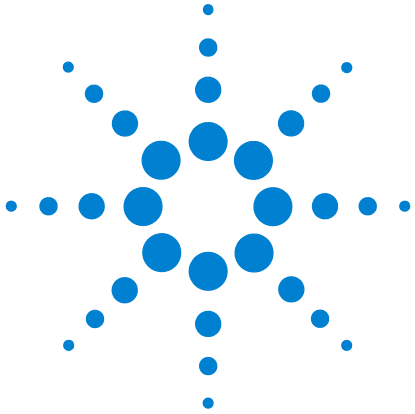

Agilent 7697A Headspace Sampler

Operation

Agilent Technologies

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1 Introduction

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This guide describes how to operate the Agilent 7697A Headspace Sampler during typical operation.

Introduction

Headspace analysis is a technique for analyzing volatile organic compounds using gas chromatography. Headspace analysis samples the ambient volume above a sample matrix, where the volatile compounds exist in gaseous form at predictable levels.

Headspace analysis is useful for situations where:

- The analyte of interest is volatile at temperatures below 285 °C (111 vial model) or 195 °C (12 vial model).
- The sample matrix is a solid, paste, or a liquid that is not easy to inject into a GC inlet.
- Sample preparation to allow easy liquid injection is currently difficult.

Headspace analysis provides several advantages over traditional injections:

- Simpler sample preparation. The sample does not need to be processed into an injectable liquid.
- Directly analyze a wide range of sample matrices (liquids, solids, and pastes).
- Solvent peak is smaller or nonexistent compared to traditional liquid injection GC techniques.
- Columns last longer, with less maintenance. The headspace volume above the sample matrix is more clean than the matrix. By injecting fewer contaminants, the analytical column lasts longer and requires less maintenance (trimming, bakeout, guard column replacement, and so forth).
- High precision.

The Agilent 7697A Headspace Sampler

The Agilent 7697A Headspace Sampler (HS) is a pressure-loop headspace sampling system with either a 12-vial or 111-vial capacity. If configured for 12 sample vials, the HS uses a single-vial oven to equilibrate the sample at the desired temperature. If configured with the 111 sample vial tray, the HS uses a 12 vial oven for equilibrating samples at temperature. Since the longest hold time in headspace analysis is typically the equilibration time, using a multi-vial oven allows the higher capacity HS to increase throughput by equilibrating multiple vials at once.

About This Manual

This manual described the concepts and tasks needed for routine headspace sampler operation.

For information needed to perform more advanced tasks and method development, see the *Advanced Operation Guide*.

Getting Familiar with the Headspace Sampler

Figure 1 111 Vial model—front view

Figure 2 12 Vial model—front view

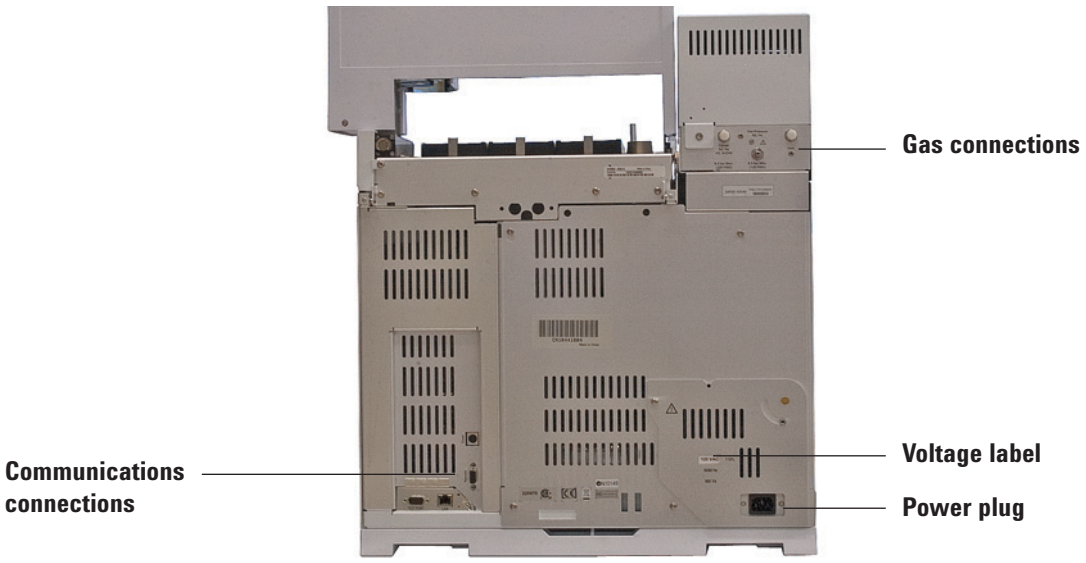

Figure 3 12 Vial model—back view (111 vial model is similar)

2
The Operation Workflow

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Method Development Workflow 15

This section describes the basic work flow for using the headspace sampler.

Routine Operation Workflow

Figure 4 summarizes the normal operating workflow for headspace analysis. This workflow assumes that the headspace sampler is set up and that the methods and samples are known.

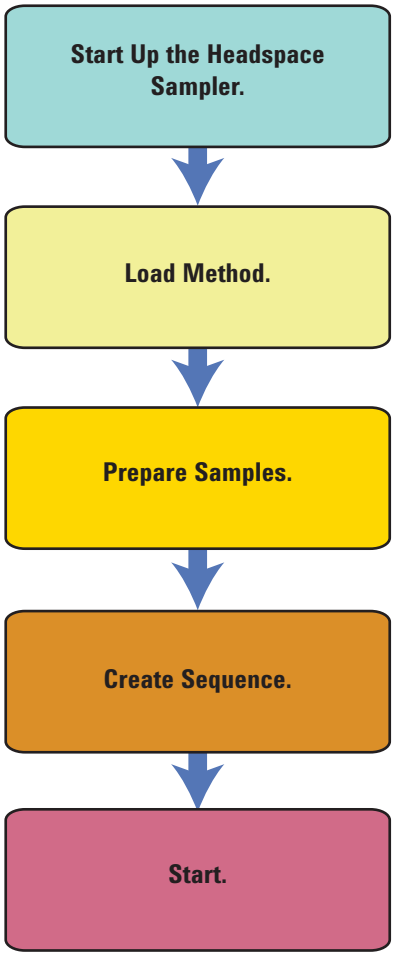

Figure 4 Routine headspace analysis workflow

Method Development Workflow

Figure 5 summarizes the workflow for developing methods. For details about method development, refer to the *Advanced Operation Guide*.

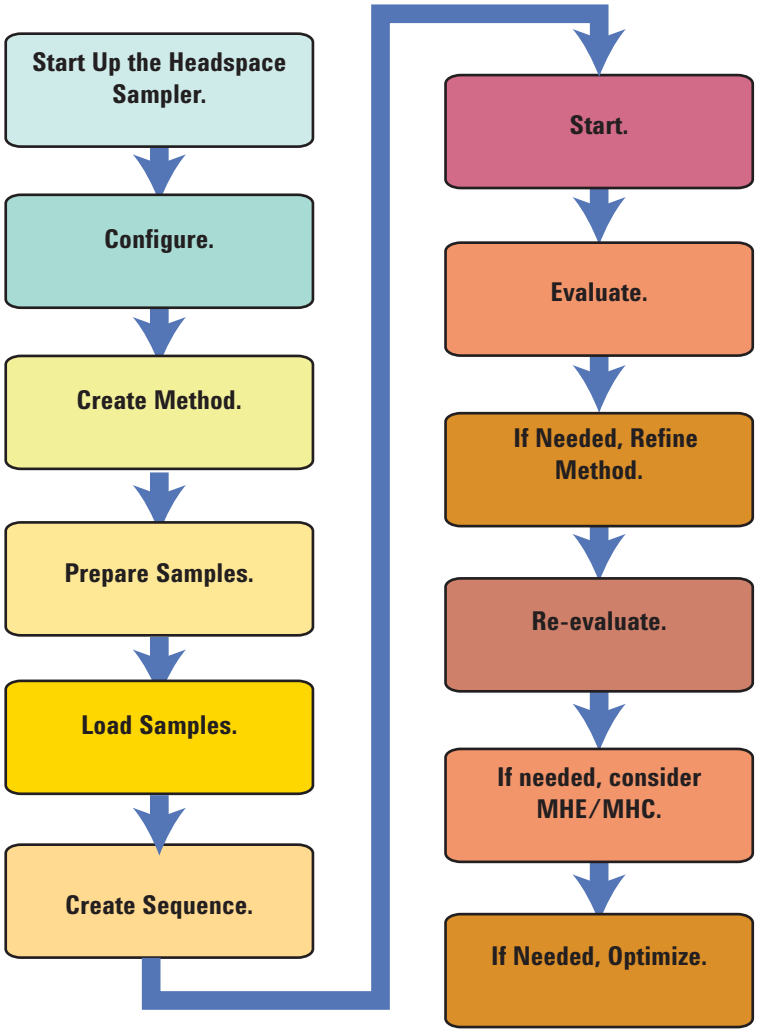

Figure 5 Workflow for method development

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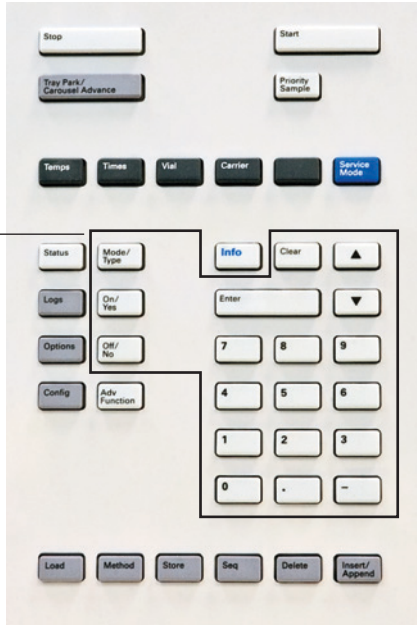
This section describes the basic operation of the Agilent 7697A HS keypad. The keypad provides access to all instrument functionality. For additional details on keypad functionality, see the *Advanced Operation Guide*.

