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# Agilent 7694 Headspace Sampler

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## Operating Manual

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## Safety Information

The Agilent Technologies 7694 Headspace Sampler meets the following IEC (International Electrotechnical Commission) classifications: Safety Class 1, Transient Overvoltage Category II, and Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the Agilent 7694 has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

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

## Safety Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use

of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

## WARNING

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

## CAUTION

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



Indicates a hot surface



Indicates earth (ground) terminal

## Sound Emission Certification for Federal Republic of Germany

Sound pressure  $L_p < 68$  dB(A)

During normal operation  
At the operator position  
According to ISO 7779 (Type Test)

## Schallemission

Schalldruckpegel  $L_P < 68$  dB(A)  
Am Arbeitsplatz  
Normaler Betrieb  
Nach DIN 45635 T. 19 (Typprüfung)

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## Contents



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**About This Manual**

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# About This Manual

The *7694 Headspace Sampler Operating Manual* explains how to use the 7694 Headspace Sampler. It includes all the basic information the user needs to develop methods, use the sampler to carry out analyses, and maintain and troubleshoot the sampler.

For site preparation requirements and checkout procedures, see the *7694 Headspace Sampler Site Preparation and Installation Manual*.

## Manual Overview

This manual contains ten chapters, a glossary, and an index.

**Chapter 1: About This Manual** provides the purpose of this manual and an overview of its contents. It identifies the intended reader and lists the conventions used throughout the manual.

**Chapter 2: Introduction to Headspace and to the 7694 Headspace Sampler** provides an overview of headspace theory and practice. It describes the instrument and its modes of operation.

**Chapter 3: The Keypad** describes the keypad, the functions each key controls, and how to enter new setpoints for each function.

**Chapter 4: Using the Adv Fctn Key** describes the advanced functions controlled by the Adv Fctn key, how to edit setpoints for these functions, and the displays that accompany each function.

**Chapter 5: Preparing a Sample** describes the preparation of a sample for analysis, including filling and sealing the sample vial, loading the sample tray, and running a checkout sample.

**Chapter 6: Entering a Method** describes the steps to follow in entering a method, including changing setpoints, storing and retrieving a method, and chaining methods.

**Chapter 7: Developing Methods** describes the parameter changes that can affect sensitivity, precision, and accuracy.

**Chapter 8: Running a Sample** summarizes operation of the headspace sampler to run a sample.

**Chapter 9: Maintaining the Instrument** describes maintenance procedures including changing the sample loop, transfer line, needle, valves, and EPROM.

**Chapter 10: Troubleshooting** lists the possible causes and solutions for chromatography problems or instrument failure.

**Chapter 11: Error Messages** describes the various error messages that accompany machine failures and the means to correct the failures.

## Intended User

This manual is intended for users who understand the basics of gas chromatography. Although the manual contains some headspace theory, it primarily provides operating, maintenance, and troubleshooting information for the 7694 Headspace Sampler.

## Conventions

The following conventions are used in this manual:

1. Braces { } enclose a generic name for which you should substitute specific information. For example, {setpoint} means you should key in the numeric value you desire for the setpoint.
2. Text in **Bold** means that you should press the key by that name.  
Be careful how you enter information. For example, the key sequence **Adv Fnc1**, **.**, **1**, **Enter** indicates that you should press the **Adv Fnc1** key, the **.** key, the **1** key, and then **Enter**. It does not mean that you should press a comma (","). There is no comma on the sampler's keypad.
3. Text in *Courier* indicates the message you will read on the sampler display.

About This Manual  
**Conventions**

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**Introduction to Headspace and to the  
7694 Headspace Sampler**

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# Introduction to Headspace and to the 7694 Headspace Sampler

This chapter briefly describes the theory and application of headspace analysis. It describes the parts of the 7694 Headspace Sampler and its modes of operation.

## Overview of Headspace Analysis

Your new headspace sampler is a tool for introducing volatiles that are contained in liquid or solid samples into a gas chromatograph. The analytical technique is called “static headspace gas chromatography.” The term “headspace” refers to the space above the liquid or solid sample that you place in a sealed vial. The term “static” means that the volatiles in the headspace of the vial are at equilibrium with the same compounds in the liquid or solid sample.

The instrument extracts a reproducible volume of the headspace and injects it into the carrier gas flowing into the gas chromatograph. Food, packaging, pharmaceutical products, soil, and water are often tested to see if they meet quality criteria or regulatory standards. Fragrances and consumer products are analyzed using headspace in order to understand their composition. Forensics laboratories use headspace for measuring the percent of ethanol in human blood to see if a driver is legally intoxicated.

The ease with which a sample forms a gaseous headspace depends on the sample’s “partition coefficient.” See “*Developing Methods*” on page 68 for a more thorough explanation. After a programmed heating time, the headspace gas is extracted from the vial and injected into a GC for analysis.

There are two means of extracting the headspace gas from the vial—single or multiple headspace extraction (MHE). With single headspace extraction, only one sample of gas is taken from each sample vial. Single headspace extraction is the standard method. It is used for samples that are readily volatile or for

which there are no matrix effects. Quantitative analysis can be performed by using suitable internal or external standards.

MHE is available only in models G1289 and G1290. MHE is used for headspace samples for which no calibration standard can be prepared easily (for example, solid samples) or for highly volatile compounds in liquids, where the method of standard addition is difficult because of losses due to evaporation. MHE takes several headspace samples from each vial. With each extraction, the concentration of the volatile compound in both the gas phase and the sample becomes smaller, although the ratio remains constant. The logs of the peak areas versus the extraction number are plotted to obtain quantitative data about the compound of interest.

MHE is often used as a calibration tool. Once the peak area versus the concentration is established for a given sample matrix, quantitative measurements of samples with a similar matrix can be carried out using a single extraction.

## **The 7694 Headspace Sampler**

The 7694 Headspace Sampler provides an automated method to run up to 44 samples consecutively without operator attention. The sampler's microprocessor optimizes the time spent for each run according to the values programmed in by the operator. Up to four different methods can be stored and used to analyze a series of samples.

Models G1289 and G1290 can also be used for multiple HS extraction using multiple septum punctures, with up to 10 extractions per sample.

You can also automate optimization routines. The unit is made up of the following components:

### **Oven (G1289/G1290)**

The oven contains a circular aluminum sample carousel that can hold up to six 10- or 20 ml sample vials. A resistance band heater is used for heating. A

motor-driven fan in the center of the carousel circulates the air to maintain constant and uniform temperature. The carousel can be shaken to help components elute into the headspace quicker. Mechanical rods move vials up for removal by a robotic arm or for sampling.

Adapters (part no. 301-017-HSP) are required in the carousel when 10 ml vials are used.

### Heated Zones (G1883)

There are three heated zones. They are the vial oven, the loop, and the transfer line. The vial oven is where the vial is heated during thermal and chemical stabilization. Setpoint temperature range is between 40 and 200°C. At this point, the needle has not pierced the septum. The loop zone is controlling the temperature of the upper chamber, the valve, and the sample loop. The setpoint range of the loop is between 50 and 200°C. It is recommended that the loop temperature is kept 15°C higher than the vial temperature for most operations.

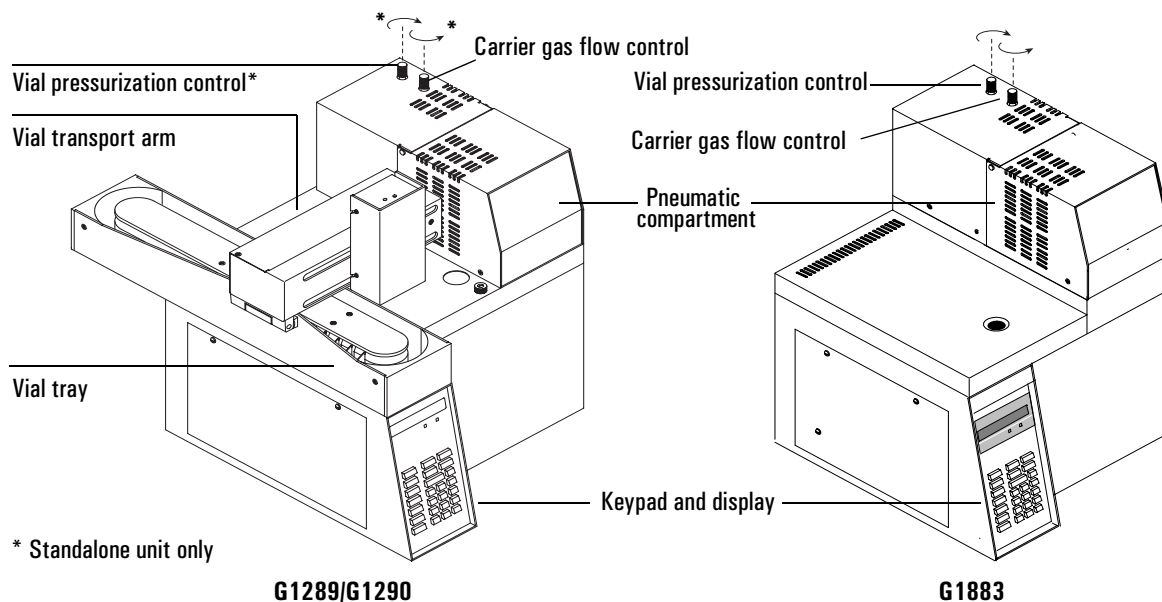
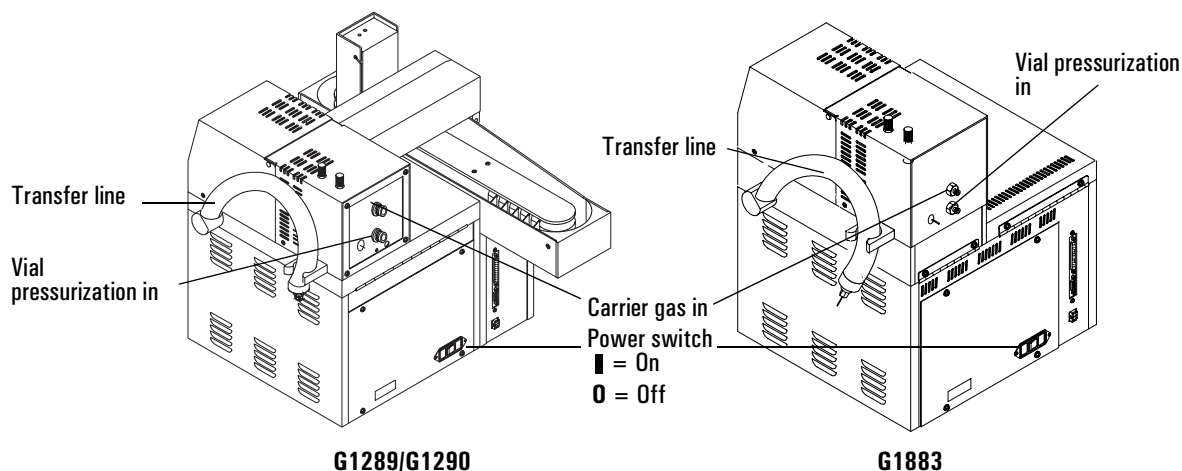


Figure 1. Overview of the 7694 Headspace Sampler (from front of instrument)





**Figure 2. Overview of the 7694 Headspace Sampler (from back of instrument)**

### **Vial Tray (G1289/G1290)**

The 44-sample vial tray is located on top of the unit to the left of the oven. The tray is constructed of a flexible belt with dividers for each vial compartment. Tray rotation is controlled via the keypad to assist filling the tray with headspace vials. The tray can move forward and backward. When you use the keypad to move the tray from position 1 to 44 (or from 44 to 1) the tray will rotate completely. Adapters (part no. 300-305-HSP) are needed in the sample tray when 10 ml vials are used.

### **Vial Transport Arm (G1289/G1290)**

A robotic arm moves samples from the vial tray into the oven and back. Vials are lifted from the tray, carried across the unit until centered above a port in the oven, and then dropped into place. The rail supporting the robotic arm swings away to allow access to the oven. Use the switch on the back of the support rail to set the arm for either 10- or 20 ml vials.

## Sampling System

The basic components include a 316–stainless–steel single–hole (side port) concentric vial–sampling needle or probe with an internal diameter of 0.5 mm, 316–SS six–port valve, nickel valve sample loop, set of two miniature solenoid valves, and nickel tubing. You can program the system’s temperature up to 200°C.

Also available is a fused silica-lined stainless steel sampling path (Accessory G1286A).

## Transfer Line

This line connects the headspace sampler with the GC. Two types are available: standard nickel and fused silica–lined stainless steel. The line is heated to a maximum temperature of 220°C. There is a removable needle for easy interfacing through GC inlet septa.

**Table 1. Transfer Lines**

Material	Length, cm	Part Number	Comment
Nickel	68	301–011–HSP	
Nickel	82	301–152–HSP	Standard transfer line
Fused silica-lined stainless steel	82	301–211–HSP	For applications requiring a highly inert non–metal flow path

## Keypad and Display

The headspace sampler has a 25–key elastomeric keypad. The display is a single–line 20–character LED dot matrix design. All functions of the sampler are controlled from the keypad with the exception of EPC, which is controlled from the GC or ChemStation.

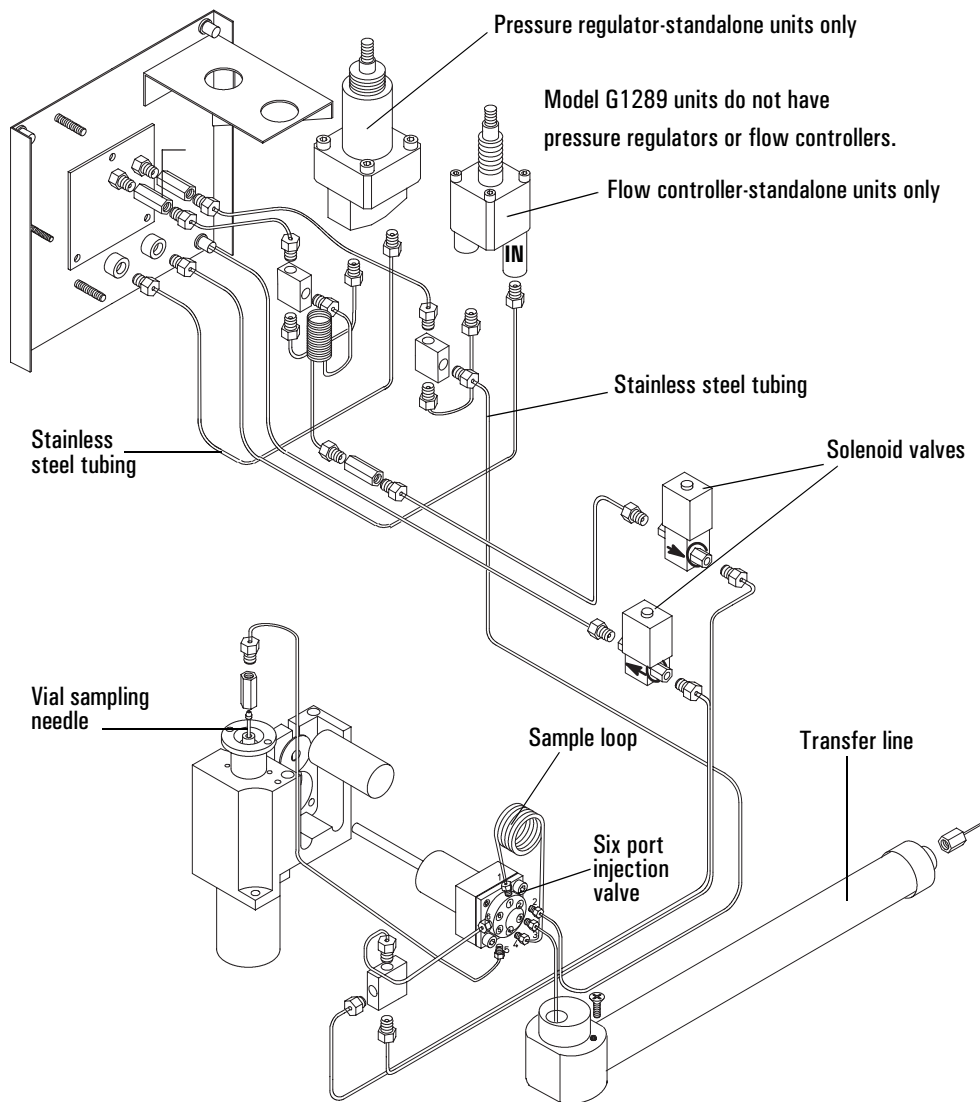
## **Pneumatics**

The G1290 and G1883 headspace samplers use two flow paths: GC carrier gas flow and flow for vial pressurization. You control the GC carrier gas flow using a manual flow controller and the vial pressurization using a manual pressure regulator.

The G1289 samplers uses the same gas streams, but all flow and pressure control is provided by the 6890 GC.

In all models, two 1/8-inch Swagelok® bulkhead fittings are provided at the rear of the pneumatic compartment for connecting lines for vial pressure and carrier gas.

Introduction to Headspace and to the 7694 Headspace Sampler  
The 7694 Headspace Sampler



**Figure 3. Pneumatic system for the 7694 Headspace Sampler**

## Principles of Operation

### General Operation

When the instrument is switched on, the sampler moves the first vial into position. (For models with a tray, the vial tray moves to the start position, placing the vial located in position one next to the robotic arm. For models without a tray (G1883), the carousel moves vial one to the start position.) The oven, sample loop, and transfer line begin heating to reach the setpoint values. Until the oven, sample loop, and transfer line setpoints are reached, the display reads 7694 Not Ready and the red “Not Ready” light is lit. If you press Start/ Stop before the machine is ready, the display reads *Waiting for Ready* and the “Not Ready” light flashes. When setpoints are reached, the display changes to 7694 Ready and the “Not Ready” light goes out.

During normal operation, when a run is in progress, the “Not Ready” light may light, depending on the values specified for the GC Cycle Time and the Vial Equil Time. When the “Not Ready” light goes on during a run, it simply means that the headspace sampler is not ready to insert the next vial into the oven because of timing restraints to maintain constant heating time.

## Power on Diagnostics

When the instrument is powered on or reset (see page 30 and page 46), it runs through a series of checks to make sure it is operating properly. The messages displayed will vary depending on your model, but will be similar to:

<b>G1289 and G1290</b>	<b>G1883</b>
Agilent 7694 or 7694 RESET	Agilent 7694E or 7694E RESET
FIRMWARE REV X.Y	FIRMWARE REV X.Y
ROM CHECK	ROM CHECK OK
RAM CHECK	RAM CHECK OK
TRAY RESET	CAROUSEL RESET
VIAL TEMP OK	VIAL TEMP PROBE OK
LOOP TEMP OK	LOOP TEMP PROBE OK
TR.LINE TEMP OK	TUBE TEMP PROBE OK
7694 NOT READY	7694E NOT READY
7694 READY	7694E READY

Other messages may appear as well. If your sampler has a tray, the sampler may move remaining vials from the carousel and display a message such as RETURNING VIALS or VIALS RETURNED. If you receive other messages, see “*Error Messages*” on page 128.

## Standby Condition

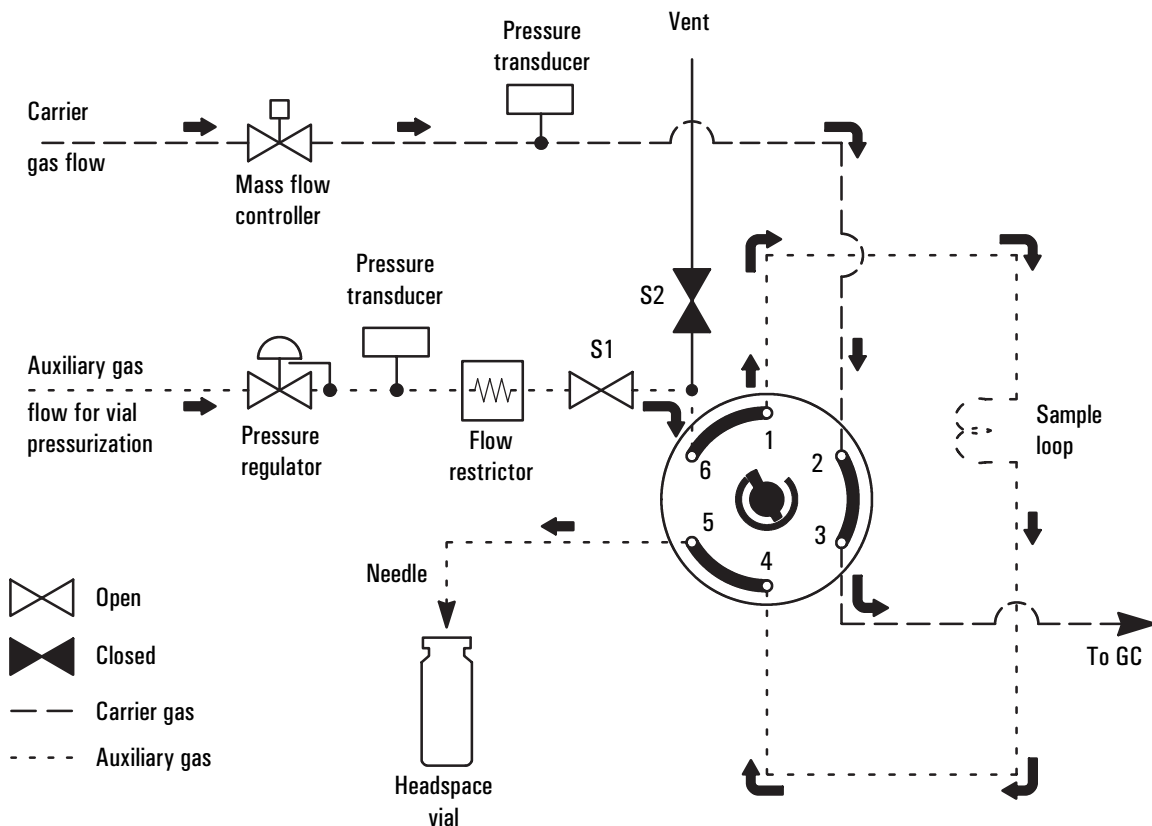
During standby, the pressurization valve is open, and auxiliary gas flushes the sample valve, the sample loop, and the sampling needle. Carrier gas is also flowing to the GC injection port through the sample valve.

