MS (or MSD)

Other systems

Use the Agilent general purpose remote start/stop cable, G1530-60560, to synchronize an Agilent product and a non-Agilent product. See Figure 8 and Figure 9.

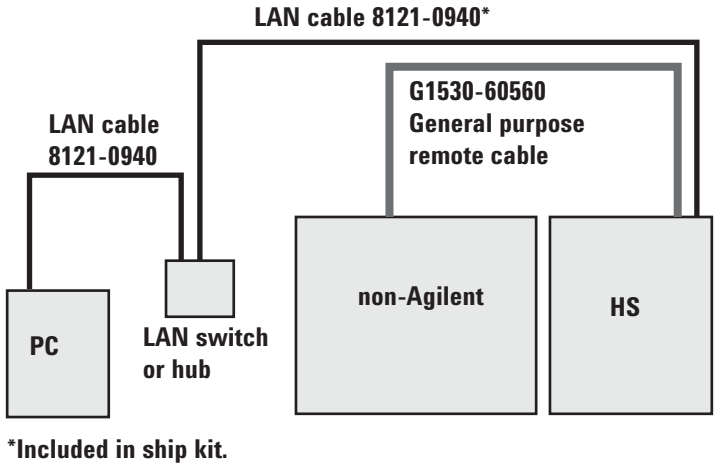


Figure 8 HS–GC (LAN)–PC–MS (or MSD)

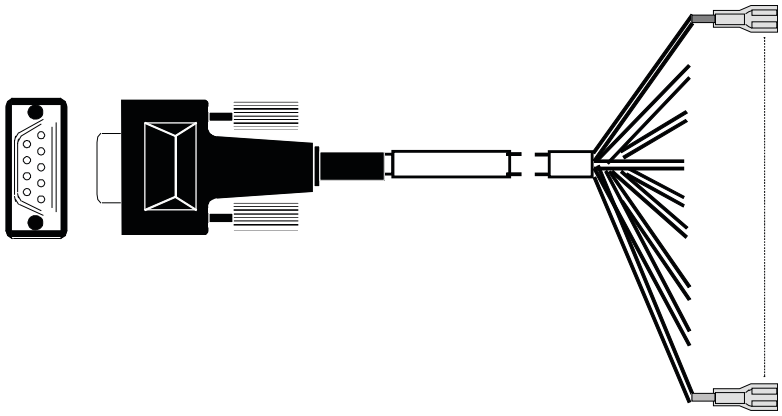


Figure 9 General purpose remote start/stop cable, Agilent product to non-Agilent product

Table 7 Cable pinouts, general purpose remote start/stop cable

Wire color	Signal name
Black	Ground
White	Prepare (low true)
Red	Start (low true)
Green	Start relay
Brown	Start relay
Blue	Not used
Orange	Ready (high true input)
Yellow	Stop (low true)
Violet	Not used

To ensure compatibility when connecting to a non-Agilent product, a detailed knowledge of signal levels, driver and receiver circuitry, and timing is necessary.

Connect Gas Supplies

NOTE Agilent recommends installing shutoff valves (not supplied) near the vial pressurization and carrier gas fittings. Using the valves provides a fast way to isolate the HS during troubleshooting and periods of inactivity.

Agilent provides hardware to plumb the instrument gases as shown in Figure 10. In this example, the HS connects to a 7890A GC through the inlet septum. The GC provides carrier gas control. The vial pressurization gas is connected to a different supply than the carrier gas.

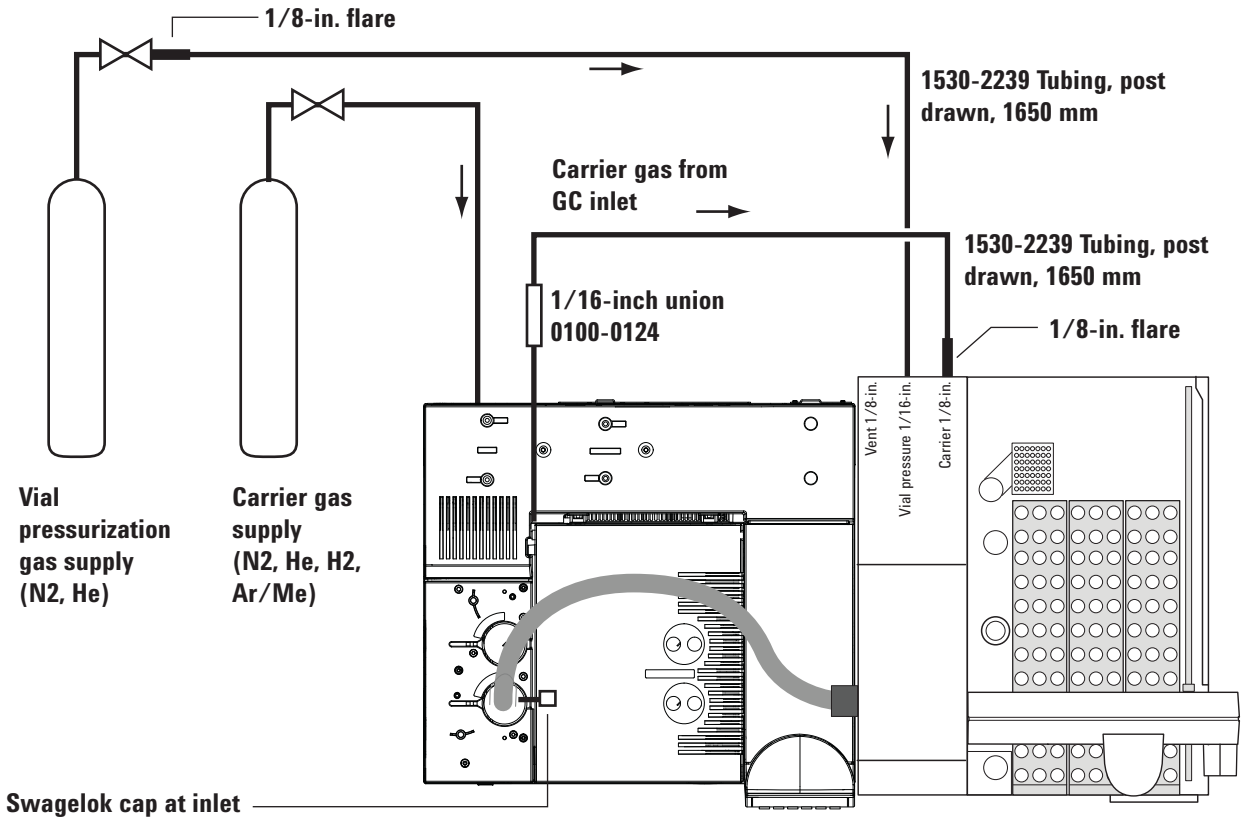


Figure 10 Standard plumbing configuration

See Table 8 for a summary of the acceptable gas types for the HS.

Table 8 Acceptable gases for HS use

Gas type	Vial pressurization	Carrier
Helium	Yes	Yes
Nitrogen	Yes	Yes
Hydrogen	No	Yes
Argon (5%)/Methane (95%)	No	Yes

NOTE

The procedures below assume the standard configuration: Plumbing as shown in Figure 10, with GC carrier gas control using the inlet EPC module. These procedures also assume that the GC is already plumbed and operating, and that the GC is plumbed for helium carrier gas.

If your setup is different, connect your gases accordingly.

Connect the vial pressurization gas

Materials needed:

- 1/8-inch Swagelok nut and ferrule set, stainless steel
- 1/16-inch Swagelok nut and ferrule set
- 1/16-inch to 1/8-inch flared stainless steel tubing, 1650 mm long

Tools required:

- 7/16-inch open-end wrenches
- 5/16-inch open-end wrenches
- Precision tubing cutter

For details on making Swagelok connections, see "Making Swagelok Connections."

WARNING

Use only helium or nitrogen as vial pressurization gas. Never use flammable gases for vial pressurization. Flammable gases create an explosion hazard if used for vial pressurization.

1 Turn off the vial pressurization gas supply.

2 Use the 1/8-inch Swagelok nut and ferrules to connect the 1/8-inch flared end of the stainless steel tubing to the gas supply.

3 Use the 1/16-inch nut and ferrules to connect the 1/16-inch end of the stainless steel tubing to the **Vial Pressure** fitting on the back of the HS.