The General Data Entry Keys

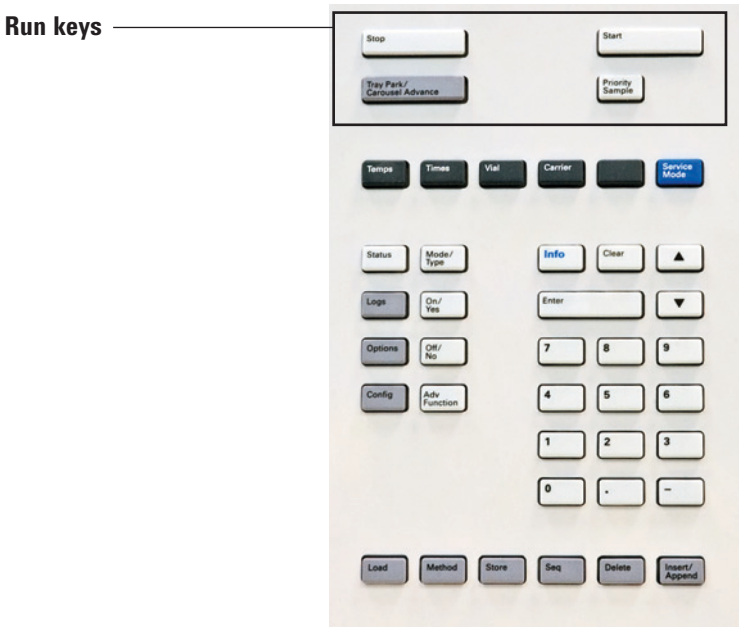
Use these keys to enter setpoints, make selections, and turn on or turn off HS components.



Mode/Type	Accesses a list of possible parameters associated with a component's nonnumeric settings. For example, when selecting the vial fill mode, press **[Mode/Type]** to list the vial fill mode options.
Clear	Removes a misentered setpoint before pressing **[Enter]**. It can also be used to return to the top line of a multiline display, return to a previous display, cancel a function during a sequence or method, or cancel loading or storing sequences and methods.
Enter	Accepts changes you enter or selects an alternate mode.
▲ ▼	Scroll keys. Use to scroll up and down through the display one line at a time. The < in the display indicates the active line.
Numeric Keys	Use to enter settings for the method parameters. (Press **[Enter]** to accept the changes.)
On / Yes **Off / No**	Use when you are setting up parameters, such as the warning beep, method modification beep, and key click, or for turning on or off a parameter or device (such as the transfer line heater).

The Run Keys

Use these keys to start sample preparation, stop preparation, and move the sample tray prior to loading sample vials.



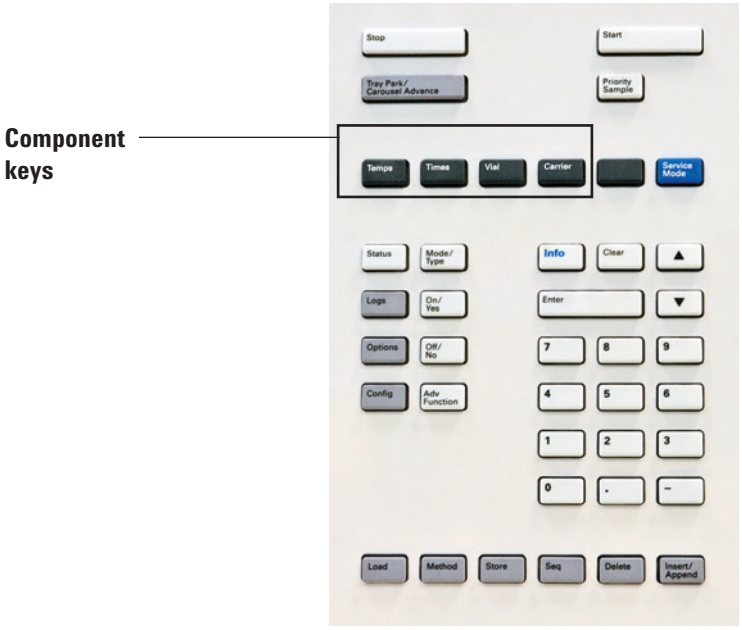
Stop	During a sequence: • Press once to pause the sequence. Current samples continue processing, but no new samples will be started. Press [**Start**] to resume. • Press twice (within 5 seconds) to abort the sequence. Any vials in the vial oven will be returned to the tray.
Start	Begin processing the next sample or sequence of samples. When processing is complete for a sample, the HS performs an injection to the GC and sends a Start Run signal.
Tray Park/Carousel Advance	• For the 111 vial model, press to park or unpark the tray. (Park the tray to load/unload vials or vial racks.) If pressed during a running sequence, the sequence pauses until the tray is unparked. • For the 12 vial model, press to advance the carousel one position.
Priority Sample	**111 vial tray model only. Standalone use only.** Press to insert a sample vial into the running sequence. Disabled when using data system control.

The Component Keys

Use these keys to access method temperatures, timing events, flows and pressures, vial fill modes, and similar parameters.

To display the current settings, press any one of these keys. More than three lines of information may be available. Use the scroll keys to view additional lines, if necessary.

To change settings, scroll to the line of interest, enter the change, and press **[Enter]**.



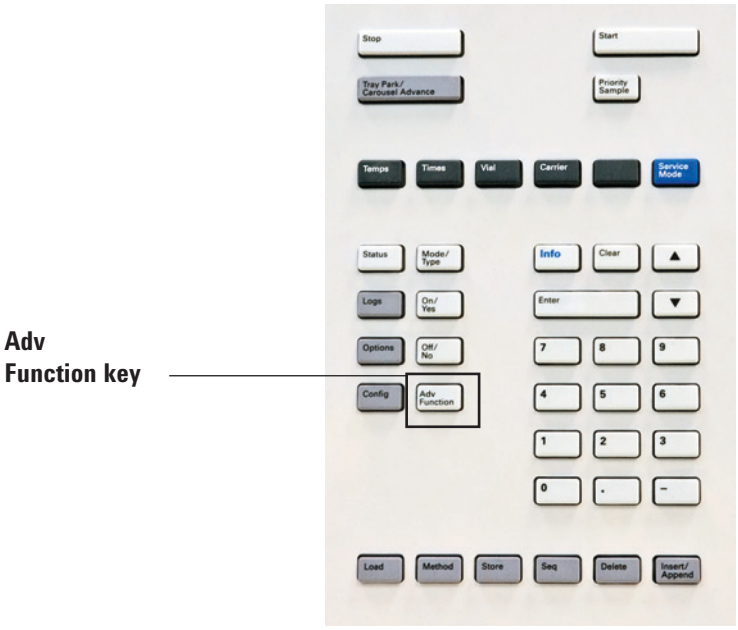
Temps	Press to set temperatures for the vial oven, sample loop, and transfer line.
Times	Press to set the times for vial equilibration, injection duration, pressure equilibration, and the GC cycle time.
Vial	Press to set vial size, vial fill mode and parameters, loop fill mode and parameters, and vial shaking.
Carrier	If using optional HS carrier gas EPC module, press to set the carrier gas control mode and the parameters for the carrier flow or pressure program. If the optional HS carrier gas control is not available, then the GC controls the carrier gas flow.

Note that editing temperatures and times from the keypad

immediately changes that setting in the current method, and the HS will begin to adjust to the new setting, if appropriate.

The Adv Function Key

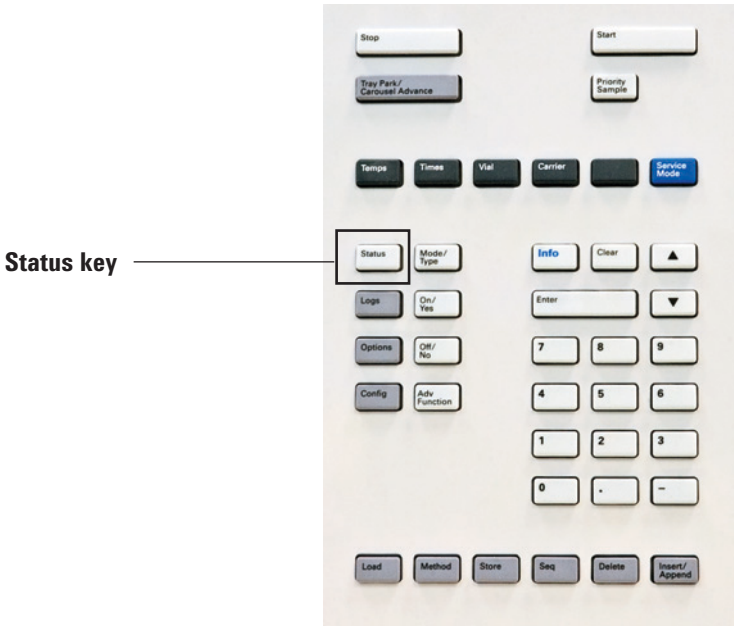
Use the [**Adv Function**] key to access advanced HS method parameters.



Adv Function	Use to access parameters for: • Sample loop and sample probe purge between sequences • Headspace extraction mode • Method sequence actions • Barcode reader settings • Parameters for method development

The Status Key

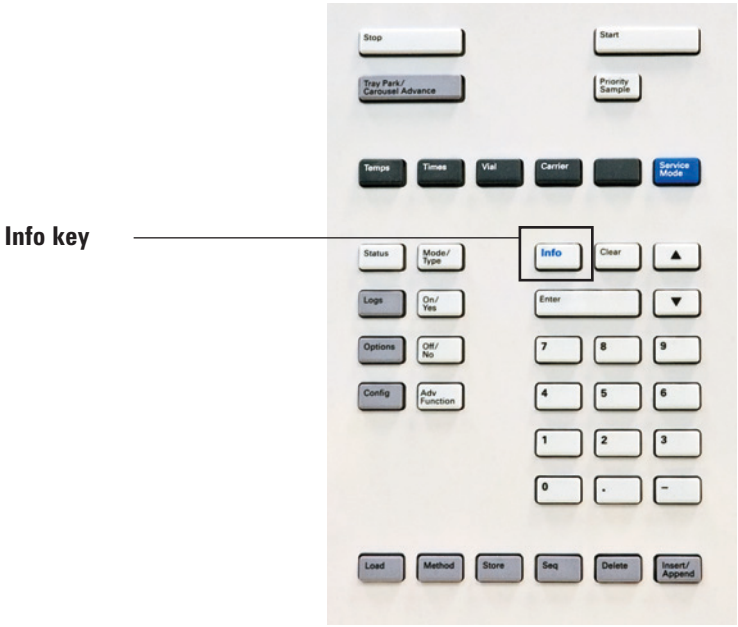
Use the **Status** key to learn about the current HS state.



