Technical Overview

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Method Transfer from the Agilent G1888A Headspace Sampler to the Agilent 7697A and 8697 Headspace Samplers

Introduction

The Agilent 7697A and 8697 Headspace Samplers have onboard electronics pneumatics control which was added to improve the overall instrument performance. The 8697 Headspace Sampler has further integrated controls between the GC and the headspace, allowing full control through the GC Touchscreen. With these additions, several significant differences can be noted between the Agilent G1888A Headspace Sampler and the 7697A and 8697 Headspace Samplers. One benefit of incorporating the onboard pneumatics control is that a GC AUX module is no longer needed to control the vial pressurization pressure.

Figure 1. Agilent G1888A Headspace Sampler.

Figure 2. Agilent 7697A Headspace Sampler.

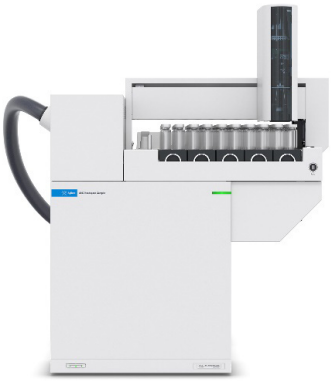

Figure 3. Agilent 8697 Headspace Sampler - XL Tray.

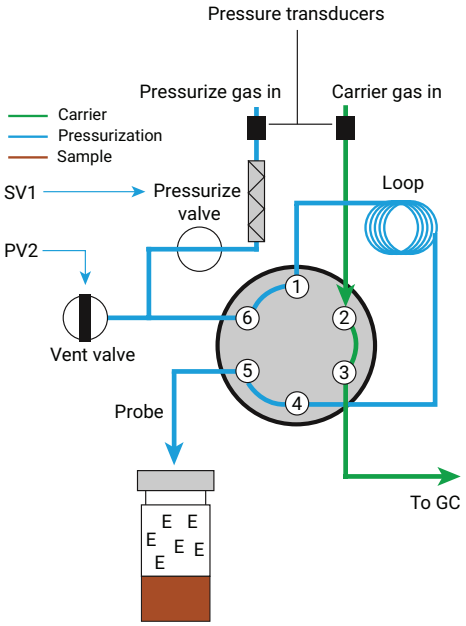

Figure 4. Agilent G1888A Headspace Sampler flow path in standby mode.

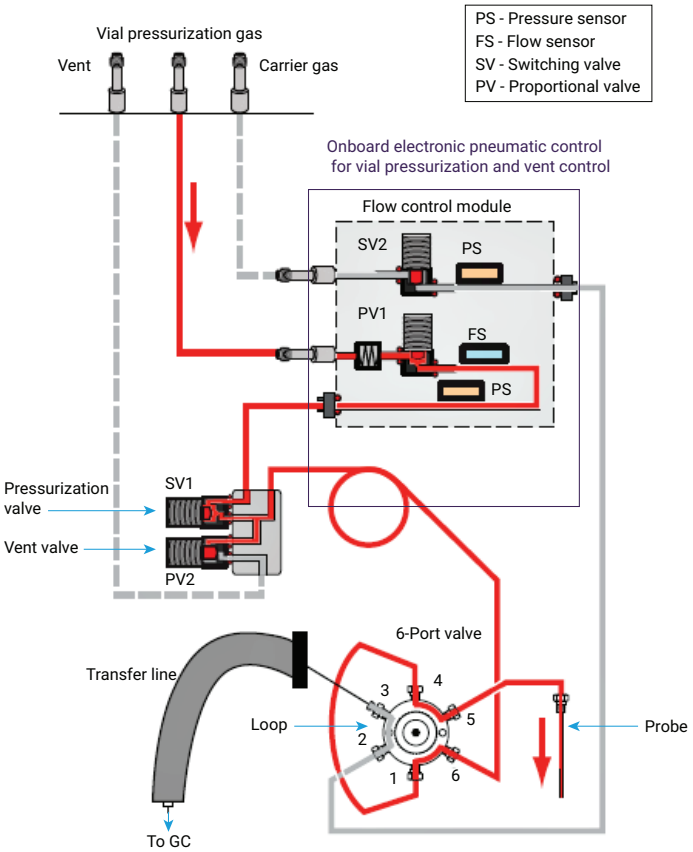

Figure 5. Agilent 7697A and 8697 Headspace Sampler flow path in standby mode (standard installation, without optional carrier PCM found in 7697A).