4 Turn on the vial pressurization gas supply and set the source pressure to 410 kPa (60 psi).

5 Check for leaks in all external fittings. Correct any leaks before continuing.

Connect the carrier gas (using GC inlet carrier control)

Materials needed:

- 1/16-inch Swagelok union, stainless steel
- 1/16-inch Swagelok cap, stainless steel
- 1/8-inch Swagelok nut and ferrule set, stainless steel
- 1/16-inch Swagelok nut and ferrule set, stainless steel
- 1/16-inch to 1/8-inch flared stainless steel tubing, 1650 mm long

Tools required:

- 7/16-inch open-end wrenches
- 1/4-inch open-end wrenches
- Precision tubing cutter

Before beginning, make sure the GC oven is cool. You will need to stop GC carrier gas flow. Stopping GC carrier flow can damage a hot column.

NOTE The procedure below assumes a 7890A GC with a multimode or split/splitless inlet. Other instrument types are similar.

NOTE This procedure assumes that you will manually splice the HS into the carrier gas stream. If you have purchased accessory G3521A, 7890A S/SL MMI Weldment for Headspace, you will skip some steps. See below. This procedure does not discuss installation using other accessories or inlet types.

1 If on, turn off the carrier gas supply.

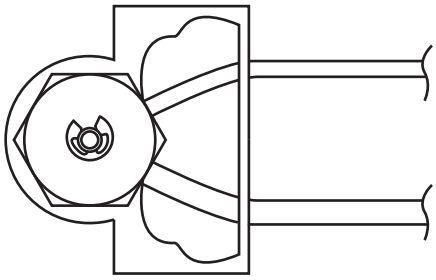
2 Remove any ALS injectors and the GC detector cover from the GC.

3 Remove the GC pneumatics cover. (You may need to disconnect any vent lines.)

NOTE If you purchased the G3521A 7890A S/SL MMI Weldment for Headspace, install it now. Skip step 4.

4 Splice into the inlet carrier gas tubing.

a Locate the inlet carrier gas line. The inlet carrier gas line is a 1/16-inch stainless steel tube that runs from the inlet EPC module to the inlet, usually beside the inlet carrier cover on the oven top. For split/splitless and multimode inlets, there is also a stainless steel septum purge vent line. The septum purge vent line is larger in diameter, and runs to the septum purge vent on the EPC module.

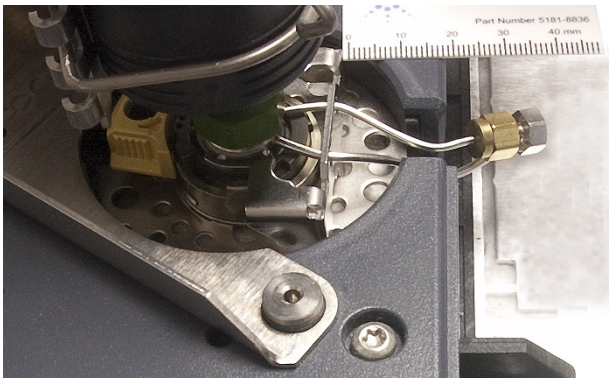


7890A Multimode inlet shown. Other inlets similar.

b Using the precision tubing cutter, cut the carrier gas line leading into the inlet at approximately 3 to 5 cm from the septum head.

CAUTION Cut the tubing as square as possible. Be careful to avoid kinks in the tubing. Avoid deforming the tubing. The cut should not be beveled or angled.

c Plug the inlet carrier line at the septum head using the 1/16-inch Swagelok nut, ferrules, and cap assembly provided in the ship kit.



5 Install the male half of a 1/16-inch union onto the open end of the carrier gas tubing coming from the inlet EPC module.

NOTE When completed, you should be able to reassemble the original carrier flow path by removing the cap from the plug, then connecting the two ends of the tubing.

6 Use the other half of the 1/16-inch Swagelok union to connect the carrier gas tubing from the EPC module (prepared in step 5) to the smaller (1/16-inch) end of the flared stainless steel tubing.

7 Use a 1/8-inch Swagelok nut and ferrules to connect the larger end of the flared stainless steel tubing to the **Carrier** fitting on the HS.

8 Turn on the carrier gas supply. Set the delivery pressure to 550 kPa (80 psi).

9 Check for leaks in all external fittings. Correct any leaks before continuing.

Connect the Transfer Line to the GC

This procedure describes installation of the fused silica transfer line provided with the instrument. If you have purchased a different transfer line, you can install it instead of the 530 µm line listed below. Refer to the Maintenance manual for part numbers and procedures.

Materials needed:

- 1/16-inch internal reducer union
- 530-µm fused silica column
- 11-mm septum
- Septum nut adapter (G3452-60835)

Tools needed:

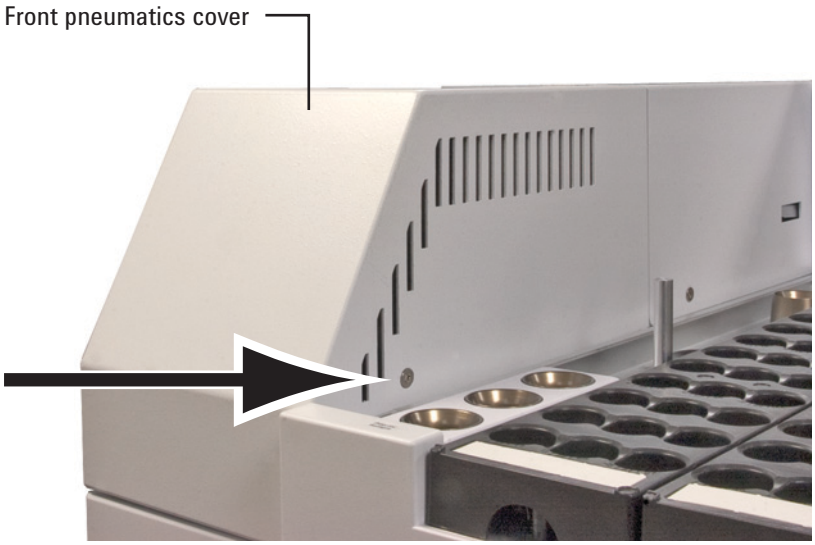
- 3/16-inch open end wrench
- 1/4-inch open end wrench
- Column cutting wafer

Install the transfer line to the Headspace

CAUTION Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

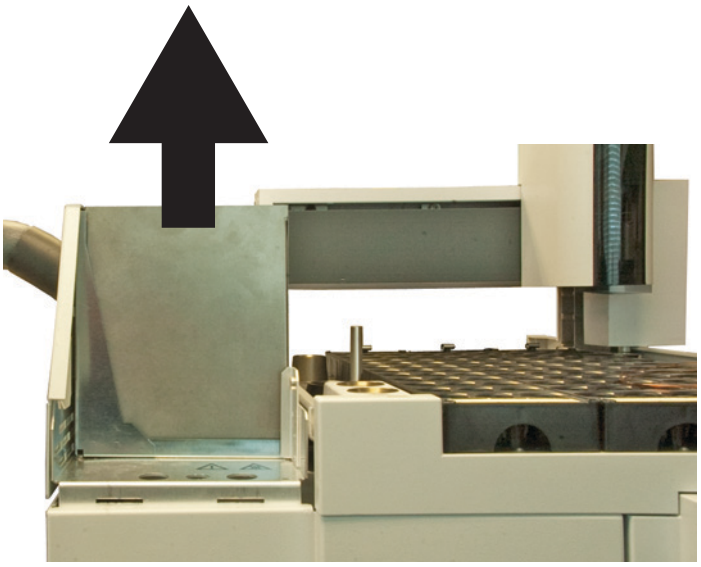
1 Remove the front pneumatics cover.

a Remove the T-20 screw that secures the cover in place.



b Pull the cover away from the chassis, then lift to remove.

2 Lift the valve thermal enclosure straight up from the HS.



3 Depending on your hardware configuration, do one of the following:

- Remove the valve cover (Figure 11). Completely loosen the two T-20 Torx screws, and remove the valve cover.
- Remove the valve/loop cover (Figure 12). Completely loosen the three T-20 Torx screws, and remove the valve/loop cover.