Status	Toggles between sequence, setpoint, and vial status information and messages. Also displays "ready," "not ready," and "fault" information. When the Not Ready status light is blinking, a fault has occurred. The order in which items appear in the scrolling display window for setpoint status can be modified. You may, for example, want to display the things you most frequently check in the top three lines so that you do not need to scroll to see them. To change the order of the Status display: **1** Press **[Config] [Status]**. **2** Scroll to the setpoint you want to appear first and press **[Enter]**. This setpoint will now appear at the top of the list. **3** Scroll to the setpoint you want to appear second and press **[Enter]**. This setpoint will now be the second item on the list. **4** Continue as above until the list is in the order you require.

The Info Key

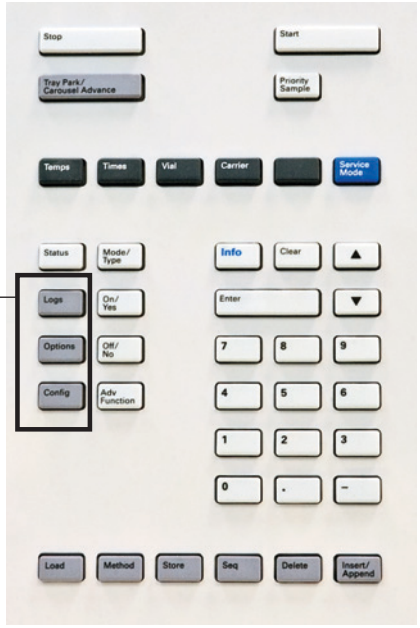
For context-sensitive help, press **[Info]**. For example, if you press **[Info]** on a setpoint entry, the help provided would be similar to: *Enter a value between 0.00 and 999.990 minutes.*



Info	Provides help for the currently shown parameter. For example, if **Vial equib time** is the active line in the display (has a **<** next to it), **[Info]** will display the valid range of times. In other cases, **[Info]** will display definitions or actions that need to be performed.

The Supporting Keys

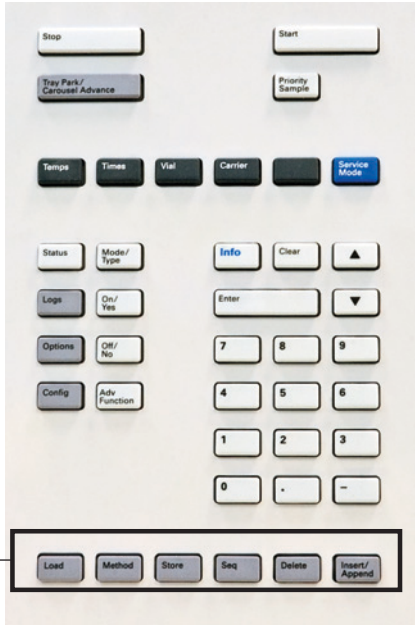
Use these keys to view and set configuration parameters, to set options, and to view instrument history data.



Logs	Toggles between the **Sequence Log**, the **Event Log**, and the **Maintenance Log**. The information in these logs can be used to support Good Laboratory Practices (GLP) standards.
Options	Accesses the instrument calibration, communications, and keyboard and display options. Scroll to the desired line and press **[Enter]** to access the associated entries. See the *Advanced Operation Guide* for details.
Config	Use **[Config]** to set up components that are not automatically detectable by the HS but are essential to preparing the sample or running method, such as gas types, carrier gas mode, loop volume, and the clock.

The Method Storage and Automation Keys

These keys are for loading and storing methods and sequences locally on your HS. They cannot be used to access methods and sequences stored by your Agilent data system.

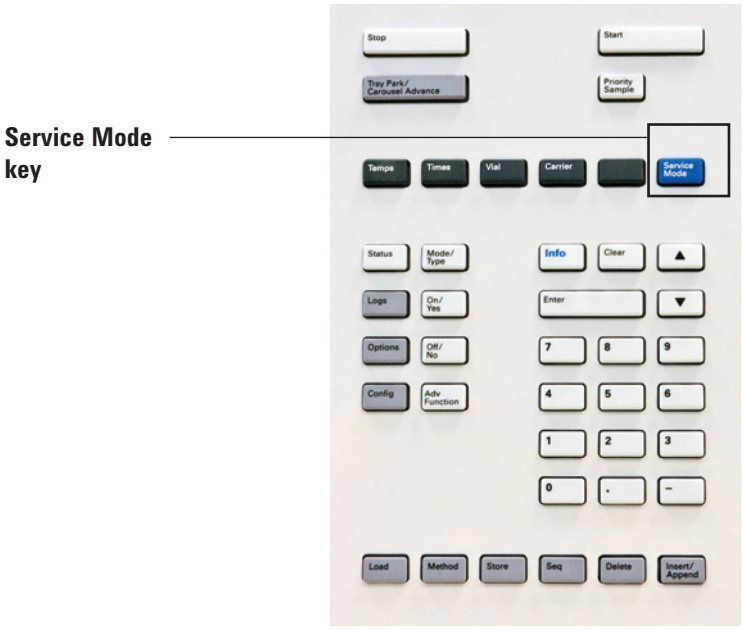


Method storage and automation keys

Load **Method** **Store** **Seq**	Use to load and store methods and sequences on your HS. For example, to load a method, press **[Load]** **[Method]** and select one from the list of methods stored in the HS. See "To Load a Method", "To Save (Store) a Method", "To Load a Sequence", and "To Save (Store) a Sequence".
Delete	Removes a method, sequence, or sequence line. See "To Delete a Sequence" and "To Delete a Method".
Insert/Append	Use to add a sample vial to a new or existing sequence.

The Service Mode Key

Use this key to access service information and procedures.



Service Mode	Use to access maintenance functions and settings, service counters, vial leak tests, and diagnostics for the HS. See the *Advanced Operation Guide* for details.

Keypad Functionality When the HS Is Controlled by an Agilent Data System

While under data system control, the keypad may be locked for general use. This locking feature prevents a user from unintended changes to the headspace method while the data system controls the instrument. If locked, the following changes occur:

- Method parameters can be viewed, but not changed.
- You cannot load, edit, or save a method.
- You cannot load, edit, or save a sequence.
- You cannot change the instrument configuration or perform advanced functions.

While under data system control, the keypad can be used:

- To view sequence status, including time data, by selecting **[Status]**.
- To view the method settings.
- To pause or abort a sequence by selecting **[Stop]**.
- To find which computer is controlling the HS by pressing **[Options] > Communication**, then scrolling. The name of the computer controlling the HS is listed below the **Enable DHCP** setting, along with the number of hosts connected to the HS.

Headspace Sampler Status

When the HS is ready to begin the current sequence, the display screen shows a Ready status, as shown below:

```
STATUS - Ready
Ready for start sequence
```

Alternatively, when a component of the HS is not ready to begin a run, the **Not Ready** indicator lights, and the status display shows Not Ready and explains why the HS is not ready.

```
STATUS - Not Ready
Oven is turned off
```

At any time, press **[Status]** to view messages explaining the current HS state, including whether the HS is ready to begin sample preparation or what conditions are currently preventing it from continuing.

Sequence status: Displays any status information related to the sequence.

Setpoint status: Displays general status information, such as readiness messages and faults states, and also lists the setting and current value data for HS setpoints.

Vial status: During sequence execution, displays details about the status of the selected vial. Use the arrow keys to select the desired vial.

Alert tones

One beep sounds when a problem exists, but the problem will not prevent the HS from executing the sequence. The HS will emit one beep and display a message. The HS can start the sequence and the warning will disappear when another sequence starts.

Fault messages indicate hardware problems that require user

intervention. Depending on the type of error, the HS emits no beep or a single beep.

A series of warning beeps sounds before a flow shutdown occurs. After a short time the component with the problem shuts down, the HS emits one beep, and a brief message displays. For example, a series of beeps sounds if the vial pressurization gas flow cannot maintain setpoint. The flow shuts down after 5 minutes. A shutdown message briefly displays. Press [**Clear**] to stop any beep. Vials in the oven continue to equilibrate, but the HS handles no additional vials and will not perform an extraction or injection.

A continuous tone sounds if using HS carrier gas control and if hydrogen flow is shut down or if a thermal shutdown occurs. During a hydrogen shutdown, all HS heaters and motors turn off.

WARNING **Before resuming operations, investigate and resolve the cause of the hydrogen shutdown. See Hydrogen Shutdown in the *Troubleshooting* manual for details.**

Error conditions

If a problem occurs, a status message appears. If the message indicates broken hardware, more information may be available.

Blinking setpoint

If the system shuts down a gas flow or the vial oven, **Off** will blink on the appropriate line of the component's parameter listing.

Status indicators

The front panel display provides five (5) indicator LEDs to show instrument status.

Tray Park	When lit, indicates the tray is parked. (111 Vial model)
Run	Lights when the HS is processing samples.
Sleep	Lights when the HS is in sleep mode. See "Resource Conservation".
Not Ready	Lights when the HS is not ready to process samples.
Service Due	Lights when an early maintenance feedback (EMF) counter has traversed a threshold, and the counter has been set to light this indicator.

Logs

Three logs are accessible from the keypad: the run log, the maintenance log, and the system event log. To access the logs, press **[Logs]** to toggle to the desired log. The display will indicate the number of entries the log contains. Scroll through the list.

Sequence log

The sequence log is cleared at the start of each new sequence. During the sequence, any deviations from the planned methods (including keypad intervention) are listed in the sequence log table.

Maintenance log

The maintenance log contains entries made by the system when any of the user-defined component counters reach a monitored limit. The log entry contains a description of the counter, its current value, the monitored limits, and which of its limits has been reached. In addition, each user task related to the counter is recorded in the log, including resetting, enabling or disabling monitoring, and changing limits or units (cycles or duration).