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### **WARNING**

Do not use hydrogen as a gas in the 7694 HS.

When the sampler is started, it moves the first vial into the oven. Equilibration time begins. To shorten the required equilibration time, agitate the vial in the oven. See “Setting Vial Parameters” on page 63.

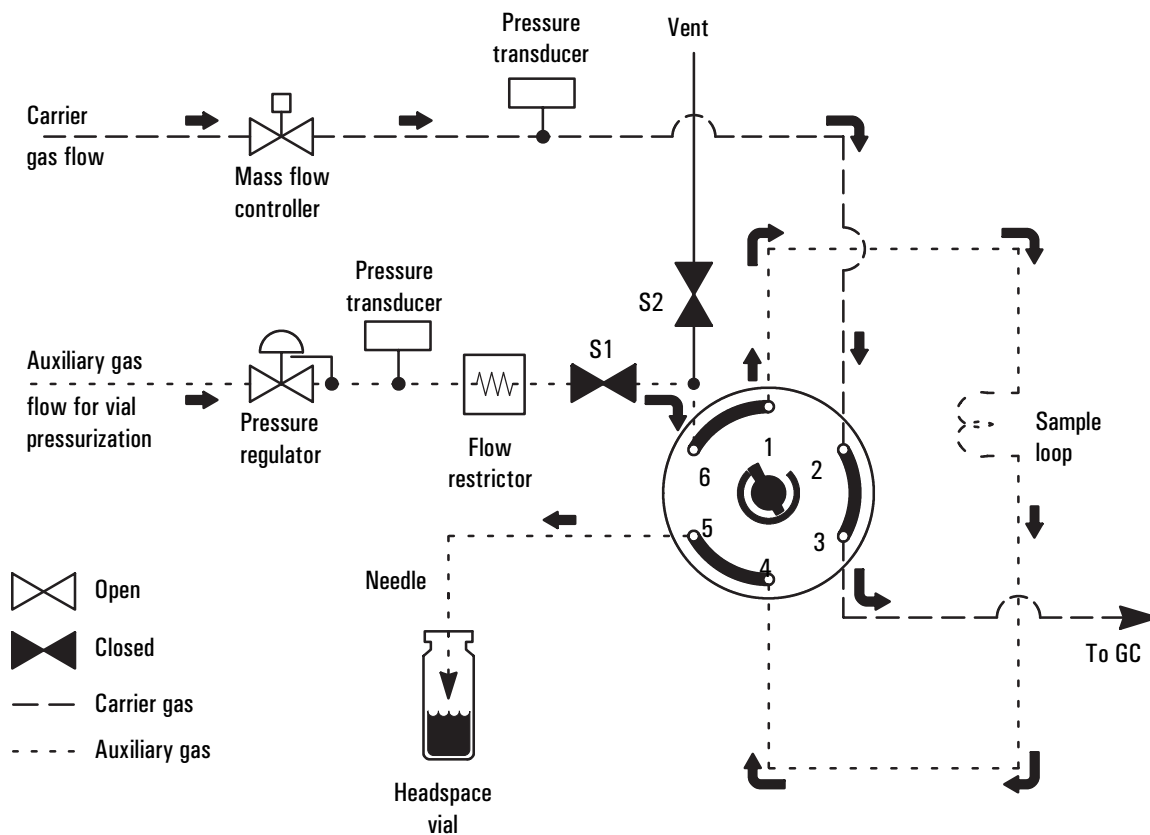


**Figure 4.** Standby condition. S1 is open.

### Vial Pressurization

At the end of the vial equilibration time, agitation stops, the vial moves in-line with the sampling needle and is raised by the mechanical rod onto the needle. The pressurization valve closes. The sampling needle pierces the septum of the

vial. The pressurization valve opens, allowing gas to enter the vial for the pressurization time. The valve then closes again.

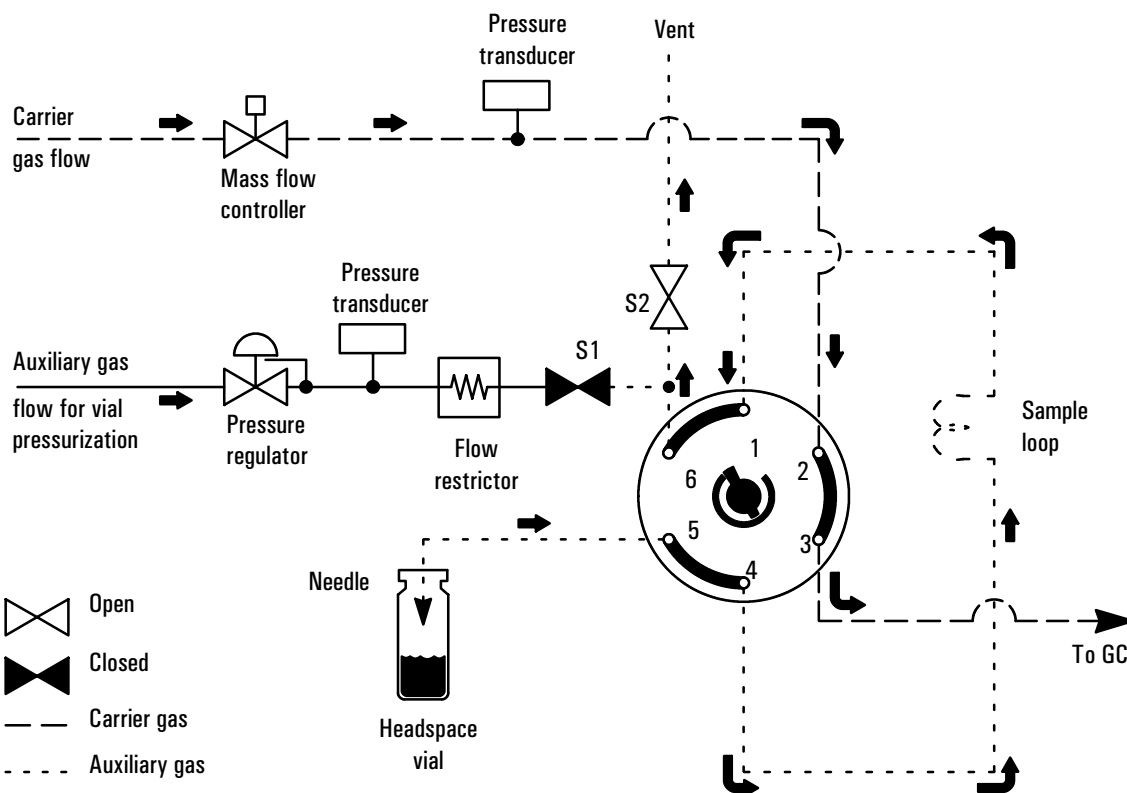


**Figure 5. Vial pressurization. S1 is closed (vial raised on needle).**

### Filling the Sample Loop

The vent valve opens and the headspace gas fills the sample loop; it then vents to atmospheric pressure through the vent outlet. A short loop-fill time (2 to 5 seconds) may leave pressure in the loop equilibrated to the vial pressure and allow more analyte to be injected. A longer loop-fill time (10 to 15 seconds) will equilibrate the loop pressure to atmospheric pressure.





**Figure 6.** Loop fill. S1 is closed. S2 is open (to fill loop).

### Loop Equilibration

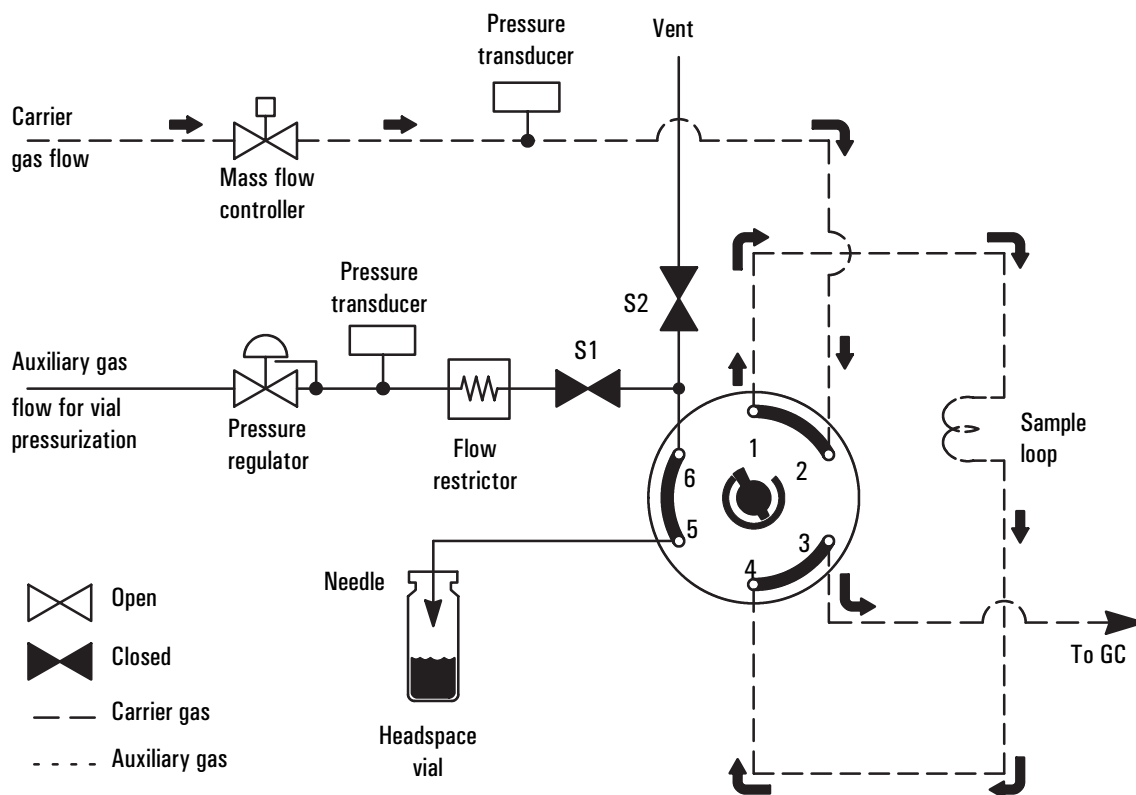
Both pressure and vent valves are closed for the length of time assigned to this setpoint.

### Injecting the Sample

The sample valve brings the sample loop in line with the carrier gas flow. The carrier gas flows through the sample loop and the transfer line into the GC inlet port. This sweeps the sample into the GC.

# Introduction to Headspace and to the 7694 Headspace Sampler

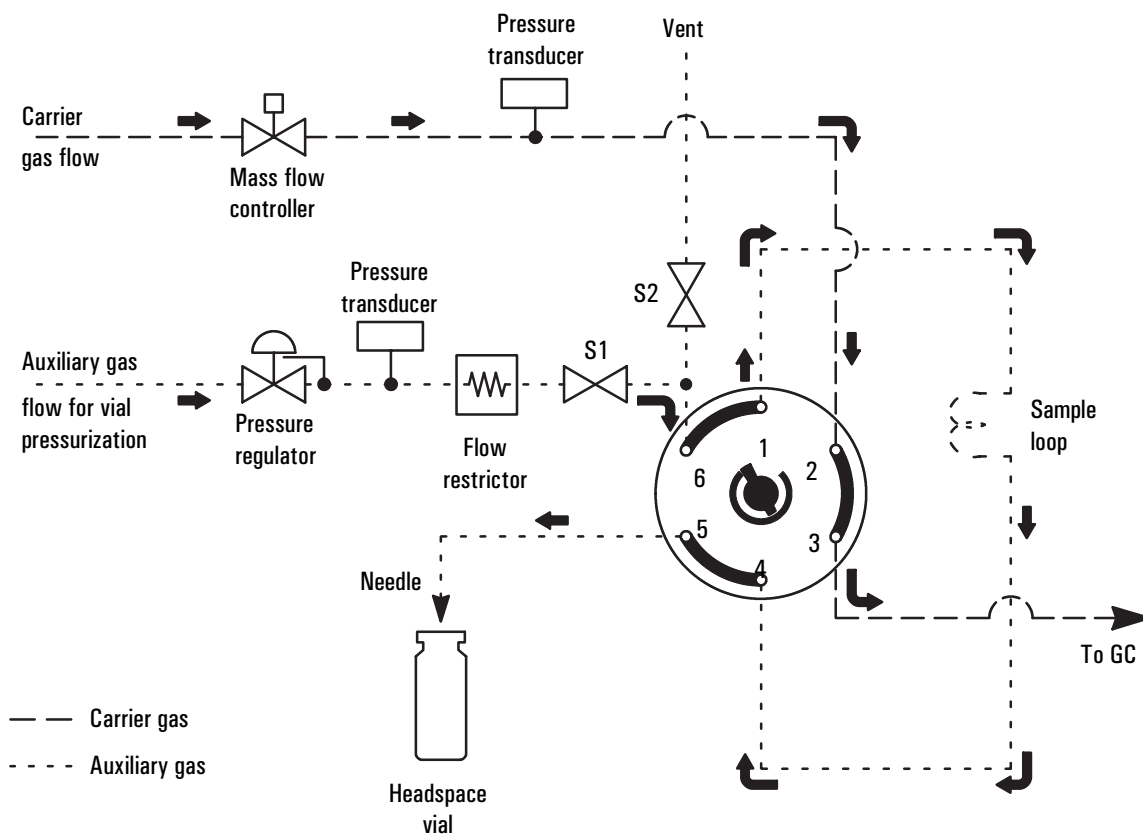
## Principles of Operation



**Figure 7. Injection. S1 and S2 are closed.**

### End of Cycle and Vial Recovery

The sample vial is lowered back into the carousel. For models with a vial tray, the carousel rotates and the robotic arm returns the vial to the tray. The vent valve opens to purge the vent line. The sampler returns to the standby configuration.



**Figure 8.** Purge. S1 and S2 are open (S2 is open for 30 seconds).

## Modes of Operation

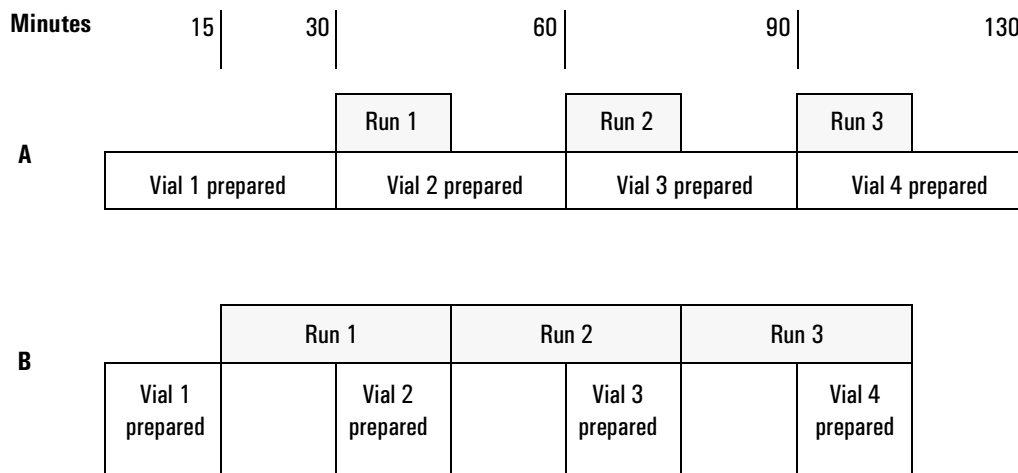
The 7694 Headspace Sampler has two modes of operation—standard headspace extraction and multiple headspace extraction.

### Standard Headspace Extraction

In standard headspace extraction mode, one extraction is made per vial. The GC run starts when the sample is injected into the GC inlet. In order to

maintain the same incubation interval during the gas chromatographic run, vials are transferred one by one into the oven and recovered after sampling. The sampler analyzes the event times and the GC cycle time, then adjusts vial delivery and recovery times to optimize overall process time. Vial agitation is allowed in this mode.

Note that the GC run cycle time is the time from injection until the system is READY for another injection. Occasionally, the time for data handling will become a factor.



The diagram above shows how the instrument actually synchronizes the availability of a vial which has been heated and is ready for injection, with the GC's readiness for another injection. For simplicity, the two cases are:

Situation **A**: total preparation time of 30 minutes, with run cycle time of 15 minutes.

Situation **B**: total preparation time of 15 minutes, with run cycle time of 30 minutes.

**Sequence of events–G1883**

The normal sequence of events is:

1. The run/method is started.
2. If agitation (shaking) is set, the carousel will shake for the entered amount of minutes.
3. The vial is lifted into the heated zone.
4. The vial is heated for the preset time.
5. A headspace injection is made.
6. The vial is returned to the carousel.

### **Sequence of events–G1289/G1290**

When a vial uses a different method from the preceding vial, maximum throughput may be effected. Runs for all vials using a particular method must be completed and the vials removed from the carousel before the new method is loaded.

The following events take place during standard headspace extraction:

1. Method loaded
2. Vial placed in oven
3. Vial heated for vial equilibration time
4. Vial sampled
5. GC started
6. Vial returned to tray

### **Multiple Headspace Extraction with Multiple Vial Puncture– G1289/G1290 only**

In multiple headspace extraction mode, up to 10 extractions can be made per vial. With multiple vial puncture, the needle is removed from the vial and the vial is returned to its original position in the oven after an extraction. For the first extraction, the vial is heated for the programmed vial equilibration time. For each successive extraction, the vial is heated for the GC cycle time. The GC is started after each sampling. Vial agitation is allowed during this mode. Sample throughput is optimized during this mode.

Multiple headspace extraction is **not** allowed if Parameter Increment (Advanced Function 2) is on.

The following events take place during multiple headspace extraction with multiple vial puncture:

1. Multiple extraction method loaded
2. Vial placed in oven
3. Vial heated for vial equilibration time
4. Vial sampled according to event times
5. GC started
6. Vial returned to slot in oven carousel
7. Second heating time started (time = GC cycle time)
8. Vial sampled according to event times
9. GC started
10. Vial returned to oven
11. Continue from Step 7 (up to number of extractions per vial)
12. Vial returned to tray

### **Multiple Vial Puncture with Constant Heating Time—G1289/G1290 only**

To perform multiple headspace extraction with multiple vial puncture where the heating time for each extraction equals the vial equilibration time, you must chain together identical methods. The number of methods equals the extractions per vial desired (maximum four). The same vial sequence is assigned to each method. A given vial is placed in the oven, sampled, then removed from the oven and placed in the sample tray. It is returned to the oven for the next extraction. Usually, four extractions per vial are adequate to allow you to obtain a good log(area) versus extraction number graph for quantitative work.

### **Multiple Headspace Extraction with Single Vial Puncture**

The use of single puncture mode is not recommended. Agilent recommends using multiple vial puncture mode.

### **Multiple Headspace Extraction for Analyte Concentration– G1289/G1290 only**

In this multiple headspace extraction mode, multiple vials are sampled to concentrate the analytes at the head of the GC column or in a cryo trap at the GC inlet. A start signal is sent to the GC only after the last vial sampling is complete. This feature is available only through ChemStation control.

Introduction to Headspace and to the 7694 Headspace Sampler  
**Modes of Operation**



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**The Keypad**

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# The Keypad

## Accessing Functions

Use the instrument keypad on the lower left front section of the instrument to view and change all settings. Pressing a key once brings up the first item in a menu of parameters connected with that key. Use the cursor keys (▲, ▼) to scroll through the menu items available for control. As you scroll to each item, the instrument display shows the current value of the setpoint for that parameter.

## Editing Setpoints

To edit a setpoint:

1. Press the key sequence for the parameter.
2. Key in the new value using the numeric keypad.

A flashing \* appears to the left of the new value as you enter it.

3. Press **Enter** to make the new value part of the active method.

If the new value is not within the allowed range, the sampler displays a message showing the allowed range. Press any non-number key to remove the error message.

At any point in a menu, press **Clear** to return to the top-level display.

See Table 2 (G1883) or Table 3 (G1289/G1290) for a quick reference of the keys and their functions.






## The G1883 Keypad

The table below lists all the keys available on your instrument, and summarizes their functions. See “*Keys and Their Functions*” on page 30 for details about key functions.

