

Notices

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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

1 Introduction



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

2

The Operation Workflow

Routine Operation Workflow [14](#)

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.

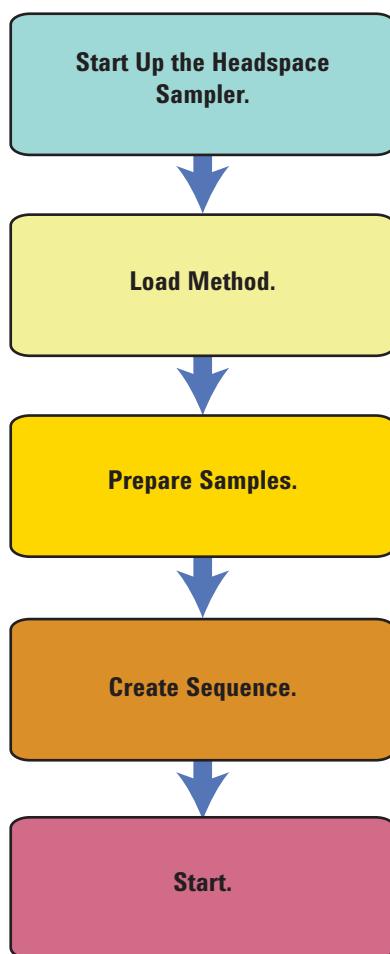


Figure 4 Routine headspace analysis workflow

Method Development Workflow

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

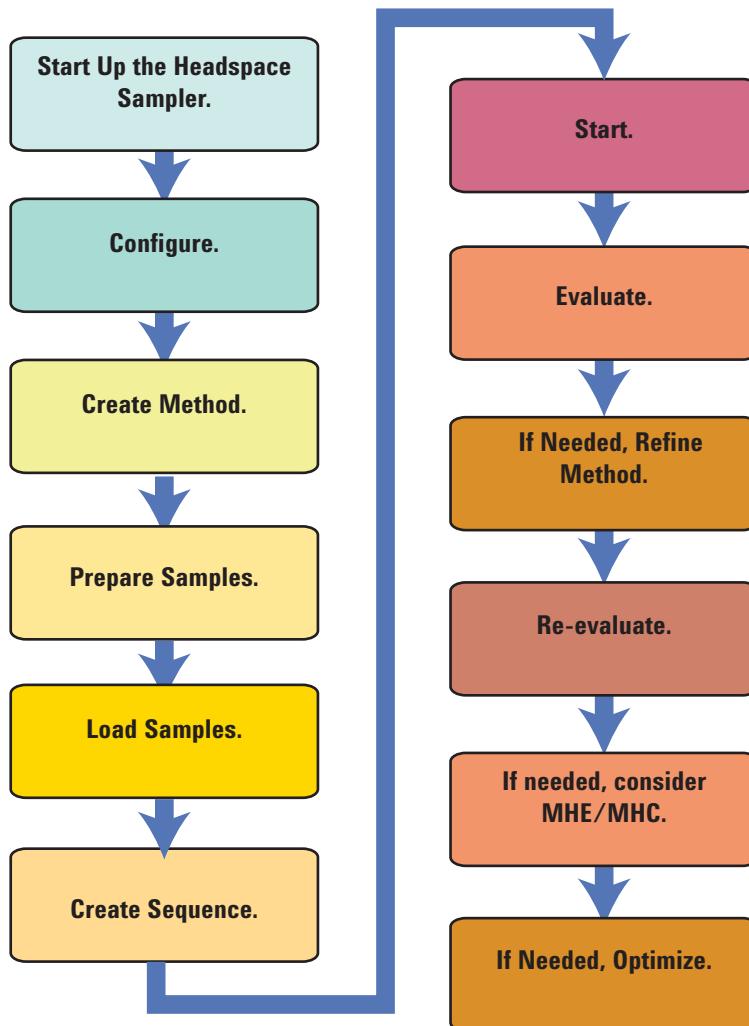


Figure 5 Workflow for method development

2 The Operation Workflow

3

The Keypad