Event log

The event log records significant events during HS operation. Some of the events also appear in the sequence log if they occur during sequence execution.

4 Consumables

Consumables for Headspace Analysis 34

This section lists commonly-used parts, such as vials and sample loops, needed for routine operation of the Agilent 7697A Headspace Sampler. Procedures for replacing these parts can be found in this manual or in the Maintenance manual.

Consumables for Headspace Analysis

The following tables provide list common supplies for the headspace sampler and headspace analysis. For the latest parts available, visit the Agilent website at www.agilent.com/chem.

Table 1 Headspace sampler parts and standards

Description	Part number
Leak test kit. Includes: • No hole ferrule • 11 mm low bleed septa, 5/pk • Headspace (blue) leak test vial • 1/8-in. nylon tube fitting plug • 1/16-in. stainless steel ZDV plug (6 port valve cap)	G4556-67010 5181-7458 5182-3413 G4556-20600 0100-2414 G6600-80039
Tray vial rack set, 7697A (3 racks)	G4564A
Tray vial rack	G4556-60019
Tray vial rack labels	G4556-90500
Universal/external split vent trap with 3 cartridges, 1/8-inch Swagelok fitting	RDT-1020
Column cutting wafer, ceramic	5181-8836
Sample probe, deactivated SN1030	G4556-60690
Sample probe, deactivated SN2000	G4556-60125
6-port valve, replacement rotor, WT series, 300 psi, 350 °C	1535-4952
Sample loop adapter: 1 ea. used with 0.025, 0.05, and 0.10 mL sample loops 2 ea. used with 0.5 and 1.0 mL sample loops	G4556-20177
Sample loop adapter: 1 ea. used with 0.025, 0.05, and 0.10 mL sample loops	G4556-20178
Standards	
GC headspace evaluation standard, 1 x 1 mL	8500-4328
Headspace OQ/PV sample	5182-9733

Table 2 Headspace sampler transfer line parts

Description	Part number
Transfer line components	

Table 2 Headspace sampler transfer line parts (continued)

Description	Part number
Ferrule, polyimide Valcon, 5/pk	
0.53 mm, 1/32 in. for tubing OD 0.50 ≤ 0.80 mm	0100-2595
0.25, 0.32 mm, 1/32 in. for tubing OD 0.25 ≤ 0.40 mm	5190-1437
Septum nut, transfer line, for split/splitless and multimode inlets	G3452-60835
Blanking nut, 1/16-inch stainless steel	01080-83202
Nut and reducing union for 6 port valve and transfer line connection	0100-2594
Transfer lines	
Deactivated fused silica, 250 µm x 5 m	160-2255-5
Deactivated fused silica, 320 µm x 5 m	160-2325-5
Deactivated fused silica, 450 µm x 5 m	160-2455-5
Deactivated fused silica, 530 µm x 5 m	160-2535-5
ProSteel deactivated stainless steel, 5 m length	160-4535-5
ProSteel sleeve for ProSteel, 5 m length	4177-0607

Table 3 Headspace sampler sample loops

Description	Part number
Sample loops, SN 2000	
0.025 mL	G4556-80101
0.05 mL	G4556-80102
0.1 mL	G4556-80103
0.5 mL	G4556-80105
1.0 mL	G4556-80106
1.0 mL, Certified	G4556-80126
3.0 mL	G4556-80108
3.0 mL, Certified	G4556-80128
5.0 mL	G4556-80109
Sample loops, SN 1030	
0.025 mL	G4556-80111
0.05 mL	G4556-80112

Table 3 Headspace sampler sample loops (continued)

Description	Part number
0.1 mL	G4556-80113
0.5 mL	G4556-80115
1 mL	G4556-80116
3 mL	G4556-80118
5 mL	G4556-80119
Adapters for sample loops	
Sample loop adapter: 1 ea. used with 0.025, 0.05, and 0.10 mL sample loops 2 ea. used with 0.5 and 1.0 mL sample loops	G4556-20177
Sample loop adapter: 1 ea. used with 0.025, 0.05, and 0.10 mL sample loops	G4556-20178

Table 4 Headspace vials and caps

Description	Part number
Certified flat bottom vials	
Certified flat bottom headspace vials, 20 mL, 100/pk	5182-0837
Certified flat bottom headspace vials, 10 mL, 100/pk	5182-0838
20 mm Headspace caps, with septa	
Certified headspace Al crimp cap, PTFE/Si septum, 20 mm,100/pk	5183-4477
Headspace vial kits	
Vial kit 20 mL Headspace crimp top, flat bottom vials, silver aluminum one-piece crimp caps with safety feature, PTFE/white silicone septa, 100/pk	5182-0840
Cappers and decappers	
Electronic crimper for 20 mm cap vials	5062-0208
Electronic decapper, for 20 mm crimp caps	5062-0210
Ergonomic manual crimper for 20 mm caps	5040-4669
Ergonomic manual decapper for 20 mm caps	5040-4671

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This section discusses sample vial selection, sample preparation, and vial handling with the Agilent 7697A Headspace Sampler.

Sample Vial Types

The headspace sampler accepts 10-mL, 20-mL, or 22-mL sample vials. Set the vial size in the method (see "To Create a Method"). The vial size can change with each new method used in a sequence, but not within a method. Using a different vial size than expected by the method causes a run-time exception.

The headspace sampler uses clear or amber glass sample vials with crimp caps, or screw-cap vials. Use amber glass vials for light-sensitive samples. Both types are available with flat or rounded bottoms. Refer to your Agilent catalog for consumables and supplies for acceptable vial types, or visit the Agilent website at www.agilent.com/chem. Incompatible sample vials can cause gripper errors (111 vial model).

Vials must conform to the specifications shown in Figure 6.

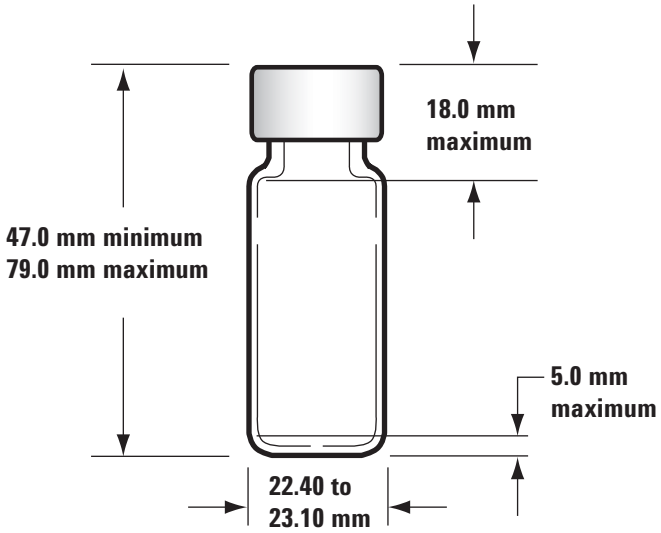

Figure 6 Supported vial dimensions

Avoid reusing vials. Reused vials may crack when heated.

Sample Vial Septa and Caps

There are two types of septa used with crimp caps and screw-on caps, each with different resealing characteristics and different resistance to solvents.

Septum material	Compatible with	Incompatible with	Resealability	Maximum temperature*
PTFE/butyl rubber	PTFE resistance until punctured, then septa or liner will have compatibility of rubber (ACN, acetone, DMF, alcohols, diethylamine, DMSO, phenols)	Chlorinated solvents, aromatics, hydrocarbons, carbon disulfide	Good	< 125 °C
PTFE/silicone rubber	PTFE resistance until punctured, then septa will have compatibility of silicone (alcohol, acetone, ether, DMF, DMSO)	ACN, THF, benzene chloroform, pyridine, toluene, hexane, heptane	Average	< 180 °C

* Approximate. Refer to manufacturer's recommendations.

Vial caps come with or without an internal safety feature that allows the vial to vent if the internal vial pressure exceeds about 310 kPa (45 psi).

In general, do not use crimp caps or septa more than once for headspace analysis.

Refer to your Agilent catalog for consumables and supplies for acceptable vial types, or visit the Agilent website at www.agilent.com/chem.

Vial Labels

CAUTION Make sure that any label and ink can withstand the oven heat without degrading.

If using labels with a 111 vial model, the label needs to conform to the dimensions below. If also using the optional barcode reader, the barcode labels must conform to the general dimensions for labels, plus the placement requirements shown.

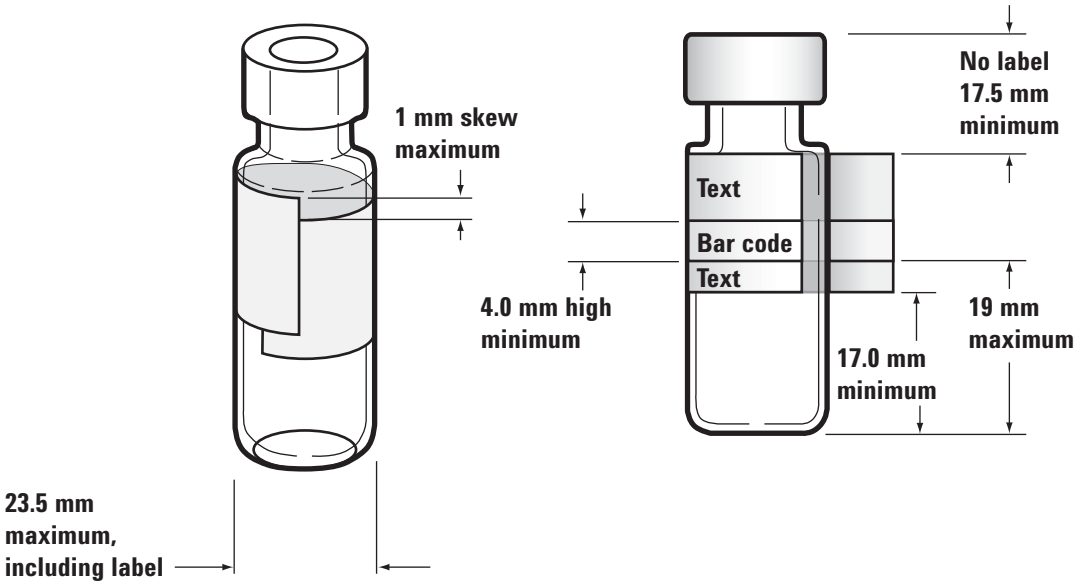

Figure 7 Vial label and barcode specifications

CAUTION Correct sample vial dimensions are critical for proper gripper operation. Vials and labels that do not meet these specifications may cause sampler errors. Service calls and repairs found to be due to vials and labels that do not meet these specifications are not covered under warranty or the service contract.