**Table 2. Quick Reference of G1883 Keys and Functions**

Key Sequence	Display	Definition/Function
<input type="button" value="Start"/>	START	Starts a run
<input type="button" value="Zone Temps"/>	VIAL	Sets heated zone equilibration temperature
<input type="button" value="Zone Temps"/> <input type="button" value="▼"/>	LOOP	Sets loop temperature
<input type="button" value="Zone Temps"/> <input type="button" value="▼"/> x 2	TR. LINE	Sets transfer line temperature
<input type="button" value="Event Times"/>	GC CYCLE TIME	Time to complete GC run
<input type="button" value="Event Times"/> <input type="button" value="▼"/>	VIAL EQ.TIME	Time vial spends heating in oven
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 2	PRESSURIZ.TIME	Time gas enters vial through needle
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 3	LOOP FILL TIME	Time when gaseous contents of vial pass through loop to vent
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 4	LOOP EQ.TIME	Time when gases in loop equilibrate
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 5	INJECT TIME	Time when gases in sample loop are swept into the GC
<input type="button" value="Vial Parameters"/>	FIRST VIAL	First vial analyzed during the method
<input type="button" value="Vial Parameters"/> <input type="button" value="▼"/>	LAST VIAL	Last vial analyzed during the method
<input type="button" value="Vial Parameters"/> <input type="button" value="▼"/> x 2	SHAKE [0, 1, 2]	Sets agitation level of oven carousel— 0 = off, 1 = low, 2 = high
<input type="button" value="Load Method"/>	LOAD METHOD	Moves a method from stored to active memory for current use
<input type="button" value="Store Method"/>	STORE METHOD	Moves a method in active memory into one of four memory storage locations
<input type="button" value="Link Methods"/>	METH #'S	Strings (chains) methods together to form a sequence












**Table 2, continued**

Key Sequence	Display	Definition/Function
		Stops and/or resets a run
		Moves carousel ahead one position
		Adds keyboard entry to current method
		Cancels a keyboard entry. Displays menu top level.
	ADV FCTN	Accesses nonroutine parameters. See <i>“Using the Adv Fctn Key”</i> on page 40.

## The G1289/G1290 Keypad

The table below lists all the keys available on your instrument, and summarizes their functions. See *“Keys and Their Functions”* on page 30 for details about key functions.

**Table 3. Quick Reference of G1289/G1290 Keys and Functions**

Key Sequence	Display	Definition/Function
	START or STOP	Starts and/or stops a run
	OVEN	Sets oven equilibration temperature
 	LOOP	Sets loop temperature
  x 2	TR. LINE	Sets transfer line temperature
	GC CYCLE TIME	Time to complete GC run
 	VIAL EQ. TIME	Time vial spends heating in oven
  x 2	PRESSURIZ. TIME	Time gas enters vial through needle

**Table 3, continued**

Key Sequence	Display	Definition/Function
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 3	LOOP FILL TIME	Time when gaseous contents of vial pass through loop to vent
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 4	LOOP EQ. TIME	Time when gases in loop equilibrate
<input type="button" value="Event Times"/> <input type="button" value="▼"/> x 5	INJECT TIME	Time when gases in sample loop are swept into the GC
<input type="button" value="Vial Parameters"/>	FIRST VIAL	First vial analyzed during the method
<input type="button" value="Vial Parameters"/> <input type="button" value="▼"/>	LAST VIAL	Last vial analyzed during the method
<input type="button" value="Vial Parameters"/> <input type="button" value="▼"/> x 2	SHAKE [0, 1, 2]	Sets agitation level of oven carousel— 0 = off, 1 = low, 2 = high
<input type="button" value="Load Method"/>	LOAD METHOD	Moves a method from stored to active memory for current use
<input type="button" value="Store Method"/>	STORE METHOD	Moves a method in active memory into one of four memory storage locations
<input type="button" value="Chain Methods"/>	METH # 'S	Strings methods together to form a sequence
<input type="button" value="Pressure"/>	CARR. PRESS	Displays current carrier gas pressure setpoint
<input type="button" value="Pressure"/> <input type="button" value="▼"/>	VIAL PRESS	Displays current vial pressurization pressure setpoint
<input type="button" value="Tray Advance"/>	TRAY POSITION	Moves slots in sample tray to their "home" position. Use cursor keys to move slots forward or backward by one position
<input type="button" value="Enter"/>		Adds keyboard entry to current method
<input type="button" value="Clear"/>		Cancels a keyboard entry. Displays menu top level.
<input type="button" value="Adv Fctn"/>	ADV FCTN	Accesses nonroutine parameters. See <i>"Using the Adv Fctn Key"</i> on page 40.

## Keys and Their Functions

### Start (G1883 only)

Press this key to start a run.

### Stop/Reset (G1883 only)

Press this key once to stop a run. To reset the unit, press the key again. The sampler aborts whatever it is doing and re-runs its startup routine. See “*Power on Diagnostics*” on page 14.

- The method cannot be resumed from the point at which you stopped it. If you want to continue the analysis from the last vial sampled, you must change the method so that it starts with the correct vial.
- If this key is pressed when the unit is not in a run, the unit will be reset.
- The last method in use before reset remains in effect.

The instrument is not ready until it reaches all setpoints.

### Start/Stop (G1289/G1290 only)

Press this key to start or stop a run. To start a run, press once. To stop a run, press the key again. The display reads:

PRESS STOP TO CONFIRM

This prevents an inadvertent run interruption. To stop, press the key again. To continue the run, press **Clear**.

- If you press the Start/Stop key while the robotic arm is moving, the display shows the message “Wait End of Movement.” Press the Stop key again after the movement is completed.
- When you stop the sampler during a run, the robotic arm automatically returns the vials in the oven to the sample tray. The method cannot be resumed from the point at which you stopped it. If you want to continue the analysis from the last vial sampled, you must change the method so that it starts with the correct vial.

## Zone Temps

Press **Zone Temps** to set temperature values for various heated zones in the sampler. Pressing **Zone Temps** brings up the following top-level display:

<b>G1289/G1290</b>		Actual	Setpoint
	OVEN	100	{120 }

<b>G1883</b>		Actual	Setpoint
	VIAL	100	{120 }

Use the cursor keys to scroll to **LOOP** or **TR. LINE** (Transfer Line) to set these temperature values. The allowed range of values is as follows:

Heated Zone	Allowed Setpoint Range*	Default Value (°C)
<b>G1883</b>		
Vial	40–200 in 1°C increments	70
Loop	50–200 in 1°C increments	50
Tr. Line	50–220 in 1°C increments	50
<b>G1289/G1290</b>		
Oven	0–200 in 1°C increments**	70
Loop	0–220 in 1°C increments**	80
Tr. Line	0–220 in 1°C increments**	90

\* The minimum oven temperature for all models is 40°C or 15°C above ambient, whichever is greater.

\*\* Do not enter a value below the minimum oven operating temperature, even though temperatures between 0–39°C are accepted.

- If a Zone Temp is changed during a run, the sampler will not display *NOT READY*, but it will adjust the zone to the new temperature value.
- As a general rule, the temperatures for the loop and transfer line should be set higher than the oven temperature to avoid condensation. Set the loop

temperature 15°C higher than the oven, and set the transfer line 25°C higher than the oven.

- To set the oven temperature at 40°C, set the loop temperature at 40°C. The minimum oven temperature is approximately 15°C above ambient.

---

**Caution**

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Oven temperature should not exceed 10°C *below* the boiling point of any solvents in the vial. Exceeding this may cause the cap and septum to leak or to pop off; the vial pressure line could also become contaminated.

## Event Times

Pressing **Event Times** brings up the first menu item:

```
Setpoint
GC CYCLE TIME
```

The cursor keys allow scrolling through the list of events. They are the following:

<b>Event</b>	<b>Allowed Setpoint Range (min. increments)</b>	<b>Default Value (min.)</b>
GC CYCLE TIME	1.0–999.9 in 0.1	25
VIAL EQ. TIME	0–999.9 in 0.1	15
PRESSURIZ TIME	0–99.99 in 0.01	0.20
LOOP FILL TIME	0–99.99 in 0.01	0.20
LOOP EQ TIME	0–99.99 in 0.01	0.05
INJECT TIME	0–99.99 in 0.01	1.00

## GC CYCLE TIME

The time required for the entire GC run to be completed. It is the sum of the GC run time and GC equilibration time (read from the GC front panel) and an estimate of the cool-down time required (if any). This time allows the 7694 Headspace Sampler microprocessor to introduce samples into the oven most efficiently and avoid injecting a sample before the GC is ready. The allowed



range of values for the cycle time is 1.0 to 999 minutes. The recommended minimum cycle time is 2.5 minutes.

### **VIAL EQ TIME (Vial Equilibration Time)**

Time the vial spends in the oven heating. The length of time you should use depends on the type of sample (solid, liquid), the amount of sample, and the partition coefficient of the analytes. The sample may or may not come to equilibrium.

- The allowed range of values is 0.00 to 999.9 minutes.
- The sampler may extend the actual equilibration time if more time is needed to put the next vial into the carousel before starting the next run.
- The recommended minimum time is 5.0 minutes.

### **PRESSURIZ. TIME (Pressurization Time)**

Time during which the vial pressurization gas is introduced into the sample vial. The allowed range of values is from 0.00 to 99.99 minutes.

### **LOOP FILL TIME**

Time in which the headspace/vial pressurization gas mixture passes through the sample loop to vent. The allowed range of values is from 0.00 to 99.99 minutes.

### **LOOP EQ. TIME (Loop Equilibration Time)**

Time to allow the analytes in the sample loop to equilibrate to the higher loop temperature and to allow pressure and flow in the loop to stabilize. The allowed range of values is from 0.00 to 99.99 minutes.

### **INJECT TIME**

Time in which the gases in the sample loop are injected into the GC. This time must be sufficient for complete sample transfer. If the time is too short, the analysis will lose sensitivity because not all the sample will be transferred. It is

not a problem if the time is longer than necessary. The allowed range of values is from 0.00 to 99.99 minutes.

## Vial Parameters

This key specifies the vial(s) analyzed during a method. Use the cursor keys to scroll through the following set of parameters.

### FIRST VIAL

Designates the first vial (1–44 for the G1289/G1290, 1–12 for the G1883) analyzed by the current method.

### LAST VIAL

Designates the last vial (1–44 for the G1289/G1290, 1–12 for the G1883) analyzed by the current method. The last vial number cannot be lower than the first vial number. The headspace sampler will give an error message and you will need to re-enter the vial number.

For models G1289/G1290, if one or more sample-tray slots is left empty between the first vial and the last vial, the instrument will check the empty positions for a vial and then move on to the next tray position.

### SHAKE [0, 1, 2]

#### G1883

Sets the agitation level of the oven carousel. Agitation is used to homogenize the sample at room temperature. Settings are as follows: 0 = off, 1 = low, 2 = high. Default is off. The stepper motor rotates the carousel at a constant frequency to agitate the vials. Agitation takes place at the beginning of a method before the first vial is heated.

If Shake 1 or 2 (low or high) is selected, then a shake time parameter will be available from the Vial Parameter function key. The display will show:

SHAKE LOW (or HIGH) MIN ###

where ### is the number of minutes from 1 to 999. If 0 is entered in the first Shake screen, it will turn shaking off.

When a multi-vial method is started, the carousel shakes for the number of minutes selected; then the first vial is lifted into the heating zone. After injection, the vial immediately returns to the carousel.

If the vial heating time is shorter than the run cycle time, then shaking resumes until the run cycle expires. Under these circumstances, the shaking time setpoint only applies to the period before the first vial begins heating. The additional shaking cannot be controlled or changed by the operator.

### **G1289/G1290**

Sets the agitation level of the oven carousel. Agitation mixes the sample in the vial for potentially faster equilibration and is used most often with liquid samples. Settings are as follows: 0 = off, 1 = low, 2 = high. The stepper motor in the oven rotates the carousel at a constant frequency to agitate the vials. Agitation takes place during the entire vial equilibration time.

### **Load Method**

Retrieves one of four stored methods and places it into active memory for use in the current analysis. See “*Loading a Method*” on page 64.

### **Store Method**

Stores the current method in one of four method storage areas. See “*Storing a Method*” on page 64.

---

**Caution**

---

Storing a method overwrites any method already stored there. There is no warning message that an overwrite is taking place.

## Chain Methods

Allows two to four methods to be used sequentially to analyze groups of samples in the tray. See “*Chaining Methods*” on page 65. Chaining methods is not allowed if Parameter Increment is on.

For the G1289/G1290, chaining methods is one means of performing multiple headspace extraction (MHE). See also “*MULTI. HS EXTR.*” on page 49.

## Link Methods

Press this key to sequentially “chain” two to four method together. See “*Chaining Methods*” on page 65. Chaining methods is not allowed if Parameter Increment is on.

## Carousel Advance (G1883 only)

This key is used to move the sample tray for vial insertion and removal. The cursor keys move the tray forward or backward by increments of one position.

## Pressure (G1883/G1290 only)

This key displays the pressure of the carrier gas or the vial pressurization gas. The carrier gas pressure is measured at the outlet of the flow controller. The vial pressurization is measured at the outlet of the pressure regulator before the in-line restrictor. It is a read-only display; no editing is allowed. Use the cursor keys to scroll the display from the carrier gas to the vial pressurization gas.

## Tray Advance (G1289/G1290 only)

This key is used to move the sample tray for vial insertion and removal. Keying in {vial position} and **Enter** moves that vial position to the “Home” position. The home position in the tray is pointed out by an arrow silk-screened on the oven. For easier access to vials for inserting and removing them, key in {position + 4} and **Enter** to bring that position to the very front of the sample

tray. The cursor keys move the tray forward or backward by increments of one position.

## **Enter**

This key is used after choosing a numeric value for a setpoint. Pressing **Enter** after making a set point change incorporates the change into the active method.

## **Clear**

The Clear key has two functions. During editing, pressing **Clear** returns the value to its previous setpoint. If not editing, **Clear** returns the display to the menu top level. You may need to press **Clear** more than once to reach the top level.

## **Numeric Keypad**

The numbered keys are used to enter setpoint values for the variable conditions of the headspace sampler. After a numeric value is keyed in, press **Enter** to make the value part of the active method.

## **Adv Fctn { }**

Makes available a series of advanced functions that are not used in routine applications. These functions are accessed by pressing **Adv Fctn**, {a number code}, and **Enter**. See *“Using the Adv Fctn Key”* on page 40 for more information.

The Keypad  
**Keys and Their Functions**

---

**Using the Adv Fctn Key**

---

## Using the Adv Fctn Key

This chapter describes the nonroutine functions controlled by the Adv Fctn key. It describes the displays, value ranges, and how to edit the setpoints for these functions.

### Using an Advanced Function

To access an advanced function setpoint or reading:

1. Press **Adv Fctn**, {**1–9**, **.1**, **.2**, **.3**}, and **Enter**.
2. Scroll to the desired subfunction, if applicable.
3. Key in the new value using the numeric keypad.

A flashing \* appears to the left of the new value as you enter it.

4. Press **Enter** to make the new value part of the active method.

If the new value is not within the allowed range, the sampler displays a message showing the allowed range. Press any non-number key to remove the error message.

At any point in a menu, press **Clear** to return to the top-level display.

See Table 4 (G1883) or Table 5 (G1289/G1290) for a quick reference of the advanced functions, and “*Advanced Function Descriptions*” on page 44 for details.



## List of Advanced Functions by Model

**Table 4. Quick Reference of G1883 Advanced Functions**

Adv Fctn + Key Sequence	Display	Definition/Function
1	GC READY TYPE	Indicates the current GC mode
2	CONST HEATING TIME	Indicates the GC is in constant heating time mode
3		Not used
4	KEYBOARD LOCK	Allows you to lock or unlock the keyboard
5	PRESSURE UNITS	Changes the pressure units
6	STABILIZE TIME	Sets wait time between oven reaching initial setpoint and first vial loading
7	PARAMETER INCR.	During method development, set to increment vial temperature or equilibration times between runs.
8	MANUAL OPERATION	Manually operate carousel advance and valves.
9	PRESSURE VALVES	Display current carrier gas or vial pressurization gas pressure.
• 2 Enter	VALVE COUNT	Displays the number of times the sampling valve has actuated.

Using the Adv Fctn Key  
**List of Advanced Functions by Model**

**Table 5. Quick Reference of G1289/G1290 Advanced Functions**

<b>Adv Fctn</b> + <b>Key Sequence</b>	<b>Display</b>	<b>Definition/Function</b>
<input type="text" value="1"/>	STABILIZE TIME	Sets wait time between oven reaching initial setpoint and first vial loading
<input type="text" value="2"/>	PARAM . INCREMENT	
<input type="text" value="2"/> <input type="text" value="Enter"/>	VIAL EQ. STEP	Sets incremental time that the vials in a series spend in the oven
<input type="text" value="2"/> <input type="text" value="Enter"/> <input type="text" value="▼"/>	OVEN TEMP STEP	Sets increment in temperature of oven for vial equilibration step in a series
<input type="text" value="3"/>	VIAL NO. AND LOC .	Displays the vial number in each oven carousel location
<input type="text" value="4"/>	KEYBOARD LOCK	Allows you to lock or unlock the keyboard
<input type="text" value="4"/> <input type="text" value="Enter"/>	KEYBOARD UNLOCKED	All keys are functional
<input type="text" value="4"/> <input type="text" value="Enter"/> <input type="text" value="▼"/>	KEYBOARD LOCKED	Numeric and enter keys are nonfunctional
<input type="text" value="5"/>	MANUAL OPERATION	The 0 and 1 keys control the motors and valves to let you check their operation. For a complete list of operations, see page 48.
<input type="text" value="6"/>	PRESSURE UNITS	Changes the pressure units
<input type="text" value="6"/> <input type="text" value="Enter"/>	PSI	Expresses pressure units in psi
<input type="text" value="6"/> <input type="text" value="Enter"/> <input type="text" value="▼"/>	BAR	Expresses pressure units in BAR
<input type="text" value="6"/> <input type="text" value="Enter"/> <input type="text" value="▼"/> x2	KPA	Expresses pressure units in KPa

Table 5, continued

Adv Fctn + Key Sequence	Display	Definition/Function
7	DEFAULT METHOD	Loads the default method into active memory
7 Enter	LOAD DEFAULT METHOD METHOD LOADED	Notifies you know that the method is now loaded
8	MULTI. HS EXTR.	Sets parameters for multiple headspace extraction
8 Enter	EXTR. PER VIAL	Specifies the number of extractions per vial (1–10). To access the next display, you must specify 2 or more extractions.
8 Enter ▼	PUNCTURE [0=S 1=M]	Specifies single or multiple puncture mode
9	HANDSHAKE MODE	Tells the 7694 whether or not to look for a ready signal from the GC before injecting
9 Enter	CHECK GC READY NO	The 7694 will not look for a signal
9 Enter ▼	CHECK GC READY YES	The 7694 will look for a signal
• 1 Enter	7694 RESET	Displays “not ready” message until setpoints are reached. 7694 RESET can be used to recover from error conditions listed in “ <i>Error Messages</i> ” on page 128.
• 2	VALVE COUNT	Use this function to view/reset the number of valve cycles.
• 3	BAUD RATE	Use this function to adjust the sampler communications rate.

## Advanced Function Descriptions

### CONSTANT HEATING TIME

**G1883**                      **Adv Fctn, 2, Enter**

This function indicates that the 7694 is always in the constant heating time mode.

### DEFAULT METHOD

**G1289/G1290**            **Adv Fctn, 7, Enter**

Pressing **Adv Fctn, 7**, and **Enter** displays the following message:

LOAD DEFAULT METHOD

Pressing **Enter** again loads a default method into the headspace sampler. The display shows the following flashing message:

METHOD LOADED

before returning to a top-level display. The following parameters are loaded in the default method:

---

<b>Oven Temp</b>	70°C
<b>Loop Temp</b>	80°C
<b>Tr. Line Temp</b>	90°C
<b>GC Cycle Time</b>	25 min
<b>Vial Eq. Time</b>	15 min
<b>Pressuriz. Time</b>	0.2 min
<b>Loop Fill Time</b>	0.2 min
<b>Loop Eq. Time</b>	0.05 min
<b>Inject Time</b>	1.0 min
<b>First Vial</b>	1
<b>Last Vial</b>	10
<b>Shake</b>	Off

---

## GC READY TYPE

### G1883                      Adv Fctn, 1, Enter

This function indicates the GC mode. Pressing **Adv Fctn, 1**, and **Enter** displays the following message:

GC IS 6890 or the current set state.

Use the scroll key to choose a different mode. The following modes are available:

GC1

GC2

5890/4890

NO GC READY

## HANDSHAKE MODE

### G1289/G1290              Adv Fctn, 9, Enter

A hardwired connection is built into the remote start cable between the 6890 or 6580 GC and the 7694 HS.

Press **Adv Fctn, 9**, and **Enter** to display the active mode. Use the scroll key to choose a different mode. The following modes are available:

CHECK GC READY              NO

The sampler will not wait for the GC ready signal.

CHECK GC READY              WAIT -L 5890

The sampler waits for 5890 GC ready signal before beginning the sequence. The headspace sampler's Sample Equilibration Time is ignored; the sequence is controlled by GC readiness.

CHECK GC READY              WAIT -H 6890

The sampler waits for 6850/6890 GC ready signal before beginning the sequence. The headspace sampler's Sample Equilibration Time is ignored; the sequence is controlled by GC readiness.

CHECK GC READY        YES -L 5890

The sequence aborts if the HP 5890 GC is not ready after the Vial Equib. time has expired.

CHECK GC READY        YES -H 6890

The sequence aborts if the 6850/6890 GC is not ready after the Vial Equib. time has expired.

Note that "L" stands for *Low* and "H" stands for *High*.

If CHECK GC READY YES is active, then the sampler will check for the presence of a ready signal from the GC just prior to injection.

If a GC ready signal is not found, the sampler will abort the run or sequence, return all vials to the tray, and display the message GC NOT READY. If CHECK GC READY NO is active, the headspace sampler will inject regardless of the ready state of the GC.