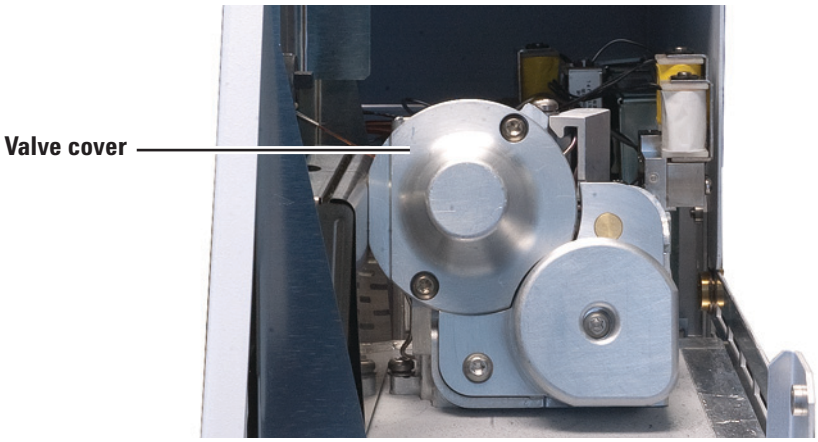
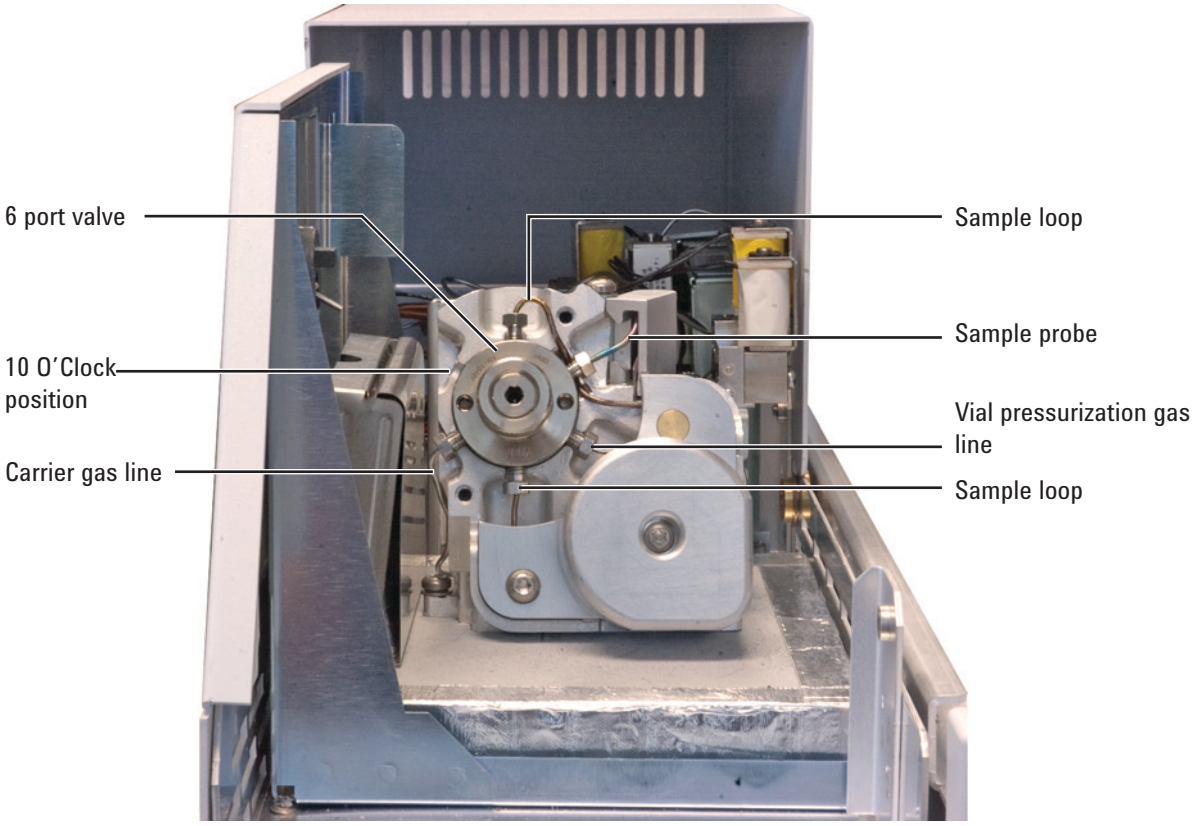


Figure 11 The valve cover

Figure 12 The valve/loop cover

4 The transfer line will install in the valve port at the 10 O'clock position, as shown in the figure below (valve port 3). Remove the 1/16-inch plug. Save the plug for future use.



5 Disassemble the 1/16-inch internal reducer provided in the HS ship kit.

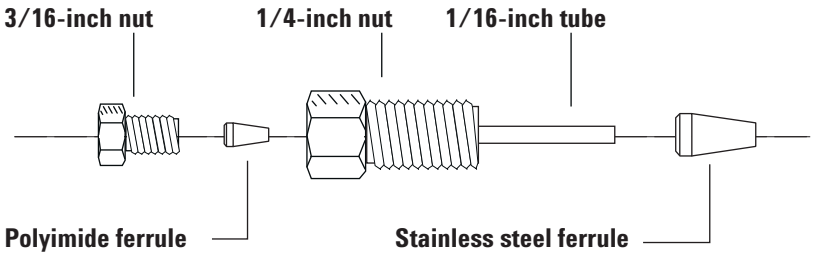
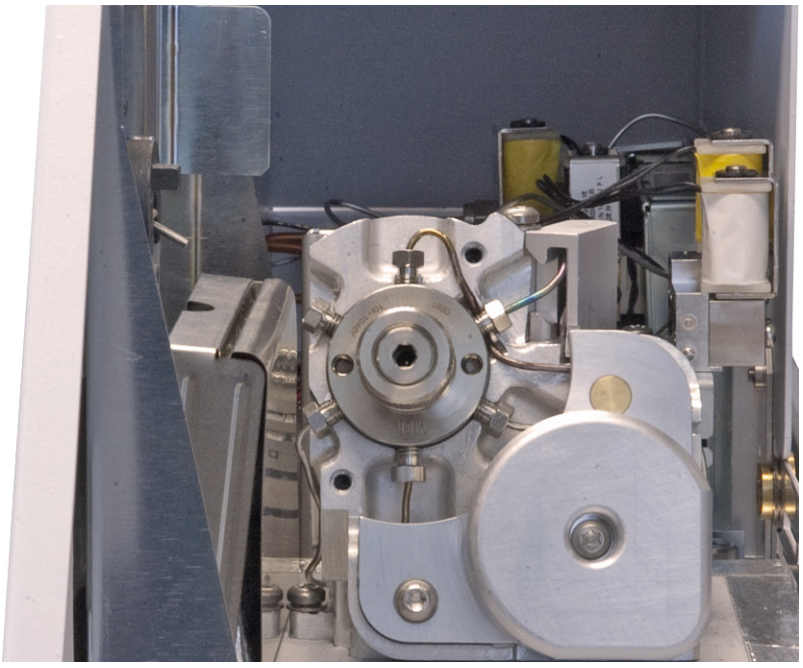


Figure 13 Internal reducer parts

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

6 Slide the stainless steel ferrule over the 1/16-inch tube end of the reducer, then install into the open valve port (at 10 O'clock). Finger-tighten, then tighten 1/4-turn more.



7 Remove the protective cap from the transfer line.

8 Depending on the type of tubing you are using in the transfer line, do the following:

Uncoated deactivated fused silica tubing: Uncoil about 1 m of the 530 µm uncoated deactivated fused silica tubing.

ProSteel tubing: Cut a ProSteel metal capillary (0.53 mm ID with maximum OD of 0.67 mm) to approximately 1 m in length using a precision tubing cutter.

9 If using ProSteel tubing and you intend to operate at temperatures 200 °C and higher, you must use the ProSteel protective sleeve with the ProSteel tubing. Without the ProSteel protective sleeve, the ProSteel tubing can permanently bind to the heated conduit tube. If using fused silica tubing, skip this step.