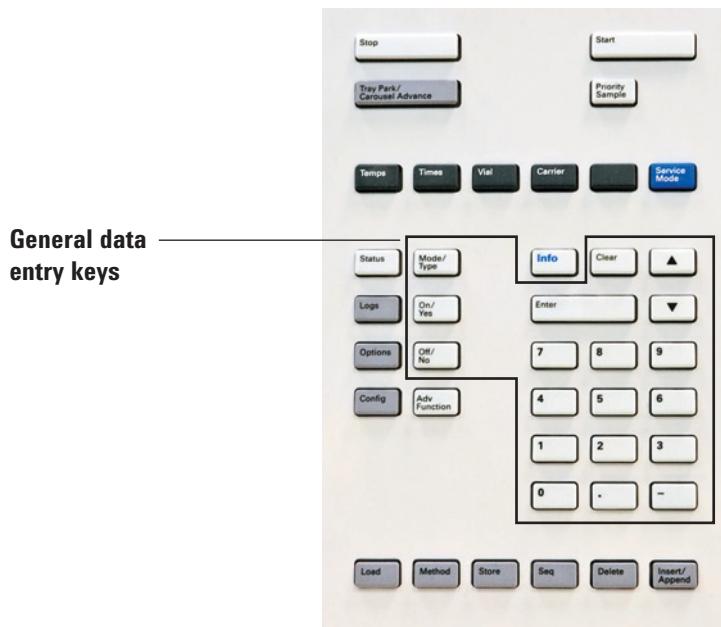
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This section describes the basic operation of the Agilent 7697A HS keypad. The keypad provides access to all instrument functionality. For additional details on keypad functionality, see the *Advanced Operation Guide*.



The General Data Entry Keys

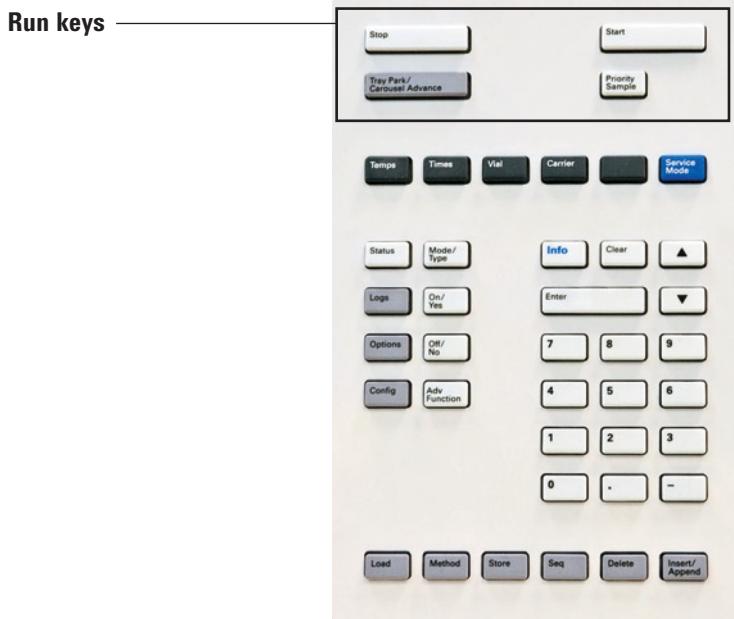
Use these keys to enter setpoints, make selections, and turn on or turn off HS components.



| | |
|---------------------|---|
| 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 | 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). |
| Off / No | |