## **7694 RESET**

**G1289/G1290        Adv Fctn, ., 1, Enter**

Select 7694 Reset to force the sampler to abort whatever it is doing and re-run its startup routine. See "*Power on Diagnostics*" on page 14 for more information.

Note:

- The instrument remains Not Ready until all setpoints for the method are reached.
- The last method in use before reset remains in effect.

Adv Fctn .1 can be used to recover from certain error conditions. These include the following:

VIAL NOT FOUND

VIAL NOT LOADED

VIAL RETAINED

See “*Error Messages*” on page 128 for descriptions of these error conditions.

### **KEYBOARD LOCK**

This function disables the numeric keypad and Enter keys.

#### **G1883                    Adv Fctn, 4, Enter**

Selecting Keyboard Lock displays the following message:

KEYBOARD UNLOCKED

This is the default condition. To lock the keyboard, press **1** (Yes).

KEYBOARD LOCKED

To unlock a locked keyboard, press **0** (No).

#### **G1289/G1290            Adv Fctn, 4, Enter**

Selecting Keyboard Lock displays the following message:

KEYBOARD UNLOCKED

This is the default condition. To lock the keyboard, scroll to the following display:

KEYBOARD LOCKED

Once the keyboard is locked, scrolling to KEYBOARD UNLOCKED will unlock the keyboard.

**MANUAL OPERATION**

**G1883 Adv Fctn, 8, Enter**

This function manually operates the vial carousel advance, and valves to check for malfunctions. Selecting Manual Operation displays the last operation used. The cursor keys will scroll through the messages.

<b>Selected Operation</b>	<b>Key Pushed</b>	<b>Result</b>
VIAL LOAD	1	To mid-position from down position (vial in heated zone)
	1 again	To top position (needle in vial) Vial goes into carousel (out of heat and needle)
PRESS. VALVE	0	Off: valve is open to flow
	1	On: valve is closed to flow
VENT VALVE	0	Off: valve is closed to flow
	1	On: valve is open to flow
SAMPLE VALVE	0	Off: the loop to pressure vent valves
	1	On: the loop is in the carrier stream
CAROUSEL POSITION	0	Move location down
	1	Move location up

**G1289/G1290 Adv Fctn, 4, Enter**

This function manually operates the vial gripper arm, tray advance, oven advance, vial loading, and valves to check for malfunctions. It is especially useful for checking that the gripper arm and gripper mechanism are properly aligned. The cursor keys scroll through the following menu of items:

- GRIPPER Y AXIS UP
- GRIPPER X AXIS 1
- TRAY ADVANCE
- OVEN ADVANCE



VIAL LOAD	DOWN
PRESS. VALVE	ON
VENT VALVE	OFF
SAMPLE VALVE	OFF
OVEN CALIBRATION	

The 0 and 1 keys activate the appropriate motors (according to the display) for x and y movement of the robotic arm, movement of the sample tray and oven carousel, and toggle the on/off states for the valves.

When you exit Adv Fctn 5, all the motors are not reset to their default states. Use **Adv Fctn, ., 1, Enter** to reset.

If the operations do not work, “*Troubleshooting*” on page 116, then call Agilent service.

### Oven Calibration

Contact Agilent service to recalibrate the oven.

### MULTI. HS EXTR.

**G1289/G1290**      **Adv Fctn, 8, Enter**

---

**Note**

---

MHE is not allowed if Parameter Increment is on.

This function sets the parameters used for multiple headspace extraction (MHE) in single-puncture or multiple-puncture (recommended) mode. Pressing **Adv Fctn, 8**, and **Enter** brings up the following display:

EXTR. PER VIAL    {4}            (example)

The user selects a value of {1-10} followed by **Enter** for the number of extractions per vial. If extractions per vial = 1 (default), then other displays associated with MHE are not available with the scroll keys.

While not recommended, you can also set single puncture mode for MHE by pressing the cursor key to bring up the following display:

PUNCTURE [0=S, 1=M] {1} (example)

Press **0**, **Enter** to enable single puncture mode or **1**, **Enter** to disable it. The display reads:

SINGLE PUNCTURE ON

or

MULTI PUNCTURE ON

Default is multipuncture mode.

The following are definitions for terminology used with Adv Fctn 8:

**Multi. HS Extr.** — Multiple headspace extraction.

**Multi. Puncture On** — Recommended. Multiple headspace extraction with multiple needle punctures of the septum. This is the default mode of the sampler.

**Single Puncture On** — Do not use MHE with single vial puncture.

**Extract Per Vial** — Number of extractions per vial for either the multiple or single needle puncture mode.

#### **PARAMETER INCR.**

Parameter increment is a method development tool that makes stepwise increases in either vial equilibration time or oven temperature automatically.

Entering a value for the Vial Equilibration Increment increases the Vial Equilibration Time by that many minutes for the second vial and each subsequent vial in the present method. The value range is 0.0 to 99.9 minutes.

Entering a value for the Vial Temp Incr increases the Vial Temperature during vial equilibration by that many degrees for the second vial and each subsequent vial in the present method. The value range is from 0 to 99 minutes.

During parameter incrementing, each vial is sampled and returned to the carousel. The sampler displays 7694 NOT READY for the duration of the GC Cycle Time between each incrementation. If Vial Temp is incremented, the display reads WAITING FOR READY until the new setpoint is reached. The default values for both displays are 0.0, which means that the parameter increment is off. The two parameters are mutually exclusive: if a nonzero number is entered into one, the other must be set to zero.

- Do not allow the maximum vial temperature to be closer than 10°C to the boiling point of any solvent in the sample.
- The **Vial Temp Incr** function increases the temperature from run to run in the headspace vial only. The loop and transfer line temperatures remain at their original setpoints. Be sure to set the loop and transfer line temperatures so they exceed the highest temperature the vial is programmed to reach. Take note of any maximum temperature on the transfer line you are using.
- Chain methods are not allowed if either the Vial Temp Incr or the Vial Equilibration Incr is set to a non-zero value.
- If your combination of vial temperature step and number of vials to run would set the oven temperature above its maximum (for example, the open temperature is 155°C, the step is 10°C, and you run 6 vials), you will see a warning message. If you start the run anyway, the analysis will stop when the oven reaches 200°C.

### **G1883                      Adv Fctn, 7, Enter**

To activate/deactivate Parameter Increment, press Adv Fctn, 7, and Enter. You will see

PARAMETER INCR. NO or PARAMETER INCR. YES

Press the **1** (YES) key to activate parameter incrementing, or **0** (NO) to deactivate it.

When enabled, you can use the cursor keys to scroll to the displays to set the vial temperature and vial equilibration time step increments:

VIAL TEMP INCR            0.0

or

VIAL EQ. INCR      0.0

Change the increments from the numeric keypad and then press the **ENTER** key to execute the change in value.

**G1289/G1290      Adv Fctn, 2, Enter**

Select Parameter Incr. and use the cursor keys to change the vial equilibrium time step (increment) and the oven temperature step:

VIAL EQUIL STEP    0.0

The cursor keys scroll to the next display:

OVEN TEMP STEP    0

## **PRESSURE UNITS**

**G1883              Adv Fctn, 5, Enter**

**G1289/G1290      Adv Fctn, 6, Enter**

This function changes the pressure units for the displays among psi, kPa, and bar. Pressing **Adv Fctn, 6**, and **Enter** displays the following message:

PRESSURE UNITS    PSI

Psi is the default for pressure units when the sampler unit is powered up. Use the cursor keys to scroll to kPa or bar. When units are shown on the display, they are active.

## PRESSURE VALVES

**G1883**                    **Adv Fctn, 9, Enter**

Press **Adv Fctn, 9, Enter** to display the sampler's pressure sensor readings for either the vial pressurization gas or the carrier gas. Use the cursor keys to scroll between the two choices. The display is read-only.

VIAL PRESS PSI    XX            (example)

CARR. PRESS PSI    YY            (example)

## STABILIZE TIME

**G1883**                    **Adv Fctn, 6, Enter**

**G1289/G1290**            **Adv Fctn, 1, Enter**

This function changes the length of time the oven equilibrates after reaching its initial setpoint before vials are loaded into the oven. Selecting Stabilize Time displays the following:

STABILIZE TIME    1.0

One minute is the default time. The allowed range of values is 0.0 to 99.9 minutes.

## VALVE COUNT

**G1883**                    **Adv Fctn, ., 2, Enter**

**G1289/G1290**            **Adv Fctn, ., 2, Enter**

Displays the number of times the sampling valve has actuated. Agilent service personnel may refer to the valve count to get an idea of the extent of the sampler's use.

**VIAL NO. AND LOC .**

**G1289/G1290      Adv Fctn, 3, Enter**

Selecting Vial No. and Loc. displays the following:

05-06-07-08-09-10                      (EXAMPLE)

It tells the user what vial number is in which position in the oven carousel. The first number on the left is the first oven carousel position, the last number on the right is the sixth oven carousel position. In this example, vial number five is in oven carousel position one and vial number 10 is in position six. This is a read-only display. No editing is possible.

---

**Preparing a Sample**

---

# Preparing a Sample

This chapter describes the steps necessary to prepare a sample for analysis, including filling and sealing a sample vial and loading the sample tray.

## Sample Preparation

The 7694 Headspace Sampler can accept either 10- or 20 ml sample vials. You cannot mix vial sizes when loading the tray. Before using 10 ml vials, you must insert adapters into the spaces in the sample tray and in the oven carousel.

Samples that will undergo headspace extraction are placed in the sampler in glass vials. You also must flip the switch on the back of the horizontal loader to the proper size setting.

In general, sample vials should be filled only about half way (although vials can be filled to the 3/4 level). This ensures that the needle will not come into contact with the solid or liquid phase during the sampling step.

The vial must be sealed properly to insure that the headspace gases do not escape prematurely. Use a crimper designed for headspace vials with 20 mm caps to seal the vials (Agilent part no. 9301-0720).

---

**Note**

---

Sampling from an incorrectly capped vial may cause the 7694 Headspace Sampler to jam during septum puncture.

Do the following to prepare a vial for sampling:

1. Place a septum in a vial cap with the Teflon side facing the vial. Take care not to contaminate the septum.
2. Place the cap upside down on a table.
3. Place the sample in the vial. The vial should not be more than 50% full.
4. Place the septum and cap together over the vial opening.
5. Place the crimper over the cap and vial.



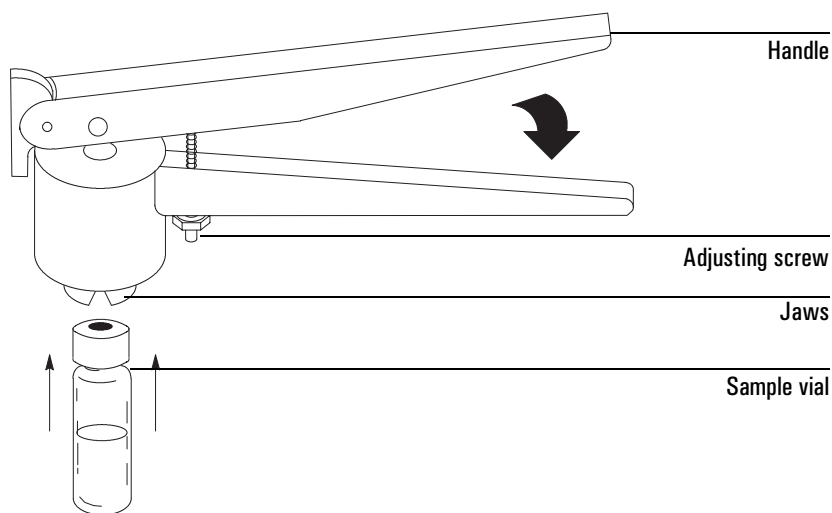
6. With slow and steady pressure, squeeze the crimper handles to seal the vial.
7. Remove the crimper.
8. Check that the seal is adequate by trying to twist the cap on the vial. It should be difficult or impossible to turn the cap. If you can easily turn the cap on the vial, re-crimp the cap. You may need to adjust the crimping tool.
9. To decap vials, use a decapper for vials with 20 mm caps (part no. 5181-1214).

---

**Note**

Vials will contain laboratory air, which may be contaminated with things such as solvents or Freon leaking from air conditioners. If contaminants will adversely affect your results, use argon to purge the vials just before capping and sealing.

---



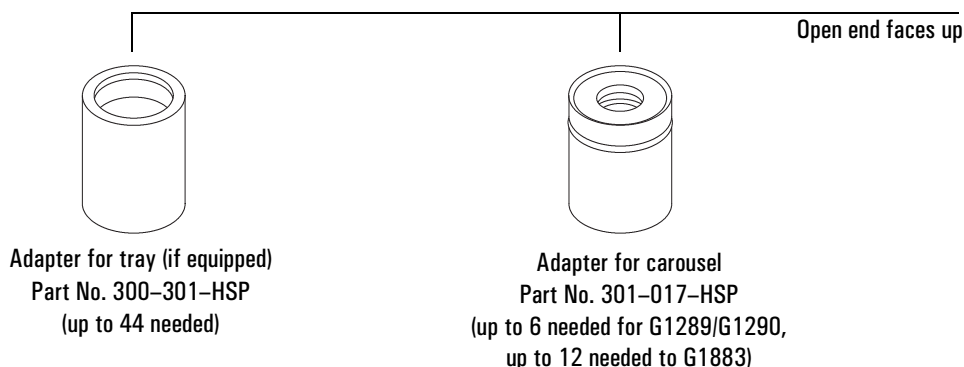
**Figure 9. Position of crimper and vials for proper capping**

## Loading the Sample Tray and/or Carousel

**G1883:** Press **Carousel Advance** and **Scroll** until the carousel rotates to the desired position.

**G1289/G1290:** Press **Advance Tray**, {position+4}, and **Enter** to move the desired loading position to the very front of the sample tray.

If you are using 10 ml vials, be sure to place inserts into the spaces in the oven carousel. Be sure the inserts are placed in the carousel with the closed end down (see Figure 10). Finally, be sure to set the vial-size switch on the back of the horizontal loader to the proper size. Using the cursor keys, advance tray positions one-by-one while adding vials and inserts until all samples are loaded into the tray.



**Figure 10. Adapters for use with 10 ml vials**

When loading a tray (if equipped) or carousel:

- Do not mix 10 ml and 20 ml vials in a tray, if equipped.
- Be sure to insert the oven adapters with the closed end down.
- To remove oven adapters, bend a large paper clip to make a hook and insert the hook into the lip on the inside of the adapter.

---

### WARNING

---

Oven adapters are hot when first removed. Place them on a heat-resistant surface. Do not touch them with bare hands until they are cooled.

---

**Entering a Method**

---

# Entering a Method

This chapter details the procedure for entering a method, including storing and retrieving a method, and chaining methods.

## Entering a Method

To enter a method for the 7694 Headspace Sampler via the keypad, setpoints must be set for the keys labeled **Zone Temps**, **Event Times**, and **Vial Parameters**. Each of these keys has a number of setpoints associated with it. The cursor keys (**▲**, **▼**) scroll up and down through the list of setpoints. To enter a value for a setpoint, scroll to that setpoint, key in the value using the numeric keypad, then press **Enter**. If the value keyed in is not within the allowed range, the sampler displays a message showing the allowed range. Pressing any non-number key removes the error message. If the value keyed in is not the one desired, pressing **Clear** returns you to the top level of the display.

## Entering Zone Temps

This key allows you to enter values for the oven temperature, loop temperature, and transfer line temperature. Press **Zone Temps**. The sampler displays the following:

<b>G1289 and G1290</b>	<b>G1883</b>
OVEN	VIAL

Key in the desired value for the oven temperature setting followed by **Enter**. The allowed range for this setting is 0-200°C (40–200°C for G1883).

---

### Caution

The oven temperature should not exceed 10°C below the boiling point of any solvent in the vial. Exceeding this may cause the cap and septum to leak or to pop off.

---

---

**Note**

Although the sampler may allow you to enter temperatures from 0°C to 39°C, the minimum oven temperature is 40°C or 15°C above ambient, whichever is greater.

---

Use the down cursor key (▼) to scroll to the following display:

LOOP

Key in the desired sample-loop temperature (within the allowed range of 0–220°C for the G1289/G1290, 50–220°C for the G1883) followed by **Enter**. Use the down cursor key (▼) to scroll to the last setpoint display:

TR. LINE

Key in the desired setpoint on the numeric keypad for the transfer line temperature followed by **Enter**. The allowed range is 0–220°C for the G1289/G1290 or 50–220°C for the G1883.

---

**Note**

As a general rule, the loop and transfer line temperatures should be set higher than the oven temperature. Set the loop temperature 15°C higher than the oven, and set the transfer line 25°C higher than the oven.

---

## Setting Event Times

Using this key, you enter values in minutes for the timed events which make up the vial sampling sequence. Pressing **Event Times** displays the first timed event:

GC CYCLE TIME

From the numeric keypad, enter a value for this event, which is the time for the total GC run to complete. (Total run includes the GC run and cool-down time and the vial equilibration time.) Press **Enter** after keying in the numeric value. Use the down cursor key (▼) to bring you to the next timed event:

VIAL EQ. TIME

The Vial Equilibration Time is the time which the sample vial spends heating in the oven to form the headspace gas. Enter the desired time from the numeric keypad followed by **Enter**.

The down cursor (▼ ) brings you to the next display:

PRESSURIZ . TIME

Pressurization Time specifies the time that gas is allowed to enter the vial through the sampling needle to build up additional pressure in the vial. Key in the desired time on the numeric keypad followed by **Enter**.

Use the down cursor (▼ ) to scroll to the next display:

LOOP FILL TIME

The Loop Fill Time is the time during which the vent valve is open. The gases in the sample vial travel through the sample loop and are vented to the outside. This fills the sample loop with a precise aliquot of headspace gas. Key in the desired time with the numeric keypad followed by **Enter**.

Use the down cursor key (▼ ) to scroll to the next display:

LOOP EQ . TIME

The Loop Equilibration Time is the time after the vent valve closes during which the gases in the sample loop equilibrate with the pressure and temperature of the sample loop. Key in the desired value using the numeric keypad followed by **Enter**.

Use the down cursor key (▼ ) to scroll to the last display:

INJECT TIME

The Inject Time is the time in which the sample valve is open and the gases in the sample loop are swept into the GC. Key in the desired value from the numeric keypad followed by **Enter**.

These times may be checked on the display and changed while the headspace sampler is running samples. Changes are applied to the next vial analyzed. You cannot change parameters for a vial currently being sampled. The keys involved are locked.

## Setting Vial Parameters

Using this key, you specify the locations in the sample tray from which vials will be taken for headspace analysis.

1. Press **Vial Parameters** to bring up the first display:

FIRST VIAL

First Vial specifies the location of the first vial to be analyzed by this method. Use the numeric keypad to key in the vial location followed by **Enter**.

2. Use the down cursor key (▼) to scroll to the next display:

LAST VIAL

Last Vial specifies the location of the last vial to be analyzed by this method. Use the numeric keypad to enter the vial location followed by **Enter**. The number of the last vial cannot be lower than the first vial number. The headspace sampler gives an error message and you will have to re-enter the vial number.

3. Use the down cursor key (▼) to scroll to the last display:

SHAKE [0,1,2]

This specifies the agitation level of the oven carousel during vial equilibration: 0 = off, 1 = low, 2 = high. Specify the level desired by keying in {0, 1 or 2} followed by **Enter**.

4. **G1883 sampler only:** After turning on agitation, set the agitation time. Use the down cursor (▼) key to scroll to:

SHAKE LOW (MIN) *setpoint*

Enter the desired setpoint of number of minutes and press **Enter** to save.

At this point, all the parameters in a method necessary to run a sample are entered. To save this method, use **Store Method**. To run a sample, make sure that vials are loaded into the proper spots in the sample tray then press **Start**.

## Storing and Loading Methods

### Storing a Method

**Store Method** places the method in active memory into one of four storage locations for future use. To store the method currently in use:

1. Press **Store Method**. This displays the following message:  
STORE METH. [1-4]
2. Enter the storage location desired {1-4} on the numeric keypad followed by **Enter**.

The method currently in use is put into the specified memory location. Parameters stored as parts of a method are Zone Temps, Event Times, Vial Parameters, and the advanced functions for Oven Stabilization Time, Pressure Units, and Multiple Headspace Extraction (G1289/G1290 only).

---

**Caution**

---

Storing overwrites any method already in that location. There is no warning message that an overwrite is taking place.

---

**Note**

---

When the 7694 HS is first turned on after a loss of the battery-backed-up RAM, the default method is loaded into all four method-storage locations.

### Loading a Method

You can retrieve a method from memory for use in the current analysis by using the Load Method key. To bring a method up from memory:

1. Press **Load Method**. The sampler displays the following message:

G1289 and G1290	G1883
LOAD METH. [1-4]	LOAD METH. [1-5]

For the G1883, you can load method 5, the default method. This method cannot be edited or chained.

2. Enter the number of the method you desire to retrieve on the numeric keypad and press **Enter**.



The method stored in the specified location is now available for current use.

---

**Note**

---

A method may not be loaded during a run.

## Chaining Methods

**Chain Methods** allows two to four stored methods to be used sequentially to run groups of samples. It provides the user with some automation. Once the chain (or sequence) is programmed in and the samples loaded, the sampler will run the sequence without further operator intervention. Method chaining is also a means of performing multiple headspace extraction (MHE).