To install the ProSteel protective sleeve:

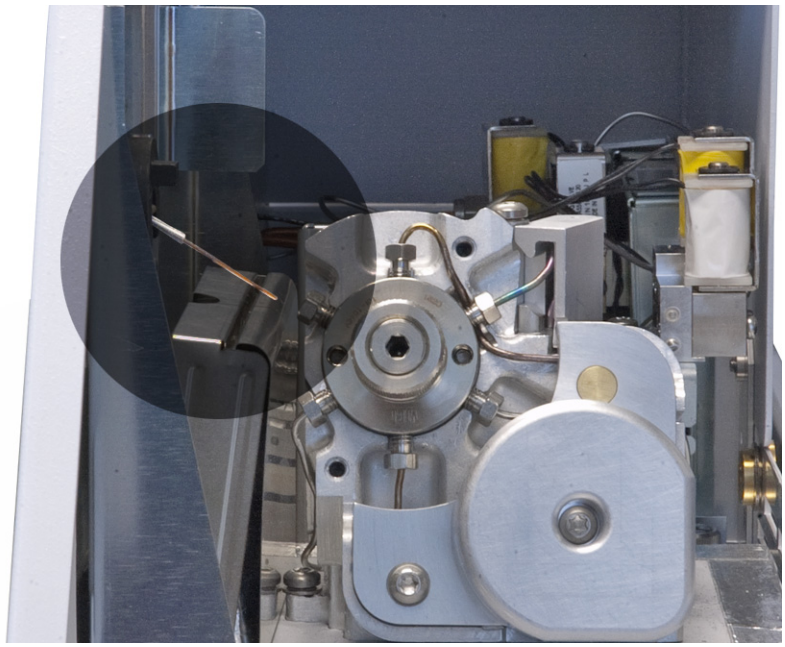
a Trim the ProSteel protective sleeve to match the length of the transfer line (approximately 1 m), plus or minus a few millimeters.

b Straighten the ProSteel tubing and protective sleeve.

c Slide the ProSteel tubing into the protective sleeve.

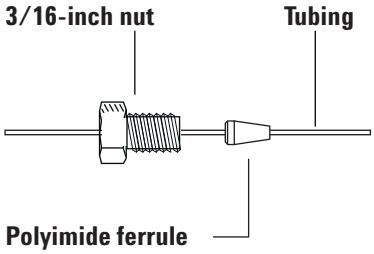
10 Working from the open end that will connect to the GC (not the end near the 6 port valve), gently slide the tubing through the transfer line until it appears out the end near the 6 port valve.

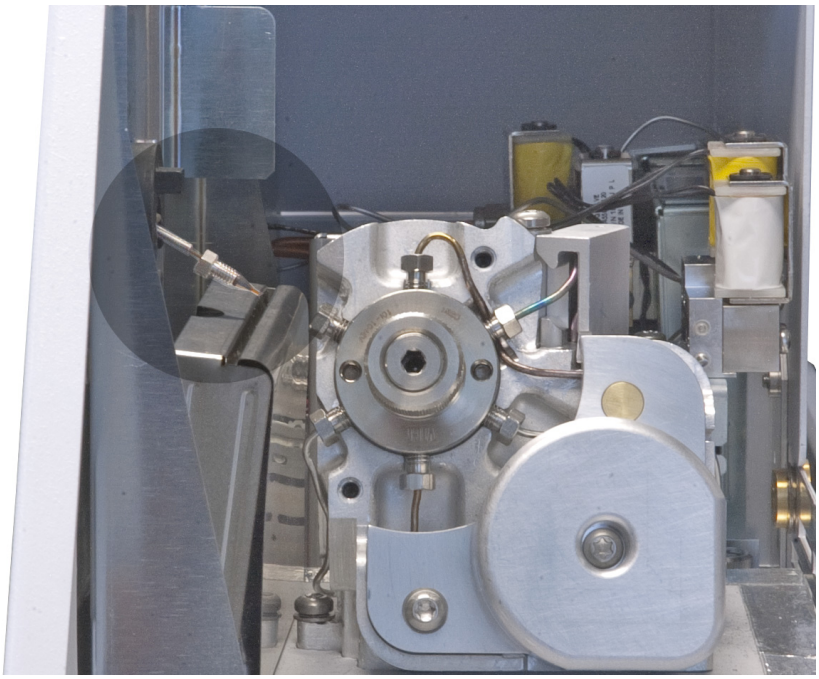
If using ProSteel tubing with protective sleeve, make sure the protective sleeve protrudes a few millimeters from both ends of the transfer line for easy removal.



11 Gently grasp the tubing at the HS end and gently push and pull the tubing to verify that it is unbroken in the transfer line. The tubing should move back and forth.

12 Assemble the 3/16-inch nut and polyimide ferrule onto the tubing as shown below. Be sure to use the appropriate nut and ferrule supplied with the capillary in use.





13 Use a column cutter to trim approximately 1 cm from the leading edge of the tubing.

NOTE For detailed instructions on how to trim a fused silica column, refer to your HS maintenance manual or the GC maintenance manual.

14 Insert the tubing into the internal reducer and loosely install the 3/16-inch nut. Do not tighten yet.

15 Gently slide the tubing into the internal reducer until it bottoms. Tighten the 3/16-inch nut finger tight, then an additional 1/4-turn. Test the connection by gently pulling on the transfer line. The transfer line should not slide out of the newly-made fittings. See Figure 14.

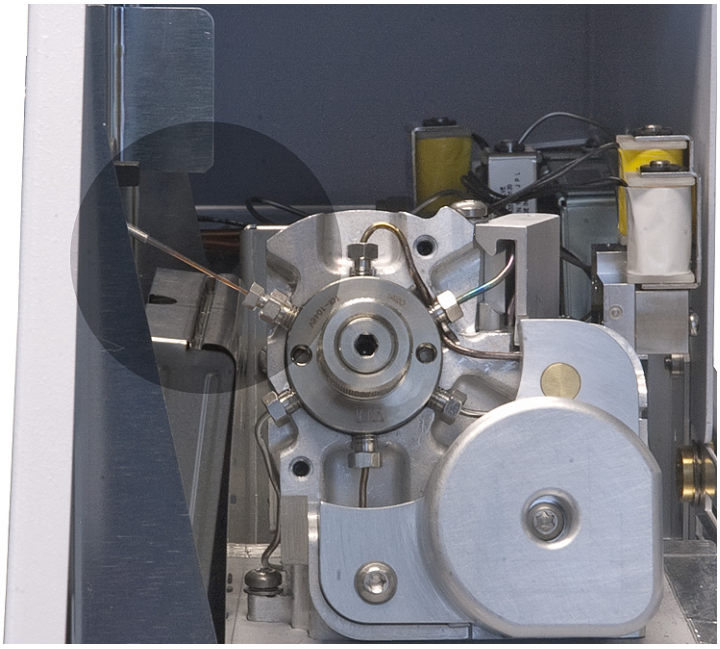


Figure 14 Transfer line installed into 6 port valve

Attach the transfer line to a GC inlet

CAUTION Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

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

Split Splitless or Multimode Inlet

1. Slide the one-piece septum nut and strain relief coupling onto the tubing until the inner metal sleeve of the transfer line bottoms-out on the strain relief.
2. Slide the septum onto the tubing until the septum is against the bottom of the one-piece septum nut and strain relief coupling.
3. Trim the tubing 35 mm past the end of the septum.
4. Install the inlet liner (with o-ring) that is appropriate for your application.
5. Insert the tip of the transfer line into the inlet.
6. Finger-tighten the strain relief coupling.

Purged Packed Inlet

1. Slide the one-piece septum nut and strain relief coupling onto the tubing until the inner metal sleeve of the transfer line bottoms-out on the strain relief.
2. Slide the septum onto the tubing until the septum is against the bottom of the one-piece septum nut and strain relief coupling.
3. Trim the tubing 2 mm past the end of the septum.
4. Install the inlet liner (with o-ring) that is appropriate for your application.
5. Insert the tip of the transfer line into the inlet.
6. Finger-tighten the strain relief coupling.

Volatiles Interface

1 Slide the transfer line nut (G2319-20210) onto the tubing.

2 Install a SilTite ferrule and make a SilTite connection as described below. Refer to Table 9 and use the SilTite ferrule appropriate for your transfer line.

Table 9 SilTite ferrules

Transfer line id	Use SilTite ferrule part number:
0.25 mm	5188-5361
0.32 mm	5188-5362
0.53 mm	5188-5363

CAUTION The SilTite ferrules are delicate. Follow the instructions in the next steps very carefully to avoid overtightening.

a Pass the transfer line tubing end through the SilTite ferrule leaving approximately 1 cm of tubing protruding beyond the ferrule. Thread the pre-swaging tool onto the transfer line nut (Figure 15) with the tubing protruding through the tool.