The Run Keys

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



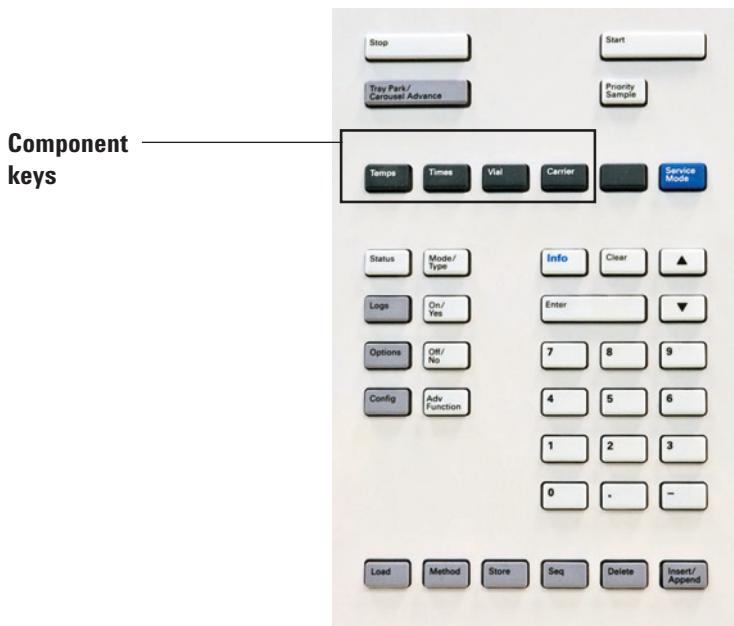
| | |
|-----------------------------------|---|
| Stop | During a sequence: |
| | <ul style="list-style-type: none"> Press once to pause the sequence. Current samples continue processing, but no new samples will be started. Press [Start] to resume. Press twice (within 5 seconds) to abort the sequence. 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 | <ul style="list-style-type: none"> 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**].



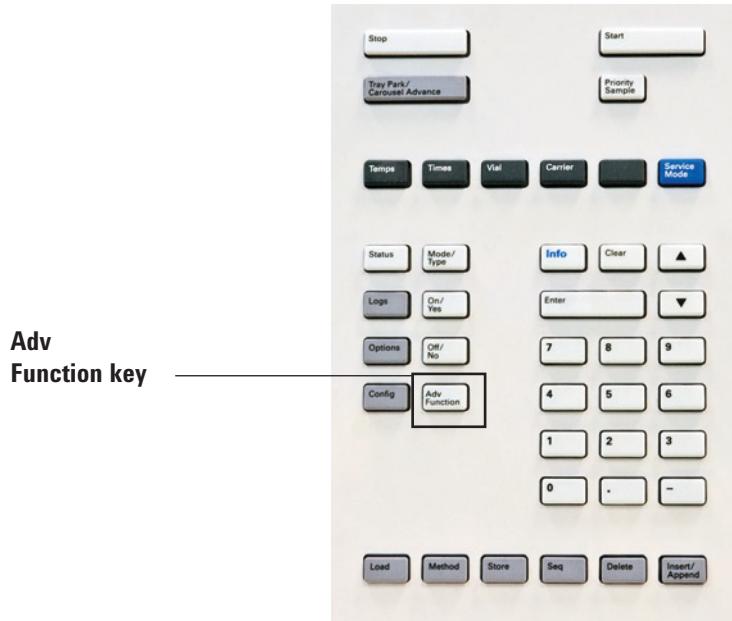
| | |
|----------------|--|
| Temps | Press to set temperatures for the vial oven, sample loop, and transfer line. |
| Times | Press to set the times for vial equilibration, injection duration, pressure equilibration, and the GC cycle time. |
| Vial | Press to set vial size, vial fill mode and parameters, 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.