### Activate a Method Chain

To activate a method chain:

1. Press **Chain Methods**. The display reads as follows:  

```
METH # 's
```
2. Press a method number {1-4}. A flashing \* appears on the display followed by the method number chosen.
3. Key in the method sequence in the order to be run.
4. Press **Enter**. The following flashing message is displayed:

```
METH CHAIN ON
```

Methods are run in order from left to right on the display. For example, keying in the following:

```
2 4 1 3   Enter
```

would have the sampler run method two, followed by method four, then methods one and three.

Press **Chain Methods** again to display the current chain sequence. This method sequence is active until you edit it or press **Load Method**.

## Deactivate a Method Chain

To deactivate a method chain:

1. Press Chain Methods. The display shows:

```
METH #++##++#
```

where # is the number of a method.

2. Press **0** (NO) to display:

```
METH CHAIN OFF
```

---

### Note

---

If Parameter Increment is active, Chain Methods is inactive. Turn off Parameter Increment to use the method chain feature.

## Using a Method Chain for MHE

Use **Chain Methods** to perform a multiple headspace extraction with multiple punctures and constant equilibration time for each extraction.

To run an MHE:

1. Key in the parameters for Zone Temps, Event Times and Vial Parameters for the method.
2. Store this method in one of the four storage locations.
3. Press the following:

```
Chain Methods {X + X + X + X }
```

where X is the location where your method is stored.

## Using a Method Chain for Running More Than 44 Samples

By chaining the same method to itself, you can run up to 88 vials. To run more than the 44 sample vials in the tray, do the following:

1. Key in the vial parameters:

First Vial: 1

Last Vial: 44

2. Press the following:

Chain Methods {X + X + X + X }

where X is the location where your method is stored.

3. Manually change the vials in the tray at the appropriate time so that you do not re-sample the same vials.

Entering a Method  
**Using a Method Chain for Running More Than 44 Samples**

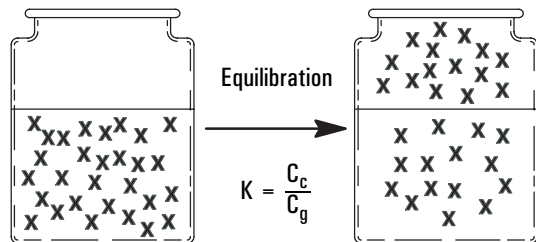
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**Developing Methods**

---

# Developing Methods

This chapter discusses various parameters that can affect the sensitivity, precision, and accuracy of an analysis.



**Figure 11. Illustration of partition coefficient**

The tendency of a material to go into the gaseous phase is the partition coefficient,  $K$ , where  $C_c$  is the concentration of the analyte in the condensed phase (the sample matrix) and  $C_g$  is the concentration of the analyte in the gaseous phase (the headspace). The partition coefficient,  $K$  is related to the degree of solubility that the analyte has in the matrix. For example, benzene is not very soluble in water, and has a  $K$  of approximately seven. Ethanol, which is very soluble in water, has a  $K$  of 7000. A high value of  $K$  implies it is difficult for the analyte to leave the matrix and go into the headspace.

$K$  is also very dependent on temperature, as demonstrated by the following equation:

$$\frac{dK}{dT} = \frac{1}{T^2}$$

Processes that reduce the value of  $K$  will increase the sensitivity of the headspace analysis. The following operations can be used to decrease  $K$ :

1. Add mineral salts to the matrix (aqueous samples)
2. Add another liquid to the matrix
3. Increase the temperature

## Varying Oven Temperature

The oven temperature can have a profound effect on the concentration of analyte that passes into the headspace gas. In general, as the oven temperature increases, the amount of gas entering the headspace from the sample increases, increasing the pressure in the vial and delivering more analyte to the GC. This increases the sensitivity of the analysis.

When you pick an oven temperature, consider the following:

- Over-pressurization of the headspace vial could be dangerous.
- Unstable compounds could degrade at elevated temperatures.

There are two ways you can program the 7694 HS to increase oven temperature in steps:

- Use Chain Methods (see “*Chaining Methods*” on page 65) and setting the oven temperature higher in each method in the chain
- Use Param Increment (see “*Advanced Function Descriptions*” on page 44).

---

**Note**

---

Do not set the oven temperature within 10°C of the boiling point of any solvents in the sample, except in special cases.

---

**Note**

---

Loop and transfer line temperatures should be set higher than the highest temperature the oven will reach in a programmed sequence.

## Varying Event Times

### Vial Equilibration Time

The time that the sample vial spends in the oven determines the amount of analyte in the headspace gas and the presence or absence of equilibrium. The 7694 HS can be programmed to analyze a series of samples to determine if equilibrium is reached.

To determine if equilibrium is reached, use Chain Methods (see “*Chaining Methods*” on page 65) to increase the Vial Equilibration Time for each method in the chain, or use Param Increment (see “*Advanced Function Descriptions*” on page 44) to increase the Vial Equilibration Time for each successive sample in the series. Then plot the peak area versus equilibration time for a certain peak. The system is at equilibrium when this curve flattens out. See Figure 12.

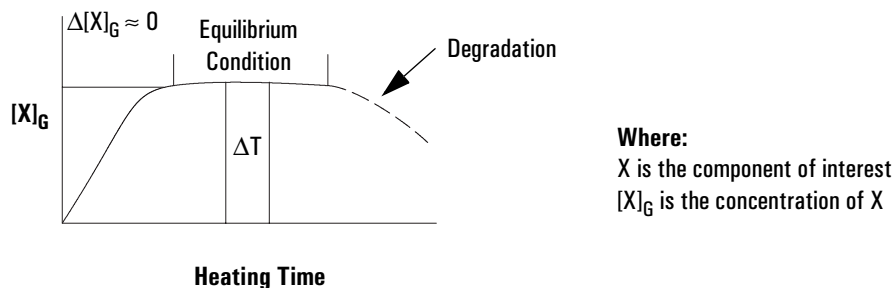


Figure 12. Equilibration curve

### Loop Fill Time

Normally, set the loop fill time to 10 seconds.

A very short loop–fill time (one or two seconds) may help increase sensitivity in headspace analysis. The headspace gas purges the sample loop, but the vent valve is closed before the sample vial falls to atmospheric pressure. The loop is maintained at the vial’s still-elevated pressure. Because the loop pressure is greater, the analyte will be more concentrated and, consequently, more sample will be injected into the GC.

### Matrix Effects

The composition of the sample matrix can affect the amount of analyte that escapes into the headspace. Adding an inorganic salt to aqueous samples increases the concentration of organic molecules in the headspace by making them less soluble in the sample matrix, increasing sensitivity. For nonaqueous



but water-miscible systems, dilute the sample with water. This decreases the solubility of most organic molecules in the sample and increases their concentration in the headspace.

Matrix effects are important when developing external standards. The composition of the matrix in the standard should closely resemble that of the sample, or accuracy suffers. The effect of the matrix on a standard can be compensated for by using the standard addition method or by quantifying results using MHE.

## Changing the Sample Loop

Although the 1 ml sample loop is usually adequate, a larger sample injection for trace analysis might be desired. A 3 ml loop is available. See “*Changing the Sample Loop*” on page 92.

---

**Note**

---

A larger sample injection may broaden some peaks, especially on a capillary column.

## Sample Size

A larger sample size may give greater sensitivity. Peak areas are often strongly influenced by the relative amount of the gas and condensed phases in the sample vial. Increasing the sample size may give corresponding increases in peak areas. If sensitivity is not an issue, small samples may be preferred because the required equilibration time is shorter.

## Vial Pressurization and Time

### Vial Pressurization

With many liquid (e.g. aqueous) samples, the pressure developed in the vial may be sufficient to ensure filling the valve loop without additional pressure.

Some samples (e.g. dry plastics) may not create enough head pressure, and do require more, in the method.

In general, 1.5 –2 atmospheres pressure in the vial will be appropriate for filling the valve loop. It may be possible to make or buy a pressure gauge fitted with a needle that could be used to determine the actual vial pressure developed under your time and temperature conditions. Set the method vial pressurization at a slightly higher value (e.g. 3–5 psi), if your measured pressure is not enough to ensure adequate loop filling.

To optimize your selection of vial pressurization, run a series of vials using different pressures, and interpret the optimum conditions from peak areas versus vial pressurization.

---

**Note**

Optimization is valid only for analysis of a specific sample at a specific oven temperature.

---

### **Vial Pressurization Time**

Typical vial pressurizations times range from 10 to 30 seconds.

## **Optimizing Carrier Gas Flow**

The 7694 Headspace Sampler carrier-gas flow rate should be set high enough to sweep the headspace sample out of the sample loop into the GC without causing peak broadening. For example, for a packed GC column that gives 15–second–wide peaks, flow would be at least 6 ml/min for a 1 ml loop. This will sweep out the loop in 10 seconds.

---

**Note**

Some detectors, such as ECDs and TCDs, are more sensitive at lower detector flow rates.

---

## **Recommended 6890/6850 GC Liner**

For the split/splitless inlet, Agilent recommends using the deactivated splitless liner, part no. 5181-8818.

Developing Methods  
**Recommended 6890/6850 GC Liner**

---

**Running a Sample**

---

# Running a Sample

To run a sample, perform the following:

1. If the transfer line is not connected to the GC, connect it.
  - If using a needle through the septum, see your installation manual.
  - If using a volatiles interface, see page 96.
2. Check your gas supplies to make sure you have sufficient gas available for all your analyses.
3. Set the flow and pressures for the headspace sampler. See page 78.
4. Set the GC conditions. See your GC operating manual.
5. Load the method or set the method chain you want to use. See page 64 or page 65.
6. Prepare the samples. See 5Preparing a Sample.
7. Load the samples into the tray (G1289/G1290) or carousel (G1883). See page 58.
8. When the GC and HS are ready, press Start.

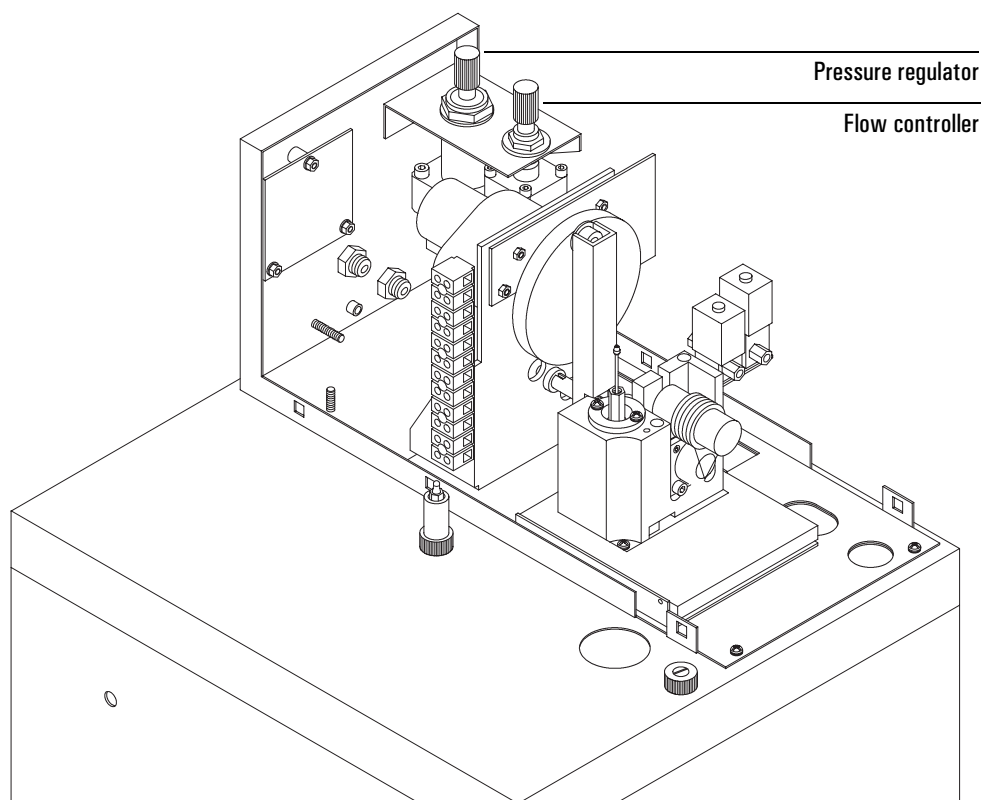
## Setting Conditions

### Flow Rates

Refer to your GC operating manual for details about its flow systems.

Two distinct versions of the 7694 Headspace Sampler exist. Models G1883 and G1290 are supplied with mechanical pneumatic controls; model G1289 is designed for use with a GC with EPC control, and so it contains no pressure regulator or flow controller. The procedure for setting flow rates differs for these two versions.

As a general rule, when you are setting flows for the headspace sampler, most of the flow should come through the transfer line from the headspace sampler.



**Figure 13. Location of flow controller and pressure regulator (G1883 and G1290 only)**

## Setting the Carrier Gas Flow Rate for the G1883/G1290

Set the carrier gas flow as follows:

1. Set the following GC parameters:

### Inlet (Volatiles Interface or Split/Splitless)

Mode	Split
Initial temperature	Per method
Pressure	Per method
Split ratio	1:1 (G1290)
Split flow	4 ml/min (G1290/G1883) at GC EPC
Gas saver	Per method
Gas type	Per method

### Column

Mode	Per method
Initial flow	4.0 ml/min
Initial pressure	Per method
Outlet pressure	Ambient (per detector)

2. Set the split flow to 4 ml/min. The GC total flow will read 11 ml/min.
3. With an electronic flow meter connected to the GC split vent, turn up the headspace carrier gas flow until the flow rate out the split vent equals the flow rate desired in the column. (Because the split ratio is 1:1, this flow rate equals the column flow rate.) See Figure 14. Remove the flow meter.

Note that the GC and Headspace pressure readings should be similar. Record the headspace pressure reading—you can use it to set up future checkout runs.

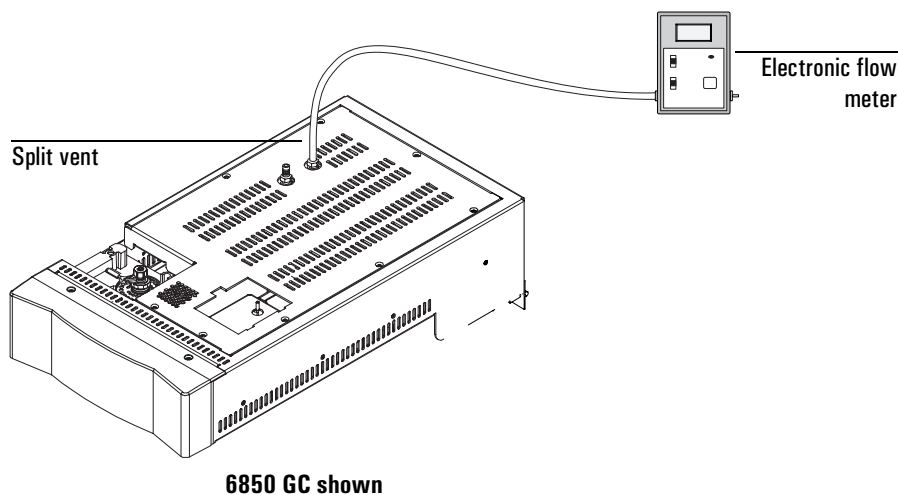
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### Caution

The added flow of the headspace sampler will cause the flow calculations of the inlet to be incorrect. If your added flow and pressure exceed the limits of the inlet, the GC may go “Not Ready.”

---





**Figure 14. Measuring the split vent flow rate**

### **Setting the Vial Pressurization Gas Pressure for the G1883/G1290**

Control the gas pressure used for vial pressurization using the pressure regulator. See Figure 3 on page 12.

To read the actual pressure value, press Pressure and use the cursor keys to scroll to the Vial Press PSI display.

### **Setting Carrier and Vial Pressurization Flows for the G1289**

Set the carrier gas flow rate directly using the GC or GC data system.

Set the vial pressure directly using the appropriate auxiliary channel in your 6890 GC.

Running a Sample  
**Setting Conditions**

---

**Maintaining the Instrument**

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# Maintaining the Instrument

This chapter describes the procedures for maintaining the instrument. Table 6 lists routine maintenance activities that should reduce unplanned repairs and maintenance. The maintenance frequency appropriate for your instrument will vary significantly depending on the sample matrix, solvents, temperatures, and sample throughput.

**Table 6. Recommended Maintenance Schedule**

<b>Maintenance Activity*</b>	<b>Frequency*</b>
Replace sample probe	3 month
Steam clean flow path	3 months
Check vial transport alignment and adjust if necessary	3 months
Check carousel alignment and adjust if necessary	6 months
PM Service and inlet maintenance	12 months
Flowpath / vent parts replacement	24 months

\* These tasks and frequencies are based on experience. They are not based on life time testing or benchmark testing.

## Common Replacement Parts and Consumables

Below is a list of commonly used parts. Also refer to the Agilent catalog for consumables and supplies.

**Table 7. Common Replacement Parts and Consumables**

Description	Comment/use	Part Number
<b>Adapters and tools</b>		
Decapper, 20-mm		5181-1214
Crimper, 20-mm		9301-0720
Tray insert, for 10 ml vial, 25/pk	For use in tray with 10 ml vials	300-301-HSP
Oven/carousel insert, for 10 ml vial, 1 each	For use in carousel with 10 ml vials	301-017-HSP
Wrenches, 7 × 8 mm, Set of 2		134-0407010-HSP
Leak test fittings	For capping the transfer line and other connections	325-0050010-HSP
<b>Vials and caps</b>		
Flat bottom, 20 ml, 23 mm × 75 mm, 144/pk		5182-0837
Flat bottom, 10 ml, 23 mm × 46 mm, 144/pk		5182-0838
One piece silver aluminum crimp caps, 20 mm, with PTFE/silicone septa, 100/pk	Preassembled. Wider temperature range septa, need higher sensitivity.	5183-4477
One piece silver aluminum crimp caps, 20 mm, with PTFE/silicone septa and safety feature, 100/pk	Preassembled. Wider temperature range septa, need higher sensitivity.	5183-4478
One piece silver aluminum crimp caps, 20 mm, with PTFE/butyl septa, 100/pk	Preassembled. General purpose.	5183-4479
One piece silver aluminum crimp caps, 20 mm, with PTFE/butyl septa and safety feature, 100/pk	Preassembled. General purpose. 45 psi.	5183-4480

**Table 7. Common Replacement Parts and Consumables (continued)**

Description	Comment/use	Part Number
<b>Vial kits</b>		
20 ml flat bottom Headspace crimp top vials, silver aluminum one piece crimp caps with safety feature, molded PTFE/butyl septa, 144/pk	General purpose	5182-0839
20 ml flat bottom Headspace crimp top vials, silver aluminum one piece crimp caps with safety feature, molded PTFE/silicone septa, 144/pk	Wider temperature range septa, need higher sensitivity.	5182-0840
<b>Sample loops</b>		
0.25 ml nickel		169-0011-HSP
0.5 ml nickel		169-0012-HSP
1 ml nickel		321-055-HSP
2 ml nickel		169-0013-HSP
3 ml nickel		321-056-HSP
1 ml Silcosteel®		321-058-HSP
3 ml Silcosteel®		321-059-HSP
<b>Probes/unions</b>		
Nickel needle assembly for G1289 or G1290		301-013-HSP
Silcosteel® needle assembly for G1289 or G1290		301-220-HSP
Nickel needle assembly for G1883		232-2790010-EHS
Silcosteel® needle assembly for G1883		232-2790012-EHS
Zero dead volume union for G1290/G1289		325-045-HSP
Union elbow for G1883		998-0000053-EHS

**Table 7. Common Replacement Parts and Consumables (continued)**

<b>Description</b>	<b>Comment/use</b>	<b>Part Number</b>
<b>Transfer line needles and unions</b>		
Transfer line needle, dia. 0.5, for on-column inlet	Nickel	301-016-HSP
Transfer line needle, dia. 0.7, for split.splitless inlet	Nickel	301-015-HSP
Transfer line needle, dia. 0.7, for split/splitless inlet	Silcosteel	301-221-HSP
Union, M5/D1.6 SS assembly	Zero dead volume union	325-961-DAN
Strain relief	Supports needle through GC inlet septum	301-205-HSP
<b>Manuals</b>		
Site Preparation and Installation		G1290-90307
Operating		G1290-90317
Service CD-ROM		G1290-90327
<b>Standards</b>		
<b>Headspace OQ/PV Standard kit:</b>		5182-9733
1 ampoule of standard, 1 ml		
1 pkg of 5 µL micro-pipettes		
Material Safety Data Sheet		
Certificate of Analysis		

## Fuse Locations

Refer to Figure 15 and Table 8 below for fuse locations and part numbers.