Loop fill behavior

The onboard pneumatics control allows the 7697A and 8697 Headspace Samplers to achieve active or passive backpressure control. The G1888A Headspace Sampler uses passive backpressure control to vent the vial contents through the sample loop down to ambient pressure. The default loop fill modes on the 7697A and 8697 Headspace Samplers are active pressure control. In this control mode, the final loop pressure is dropped to sweep sample from the headspace vial through the sample loop and out the vent before injection. The final pressure setpoint of the 7697A and 8697 Headspace Samplers is dependent on the initial vial pressure. Using the active backpressure control allows the 7697A and 8697 Headspace Samplers to achieve higher responses than using passive control. See Figure 6 and Table 1 for results which compare active and passive backpressure control.

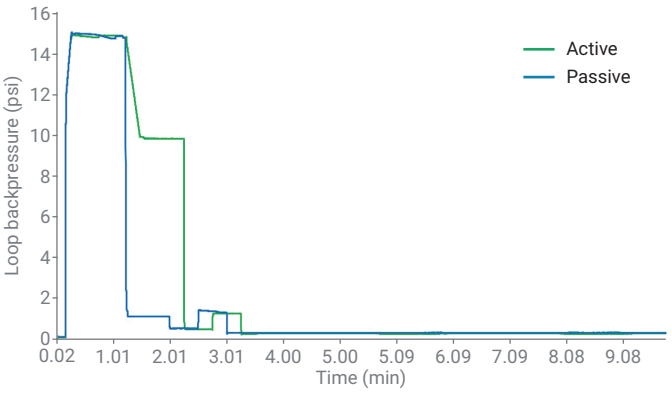

Figure 6. Active versus passive backpressure control.

Table 1. Comparison of active versus passive backpressure control.

Analyte	Active Area (pA*s)	Passive Area (pA*s)	Passive Area as % of Active Area
Ethanol	357.98	251.93	70.38
n-Propanol	701.04	458.82	65.45

Impact of active backpressure and modifications

Operating the 7697A or 8697 Headspace Sampler in the active backpressure control can occasionally cause a method developed on a G1888A Headspace Sampler to develop bad peak shape. If this happens, the final backpressure can be set to a lower value by changing the loop fill mode from Default to Custom, or a lower column loading can be achieved by increasing the split ratio, if applicable. Entering a final loop pressure of 0 psig allows the final vial pressure to be subject to fluctuations in ambient pressure, and is equivalent to the passive backpressure control found in the G1888A. A low pressure, for example 2 psig, could be used where a lower response is expected than under default conditions but the atmospheric pressure compensation would still be active.

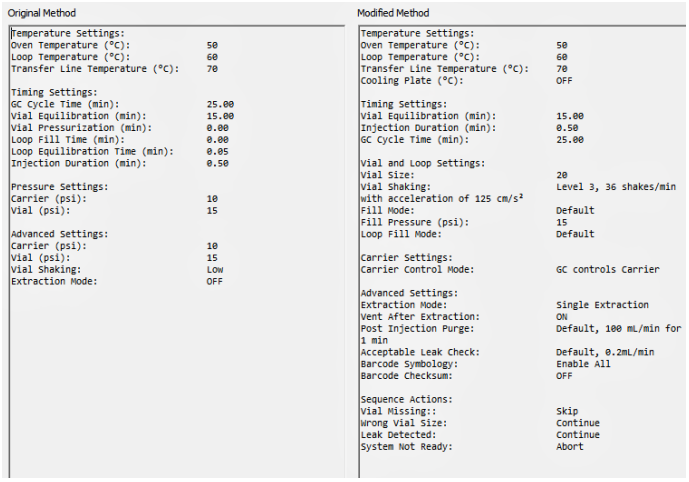

Figure 7. Agilent Method Development Viewer transitioning G1888A setpoints to 7697A and 8697 setpoints.

Table 2. Converting G1888A Advanced Functions to Agilent 7697A and 8697 features.

G1888A Advanced Function Number	Display	Equivalent Function
1	STABILIZE TIME	Not Included
2	PARAM. INCREMENT	Method > Miscellaneous (Method Development) on data system or 8890 Browser Interface
3	VIAL NO. AND LOC.	Data system or 8890 Browser Interface
4	KEYBOARD LOCK	Data system or 8890 Browser Interface
5	MANUAL OPERATION	Data system or 8890 Browser Interface
6	PRESSURE UNITS	System Settings > System Setup on 8890 Touchscreen
7	STORED METHOD	Data system or 8890 Browser Interface
8	MULTI. HS EXTR.	[Adv Function] > Extraction Mode on 7697 touchpad; Settings > Configuration > Headspace on 8890 Touchscreen for 8797 and 7697A
9	CHECK FOR READY	Miscellaneous > Sequence Actions on 8890 Touchscreen
10	RESET	Stop on 8890 Touchscreen
11	VALVE COUNT	Maintenance > Headspace > Early Maintenance Feedback (EMF) on 8890 Touchscreen
12	VIAL SIZE	Automatically detected
13	ZONE CALIBRATION	Temperatures > Oven, Loop, Transfer Line on 8890 Touchscreen
14	LAN CONFIGURATION	System Settings > Network on 8890 Touchscreen
15	MAC ADDRESS	System Settings > Network on 8890 Touchscreen
16	ENABLE RS-232	Not Included
17	PURGE VALVES	Venting and Purging > Purge Flow Mode, Purge Flow, Purge Time on 8890 Touchscreen
18	LEAK TEST	Diagnostics > Diagnostic Tests > Headspace > Restriction and Pressure Decay Test on 8890 Touchscreen
19	DIAGNOSTIC	Diagnostics > Diagnostic Tests on 8890 Touchscreen