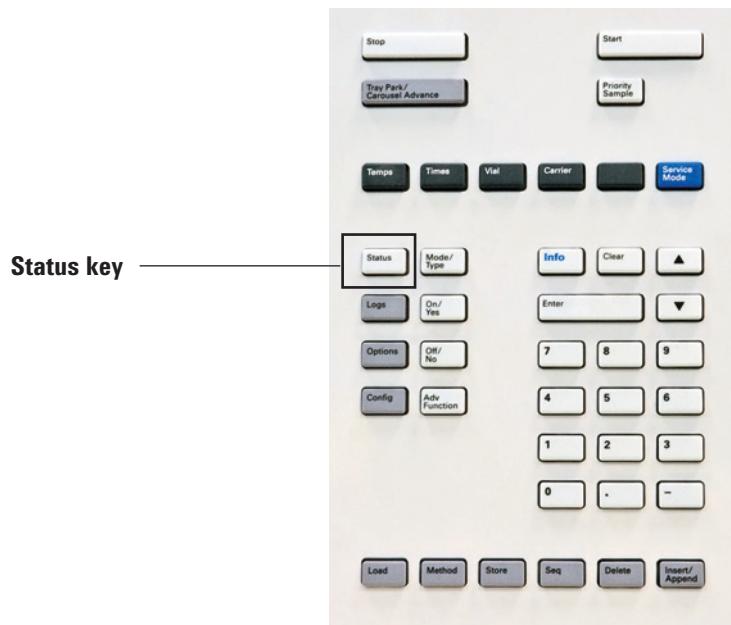
Adv Function

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

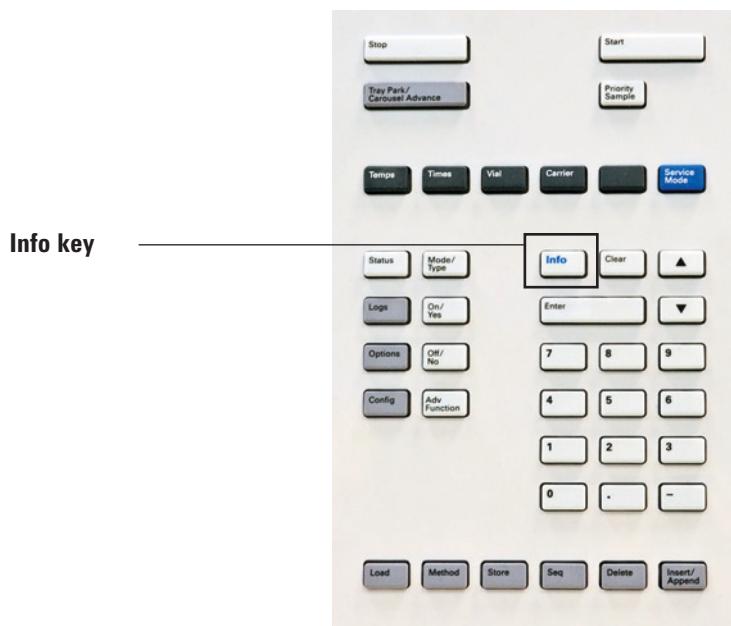
Use the **Status** key to learn about the current HS state.



| | |
|---------------|---|
| 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.*

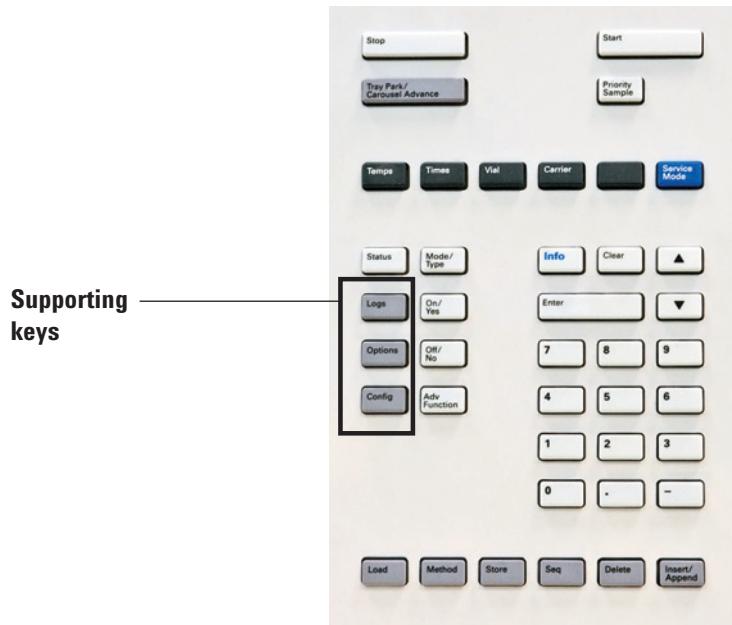


Info

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 <i>Advanced Operation Guide</i> 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.