**WARNING** Fuse replacement should be performed only by qualified service personnel.

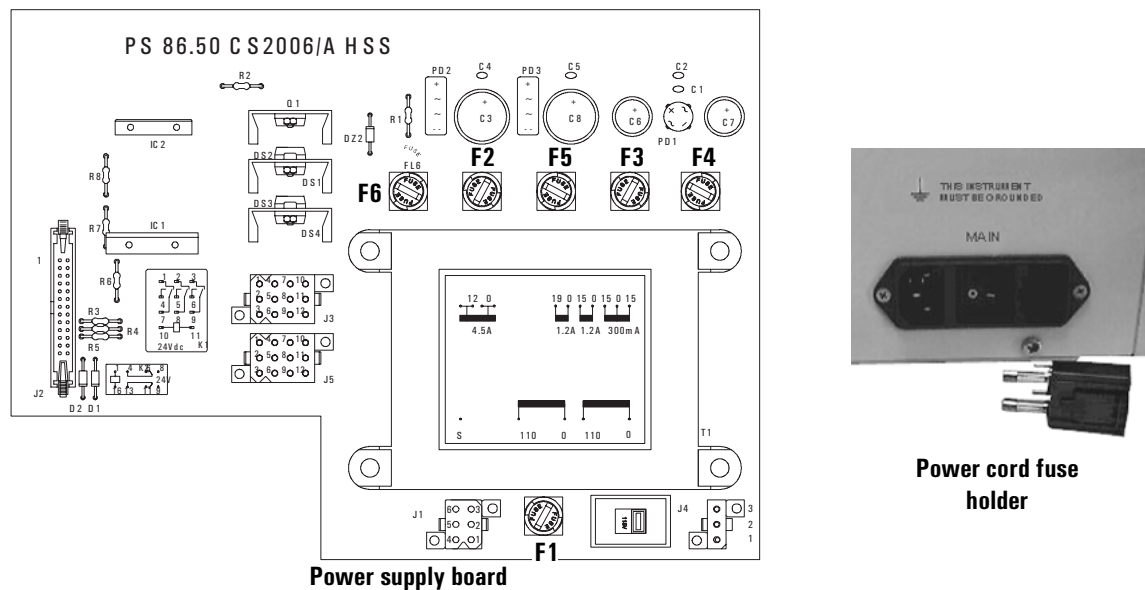


Figure 15. Fuse locations on the power supply

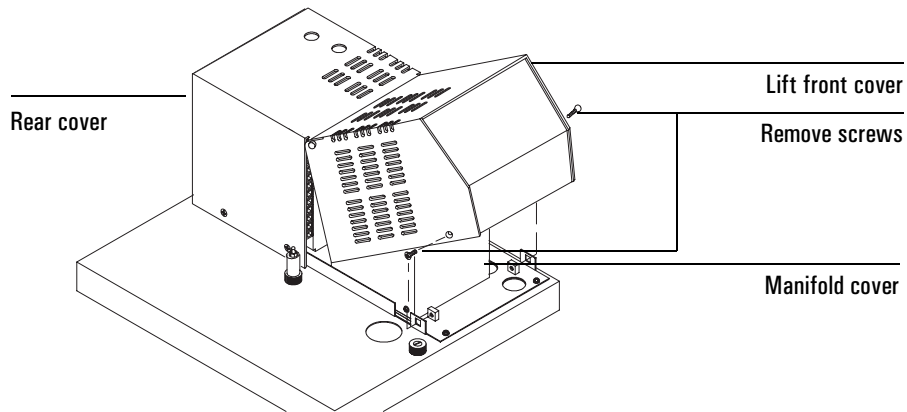
Table 8. Fuse Part Numbers

Fuse Location	Voltage	Description	Part No.
F1	230	0.5 A, 5×20	384-1695004-HSP
F1	115	1 A, 5×20 T	384-1595011-HSP
F2	all	2 A, 5×20 T	384-1695001-HSP
F3, F4	all	0.5 A, 5×20	384-1695004-HSP
F5	all	2 A, 5×20 T	384-1695001-HSP
F6	all	6 A, 5×20 F	384-1295001-HSP
Power cord fuse holder	230	3 A, 5×20 T	384-1695003-HSP
Power cord fuse holder	115	6 A, 5×20 F	384-1295001-HSP



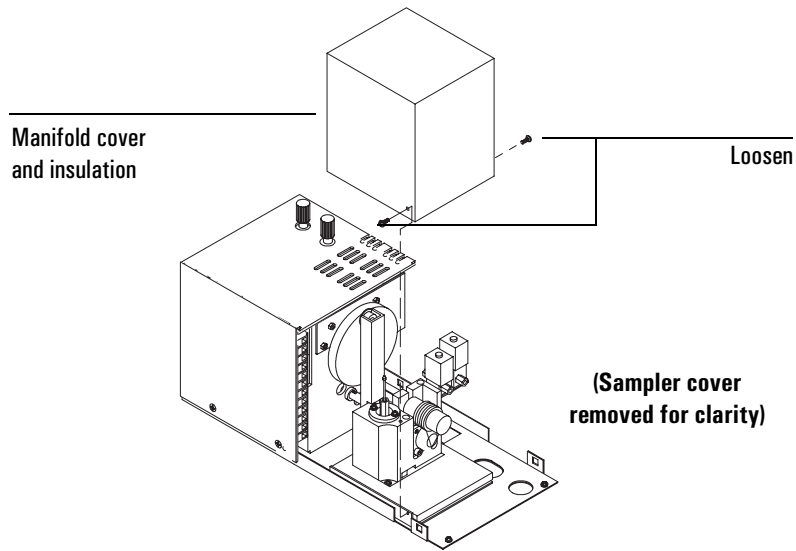
## Changing the Sampling Needle

1. Remove the two screws on the sides of the sampler cover shown below and lift the cover. Support the cover while working in the sampler area.



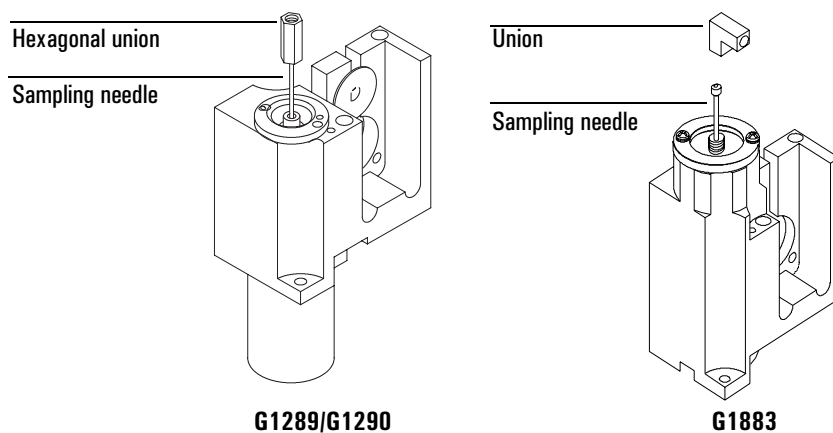
**Figure 16. Removing the sampler cover**

2. Loosen the two screws on the manifold cover (it is a stainless steel box) and insulation from the valve compartment.



**Figure 17. Removing the manifold cover and insulation**

3. The sampling needle is located inside the square aluminum block. It is connected to the sampling valve through a 1/16-inch nickel tube and hexagonal union.
4. Support the hexagonal union with a 7-mm wrench and remove the nut with another 7-mm wrench. Then, loosen and remove the hexagonal union. Lift out the needle.

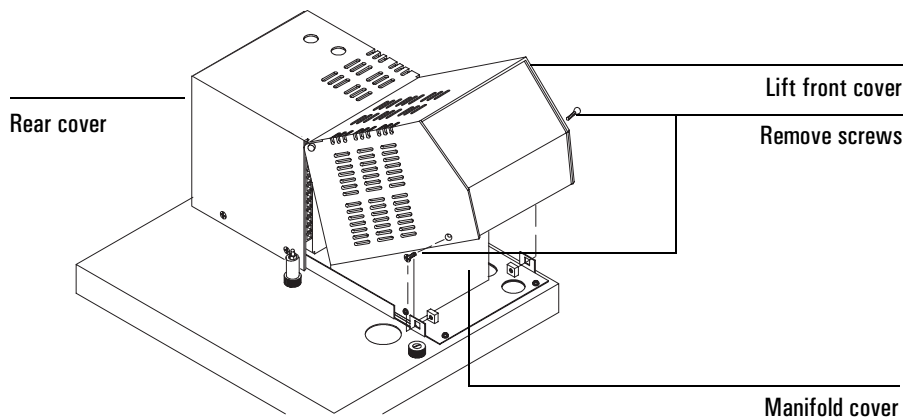


**Figure 18. Position of vial sampling needle**

5. Replace the sampling needle, tighten the fittings, and perform a leak test before replacing the insulation and cover.

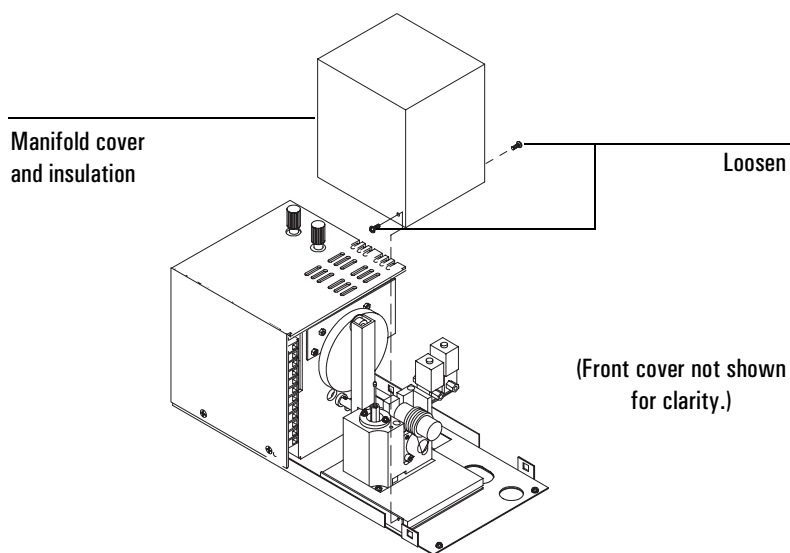
## Changing the Sample Loop

1. Remove the two screws on the sides of the sampler cover shown below and lift the cover. Support the cover while working in the sampler area.



**Figure 19. Removing the sampler cover**

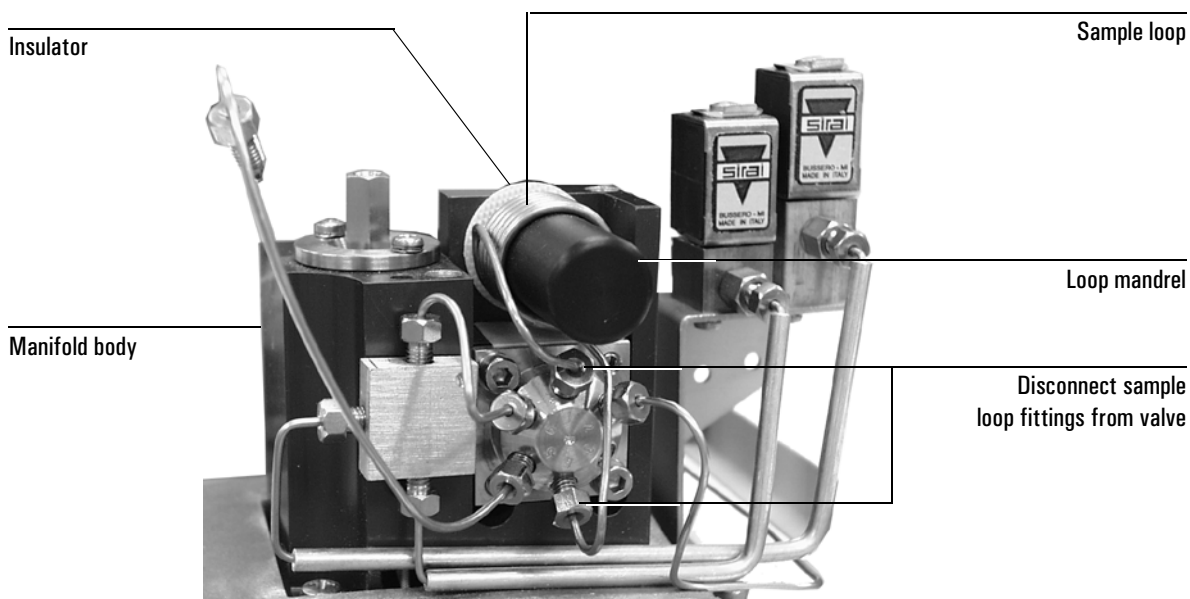
2. Loosen the two screws on the manifold cover (the stainless steel box) and remove it (and the insulation inside it) from the valve compartment.



**Figure 20. Removing the manifold cover and insulation**

3. Use a 7-mm wrench to loosen the two nuts connecting the sample loop to the six-port valve. See Figure 21.

## Maintaining the Instrument Changing the Sample Loop



**Figure 21. The Sampling System**

4. Use a 7-mm wrench to loosen the bolt holding the loop mandrel to the manifold body, and remove the loop mandrel.
5. Slide the loop off the mandrel.
6. Slide the new loop over the mandrel and tighten the nuts to the six-port valve. Reinstall the mandrel. Position the fiberglass insulator between the manifold body and the loop mandrel.

---

### Note

When installing a Silcosteel<sup>®</sup>-treated sample loop, take care not to bend the loop when installing it or you will break the coating and compromise inertness.

7. Perform a leak test on fittings before replacing the box and cover.

## Steam Cleaning the Pneumatic System

Steam cleaning removes components of the sample that could stick to the surfaces of the flowpaths and valves. For example, polar compounds tend to stick to these surfaces and may appear as carryover when running solvent or air blanks after samples. For safety reasons, Agilent only recommends water as the cleaning agent.

The success of the cleaning procedure will vary depending on your application. Check the results to assess the value of steam cleaning.

---

### Caution

Disconnect the transfer line from the gas chromatograph or 5973 MSD. Injecting water vapor into the GC will damage the column. Injecting water vapor directly into the MSD may shorten the life of analyzer components. You may have to cool down the GC or vent the MSD before disconnecting the transfer line.

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### WARNING

Use vial septa that can handle the temperature and pressure associated with this procedure. Agilent recommends septa part number 9301-0719, tan PTFE/white silicone septa,  $-60^{\circ}\text{C}$  to  $125^{\circ}\text{C}$ . These are the septa provided in the part number 5182-0840 preassembled 20-ml vial kits.

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1. Prepare the GC or MSD.
2. Prepare 23 vials: 20 vials with 1 ml of distilled water and 3 air blanks.
3. Disconnect the transfer line. Set the carrier flow so that it is less than 100 ml/min.
4. Enter the following parameters on the headspace sampler keypad:

Function	Parameter Settings
Zone Temps	125°C, 140°C, 150°C
Event Times (minutes)	2.5, 15.0, 0.0, 0.2, 0.2, 1
Vial 1	1, 23, 0 (off)

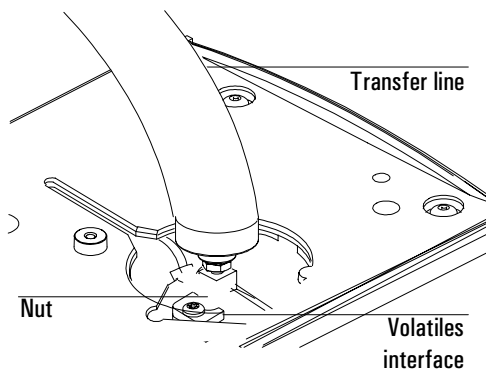
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5. Start. It takes about 70 minutes to complete.

## Installing the Transfer Line onto the Volatiles Interface

If you are using your headspace sampler with a 6890 and a volatiles interface, install the transfer line onto the interface as follows:

1. Install an appropriate-sized male nut and steel ferrule onto the transfer line.
2. Connect the transfer line to the interface. Tighten the nut 1/4-turn past finger tight. Do not overtighten. If the nut leaks, tighten an additional 1/8-turn.



3. Check for leaks. See your 7694 Headspace Sampler Site Prep and Installation Guide for details.



## Aligning the Vial Loader (G1289/G1290 only)

Every three months, Agilent recommends that you check the vial loader to see if it needs adjustment. This helps prevent vial handling problems.

This procedure uses the Advanced Function keys to pick up and move a vial from the sample tray to the oven port. At the steps where the fork picks up or releases the neck of a sample vial, you will check to see if the fork and vial are lined up properly.

If there is a misalignment between the vial and the fork, you will adjust one or more of the six potentiometers that control the positioning of the loaders and the vial gripper fork. These potentiometers are located in the horizontal loader arm. You will need to remove the horizontal loader back cover to adjust them.

The procedure to adjust the potentiometers is described below. The procedure to check vial loader operation starts on page 99.

### Adjusting the potentiometers

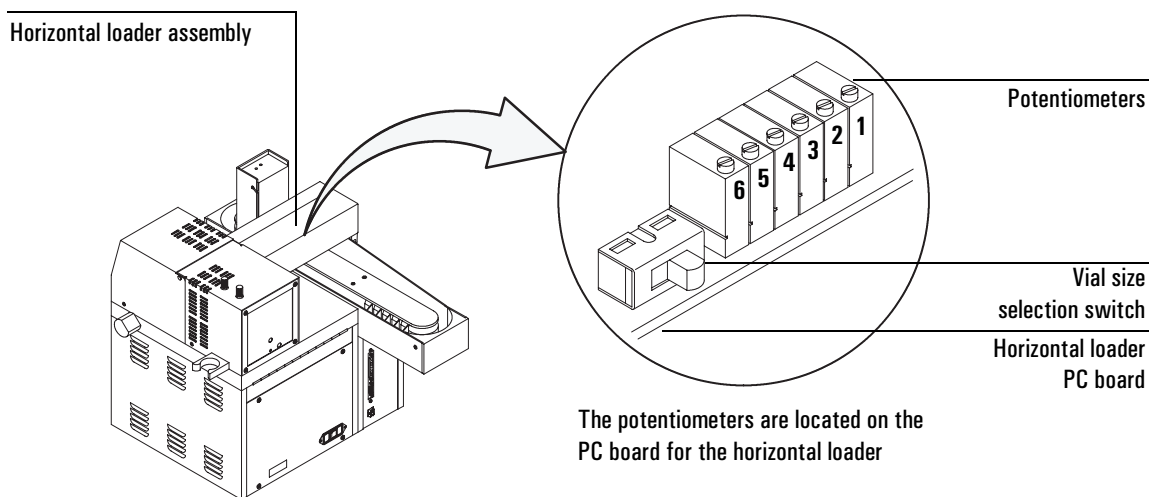
Remove the back cover by removing the three screws.



Figure 22. Removing the horizontal loader back cover

**Maintaining the Instrument**  
**Aligning the Vial Loader (G1289/G1290 only)**

The potentiometers are located on a small printed circuit board and are labelled P1 through P6. See Figure 23. Use the procedure on page 99 to identify which potentiometer you need to adjust. P1 and P2 adjust the height of the fork; these are the y-axis adjustments. P3 through P6 adjust the horizontal position of the fork at four different locations; these are x-axis adjustments.



**Figure 23. Potentiometers controlling the vial gripper arm**

To determine if you need to turn a potentiometer clockwise or counterclockwise, refer to Table 9. Then use a small flathead screwdriver to turn the adjusting screw.

**Table 9. Vial Loader Potentiometer Adjustments**

Potentiometer	Position	Display Reads	Desired Results	Adjustment
P1	Fork Down	GRIPPER Y AXIS DOWN	Fork grabs center of vial neck	Clockwise = UP
P2	Fork Up	GRIPPER Y AXIS UP	Vial clears all obstacles during delivery	Clockwise = UP
P3	Oven Load (4)	GRIPPER X AXIS 4	Successfully drops or picks up vial at oven port	Clockwise = LEFT
P4	Oven Wait (3)	GRIPPER X AXIS 3	Loader waits between oven and tray, no interferences	Clockwise = LEFT
P5	Tray Load (2)	GRIPPER X AXIS 2	Successfully drops or picks up vial at sample tray	Clockwise = LEFT
P6	Rest Set (1)	GRIPPER X AXIS 1	Loader rests over center of tray, no interferences	Clockwise = LEFT

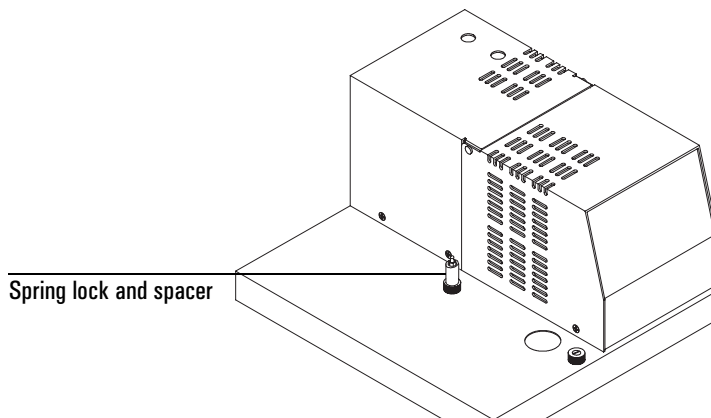
## Procedure

1. Place a capped sample vial in sampler tray position 1.
2. Press **Adv Fctn, 5, Enter**.
3. Check vial pickup from the tray.
  - a. Scroll until the display reads Y AXIS. Press **0** to lower the fork.
  - b. Scroll down until the display reads X AXIS. Press **1** to move the fork to the right. It should snap onto the middle of the vial's neck. Observe this movement by pressing **0** and **1** to move the fork back and forth. If the fork is too high or too low, adjust potentiometer P1. If the fork moves too little or too far to the left, adjust P5.
  - c. Scroll up until the display reads Y AXIS. Press **1** to raise the vial. If the vial does not clear the tray when lifted, adjust potentiometer P2. The bottom of the vial should not strike anything.
4. Check vial delivery to the oven port.
  - a. Scroll down until the display reads X AXIS. Press **1** to move the vial to the left. Press **1** again to position the vial over the oven port.
  - b. Scroll up until the display reads Y AXIS. Press **0** to lower the vial into the oven port. The vial should be placed in the center of the oven port,

and should not touch the walls. Press **1** then **0** to move the fork up and down, and watch the motion. Repeat several times. If the vial appears to be too far right or left from the oven port, adjust P3.