Filling Sample Vials

In general, fill sample vials about half way. Although sample amounts can vary depending on the analysis, do not fill vials more than the amount shown in Figure 8. Filling the vial correctly ensures that the sampling probe will not contact with the matrix during sampling. If you need more sample, use a larger vial or optimize the method to improve results. See the *Advanced Operation Guide* for recommendations.

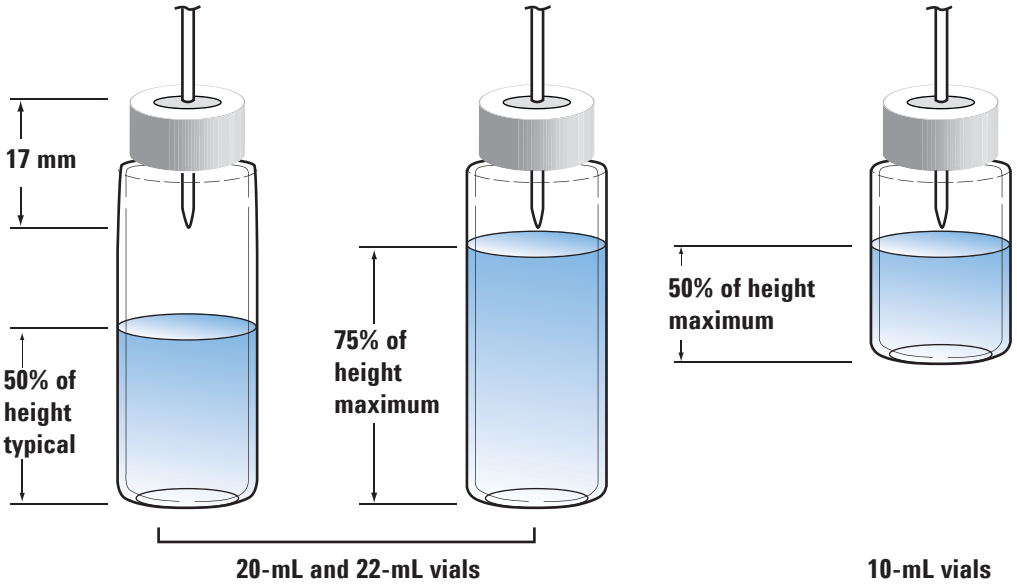

Figure 8 Vial fill limits

To Cap a Sample Vial

The vial must be sealed properly to insure that the headspace gases do not escape prematurely. For crimp top vials, use a crimper designed for headspace vials with 20-mm caps to seal the vials. Screw caps and screw top vials are also available. See "Consumables for Headspace Analysis".

1. Before beginning, clean the inside surfaces of the crimper jaws.
2. If using separate septa and caps, place a septum in a vial cap with the PTFE side facing the vial. Take care not to contaminate the septum.
3. Place the cap upside down on a table.
4. Place the sample in the vial. (Most vials should not be more than 50% full, but some vials can reach 75% full. See "Filling Sample Vials".)
5. Place the septum and cap assembly over the vial opening.
6. Lift the vial into the crimper.
7. With slow and steady pressure, squeeze the crimper handles to seal the vial. (Squeeze the handle until it reaches the adjustment screw.)

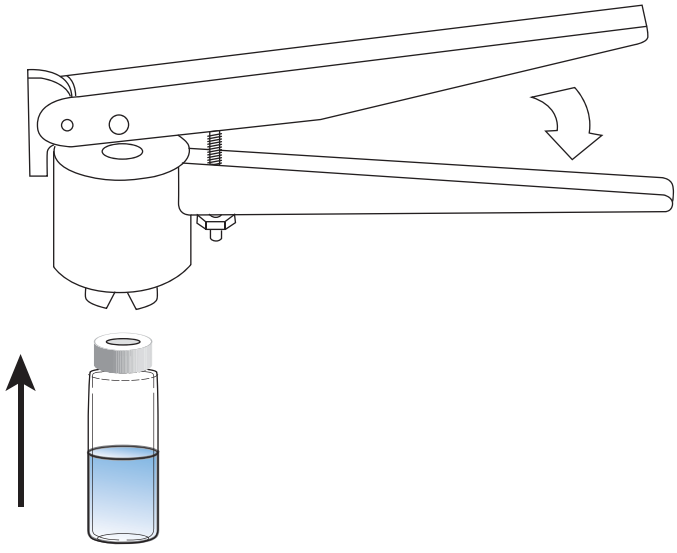

Figure 9 shows proper and improper vial caps.

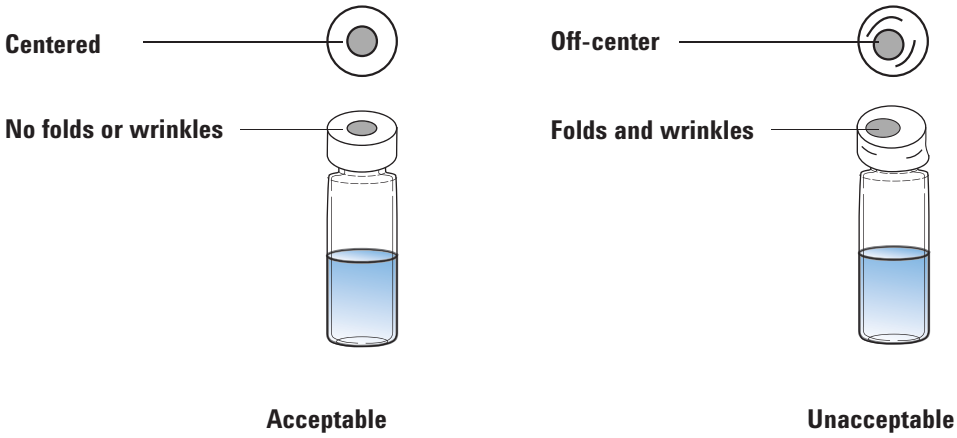

Figure 9 Acceptable and unacceptable vial caps

Check each vial for proper crimping:

- Be sure there are no folds or wrinkles on the part of the cap that wraps under the neck of the vial. To remove folds or wrinkles, turn the vial about 10° and crimp it again. Adjust the crimper for a looser crimp by turning the adjusting screw clockwise.
- The cap should be finger-tight. If the cap is loose, adjust the crimper for a tighter crimp by turning the adjusting screw counterclockwise. Crimp the cap again. If the cap is too tight, the septum will distort and the vial may leak.
- Be sure that each cap has a flat septum centered over the top of the vial.
  - If the septum is not flat, remove the cap, turn the crimper adjusting screw clockwise, and try again.
  - If the cap is not centered, remove the cap and make sure the new cap is flat on the top of the vial before you squeeze the crimper.

To Park or Unpark the Tray (111 Vial Model)

Parking the tray moves the tray gantry to a safe position. When parked, you can load vials into the racks, or install and remove racks from the HS.

Press **[Tray Park/Carousel Advance]** to park the tray. The display reads:

```
          TRAY PARKED
Tray Park - Unpark tray
Start - Start sequence
```

Press **[Tray Park/Carousel Advance]** to unpark the tray and ready it for use. The display reads:

```
         SEQUENCE IDLE
Start - Start sequence
Tray Park - Park tray
```

You cannot start a sequence if the tray is parked.

Parking the tray during a sequence pauses the sequence. Current vials continue to process, but no new vials are started.

To Install a Vial Rack (111 Vial Model)

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

CAUTION When loading a tray rack with sample vials, avoid excessive tray motion. If the sample coats the septum or coats the vial more than typical, this may change results.

2 While holding up the front end of the rack, slide the rack back and under the mounting clip on the HS top. Then, lower the front of the rack in place.

When installed correctly, a green LED on the tray rack lights.

Figure 10 Tray vial positions

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

To Load a Sample into the Tray (111 Vial Model)

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

2 Place the capped sample vials into the tray as desired. See Figure 11.

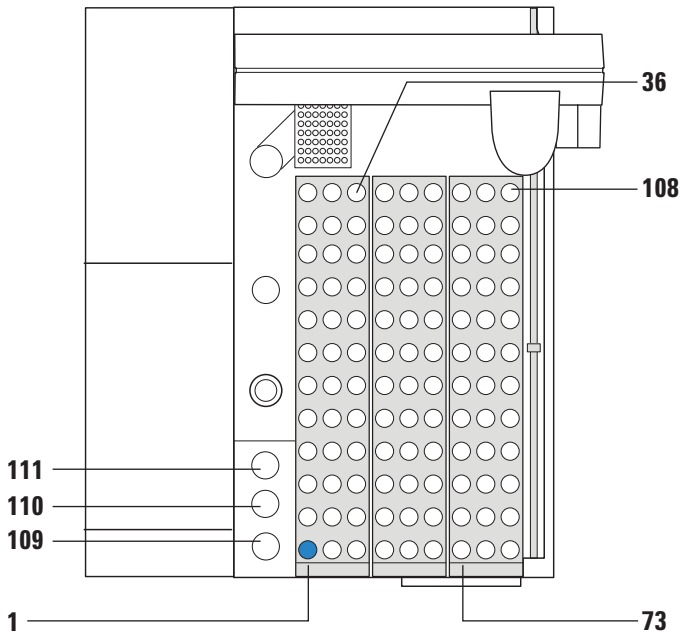

Figure 11 Tray vial positions

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

To Load a Sample into the Tray (12 Vial Model)

1 Open the tray cover. See Figure 12.

Figure 12 Opening the tray cover

2 A label to the left of each vial position denotes the number of that position. See Figure 13.

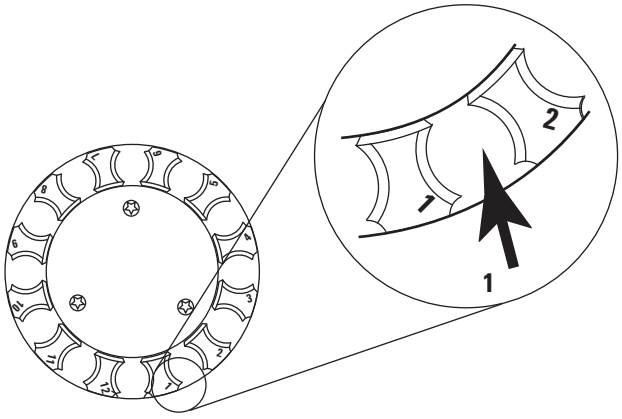

Figure 13 12 Vial tray positions

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

4 Place the capped sample vials into the tray as desired.

6 Methods

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To Edit a Method 57
To Load a Method 58
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To Determine the GC Cycle Time 60

This chapter defines the headspace sampler method, and describes how to load, save, create, and edit a method using the HS front keypad. If using an Agilent data system, most likely you will use the data system to create methods. Refer to the data system help and documentation for details, and to the *Familiarization Guide*.