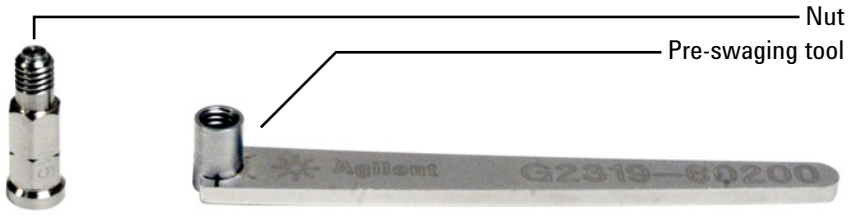


Figure 15 Volatiles interface nut and pre-swaging tool (G4556-67018)

b Using a wrench and ferrule pre-swaging tool, tighten the nut a little at a time, occasionally checking to see if the ferrule is gripping the tube. When the ferrule just starts to grip, notice position of the nut and then tighten by turning 45 to 60 degrees of rotation, but no

more than 60 degrees (one flat). If you can pull the transfer line tubing free, it is not tight enough.

c Remove the pre-swaging tool.

d Using a ceramic column cutter, trim the tubing at the small end of the ferrule leaving approximately 0.3 mm of tubing extending beyond the ferrule.

It is important that the tube end does not extend beyond 0.5 mm from the end of the ferrule.

e Check the end of the tube with a magnifier. The end of the tube need not be perfectly square, but should not have cracks which extend under the ferrule.

3 Screw the nut onto the volatiles interface.

Cool On-Column Inlet

1 Trim the tubing so that it extends 42 mm out of the inner metal sleeve on the transfer line.

2 Slide the tubing through the retaining nut on the cool on-column inlet.

3 Finger-tighten the retaining nut. Pull the transfer line gently to be sure the ferrule has formed a tight seal with the transfer line. Tighten the retaining nut further, if necessary.

Install the transfer line support bracket

Install the transfer line support bracket as described below. Use this bracket in all configurations, and for all Agilent inlet types and GCs.

1 If necessary, remove any existing ALS post from the mounting hole next to the transfer line.

2 Place the transfer line support bracket on the inlet carrier cover. The locator holes in the support bracket should align over the holes for the inlet ALS injector post and the raised mounting detail. See the figures below.

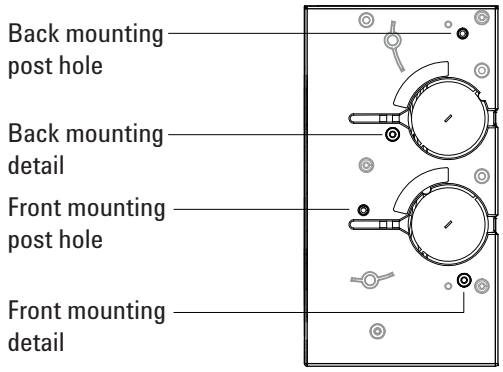


Figure 16 Mounting location for support bracket, 7890A inlet carrier cover shown

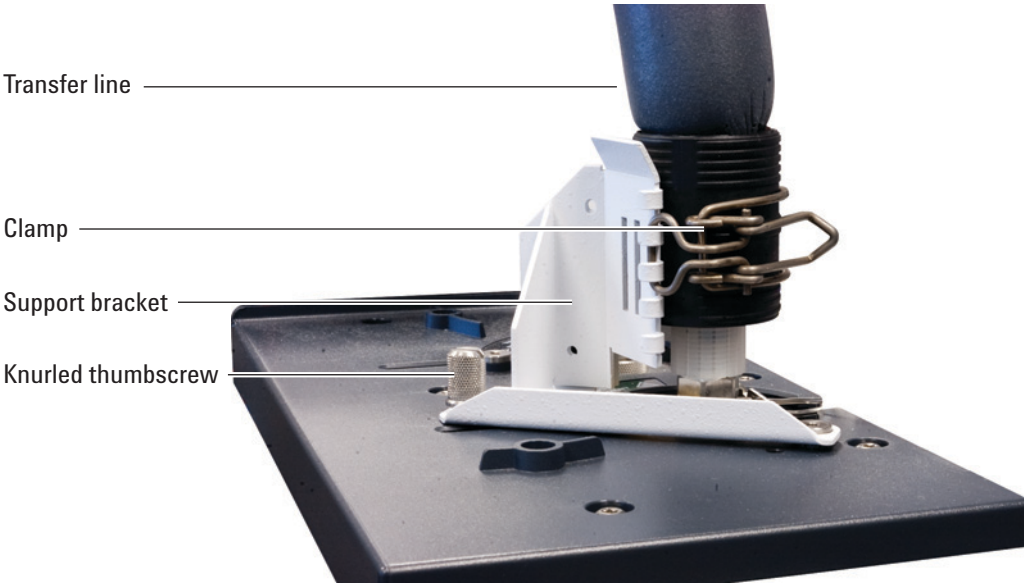


Figure 17 Transfer line installed onto 7890A inlet

3 Locate the knurled thumbscrew provided in the HS ship kit transfer line support assembly.

4 Install the knurled thumbscrew into the inlet cover through the transfer line support bracket.

5 Install the clamp onto the bracket. Align the clamp so that it supports the transfer line as high as possible above the GC as shown in Figure 17.

Turn On the Headspace Sampler

1 Turn on the gas supplies.

2 Turn on the headspace sampler.

After power up, the display should be similar to the following:

```
Agilent 7697A
A.01.01

Power on successful
```

Now that the gases and HS are turned on, the vial pressurization gas will begin to purge the HS sampling system.

Set the IP Address

For network (LAN) operation, the HS needs an IP address. Typically enter the IP address using the HS keyboard. See your LAN administrator.

To set the LAN address at the keyboard

1. Press **[Options]**. Scroll to **Communications** and press **[Enter]**.
2. Scroll to **IP**. Enter the numbers of the HS IP address, separated by dots, and press **[Enter]**. A message tells you to power cycle the instrument. Do not power cycle yet. Press **[Clear]**.
3. Scroll to **GW**. Enter the Gateway number and press **[Enter]**. A message tells you to power cycle the instrument. Do not power cycle yet. Press **[Clear]**.
4. Scroll to **SM** and press **[Mode/Type]**. Scroll to the appropriate subnet mask from the list given and press **[Enter]**. A message tells you to power cycle the instrument. Do not power cycle yet. Press **[Clear]**.
5. Scroll to **Reboot instrument**. Press **[On/Yes]** and **[On/Yes]** to power cycle the instrument and apply the LAN setpoints.

To use a DHCP server

Note that DHCP is not supported by all Agilent data systems.

1. Press **[Options]**. Scroll to **Communications** and press **[Enter]**.
2. Scroll to **Enable DHCP** and press **[On/Yes]**. When prompted, turn the instrument off and then on again.

Configure the Headspace Sampler

Next, enter details that describe the gas types, vial sizes, sample loop size, and other important information.

1 Press **[Config]**.

2 Use ⇑⇓ to scroll to **Vial Gas type** and press **[Mode/Type]**.

3 Use ⇑⇓ to select the vial pressurization gas type from the list, then press **[Enter]**.

4 Scroll to **Loop Volume (mL)**. Press **[Mode/Type]**. Select **1.0** as the sample loop volume, in mL, then press **[Enter]**. (All headspace samplers ship with a 1 mL sample loop installed.)

5 If you ordered the optional G4562A Carrier Gas EPC Module Accessory, scroll to **Carrier**. Press **[Mode/Type]**. Select the correct control mode for checkout:

- If the HS will provide all carrier gas to the GC, select **GC Control**. (For example, you did not plumb carrier gas to the GC.)
- If the GC will provide its own carrier gas flow, select **GC + HS Control**. In this case, the HS will provide enough additional carrier gas flow during injection to push the checkout sample into the GC inlet.

6 Scroll to **APG polarity**. If needed, press **[Mode/Type]** to change.

- For Agilent GCs, use **Active high**.
- For non-Agilent GCs, refer to the GC documentation.

2
First Startup

This section describes how to start up the Agilent 7697A Headspace Sampler for the first time and verify its performance. This section describes how to get started using both the front panel and an Agilent data system. While the specific software described is the Agilent ChemStation, version B.04.03, the instructions apply generally to any Agilent data system. Refer to the data system's help.

Only perform the sections that apply to your setup.

Tools and Materials Required

Materials needed:

- Headspace checkout sample, 5182-9733
- Flat bottom headspace vial, 20 mL
- Crimp cap, headspace, 20 mm septum

Tools required:

- Ergonomic manual crimper for 20 mm caps
- Forceps or tweezers

The checkout sample kit, part number 5182-9733, contains 5 mL of sample and a box of 5-µL micropipettes.

The sample composition is:

- 9.59 ± 0.05% n-nonane (w/w)
- 0.348 ± 0.002% 1,2-dichlorobenzene (w/w)
- 0.322 ± 0.002% nitrobenzene (w/w)
- 0.241 ± 0.001% tert-butyldisulfide (w/w)

in dodecane matrix.