5. Check vial release and pickup at the oven port.

Scroll down until the display reads VIAL LOAD. Press **1** to move the elevator to the “up” position. Scroll up until the display reads Y AXIS. Press **0** to move the vial down into the oven port. Scroll down until the display reads X AXIS. Press **0** then **1** to move the fork left and right. The fork should cleanly release the vial, then grab it again. If the fork is too high or too low, adjust the spring lock and spacer on the top of the oven cover. If you make an adjustment to the spring lock, repeat step 3b.



**Figure 24. Spring lock and spacer**

## Other vial handling adjustments

In addition to the potentiometers, also check or adjust the following:

- Vial size selection switch on the back of the vial transport arm
- Carousel adjustment (see “*Checking carousel adjustment*” on page 101)
- Elevator adjustment (contact Agilent service)
- Sampler tray sensor adjustment (contact Agilent service)

## Checking carousel adjustment

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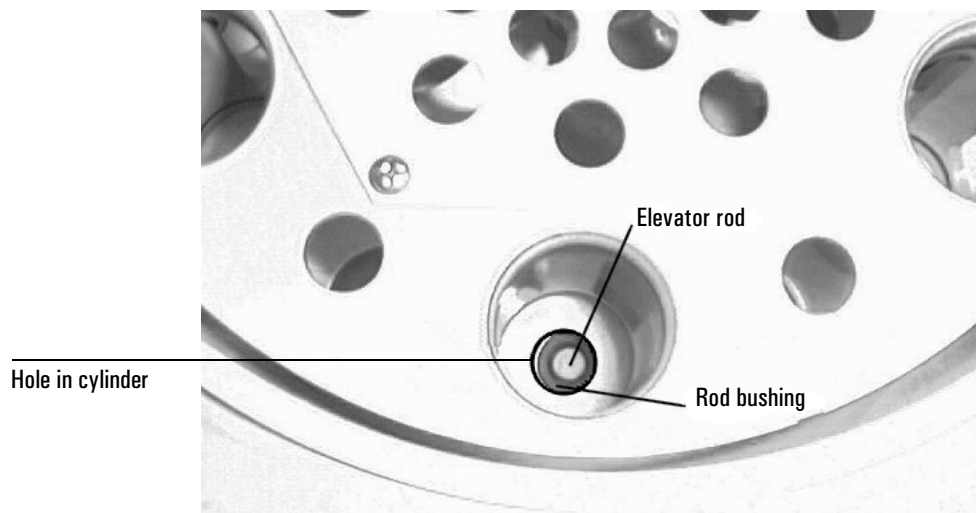
**WARNING**

The surfaces of the oven, carousel, and oven lid may be hot and could burn you. Before you begin, set the oven and loop temperatures to 30°C, swing the vial transport arm out of the way and open the oven cover. Allow the oven to cool to a safe temperature. Note that vials may remain hot even if the oven has cooled.

---

Check carousel adjustment to help prevent vial handling problems.

1. Turn off the headspace sampler, then turn it back on.
2. During startup, the sampler will reset the carousel position so that carousel position 1 is directly over the elevator rod and under the oven port in the cover. If the position is off by a few degrees, as shown below, you may have intermittent vial handling problems when the vial is pushed up onto the sampling probe.

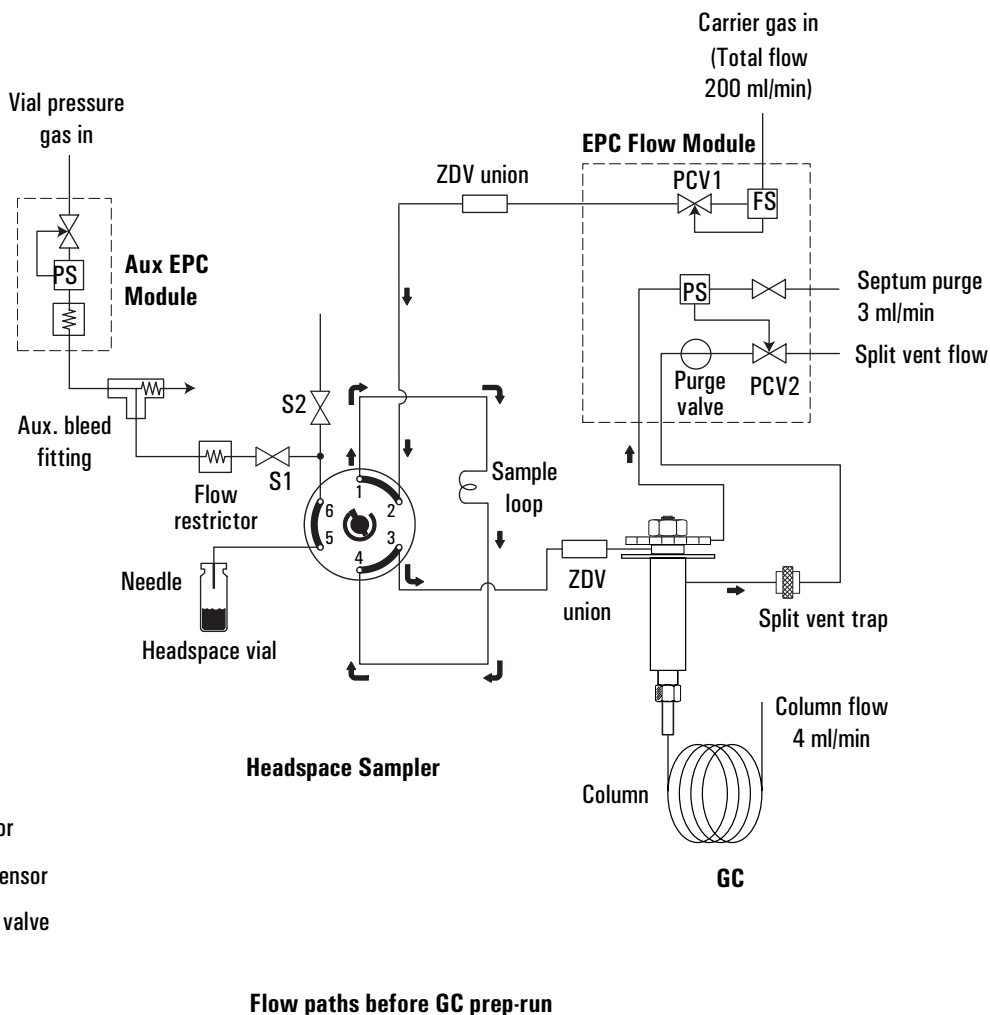


**Figure 25. Misaligned carousel**

If the elevator rod and bushing do not line up exactly under the hole in the carousel cylinder, call Agilent service.

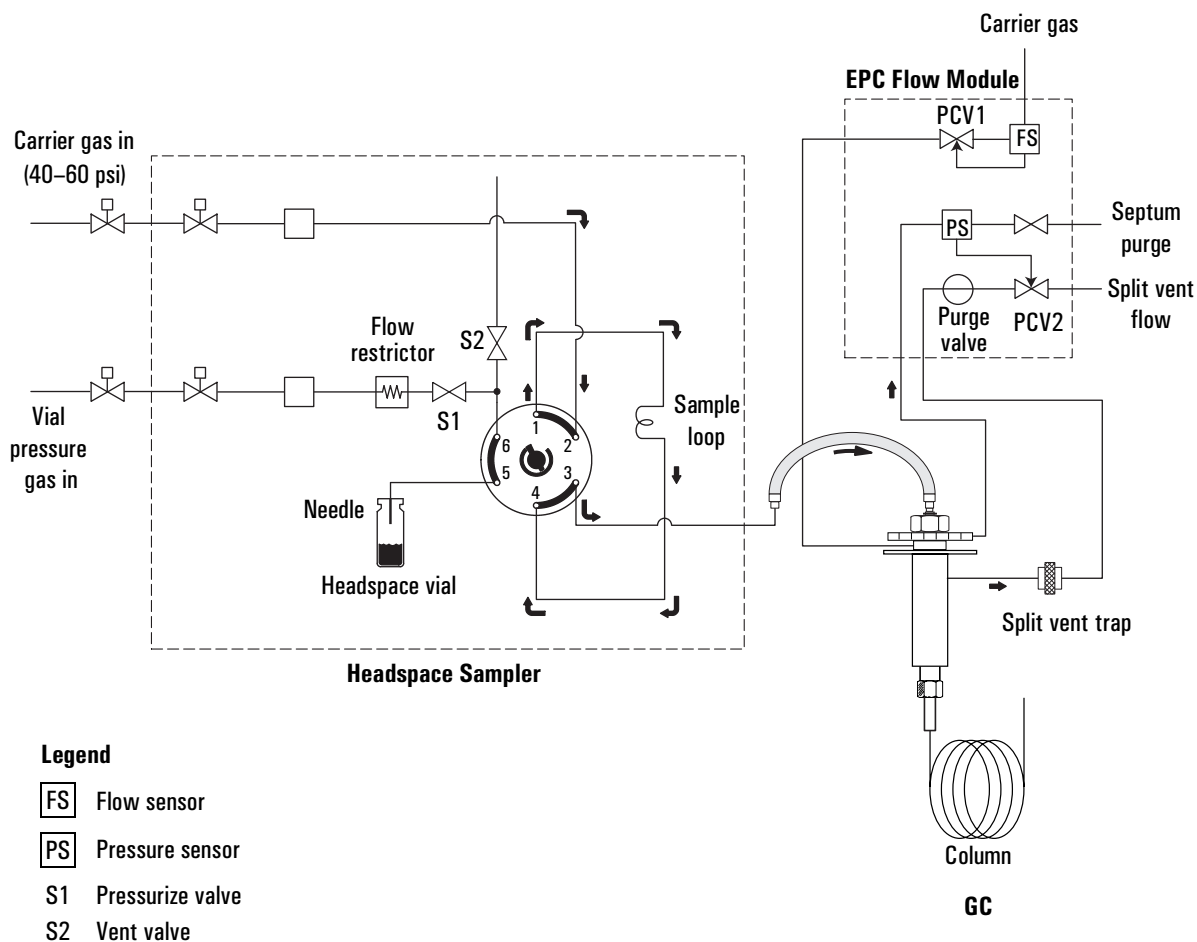
## Leak Testing: General Information

Refer to Figure 26 and Figure 27, which show the flow paths for all gases used in each type of headspace installation.



**Figure 26. Flow paths for a G1289 to 6890 GC installation**

Maintaining the Instrument  
**Leak Testing: General Information**



Flow paths before GC prep-run

**Figure 27. Flow paths for a G1290/G1883 to GC installation**

In general, if there is a leak, check the following locations:

- Zero dead volume (ZDV) unions
- Auxiliary bleed fitting



- Sample loop connections
- Connections to the sampler from the GC or gas supply
- Remaining valve connections

## **Leak Testing a G1289 to 6890 GC Interface**

### **Carrier Gas Flow Path**

Perform this leak test to check the integrity of the entire carrier gas flow path between the GC and the headspace sampler.

1. Install a checkout column, part number 19091J-413, in the GC.

2. Set the following conditions on the 6890 GC:

<b>Setting</b>	<b>Value</b>
<b>Oven</b>	
Initial temp	40°C
Ramps	Off
Equilibration time	0.10 min
Run time	10 min
<b>Inlet</b>	(PTV or split/splitless)
Mode	Splitless
Initial temp	250°C
Pressure	25.00 psi
Purge flow	200.0 ml/min
Purge time	1.00 min
Gas saver	Off
<b>Column</b>	
Model number	19091J-413
Max temperature	325°C
Nominal length	30 m
Nominal diameter	320.00 µm
Nominal film thickness	0.25 µm
Mode	Constant pressure
Pressure	25 psi
Outlet pressure	Ambient
Nominal initial flow	3.9 ml/min
<b>Aux Pressure (Vial Pressurization channel)</b>	
Gas type	Helium
Initial pressure	15 psi (On)
Initial time	0.00 min
Rate #1	0.0 (Off)

3. Observe the column flow rate from the GC front panel. The flow rate should be close to the nominal, approximately 4 ml/min.
4. The inlet Total Flow at the GC front panel should read the sum of the purge flow (200 ml/min) + the column flow (4 ml/min) + the septum purge flow (3 ml/min), or approximately 207 ml/min.
5. Press the **Prep Run** key on the 6890. (If another temperature or flow is not ready, you may need to press Prep Run twice.)  

The capillary inlet purge valve shuts off and the inlet switches to forward pressure control. The inlet EPC module flow sensor becomes read-only.
6. Check the total flow reading for the inlet. Now that the purge flow is turned off, the total flow should drop to the column flow (4 ml/min) + the septum purge flow (3 ml/min), or approximately 7 ml/min. If the flow rate is much higher, there is a leak in the carrier gas flow path between the 6890 inlet flow module and the detector column fitting. Continue with the next step to get more information.
7. On the headspace sampler, press **Adv Fctn, 5, Enter**, and scroll to Sample Valve. Turn it on using keys **0** and **1**. Observe the total flow rate. If the flow rate changes when the sample loop is taken out of the flow path, then one (or both) of the fittings on the sample loop is leaking. Tighten any leaking fitting.
8. If the total flow rate indicates a leak, check each connection in the path and tighten if necessary.

---

**Caution**

Do not tighten Swagelok fittings more than approximately 1/4-turn. If needed, remake the connection.

---

### Carrier Flow Path Pressurization Test

This test is an alternate to the test described above.

1. Set the three heated zones to ambient temperature.
2. De-configure the GC column assigned to the inlet used with the headspace sampler.
3. Cap the column fitting with a “no-hole” ferrule (part no. 5181-7458).
4. Cap the inlet’s septum purge fitting with a 1/8-inch Swagelok cap.

5. Set the following GC conditions:

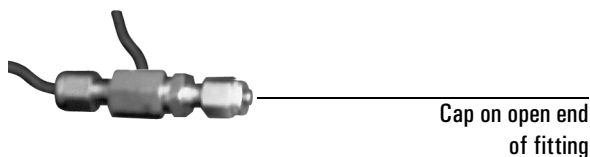
Setting	Value
Inlet	(PTV or split/splitless)
Mode	Splitless
Total flow	60 ml/min
Pressure	25.00 psi

6. Allow the system at least 60 seconds to equilibrate.
7. Turn the inlet pressure Off.
8. Monitor the pressure on the GC front panel display. It should equilibrate close to the setpoint pressure and then hold. It should drop less than 2 psi in 5 minutes.
9. Using MANUAL OPERATION, switch the sample valve to **1** or **ON** to include the sample loop.
10. Repeat steps 5–8.
11. After the test, remove the plug in the column fitting. The flow path should instantaneously de-pressurize.
12. Using MANUAL OPERATION, switch the sample valve to 0 or OFF to reset the sample loop.

## Vial Pressurization Gas (Aux) Flow Path

### Leak test

1. Prepare a new, empty 20 ml headspace vial. Make sure that it is capped properly. See “*Sample Preparation*” on page 56.
2. Manually install the headspace vial in the oven slot under the headspace probe.
3. Using MANUAL OPERATION, load the vial onto the probe and make sure the Pressurize Valve is “ON” and the Vent Valve is “OFF”.
4. Pressurize the vial to 20 psi using the Aux control on the GC.
5. Wait at least one full minute, then cap off the aux bleed fitting.



**Figure 28. Cap the aux bleed fitting**

6. Monitor the pressure on the front panel display—it should equilibrate at some pressure lower than 20 psi and then hold. The pressure will drop slightly at first, then stabilize. At this point, it should drop less than 1 psi in 5 minutes. If the unit cannot maintain pressure, there is a leak in the vial pressurization flow path.
7. Using MANUAL OPERATION, switch the sample valve and repeat steps 2–6. If the unit cannot maintain pressure, there is a leak at the sample tube fittings.
8. De-pressurize the system by opening the vent valve from MANUAL OPERATION.

### **Correcting leaks**

1. If the headspace sampler–GC system failed the test, isolate the source of the leak by using MANUAL OPERATION to toggle the Pressurize Valve to “OFF.”
2. Repeat steps 4–6 under “*Leak test*” on page 108” above.

If the unit passes the test, the leak is in the headspace unit at either the Vent Valve, the connections on the sampling valve, probe connections, or vial cap/septum.

If the unit fails the test, the leak is in the Aux control module, the Swagelok fittings on the back of the sampler, or the auxiliary bleed fitting.

## Leak Testing a G1290/G1883 to GC Interface

For the G1290/G1883, you can check the carrier gas flow system for leaks by performing a pressure decay test.

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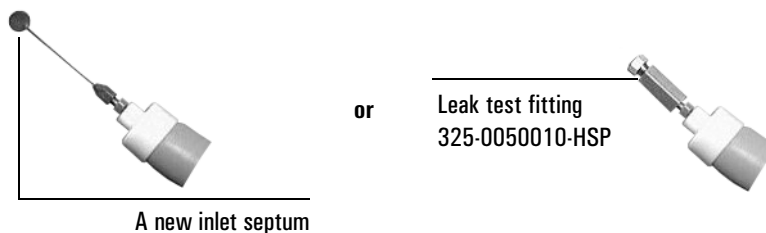
### WARNING

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The heated zones of the unit can burn your skin. Allow the zones to cool off before performing the test.

### Prepare the Sampler

1. Set the three heated zones to ambient temperature.
2. Detach the transfer line from the inlet or interface and plug it with either a new inlet septum or a leak test fitting as shown in Figure 29.



**Figure 29. Plug the transfer line**

3. Remove the pneumatics cover and loop oven cover to give you access to all of the tubing, valve, and manifold connections.

### Carrier Gas Flow Path

1. Open the Mass flow controller full counter-clockwise (CCW) until the carrier flow path pressurizes to line pressure. The carrier pressure should read between 40 to 60 psi, about 4 bar.
2. Shut off the ball valve installed between the source and the headspace sampler. An alternative would be to turn the Mass Flow Controller full clockwise (CW).

---

**WARNING**

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Turning the valve may not completely shut off flow, depending on the setting of the knob on the stem. Also excessive pressure on the stem can damage the flow controller if the knob is set to allow full travel.

3. Using MANUAL OPERATION, switch the sample valve to **0** or OFF. (See “MANUAL OPERATION” on page 48 for details.)
4. Monitor the pressure on the front panel display—it should equilibrate at some pressure close to the line pressure and then hold. It should drop less than 2 psi in 5 minutes. If the pressure drop is much greater, there is a leak.
5. Using MANUAL OPERATION, switch the sample valve to 1 or ON to include the sample loop.
6. Repeat steps 2–5.
7. After the test, remove the septum or plug. The flow path should instantaneously de-pressurize. If it does not, there may be a restriction.
8. Using MANUAL OPERATION, switch the sample valve to 0 or OFF to reset the sample loop.

### Vial Pressurization Flowpath

1. Prepare a new, empty 20 ml headspace vial. Make sure that it is capped properly. See “Sample Preparation” on page 56.
2. Manually install the headspace vial in the oven slot under the headspace probe.
3. Using MANUAL OPERATION, load the vial onto the probe and make sure the Pressurize Valve is “ON” and the Vent Valve is “OFF”.
4. Pressurize the vial to 20 psi by adjusting the pressurize regulator.
5. Wait at least 1 minute to allow the system to equilibrate, then shut off the pressurize regulator (full CCW).
6. Monitor the pressure on the front panel display. It should equilibrate at some pressure lower than 20 psi and then hold, dropping less than 5 psi in 10 minutes. If the pressure drop is much greater, there is a leak.
7. Using MANUAL OPERATION switch the sample valve and repeat steps 2–6.

8. De-pressurize the system by opening the vent valve from MANUAL OPERATION.

## Flow Testing the Vent Valve and Associated Flow Path

Perform this test only after the system (all models) passes the carrier and pressurize vial tests.

1. Prepare a new, empty 20 ml headspace vial. Make sure that it is capped properly. See “*Sample Preparation*” on page 56.
2. Manually install the headspace vial under the probe in the headspace oven.
3. Set the Vial Pressure to 20 psi.
4. Place the vent exit tube from back of headspace in a beaker of water.
  - If needed, extend the tube using 1/8-inch PTFE or similar tubing.
5. Using MANUAL OPERATION, load the vial onto the probe, set the vent valve to “OFF” and set the pressurize valve to “ON.” (See “*MANUAL OPERATION*” on page 48 for details.)
6. Wait **at least** 20 seconds for the vial to pressurize.
7. Using MANUAL OPERATION, turn Pressurize Valve “OFF” and set the vent valve to “ON.”
8. Observe the vent tube in the water. The vent tube should produce bubbles immediately when the vent is turned “ON” and the bubbling should stop within 10–15 seconds. If the bubbling lasts much longer, the vent valve or associated flowpath may be restricted. Contact Agilent service.

Note that if the vent flow path is restricted, you may be overpressurizing your sample vials. When the problem is fixed, your sensitivity may appear to decrease because you are now injecting the appropriate amount of sample for your method.



## Tightening the PTV Septumless Head Using the Leak Test Procedure

If you are using a PTV inlet with a septumless head, you can use the leak test procedure described in “*Carrier Gas Flow Path*” on page 105 or “*Carrier Gas Flow Path*” on page 110 to help you properly tighten the septumless head onto the needle. As you tighten the fitting on the needle, monitor the flow rate on the GC front display. When the flow rate indicates there is no leak, the connection is tight enough.

Maintaining the Instrument  
**Tightening the PTV Septumless Head Using the Leak Test Procedure**

---

**Troubleshooting**

---

# Troubleshooting

This chapter discusses the possible causes and the solutions for chromatography problems or instrument failures.