What Is a Method?

A method is the collection of setpoints needed for the headspace sampler to prepare a sample and inject it into the gas chromatograph. The Agilent 7697A Headspace Sampler can store up to 32 methods internally, plus several built-in methods. The primary parts of the method control the parameters for:

- **Temperatures** for the vial, sample loop, and transfer line
- **Times** for equilibration and injection, plus the GC cycle time (used for sample overlap and throughput calculations)
- **Vial** settings for vial size, filling, shaking, and venting after the injection
- **Carrier** gas settings, for controlling carrier gas flows during the injection and GC run (if using the optional EPC module)

The parameters available depend on the instrument model and current configuration. For complete details about all possible method parameters, see the *Advanced Operation Guide*.

Built-in methods

In addition to the 32 user-definable methods, the HS also stores 6 special methods:

Method	Comments
Default	A basic method, useful as a basis for new methods. Not editable.
Checkout	The parameters needed for initial performance verification. Not editable.
Qualification	The parameters needed for Agilent's performance verification and qualification service. Not editable.
MeOH in H2O	Basic starting parameters for analyzing methanol in water. Not editable.
Sleep	Use the **Sleep** method to load settings for periods of inactivity. The sleep method is editable. To schedule the instrument to load the sleep method at a given time of day, see the *Advanced Operation Guide*.

Method	Comments
Wake	Use the **Wake** method to load settings after the sleep period ends, for example, to warm the vial oven or increase gas flows. If desired, the wake method can be simply the last analytical HS method run. The wake method is editable. To schedule the instrument to load the wake method at a given time of day, see the *Advanced Operation Guide*.

Data system methods

If using an Agilent data system, you will primarily use the data system methods, rather than the methods stored locally in the HS. Data system methods save parameters for all associated instruments and devices, such as the GC and HS, plus all data analysis and reporting settings.

When running a sequence, the data system will download the setpoints to the HS as needed. HS data system method parameters appear on the HS display as a **HOST** method. The HS does not permanently store HOST methods. HOST methods only remain until overwritten or until the HS is turned off.

Method Parameter Summary

This section lists method parameters, along with a brief description of each one. For more detailed information, see the *Advanced Operation Guide*.

Table 5 Common method parameters

Parameter	Enabled in modes	Description
[Temp] key		
Oven		Oven temperature for vial equilibration.
Loop/Valve		Temperature of the sample loop and valve.
Transfer line		Temperature of the transfer line (isothermal).
[Time] key		
GC cycle time		Time for the GC to become ready after an injection.
Vial equib time		Time for the vial to equilibrate in the oven.
Pres equib time		Time to allow for the pressure in the vial to stabilize after initial vial pressurization.
Inject time		Amount of time to sweep the sample loop vapors into the GC inlet.
[Vial] key		
Fill mode		Select how to pressurize the vial.
Fill pressure	Fill modes: Flow to pressure and Fill to pressure	Target sample vial final pressure.
Fill flow	Fill mode: Flow to pressure	Flow rate used to pressurize the vial.
Fill volume, mL	Fill mode: Constant volume	Specific volume of gas with which to pressurize the vial.
Loop fill mode		Select how the HS will fill the sample loop with gas after pressurizing and puncturing the sample vial.
Loop ramp fill rate	Loop fill mode: Advanced	How quickly to fill the sample loop.
Loop final pressure	Loop fill mode: Advanced	Final target pressure for the filled sample loop.
Loop equilibration	Loop fill mode: Advanced	Time set for the sample loop to stabilize after pressurization.
Vent after extraction		After the last extraction, and while the sample transfers to the GC, vent residual vial pressure to atmosphere. (Other venting is provided during concentrating extractions under the [**Adv Function**] key.)

Table 5 Common method parameters (continued)

Parameter	Enabled in modes	Description
Vial size		Select the sample vial size for all vials using this method.
Shaking		Set the level of shaking for the sample during equilibration in the oven. (111 vial model only)
[Carrier] key		
Pressure (read only display)	Carrier control: GC Control	For other carrier parameters, see the *Advanced Operation Guide*.
[Adv Function] key		See the *Advanced Operation Guide*.
Extraction mode		Set the type of extraction for the method, Single, Multiple, or Concentrated.
# of extractions	Concentrated extractions	Enter the number of extractions to concentrate.
Vent between extractions	Concentrated extractions	On or Off. Vent vial between concentrating extractions.
Purge flow		Purge sample probe and loop with vial pressurization gas after removing the vial from the probe.
Purge time		
Barcode reader		Requires the 111 vial tray and optional barcode reader. Select to set barcode reader parameters.
Sequence actions		Set how the HS should handle unexpected sequence issues, such as a missing vial or vial size mismatch.
Method development		Access parameters to use when developing methods.
[Adv Function] > Barcode reader		Requires the 111 vial tray and optional barcode reader.
BCR action		Set whether and how to proceed if a barcode error occurs.
BCR symbology		Set the expected symbology for barcodes used with this method.
Enable BCR checksum		Expect barcodes to use a checksum feature (availability also depends on barcode symbology).

To Create a Method

To create a basic method, you will access the method parameters using the 4 component keys, in order: **[Temps]**, **[Times]**, **[Vial]**, and **[Carrier]**. For a listing of commonly-used parameters, see "Method Parameter Summary".

1 When the HS is not processing samples, press **[Temps]**. The display shows the current oven, loop/valve, and transfer line temperature setpoints and actual temperatures.

   a Scroll to **Oven**.

   b Use the keypad to enter the new oven setpoint, then press [**Enter**].

   c Enter the desired **Loop/Valve** temperature, then press [**Enter**].

   d Enter the desired **Transfer line** temperature, then press [**Enter**].

   Temperature setting is complete.

2 Press [**Time**].

   a Scroll to **GC cycle time**.

   b Use the keypad to enter the new **GC cycle time**, then press [**Enter**]. To determine the correct value to input, see "To Determine the GC Cycle Time".

   c Enter the desired **Vial equib time**, then press [**Enter**].

   d Enter the desired **Pres equib time**, then press [**Enter**].

   e Enter the desired **Inject time**, then press [**Enter**].

   Time setting is complete.

3 Press [**Vial**].

   a Scroll to **Fill mode**.

   b Press [**Mode/Type**] to display the selections for the vial fill mode. Scroll to the desired fill mode, then press [**Enter**].

   c Enter any vial fill mode parameters, pressing [**Enter**] after each.

NOTE If using **Constant volume** loop fill, the HS determine loop fill. Skip steps d and e.

d Scroll to **Loop fill mode**, then press [**Mode/Type**] to display the selections for the sample loop fill mode. Select the desired loop fill mode.

e Enter any sample loop fill mode parameters, pressing [**Enter**] after each.

f Scroll to **Vent after extraction**. Press [**On/Yes**] to vent the sample loop after the last each extraction, or [**Off/No**] to leave the loop as-is.

g Scroll to **Vial size**. Press [**Mode/Type**] to select the vial size from a list, then press [**Enter**].

h Scroll to **Shaking**. Press [**On/Yes**] to enable, then press [**Mode/Type**] to select the level of shaking desired. Press [**Off/No**] to disable shaking for this method.

Vial settings are complete.

4 Press [**Carrier**]. If not using an optional EPC carrier gas module, these settings are disabled and the HS uses GC carrier gas control. If using the optional EPC carrier gas module, you can set parameters as defined by the configured carrier gas control mode.

Set carrier gas parameters using the same process as the other method parameters.

Carrier gas settings are described in the *Advanced Operation Guide*.

5 Basic method parameters are complete. Save the method. (See "To Save (Store) a Method".)

In addition, the **Adv Function** key provides access to other method parameters, including multiple headspace extraction modes and functions useful during method development.

See the *Advanced Operation Guide* for more information.

To Save (Store) a Method

To save a method:

1. Press [**Method**].
2. Scroll to the method number to save.
3. Press [**Store**]. When prompted, press [**On/Yes**] to save, or [**Off/No**] to return to the selection list.

Alternately:

1. Press [**Store**].
2. When prompted, press [**Method**].
3. When prompted, scroll to the desired method number to save.
4. Press [**Enter**].
5. When prompted, press [**On/Yes**] to save, or [**Off/No**] to return to the selection list.

To Edit a Method

To edit a method:

1. Load the desired method. See "To Load a Method".
2. Edit the method parameters as desired. See "To Create a Method".
3. When done, save the method. See "To Save (Store) a Method".

To Load a Method

To load a method:

1 Press [**Load**].

2 When prompted, press [**Method**].

3 When prompted, select the method to load from the list, then press [**Enter**].

4 When prompted, press [**On/Yes**] to load the selected method, or [**Off/No**] to return to the selection list.

Alternately:

1 Press [**Method**].

2 Scroll to the desired method.

3 Press [**Load**]. When prompted, press [**On/Yes**] to load the selected method, or [**Off/No**] to return to the selection list.