Load the Checkout Method in the HS

Using the HS front panel, load the checkout method.

1 Press **[Load][Method]**.

2 Scroll to select **Checkout**, then press **[Enter]**.

The checkout method will load. The HS will begin heating and adjusting flows to the initial conditions.

Once the front display reads **Ready**, let he HS sampler stabilize for 25 to 30 minutes before running the checkout sample. This hold time ensures thermal stability and that any residual moisture or other contaminants are swept from the system.

For reference, Table 10 lists the headspace sampler checkout setpoints and configuration.

Table 10 Headspace checkout parameters

Configuration	
Vial Gas type	He
Loop Volume (mL)	1
Advanced Functions	
Extraction mode	Single
Purge flow	100.0
Purge time	1.000
Temperature	
Oven	100.0 °C
Loop/Valve	110 °C
Transfer line	115 °C
Time	
GC cycle time	4 min. (FID, NPD) 5 min. (µECD) 6.5 min. (MS, MSD)
Vial equib time	7 min.
Pres equib time	0.1 min.
Inject time	0.5 min
Vial	

Table 10 Headspace checkout parameters (continued)

Fill mode	Flow to pressure
Fill pressure	103.4 kPa (15 psi)
Fill flow	50 mL/min
Loop fill mode	Default
Vent after extraction	Yes
Vial size	20 mL
Shaking	1

Make the First Connection

If using an Agilent data system, perform this step. If operating the HS in standalone mode, skip this section.

1 Open the online session for the GC.

2 As the GC session opens, you will be prompted to configure the instrument. Select **Yes**. The instrument configuration utility opens.

   If not prompted, open the utility from the **Instrument** menu.

3 In the left pane, double-click on the 7697A instrument. The instrument icon moves to the right hand list.

4 Click **Configure**. In the dialog that opens, enter the following:

   - In the **Connect Info** group, enter the **Instrument Name**, then enter the instrument IP address or host name.
   - Enter your license key in the **Instrument License** field. You will see the **Version Information** for the instrument appear (instrument serial number, firmware version, software version, and so forth).
   - Enter any **Notes** as desired.

Set the Checkout Method Parameters

Next, set the rest of the checkout parameters.

If using an Agilent data system

If using an Agilent data system, upload the current HS parameters into the data system method. In the ChemStation, select **Method and Run Control** view.

1. Select **Instrument > Upload method from Agilent 7697A**.
2. Save the method using a new name.
3. Select **Instrument > Edit Parameters**, then enter the GC parameters listed in the next section.

Apply the method to the GC and save it.

Checkout method GC parameters

Enter the following GC setpoints, either in the data system (if used) or at the GC using the keypad.

NOTE The table lists the settings for a 7890A GC. Other GC types use similar settings.

Table 11 GC checkout method

Oven	
Initial temperature	100 °C
Initial time	4.00 min
Rate	0 (Off)
Equilibration time	1.00 min
Run time	4.00 min
Inlet (multimode, volatiles interface, or split/splitless)	
Mode	Split
Initial temperature	250 °C (On)
Pressure	134.4 kPa (19.5 psi)
Split ratio	20:1
Split flow	80 mL/min

Table 11 GC checkout method (continued)

Total flow	87 mL/min
Gas saver	Off
Column	
Mode	Constant flow
Initial flow	4.0 mL/min
Initial pressure	134.4 kPa (19.5 psi)
Average velocity	60 cm/s
Detector parameters–FID	
Temperature	300 °C
Hydrogen (fuel) flow	30 mL/min
Air flow	400 mL/min
Makeup mode	Constant makeup
Makeup flow	25 mL/min
Detector parameters–NPD	
Temperature	300 °C
Hydrogen (fuel) flow	2.0 mL/min
Air flow	60 mL/min
Makeup mode	Constant makeup
Makeup flow	8 mL/min
Detector parameters–µECD	
Temperature	250 °C
Makeup mode	Constant makeup
Makeup flow	30 mL/min
MS or MSD	
Solvent delay	3 min
Run time	6.5 min
Headspace event time for GC cycle time	7 min
Scan range	45 to 300
Method used	Atune file
Inlet pressure	82.8 kPa (12 psi)
Column flow	1.1 mL/min

Table 11 GC checkout method (continued)

Split ratio	80:1
Injection source	Headspace sampler (front)
Injection volume	5 µL

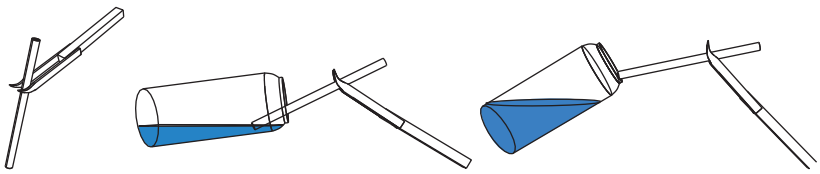
Prepare the Checkout Sample

While the instrument comes to equilibrium, prepare the checkout sample. The checkout method calls for a 5-µL sample in a 20-mL headspace vial.

1 Snap the top off the checkout sample ampoule. Transfer the contents to a screw-cap bottle.

2 Dispense one capillary pipette.

3 Hold the pipette with clean tweezers. With the checkout sample bottle and the pipette as close to horizontal as practical, dip the end of the pipette into the sample. See Figure 18.



(1) Grasp (2) Fill (3) Wipe

Figure 18 Filling the pipette

4 The pipette will fill by capillary action. When it has filled completely, pull the pipette out of the sample. Wipe the bottom edge of the pipette against the edge of the vial to remove any droplets on the outside.

5 Drop the filled pipette into a 20-mL headspace vial. Crimp a cap on the vial.

6 Cap the checkout sample vial.

Run the Checkout Sample

After the HS has been Ready for 25–30 minutes, it has been purged and is sufficiently stable to run the checkout sample.

1 Load the checkout sample into the HS.

For the 111 vial model:

a Press **[Tray Park/Carousel Advance]** to “park” the tray (move the gantry to a rest position for easy access to the vial racks).

b Place the capped sample vial in position 1.

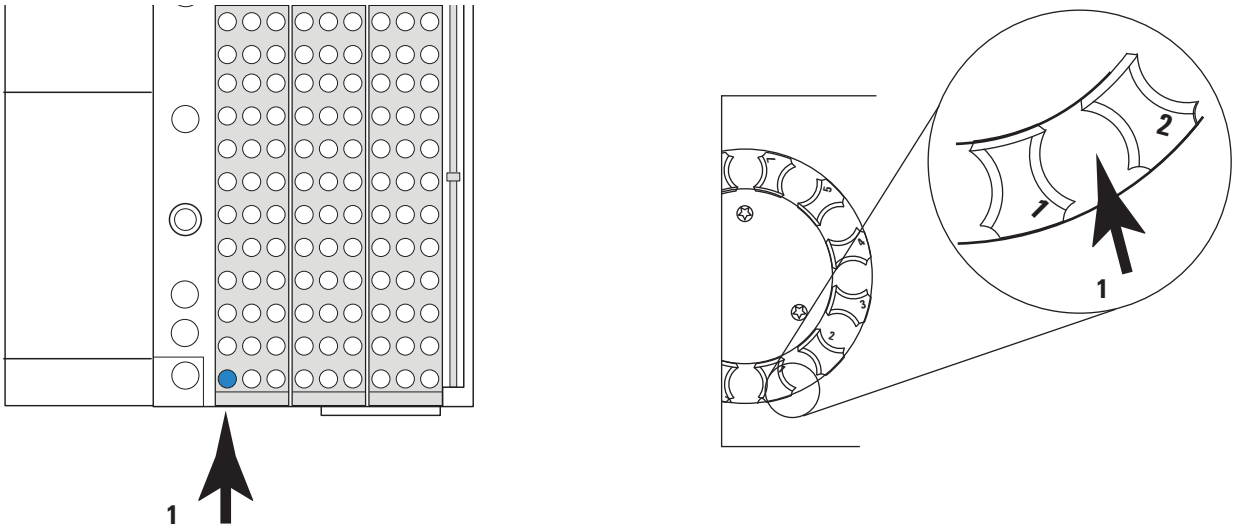


Figure 19 Tray vial position 1

c Press **[Tray Park/Carousel Advance]** to prepare the tray for use.

For the 12 vial model:

a Open the tray cover. A label to the left of each vial position denotes the number of that position. See Figure 19 and Figure 20.

b If vial position 1 is not available, press **[Tray Park/Carousel Advance]** to rotate the tray.

c Place the capped sample vial in position 1.