## Chromatography Problems

### No Peaks

#### Probable Causes

- Vial cracked
- Vial cap leaking
- Sample vial empty.
- 10 ml vial used without adapter.
- Method not sensitive enough.
- Leaks in the system.
- Needle broken/clogged.
- Sample condensed in the loop or transfer line.
- Sample loop not filled with sample.
- Inject time too short.
- Sample did not create headspace.
- Incorrect vial sampled.
- Vial not punctured (may be due to mechanical failure or vial elevator jam).
- Transfer line plugged.

#### Suggested Actions

1. Check that the proper vial is sampled and that the vial contains sample.
2. Use adapters with 10 ml vials.

3. Edit the method parameters to increase the method sensitivity. See Chapter 7, *“Developing Methods”*.
4. Check the system for leaks.
5. Check the sample vials for a proper seal (see *“Sample Preparation”* on page 56).
6. Check the condition of the needle. Replace it if necessary (see *“Changing the Sampling Needle”* on page 89).
7. Check that the sample loop and transfer line temperatures are higher than the oven temperature.
8. Increase the vial pressurization to insure that the loop is filled.
9. Check the Event Times. Increase the Inject Time.
10. Check that the oven temperature is set high enough to volatilize the compound of interest.
11. Make sure that the transfer line is hooked up to the GC and inlet in an appropriate manner.
12. Check the disk needle guide, circle clip, and spring in the injector slot for wear. Replace them if necessary.
13. Make sure the transfer line is not plugged.
14. Make a syringe injection to be sure that the column and detector are working properly.
15. Call Agilent service.

## **Bad Data (Bad Peaks)**

### **Probable Causes**

- GC conditions wrong for the sample type.
- Sample not at equilibrium.
- Too much sample injected into the GC.
- Flow too low.
- Sample condensing in the loop, transfer line, or valves.
- Leaks in the system.

- Sample contaminated.
- Sample decomposing. Oven temperature is too high.
- Incomplete sample transfer.
- System contaminated.

### **Suggested Actions**

1. Check the GC settings.
2. Increase the Vial Equilibration Time.
3. Use a smaller sample loop. See *“Changing the Sample Loop”* on page 92.
4. Use split injection.
5. Increase the Vial Pressurization Time to dilute the sample.
6. Check that the carrier–gas flow rate is high enough for the column type. See *“Developing Methods”* on page 70.
7. Check that the loop and transfer line temperatures are higher than the oven temperature.
8. Check the system for leaks.
9. Lower the oven temperature.
10. Check the sample preparation procedure to check for sources of contamination. See Chapter 5, *“Preparing a Sample”* for more information.
11. Increase the Inject Time.
12. Check the agitation level to prevent splashing material on the septum.
13. Use a heat gun to drive any contamination out of the valves or tubing while checking the detector response to confirm the location of contamination.
14. Steam clean the system. See *“Steam Cleaning the Pneumatic System”* on page 95.
15. Change the valves, regulators, and tubing. Refer to your service manual or contact Agilent service.
16. Call Agilent service.

## Unwanted Peaks Present

### Possible Causes

- Dirty vials or septa.
- Dirty laboratory air sealed in the vial with sample.
- Carryover from the previous sample.
- Contaminated GC system.
- Dirty column and/or ferrules.

### Suggested Actions

1. Bake the vials and septa in oven.
2. Flame the vials before use.
3. Wash the vials and septa and then dry them in the oven.
4. Purge the vial with argon during sample preparation.
5. Steam clean the headspace sampler. See “*Steam Cleaning the Pneumatic System*” on page 95.
6. Perform a blank GC run; clean or bake out the GC.
7. Heat the graphite GC–column ferrules with a flame before use.
8. Call Agilent service.

## Vial Handling Problems (G1289/G1290 only)

### Gripper Arm Doesn't Pick Up Sample Vial

#### Probable Causes

- Arm movements out of adjustment.
- Spring lock and spacer out of adjustment.
- Carousel out of adjustment. See “Checking carousel adjustment” on page 101.
- 10 ml vials used without adapters.

- Selector switch on the back of the horizontal loader not set for the vial size being used.
- Elevator assembly height incorrect.
- Oven adapter for 10 ml vial inserted upside down.
- Servomotor on vertical loader defective.
- Microswitch on fork defective

### **Suggested Actions**

1. Check that vials are loaded in the proper locations in the sample tray.
2. Check that the method specifies the proper vial locations.
3. Insert tray adapters (part no. 300-305-HSP) in the sample tray for 10 ml vials.
4. Check the setting of the vial-size selector switch.
5. Adjust the height of the elevator assembly. See your service manual or contact Agilent service.
6. Insert oven adapters correctly.
7. Check the operation of the servomotor. Replace it if necessary. See your service manual or contact Agilent service.
8. Check that the vial sensor microswitch is actuating properly.
9. Call Agilent service.

## **Sample Vial Doesn't Drop Down Into Oven**

### **Probable Cause**

- Horizontal gripper arm movements are out of adjustment.

### **Suggested Actions**

1. Adjust the arm movements. See “*Aligning the Vial Loader (G1289/G1290 only)*” on page 97.
2. Call Agilent service.



## Vial Not Raised and Lowered in Oven

### Probable Causes

- Belt on the vial elevator is loose or broken.
- Vial elevator motor is at fault. Check the voltage on J9 pins 3 and 9 on the main board:  $V > 20\text{ V}$  or  $< -20\text{ V}$ .

### Suggested Actions

1. Check the belt. Replace it if necessary. See your service manual or contact Agilent service.
2. Exchange the motor.
3. Call Agilent service.

## Vial Not Loaded/Vial Retained Warning

### Probable Causes

- Robotic arm microswitch out of adjustment.
- Vial remains in robotic arm.

### Suggested Actions

Contact Agilent service.

## Vial Jam Problems (G1289/G1290 only)

In certain situations, vials will jam when the unit attempts to drop one vial on top of another in the oven or tries to place a vial in the sample tray when a vial is already in that location. Vial jam problems can also result from misalignment of the robotic arm or oven carousel. This may cause the motor for the oven carousel or the robotic arm to jam.

## Recovering from vial jam problems

The headspace sampler attempts to recover from vial jams and continue where it left off. Sometimes this is not possible. The simplest way to recover from a vial jam problem is as follows:

1. Determine which vial was the last one that was **completed** (the sample was run and you have believable run data).

---

### WARNING

---

Vials removed from the oven are hot!

2. Clear the jam.

Note that if the robotic arm jams in the x-axis, a protection circuit may engage, disabling the arm for 10 to 15 minutes until the circuitry cools down. After this time has elapsed, press **Adv Fctn**, **.**, **1**, and **Enter** to reset the sampler.

3. Turn the sampler off, then on again. The sampler should return all vials back into the tray in their appropriate positions.

If it does, you have recovered from the vial jam.

If it does not, turn the sampler off and continue with the next step.

4. Remove all vials from the oven and the gripper. Place into the tray any vials from the oven or gripper that still need to be run.
5. Press the **Clear** key and turn the power on. Continue to hold the **Clear** key until you see the message *Remove all vials!!!*. At this point, the instrument is reset. Adjust the method for the new range of vial locations, then restart the run.

## Mechanical/Electrical Problems

### Instrument Resets Continuously

#### Probable Causes

Shorted temperature sensor

### **Suggested Actions**

Call Agilent service.

### **No Power**

#### **Probable Causes**

- No power to the unit.

#### **Suggested Actions**

1. Verify that the power cord is connected to the 7694 Headspace Sampler and to a live power source.
2. Call Agilent service.

### **Oven Carousel Won't Rotate Properly**

#### **Probable Causes**

- Loose or broken belt in the oven assembly.
- Check the stepper motor. Measure R across the left or right pair of wires in the motor connector: R = 6-7 ohms.
- Dirty carousel sensor eye.

#### **Suggested Actions**

1. Tighten or replace the belt in the oven assembly. See your service manual or contact Agilent service.
2. Replace the stepper motor if necessary.
3. Clean the sensor eye with an alcohol swab.
4. Call Agilent service.

### **Oven Carousel Won't Agitate**

#### **Probable Causes**

- Agitation set off.
- Loose or broken belt in the oven assembly.

- Check the stepper motor. Measure R across the left or right pair of wires in the motor connector: R = 6–7 ohms.

### **Suggested Actions**

1. Set agitation on with Vial Parameters.
2. Tighten or replace the belt in the oven assembly. See your service manual or contact Agilent service.
3. Replace the stepper motor if necessary.
4. Call Agilent service.

## **Sample Tray Doesn't Stop at Correct Location (G1289/G1290 only)**

### **Probable Causes**

- Optical sensor is blocked or broken.
- Faulty chip on the mother board.
- Faulty encoder on the tray motor.

### **Suggested Actions**

1. Make sure that the tab is in contact with the optical sensor.
2. Clean the sensor with an alcohol swab.
3. Call Agilent service.

## **Sampling Sequence Won't Start**

### **Probable Causes**

- The 7694 Headspace Sampler not ready.
- Oven stabilization time not expired.
- No vial in tray (G1289/G1290 only).
- Setpoints not reached.

### **Suggested Actions**

1. Check the 7694 Headspace Sampler setpoints.

2. Oven stabilization time may be set too long. Check the `STABLIZE TIME` advanced function.
3. Check that the vials are loaded into the proper locations in the tray or carousel.
4. Check that the method specifies the proper vial locations.
5. Wait for the `7694 READY` display before pressing **Start**.
6. Call Agilent service.

## Second Method in Sequence Won't Start

### Probable Causes

- Chain Methods Off.
- Parameter Increment on.
- Incorrect sequence entered.
- Vials not loaded correctly.
- Setpoints for second method not reached.

### Suggested Actions

1. Chain Methods On.
2. Turn off Parameter Increment.
3. Check the sequence entered.
4. Check that the vials are loaded in the sample tray correctly.
5. Check that the setpoints for the second method are reached.
6. Call Agilent service.

## Zones Won't Heat

### Probable Causes

- Oven temperature sensor defective.
- Heater band/vial heater not working.
- Fuse blown.

### **Suggested Actions**

1. Measure R = 111 ohms at connector M1 (pins 9, 10, 11, 12). Replace the sensor if necessary. See your service manual or contact Agilent service.
2. Check the “heaters” line on J3 on the MK12 connector (pins 9, 10, 11, 12) Replace the heater if necessary.
3. Have a qualified service technician check the fuses.
4. Call Agilent service.

### **Pressures Are Wrong**

#### **Probable Causes**

- Leaks in system.
- Gas supply exhausted.

#### **Suggested Actions**

1. Check the system for leaks.
2. Replace the gas cylinder.
3. Call Agilent service.

---

**Error Messages**

---

# Error Messages

This chapter describes various error messages that accompany machine failures and the means to correct the failures.

Most errors cause the sampler to stop running the current method. You may need to remove vials from the carousel, re-load vials into the carousel, or change the method if you want to continue your analysis from the point at which it stopped.

## Error Message Listing

**Agilent 7694 or 7694 RESET**

**Agilent 7694E or 7694E RESET**

### Probable Causes

The sampler rebooted due to a power failure (or user input).

### Comments

Power to the 7694 Headspace Sampler may be lost during a run. The sampler contains a battery backup which will retain methods in storage. When power is restored, the headspace sampler runs through a series of diagnostic tests. (See *“Power on Diagnostics”* on page 14 for more information.)

The method in use when the power was lost will be reloaded into the active memory. If you press **Start** the sampler will run the method starting with the first vial specified. If you wish to start the run from the point at which the run was interrupted, you will have to edit the number entered for First Vial.



## **ALARM STEPPER errors**

### **Probable Causes**

- Carousel jammed because of mishandled vial or broken glass or other foreign bodies.
- Carousel out of alignment. See “Checking carousel adjustment” on page 101.
- Carousel drive mechanism failed.

### **Suggested Actions**

1. Remove all vials from the carousel and gripper. Clean out the oven/carousel. Power cycle the headspace sampler and *hold down the clear key* until the tray is reset.
2. Call Agilent service.

## **ALARM TEMP1 (OVEN)**

## **ALARM TEMP2 (LOOP)**

## **ALARM TEMP3 (TUBE)**

### **Probable Causes**

- The temperature sensor for the heated zone noted is failed, shorted, or disconnected.
- The temperature for the zone is too high, indicating a bad PC board or sensor.

Oven > 220°C

Loop > 240°C

Tube > 240°C

### **Suggested Actions**

Call Agilent service.

The microprocessor immediately switches off the heater associated with the failed sensor. You must correct the problem before you can continue to use the sampler.

### **CAROUSEL RESET**

#### **Probable Causes**

The sampler was reset.

#### **Suggested Actions**

See “*Agilent 7694 or 7694 RESET Agilent 7694E or 7694E RESET*” on page 128.

### **PRESS STOP TO CONFIRM**

The sampler is waiting for confirmation before changing a setpoint.

### **RAM CHECK ERR**

### **PRESS CLEAR TO RESET**

#### **Probable Causes**

The RAM check value is different from the stored value.

#### **Suggested Actions**

Press Clear to continue. The instrument is stopped and you must totally reset it. This resets the microprocessor and automatically initializes all the data (method parameters, advanced function values, flags and all the variables) with default values. If the unit fails to reset, try turning the power off, then on. If this fails, contact Agilent service.

## **REMOVE ALL VIALS!!!**

### **Probable Causes**

The sampler was reset. The sampler does not know if the vials still in the carousel are the ones originally associated with your method.

### **Suggested Actions**

Remove all vials from the carousel and re-load the carousel according to the current method. See also “*Agilent 7694 or 7694 RESET Agilent 7694E or 7694E RESET*” on page 128, and “Vial Jam Problems (G1289/G1290 only)” on page 121.

## **ROM CHECK ERR PRESS CLEAR TO RESET**

### **Probable Causes**

The ROM check value is different from the stored value.

### **Suggested Actions**

Press **Clear** to continue. The instrument is stopped and you must totally reset it. This resets the microprocessor and automatically initializes all the data (method parameters, advanced function values, flags and all the variables) with default values. If the unit fails to reset, try turning the power off, then on. If this fails, contact Agilent service.

## **TRAY RESET**

### **Probable Causes**

The sampler was reset.

### **Suggested Actions**

See “*Agilent 7694 or 7694 RESET Agilent 7694E or 7694E RESET*” on page 128.

## **UP MOTOR ERROR**

### **Probable Causes**

The elevator assembly that lifts the vial onto the sampling needle is unable to reach the needle.

### **Suggested Actions**

Remove all vials from the oven and the gripper. Power cycle the headspace sampler and *hold down the clear key* until the tray is reset.

## **VIAL NOT FOUND**

### **Probable Causes**

- The robotic arm is unable to retrieve a vial from the oven
- The headspace sampler is unable to load a vial into the oven

### **Suggested Actions**

1. Manually remove any vials that are in the oven carousel.

---

### **WARNING**

Vials in the oven may be *hot*.

2. Power cycle the headspace sampler and *hold down the clear key* until the tray is reset.

To remove a vial from the robotic arm, see “*Vial Not Loaded/Vial Retained Warning*” on page 121.

See also “Vial Jam Problems (G1289/G1290 only)” on page 121.

## **VIAL RETAINED**

### **Probable Causes**

The 7694 HS was reset with a vial still held by the robotic arm. This may happen after a power failure or a manual reset.

### **Suggested Actions**

1. Manually remove the vial that is held in the robotic arm. See “*Vial Not Loaded/Vial Retained Warning*” on page 121, or remove the cover from the vertical loader and pull the vial out of the gripper.

---

### **WARNING**

---

Vials in the oven may be *hot*.

2. Power cycle the headspace sampler and *hold down the clear key* until the tray is reset.

## **Recovery from an Automation Error (G1289/G1290)**

If an automation error occurs, there are two recovery modes.

If you want to reload the default method, hold **Clear** during power-up. The following message is displayed.

```
LOAD DEFAULT METHOD  
TRAY RESET
```

If you want to reset the counter, hold **Adv Fctn** during power-up.

Error Messages  
**Recovery from an Automation Error (G1289/G1290)**

# Glossary

**accuracy** The difference between a measured quantity and its true value

**analyte** The compound of interest in an analysis

**calibration curve** A graph showing the relationship between the amount of a compound and its response from an analytical technique. In gas chromatography, that response is the peak area.

**carrier gas** The gas that flows through the chromatographic column and moves the sample components when they are not retained by the stationary phase.

**chromatographic column** A tube (e.g. of glass, stainless-steel or plastic) generally cylindrical, containing the stationary phase and provided with an inlet and an outlet for the mobile phase

**chromatography** A separation method in which the sample components' different affinities toward two phases, one of which is immobile (the stationary phase) and the other which moves (the mobile phase), are taken advantage of

**column** See **chromatographic column**

**constant heating mode** In HS sampling, a setting which heats the contents of each sample vial for an identical length of time for each extraction

**distribution coefficient** See **partition coefficient**

**elastomeric** displaying elastic qualities similar to rubber

**Electron Capture Detector (ECD)** A gas phase detector that uses a radioactive source and detects a component by its ability to capture low-energy electrons

**eluate** The material exiting a column in chromatography

**equilibrium** In HS sampling, the point at which molecules diffuse out of the sample into the headspace at the same rate at which they diffuse from the headspace back into the sample

**external standard** A sample containing a known quantity of the component to be quantified

**Flame Ionization Detector (FID)** An ionization detector consisting of a hydrogen burner whose flame burns between two electrodes with potential difference between them. When the column effluent gas is burned with air or oxygen in the hydrogen flame, an increased ionization current is obtained as a result of ionization of the solute molecules.

**flow rate** The amount of the carrier gas passing through the column per unit time

**Flame Photometric Detector (FPD)** A detector that measures the intensity of the light (at fixed wavelength) that accompanies the combustion of the eluate in a hydrogen-rich flame

**gas chromatography (GC)** Chromatography in which a gas is the mobile phase

**headspace (HS)** The gaseous space in a sealed vial above the solid or liquid sample in that vial. The headspace is composed of volatile compounds contained in the sample.

**injection** In chromatography, introduction of a sample to a column. See also **split injection** and **splitless injection**.

**internal standard** A compound added in known concentration to the sample, for example, to eliminate the need to measure the size of the sample in quantitative analysis

**matrix** The main constituents of a sample other than the detected and/or determined component

**matrix effect** The disturbing influence of other components of the analyzed sample on the detection and/or determination of an analyte

**menu** A list of items that represent commands for the software to execute

**method** The parameters defined for conducting the sample equilibration and sampling



**multiple headspace extraction** An analytical technique that takes more than one headspace sample from each sample vial, followed by chromatographic analysis of each extracted sample. By summing up the peak area for each compound, the total amount of that compound in the sample can be determined, eliminating the effect of the matrix. See **single headspace extraction**.

**multiple puncture** Multiple headspace extraction performed by puncturing the septum separately for each extraction. See **single puncture**.

**partition coefficient**  $K$  (in gas chromatography.) The ratio of the amount of a component per unit volume of the stationary phase ( $\text{g cm}^{-3}$ ) to its amount per unit volume of the mobile phase at constant temperature and at equilibrium. The partition coefficient is assumed to be independent of concentration at the concentration prevailing in gas chromatography, and is related to the volume  $V_N$  by

$$K = \frac{VN}{V_L} = \frac{VN\delta_L}{m_L} = \frac{V_g T\delta_L}{m_L}$$

where:

$V_L$  = volume of the liquid stationary phase

$\delta_L$  = density of the liquid

$m_L$  = mass of the liquid

$V_g$  = specific retention volume

**phase ratio** The ratio of the volume of the mobile phase to the volume of the stationary phase in a column

**precision** The agreement between the results of repeated data

**quantitative analysis** Analysis performed for the purpose of determining the amount of a compound in a sample

**retention time** The time interval from the moment of injection of a sample into the column to the appearance of the peak maximum of the given component in the eluate

**sampling sequence** In headspace analysis, the steps involved in removing the sample from the vial and injecting it into the GC column. These steps include puncturing the septum, pressurizing the vial, venting the vial and injecting the headspace gas into the GC.

**scroll** To move text or graphics to see parts of the file that cannot be seen on the display

**sensitivity** (of analytical method). The ratio of the analytical signal increment to the corresponding increment of concentration (or contents) of the determinant

**setpoint** Numerical value for an analytical step entered into a method

**single headspace extraction** The standard method of headspace analysis. One headspace sample is taken from each sample vial being analyzed. See **multiple headspace extraction**.

**single puncture** Multiple headspace extraction performed by piercing the septum with the needle only once. The needle remains in the vial for all the heating and sampling steps until it is withdrawn after the last extraction. See **multiple puncture**.

**split injection** A injection method that allows very small amounts of sample to be introduced to capillary columns. A homogeneous sample is split into two portions in a known ratio; the smaller portion is introduced into the column and the major part is allowed to escape.

**splitless injection** An injection with the entire injected volume applied to the column

**standard addition** See **internal standard**

**Thermal Conductivity Detector (TCD)** A gas phase detector based upon changes in the thermal conductivity of the gas stream. The thermal

conductivities of hydrogen and helium are six to ten times greater than most organic compounds. The presence of even small amounts of organic materials causes a large decrease in the thermal conductivity of the column effluent.

**thermostated** Control of the temperature is provided by a thermostat

**top-level display** The main item in a menu. This is the display that appears first when the menu item is first accessed.

**vapor pressure** The pressure exerted by a vapor in equilibrium with its solid or liquid form.



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