To Delete a Method

You can delete only the internal, user-defined methods. To delete a method:

1 Press [**Method**].

2 Scroll to the user-defined method to delete.

3 Press [**Delete**]. When prompted, press [**On/Yes**] to confirm.

Alternately:

1 Press [**Delete**].

2 When prompted, press [**Method**].

3 When prompted, scroll to the method number to delete, then press [**Enter**].

4 When prompted, press [**On/Yes**] to delete the selected method, or [**Off/No**] to return to the selection list.

To Determine the GC Cycle Time

The HS uses the **GC cycle time** to calculate throughput and timing. An accurate **GC cycle time** is crucial to optimizing throughput and for correctly processing samples.

If the **GC cycle time** is too long, this can cause:

- Lowered throughput. Vials wait longer than needed before processing.

If the **GC cycle time** is too short, this can cause:

- Sequence faults. Vials may be processed too early and may sit too long while waiting for the GC to become Ready.

It is better to enter a longer time than needed than to enter too short a time and possibly reduce sample quality.

The **GC cycle time** is the GC run time plus any additional time needed for the GC to execute any post-run program, then return to a ready state.

To determine the GC cycle time

To determine the **GC cycle time**, program the GC to perform a sequence of two or three blank (no injection) runs.

- If using a data system, you can determine the cycle time from the data system sequence log. Compare the start times for the runs. A good **GC cycle time** is the average difference between the start times, plus 0.2 to 0.5 minutes.
- If not using a data system, watch the GC. Count the time between the start of the first run and when the GC becomes Ready for the second run. Then, add 1 to 2 minutes.

You can also *estimate* the **GC cycle time** without making a run. By adding the GC oven program duration and the duration of any post-run programs, you can get close to the true cycle time. However, temperature programming and cryogenic operation can make estimation more difficult. Add extra time.

Also consider time for data processing. While in most cases data processing is not a problem, a very busy data system may need extra time between samples.

Validating the GC cycle time

Look at the GC run time. The **GC cycle time** cannot be less than the total run time.

Look at the GC post run program duration. The **GC cycle time** cannot be less than the total run time plus the post run program duration.

6 Methods

7 Sequences

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This chapter defines the headspace sampler sequence, and describes how to load, save, create, and edit a sequence.

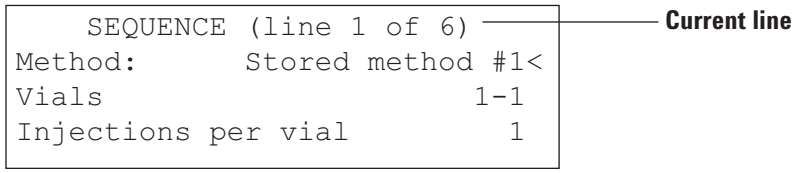
What Is a Sequence?

A sequence for the 7697A Headspace Sampler is an ordered series of sample vials to prepare and inject, including the method needed to prepare each vial. For the 7697A HS:

- All sample processing occurs in a sequence. To run a sample, there must be a sequence defined.
- A sequence can skips vials.
- A sequence can run a vial more than once.
- A sequence does not require any particular vial order. Running vials 1, 49, 5, 2, 3, and 101 is valid.

A sequence consists of a series of *lines*. Each line contains a range of vials, the method number to use for these vials, and the number of injections to make using each vial.

To view a sequence, press [**Seq**].



Use the scroll keys to view all lines in the sequence.

Sequences, extraction modes, and vial punctures

In the sequence you can specify the same vial in as many entry lines as desired. How the HS sampler processes the vial depends on the method Extraction mode and the sequence:

- **Extraction mode** is **Single**.

  If a vial appears more than once in a sequence, or if the number of injections is greater than one, the vial is completely reprocessed for each entry or injection.

- **Extraction mode** is **Multiple**.

  If the number of injections per vial is greater than 1, the HS will puncture the vial once, then make the extractions and injections. The HS will *not* perform multiple vial punctures.

When more than one consecutive entry exists for a vial, and the entries use the same method, the HS will puncture the vial once, then make the extractions and injections. The HS will *not* perform multiple vial punctures.

- **Extraction mode** is **Concentrated**.

  If a vial appears more than once in a sequence, or if the number of injections is greater than one, the vial is completely reprocessed for each entry or injection.

See also "To Create a Sequence" and "Sequences and Throughput (111 vial model)".

How many sequences can the HS store?

The HS can store up to 9 sequences internally. In addition, the HS can temporarily store sequences that are downloaded from the data system.

Priority samples

A *priority sample* is a vial that you want to run as soon as possible, before other vials in the currently-running sequence. Use the priority sample feature to insert one or more special vials into the current sequence without stopping the sequence, editing it, and restarting it.

This feature is available only for the 111 vial model, and only when operating in standalone mode. (The feature is not needed when using an Agilent data system. See "Priority samples using an Agilent data system".)

Priority sample locations

The priority sample locations are vial locations 109, 110, and 111. See Figure 14 below.

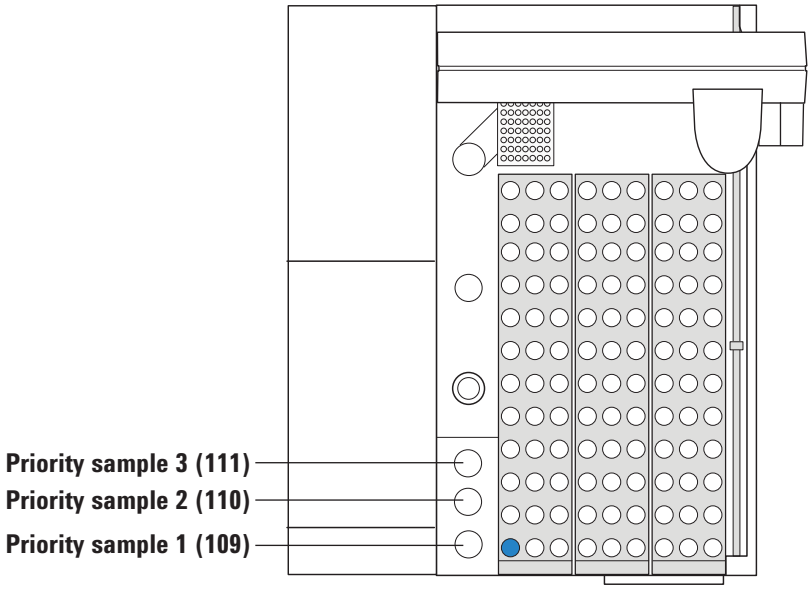


Figure 14 Priority sample locations

How the HS processes priority samples

The first time you press [**Priority Sample**] during a running sequence, the HS checks the first priority sample location (Figure 14). It takes the vial from that location and inserts it into the current sequence at the next appropriate time. (The HS does not interrupt any ongoing vial moves or processing.)

- If the vial is not present, a vial not found error occurs. (See the *Advanced Operation Guide* for information about how to set HS handling of missing vials.)
- The HS continues to process any vial it is handling before it looks for the new priority sample vial.
- **The HS uses the method of the last vial placed in the oven to process the priority sample vial.** If you wish to use another method, you must interrupt the current sequence.
- You may insert only three priority samples per sequence.
- Avoid using sample vial positions 109–111 if you might use priority samples. If you use positions 109–111 in your normal sequences, you will need to replace the normal sequence vials with the priority vials, run the priority vials, then put the normal sequence vials back.

Consecutive presses of [**Priority Sample**] cycle through the priority locations.

1 The first time you press [**Priority Sample**], the HS checks for and runs the vial in position 1.
   - The **Vial Status** names this vial **P 1**.

2 The second time you press [**Priority Sample**], the HS checks for and runs the vial in position 2.
   - The **Vial Status** names this vial **P 2**.

3 The third time you press [**Priority Sample**], the HS checks for and runs the vial in position 3.
   - The **Vial Status** names this vial **P 3**.

If you press of [**Priority Sample**] a fourth time, an error message appears. You may only insert 3 samples into the sequence using this feature. If you need to make a fourth change, stop the sequence and edit it to add the needed sample.

Priority samples using an Agilent data system

The Agilent data system already provides flexibility for editing a running sequence. When using an Agilent data system, the priority samples feature is disabled. To insert a new sample into the running sequence, simply edit the sequence to insert the new sample(s). Refer to the data system's online help for details.

Sequences and Throughput (111 vial model)

The HS optimizes throughput by checking the temperature and shaking parameters for the vials specified in the current sequence. When consecutive vials share the same temperature and shaking settings, the HS will examine the timing parameters for the samples, then calculate the best times to place each vial into the oven. This approach maximizes the number of vials equilibrating at a time.

Vials without the same temperature and shaking parameters will not be handled until the preceding samples leave the oven.

For more information, see the *Advanced Operation Guide*.

To Create a Sequence

To create a new sequence:

1 Press [**Seq**].

2 Select the method for the first vial range.

   a Press [**Mode/Type**].

   b Scroll to the desired method.

   c Pres [**Enter**] to select this method.

3 Enter the range of vial to prepare with this method.

   - For a simple range, for example vials 1 through 5, press [**1**][–][**5**][**Enter**].
   - For a single vial, enter the vial twice, for example, [**6**][–][**6**][**Enter**].

4 Enter the number of injections per vial, then press [**Enter**].

   The sequence is created. You can now store the sequence (see "To Save (Store) a Sequence"), run it (see "To Run a Series (Sequence) of Samples"), or continue to add additional lines as described below.

5 To enter another range of vials, press [**Insert/Append**]. When prompted, select where to add the new sequence line then press [**Enter**]:

   - **Append to sequence**: add the new line to the end of the sequence
   - **Insert into sequence**: add the new line before the current sequence line

6 Enter the method, vial range, and number of injections per vial for the new line.

7 Repeat step 5 and step 6 as needed.

You can now store the sequence (see "To Save (Store) a Sequence") or run it (see "To Run a Series (Sequence) of Samples").

To Save (Store) a Sequence

Storing a sequence saves the current sequence to nonvolatile memory. You do not have to be viewing the current sequence to save it.

If editing the sequence, save it as follows:.