Figure 20 Opening the tray cover

2 Start the sample run.

If using an Agilent data system:

a Set the injection source to be the HS.

b Create a 1 line sequence to run the checkout sample (vial 1) once, using the checkout method.

c Set up the report so that the correct signal output becomes part of the report.

d Start the sequence.

If using the HS standalone:

a Press **[Sequence][Load/Append]**. This creates the first line of the sequence. Note that the default new sequence line selects the current method, vial range 1 to 1, and 1 injection per vial. The sequence is complete.

b When the GC is ready, press **[Start]** on the HS keyboard.

Compare Results

When the run completes, compare the output chromatogram against the examples below. Is correctly installed and operating properly, the output chromatogram should be similar to the example here.

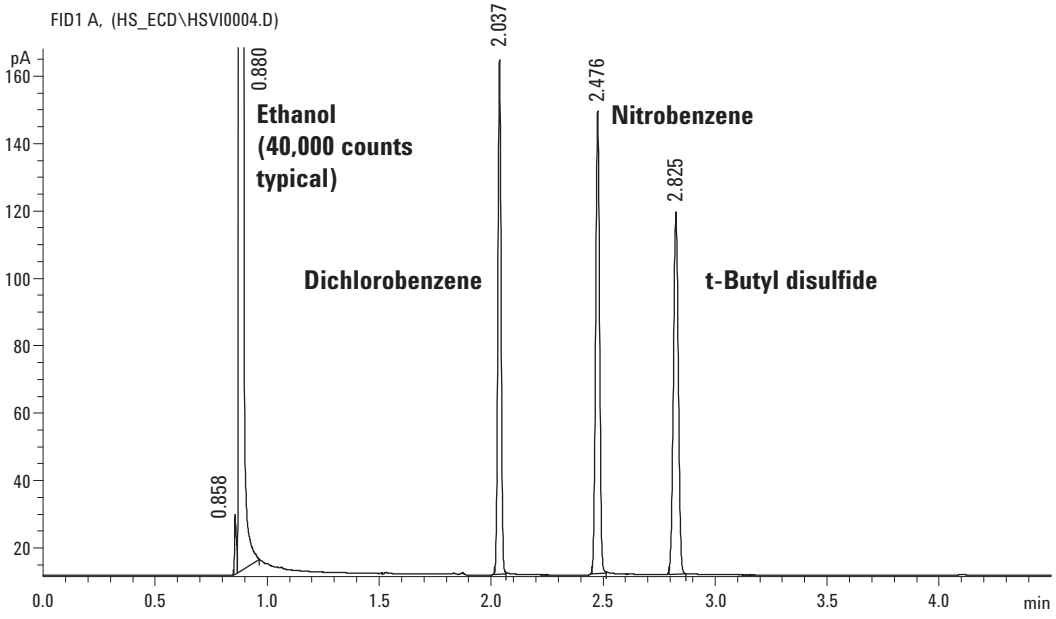


Figure 21 FID checkout chromatogram

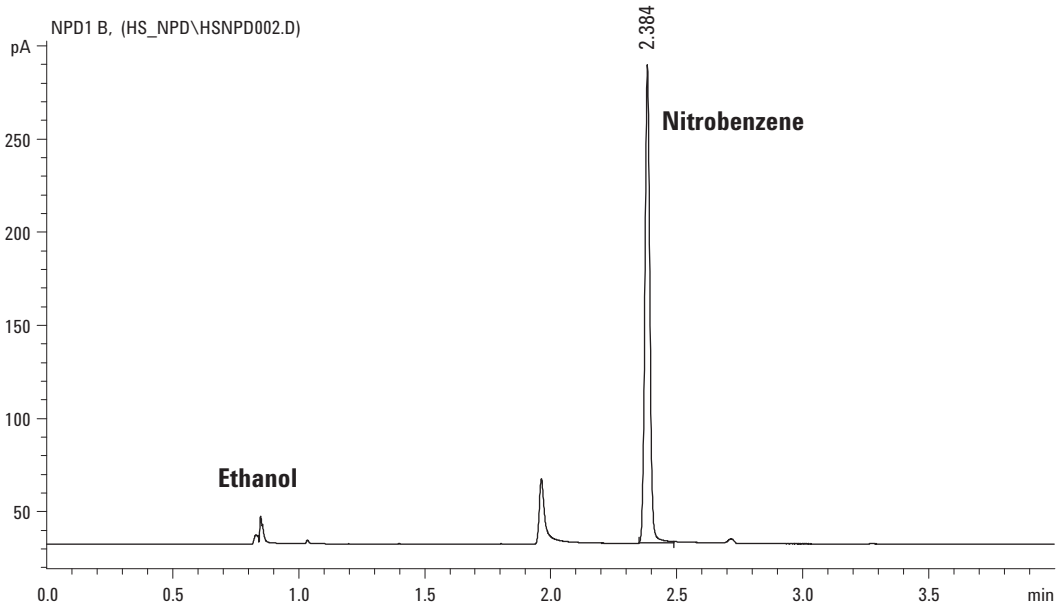


Figure 22 NPD checkout chromatogram

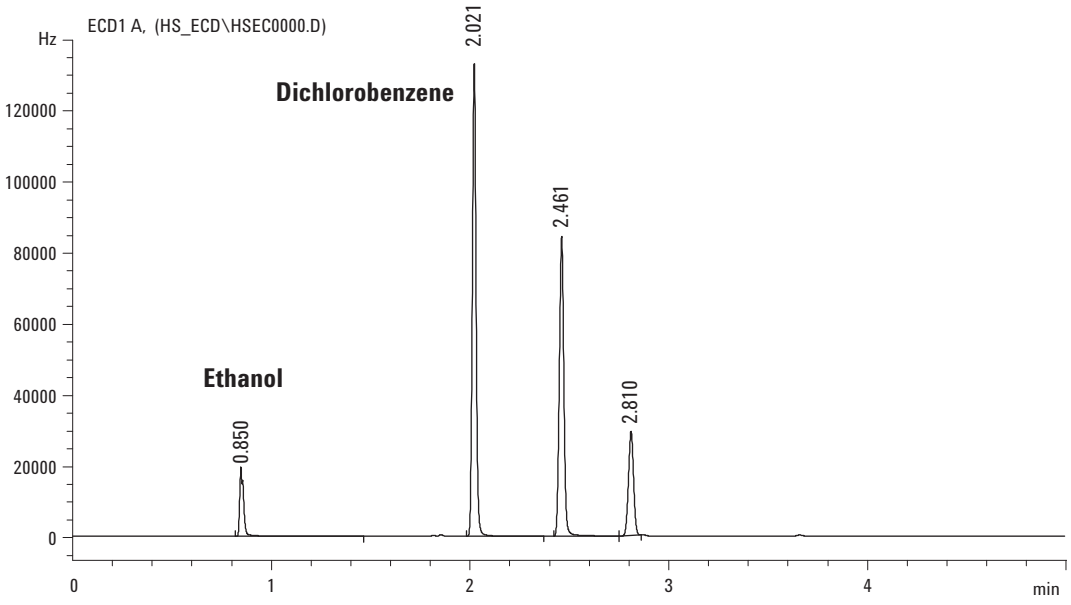


Figure 23 µECD checkout chromatogram

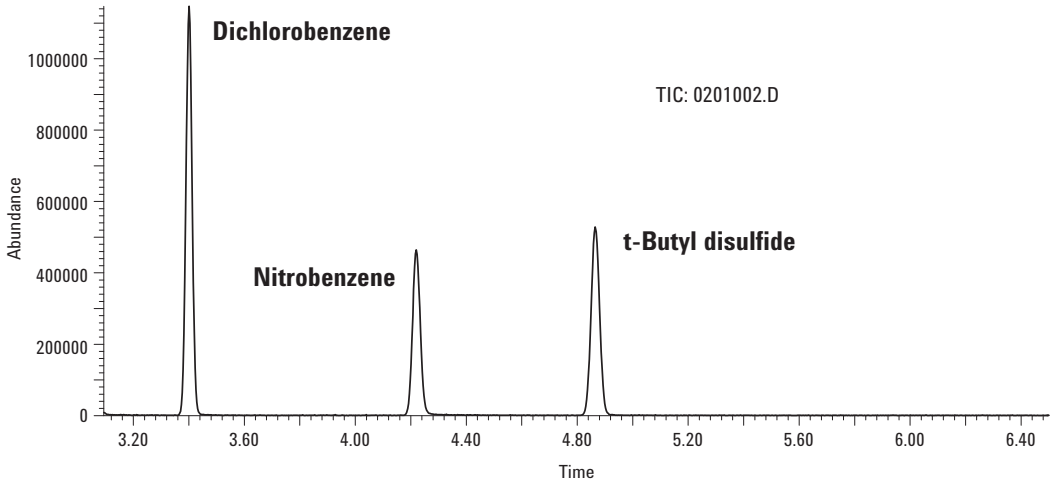


Figure 24 MS and MSD checkout chromatogram

Prepare for Your Method (Optional)

At this point, installation is complete. However, if you have purchased an optional sample loop, install it now before continuing. See the Maintenance guide for details.