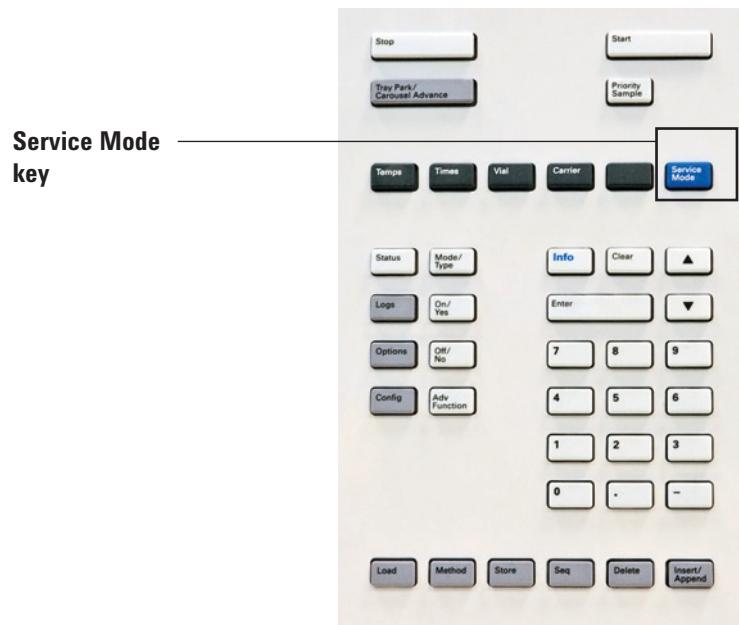


Method storage and automation keys

| | |
|----------------------|--|
| Load | Use to load and store methods and sequences on your HS. |
| Method | |
| Store | 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 ”. |
| Seq | |
| 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:

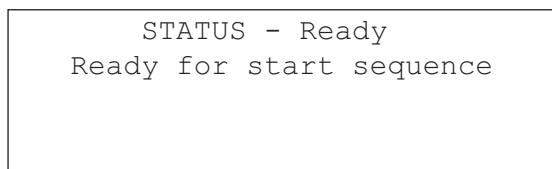
- Method parameters can be viewed, but not changed.
- You cannot load, edit, or save a method.
- You cannot load, edit, or save a sequence.
- You cannot change the instrument configuration or perform advanced functions.

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

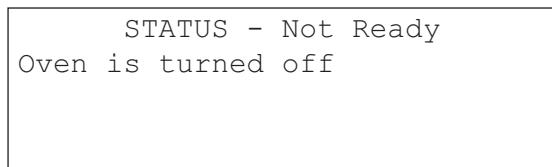
- To view sequence status, including time data, by selecting [**Status**].
- To view the method settings.
- To pause or abort a sequence by selecting [**Stop**].
- To find which computer is controlling the HS by pressing [**Options**] > **Communication**, then scrolling. The name of the computer controlling 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:



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.



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

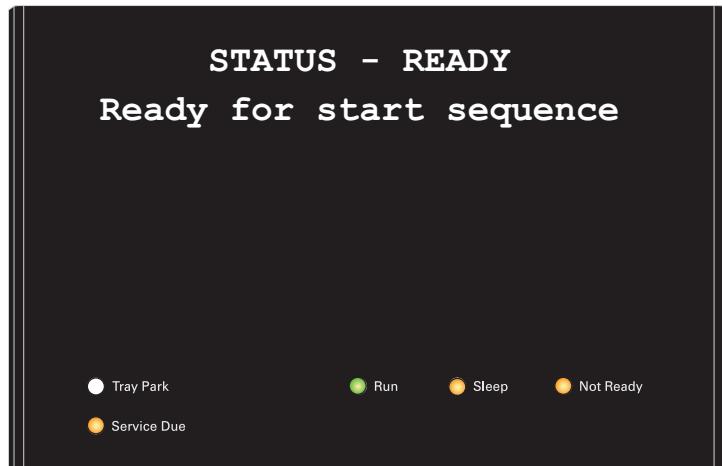
If a problem occurs, a status message appears. If the message indicates broken hardware, more information may be available.

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.