1 Press [**Store**].

2 When prompted, press [**Sequence**].

3 When prompted, scroll to the desired sequence number to save.

4 Press [**Enter**].

5 When prompted, press [**On/Yes**] to save, or [**Off/No**] to return to the selection list.

Alternately:

1 Press [**Seq**].

2 Scroll to the sequence number to save.

3 Press [**Store**]. When prompted, press [**On/Yes**] to save, or [**Off/No**] to return to the selection list.

To Load a Sequence

To load a sequence:

1 Press [**Load**].

2 When prompted, press [**Seq**].

3 When prompted, select the sequence to load from the list, then press [**Enter**].

4 When prompted, press [**On/Yes**] to load the selected sequence, or [**Off/No**] to return to the selection list.

Alternately:

1 Press [**Seq**].

2 Scroll to the desired sequence.

3 Press [**Load**]. When prompted, press [**On/Yes**] to load the selected sequence, or [**Off/No**] to return to the selection list.

To Edit a Sequence

To edit a sequence:

1 If desired, load the sequence to edit.

2 Scroll through each line of the sequence.

- Change the method, vial range, and injections per vial for each line as desired.
- Delete the entire current line by pressing [**Delete**]. Press [**On/Yes**] to confirm, or [**Off/No**] to cancel.

To Delete a Sequence

To delete a sequence:

1 Press [**Delete**].

2 When prompted, press [**Seq**].

3 When prompted, scroll through the list of sequences to the desired sequence. Press [**Enter**] to select this sequence.

4 When prompted, press [**On/Yes**] to confirm and delete the sequence, or [**Off/No**] to cancel.

Alternately:

1 Press [**Seq**].

2 Scroll to the user-defined sequence to delete.

3 Press [**Delete**]. When prompted, press [**On/Yes**] to confirm, or [**Off/No**] to cancel.

Method Sequence Actions

When the HS encounters certain problems during a sequence, it has the ability to skip vials, continue anyway, or pause the sequence. The settings to control HS behaviors during sequence execution are called *sequence actions*. Sequence actions are part of the HS method, and therefore can change from sample to sample during sequence execution. Use **Sequence Actions** to specify what the HS should do when it encounters issues such as a vial size mismatch, missing vial, barcode error, and similar issues. For more details, see the *Advanced Operation Guide*.

8 Running Samples

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To run samples on the Agilent 7697A Headspace Sampler, create and run a sequence.

To Run a Series (Sequence) of Samples

1. Create or load the first method. (This prepares the HS temperatures and flows for the first sample.)
   - If needed, create the method. See "To Create a Method".
   - If already created, load the existing method. See "To Load a Method".
2. Prepare the sample vials.
3. Load the sample vials into the tray. See "To Load a Sample into the Tray (111 Vial Model)" or "To Load a Sample into the Tray (12 Vial Model)".
4. Create or load the sequence to run.
   - If needed, create the sequence. See "To Create a Sequence".
   - If already created, load the existing sequence. See "To Load a Sequence".
5. Press [**Start**].

   When the HS becomes ready (all temperatures and flows are at method setpoint), the HS will begin processing the samples.

   To view the progress of the vials, press [**Status**]. See also "To View Vial and Sequence Status".

To Pause a Running Sequence

Press [**Stop**] once.

- All vials currently in process will continue processing through injection (and return to the tray).
- No more vials will start processing.

To Stop a Running Sequence

To stop a running sequence, press [**Stop**] twice within 5 seconds. The current sample immediately aborts. All processing stops. All vials are returned to the tray (111 vial model). No further vials are processed.

To Run a Priority Sample During a Sequence

NOTE This feature is available only with the 111 vial model, and only when operating in standalone mode. If using an Agilent data system, edit the running sequence instead.

You may run only 3 priority samples during a sequence. You should avoid using sample vial positions 109–111 if using priority samples.

To run a priority samples during a sequence:

1 Check the status of the sequence. Find the vial last placed into the oven. (Press [**Status**]. See "To View Vial and Sequence Status".) The HS will use this vial's method to process the new sample.

2 Place the new sample vial in the next priority sample position. Start with priority sample position 1. See Figure 15.

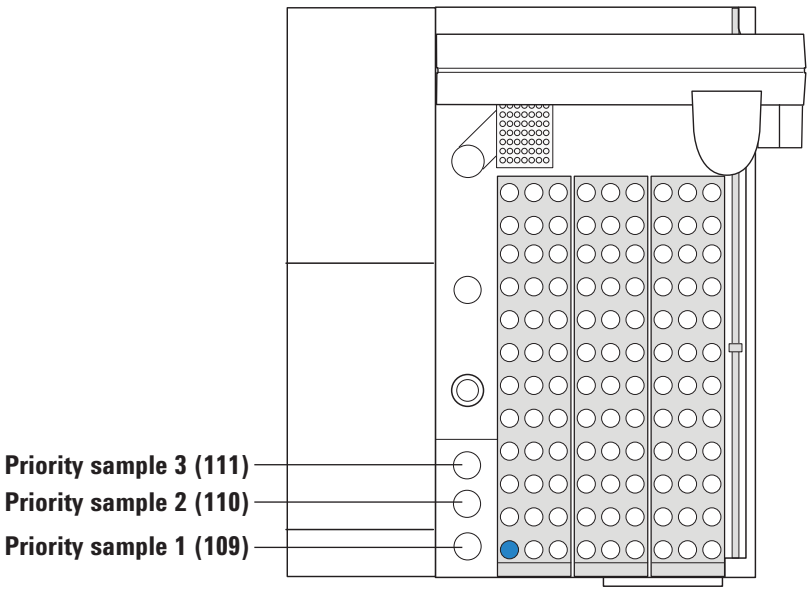
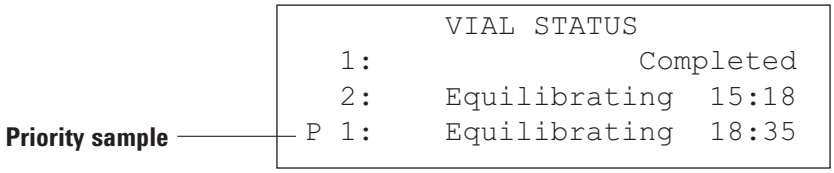


Figure 15 Priority sample locations

3 Press [**Priority Sample**].

4 The new vial will be processed using the method for the last vial placed into the oven.

- The tray will check the first priority sample position for a sample vial as soon as it is ready to load another sample into the oven.
- To track the vial status, press [**Status**] until the display reads **VIAL STATUS**. The vial number will show as **P *n***, where n is 1, 2, or 3.



To View Vial and Sequence Status

While a sequence is running, press [**Status**] to view the status of the sequence and the individual vials. The [**Status**] key cycles through displays for readiness state, temperatures and setpoints, and vial status.

Readiness status

This display shows the overall state of the headspace sampler, along with messages regarding system readiness. These messages can include warnings, faults, or other conditions that prevent the HS from becoming ready.

```
        STATUS - Ready
   Ready for start sequence
```

Setpoint status

This display lists the setpoints and current values for HS temperatures, flows, and pressures. To change the information shown in this list, see the "The Status Key".

```
              STATUS
Oven temp          100.0  100.0
Loop temp          110.0  110.0
Transfer line      115.0  115.0
```

Vial status

This display shows the status information for the next few vials listed in the sequence. The HS will display:

- Time until a vial operation is scheduled to occur.
- Current or next vial operation (for example, equilibrating, injecting, loading, and so forth).
- Completed.

```
          VIAL STATUS
   1:                Completed
   2:    Equilibrating  15:18
 P 1:    Equilibrating  18:35
```

Sequence Control with an Agilent Data System

In general, when using an Agilent data system, use the data system for starting, stopping, and pausing a sequence. However, an Agilent data system can deactivate the [**Start**] key.

Otherwise, pressing the [**Start**] and [**Stop**] keys on the HS provides the same functionality when in standalone control or data system control. Note that

9 Configuration

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This section describes how to configure the Agilent 7697A Headspace Sampler before use.

What is Configuration?

Most of the hardware used in the headspace sampler (HS) is known by the programs that control it. For example, the HS can detect whether or not a transfer line is installed, and it knows whether it has a 12 vial or 111 vial tray. However, some settings, such as gas types, sleep and wake modes, sample loop size, and similar are not detected. The HS stores your inputs for these items as settings. *Configuration* is the process of making these settings. The *current configuration* is the collection of these settings at any given time.

As part of installation, you configured the HS. However, you will need to reconfigure the HS whenever you want to do any of the following:

- Change a gas type
- Add an accessory, such as the optional EPC module or barcode reader
- Change the sample loop
- Change the carrier gas control mode
- Enable, disable, or change the resource conservation settings
- Change the standby flow (the purge flow used to keep the system clean when between sequences)

To Configure the Headspace Sampler

Press [**Config**] to access the configuration parameters. Table 6 lists the most common parameters, along with brief descriptions. Refer to the *Advanced Operation Guide* for complete information.

Table 6 Most common configuration parameters

Setting	Comments
Vial Gas type	Use the [**Mode/Type**] key to select the correct gas type.
Loop Volume (mL)	Enter the sample loop volume, in mL, and press [**Enter**].
Carrier Gas type	If available. Use the [**Mode/Type**] key to select the correct gas type.
Carrier	Use the [**Mode/Type**] key to select the desired carrier gas control mode. If the optional EPC module is not installed, this mode is automatically set to **GC Control**.
Standby flow	Input the desired flow used to purge the sampling probe between sequences, then press [**Enter**]. To turn the flow off completely (not recommended), press [**Off/No**].
Status	Scroll to each setpoint that you want to appear and press [**Enter**]. Select parameters in the order you wish them to appear. For example, select the parameter you want to appear at the top of the list first,
Clock	Select [**Clock**] to access of list of parameters that allow you to set the current time, current date, time zone, and desired date format. See the *Advanced Operation Guide*.
APG polarity	See *Installation and First Startup*.
Instrument Schedule	See Resource Conservation and the *Advanced Operation Guide*.

Resource Conservation

The Agilent 7697A Headspace Sampler provides the following features to conserve resources:

- Instrument schedule (sleep and wake methods): Load a method at a specified time during the day to reduce flows and temperatures, then load another method to restore them before operation.
- Control over standby purge flow.
- More advanced resource conservation features are available if using an Agilent data system. See the data system help.

For details, refer to the *Advanced Operation Guide*.

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