Also consider your method's vial pressures and carrier gas pressures. Make sure that the gas supplies are set to provide at least 138 kPa (20 psi) higher than the highest pressure needed (for vial pressurization or for the GC column carrier flow during temperature programming), up to the maximum input pressure of 828 kPa (120 psi).

A
Making Swagelok Connections

The gas supply tubing is attached with Swagelok fittings. If you are not familiar with Swagelok connections, review the following procedures.

Making Swagelok Connections

Objective

To make a tubing connection that does not leak and that can be taken apart without damaging the fitting.

Materials needed:

- 1/8-inch (or 1/4-inch, if used) preconditioned copper tubing
- 1/8-inch (or 1/4-inch, if used) Swagelok nuts
- Front and back ferrules
- Two 7/16-inch (for 1/8-inch nuts) or 9/16-inch (for 1/4-inch nuts) wrenches

1 Place a Swagelok nut, back ferrule, and front ferrule to the tubing as shown in Figure 25.

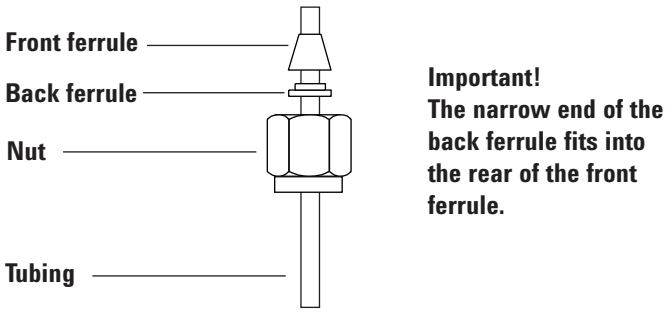


Figure 25 Swagelok nuts and ferrules

2 Clamp a stainless steel plug or similar fitting in a bench vise.

CAUTION Use a separate stainless steel fitting in a vise for initial tightening of the nut. Do not use an inlet or detector fitting. Strong forces are required to properly set the ferrules, and damage to an inlet or detector fitting is very costly to repair.

3 Push the tubing into the stainless steel plug (see Figure 26).

4 Make sure that the front ferrule is touching the plug. Slide the Swagelok nut over the ferrule and thread it onto the plug.

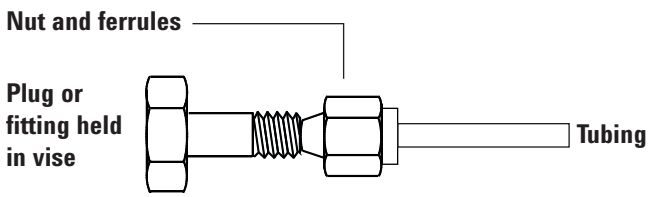


Figure 26 Assembling the fitting

5 Push the tube fully into the plug, then withdraw it approximately 1 to 2 mm (see Figure 27).

6

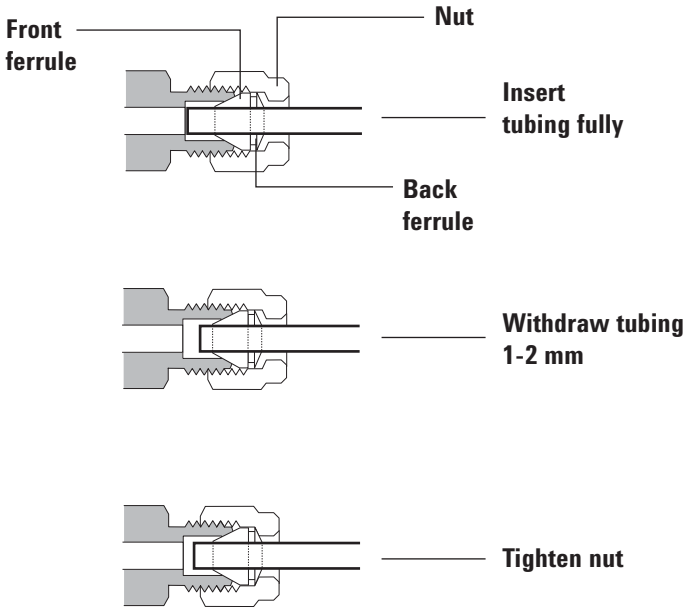


Figure 27 Insert the tubing

7 Finger-tighten the nut.

8 Mark the nut with a pencil line (see Figure 28).

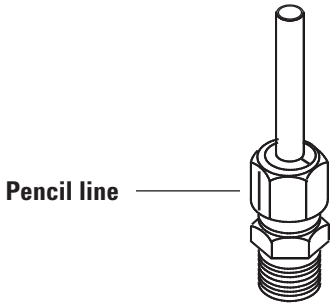


Figure 28 Marking the fitting

9 For 1/8-inch Swagelok fittings, use a pair of wrenches to tighten the fitting 3/4 of a turn (see Figure 29).

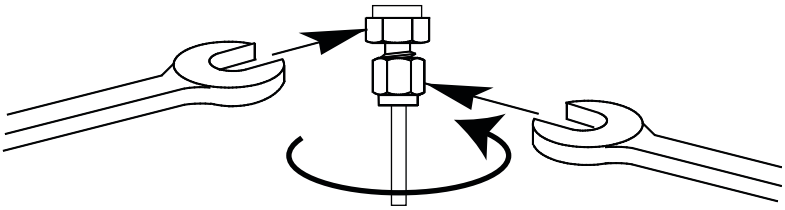


Figure 29 Final tightening

10 Remove the plug from the fitting. To connect the tubing, with nut and ferrules, to another fitting, finger-tighten the nut, then use a wrench to tighten it 3/4 (1/8-inch fittings) of a turn.

11 Both correctly- and incorrectly-swaged connections are shown in Figure 30. Note that the end of the tubing in a correctly-swaged fitting is not crushed and does not interfere with the action of the ferrules.

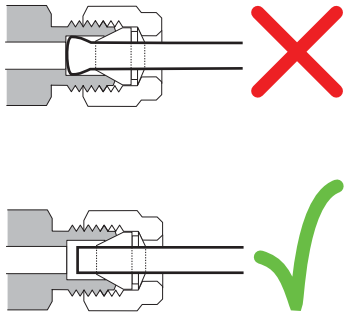


Figure 30 Completed fitting

Using a Swagelok Tee

To supply gas from a single source to more than one input, use a Swagelok Tee.

NOTE Do not combine valve actuator air with flame ionization air. The valve action will cause major upsets in the detector signal.

Materials needed:

- 1/8-inch preconditioned copper tubing
- Tubing cutter
- 1/8-inch Swagelok nuts and front and back ferrules
- 1/8-inch Swagelok Tee
- Two 7/16-inch wrenches
- 1/8-inch Swagelok cap (optional)

1 Cut the tubing where you want to install the Tee. Connect the tubing and Tee with a Swagelok fitting. See Figure 31.

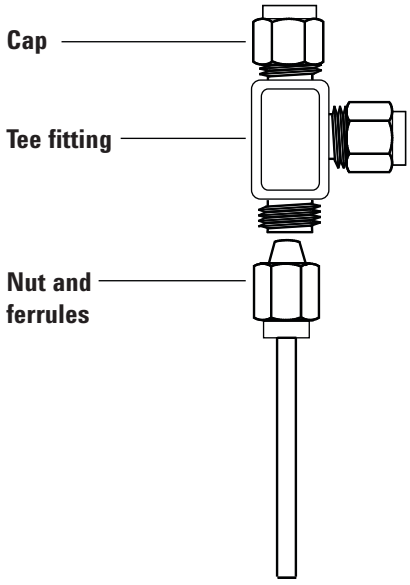


Figure 31 Swagelok tee

2 Measure the distance from the Tee to the instrument fittings. Attach copper tubing to the open Tee ends with Swagelok fittings.

Agilent Technologies