Agilent Headspace Sampler Method Development Viewer

Using the Agilent 7697A and 8697 Headspace Sampler Method Development Viewer, located in the Headspace Driver, the user will notice some differences in parameters available for the G1888A Headspace Sampler versus the 7697A or 8697 Headspace Sampler.

Temperature settings

Both systems require acceptable oven, loop, and transfer line temperatures. When using the 7697A or 8697 samplers, the user should take into consideration the recommendations in the white paper "Thermal Zone Considerations for the Agilent 7697A Headspace Sampler" (publication number 5990-9892EN) when determining the appropriate temperature zone setpoints.

Timing settings

When converting from a G1888A Headspace Sampler to a 7697A or 8697 Headspace Sampler, timing settings such as Vial Equilibration, Injection Duration, and GC Cycle Time should not be affected. On the 7697A and 8697 Headspace Sampler, there is an additional parameter: pressure equilibration. Pressure equilibration is the time allotted for the vial to equilibrate at pressure during vial pressurization. The default value that can be used is 0.10 minutes. The equivalent parameter can be set in the software as hold time. Vial pressurization, loop fill, and loop equilibration times are now automatic, but can be set to custom values by the user.

Vial and loop settings

The 7697A and 8697 Headspace Samplers require specific vial and loop settings such as Vial Size, Fill Mode, Fill Pressure, and Loop Fill Mode. These setpoints determine how the system maintains active backpressure control.

The default fill mode is Fill at flow to pressure. The headspace uses a fixed flow rate to pressurize the vial to a specified level. In the default Loop Fill Mode, the headspace uses the initial vial pressure to calculate an optimum flow rate and final vial pressure for filling the sample loop. The user can learn more about the different modes available for each of these parameters in the operation manual for their model of headspace sampler.

The shaking speeds on the 7697A and 8697 Headspace Samplers take into account both acceleration and frequency. In terms of acceleration, the 7697A and 8697 Headspace Sampler shaking level 4 is equivalent to the G1888A Headspace Sampler high setting, and shaking level 3 is equivalent to the G1888A low setting.

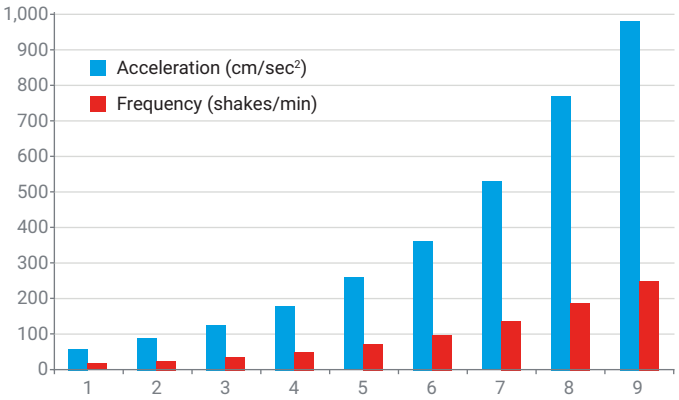

Figure 8. Graph of acceleration frequency for the nine levels of shaking available on the 7697A and 8697 Headspace Samplers.

Carrier settings

GC control of carrier gas is the default installation mode for the G1888A, 7697A, and 8697 Headspace Samplers. The G1888A and 7697A have optional configurations allowing carrier gas control by the headspace, but the 8697 does not. The G1888A can be set for Manual Pressure Control (MPC) to allow the HS to control the carrier gas pressure. The 7697A with the optional EPC Module also has the GC + HS Control mode, where the flow is controlled by the HS. Any method using HS control must have the flow included in flow rate calculations for method transfer to a 7697A or 8697.

For 8697 users, carrier control is through the GC only. If any prior configuration had the headspace providing carrier gas flow in addition to the GC inlet carrier gas flow, method setpoints will need to be updated to achieve the same inlet conditions with the single carrier gas flow from the GC.

Advanced settings

Three extraction modes are available on the 7697A and 8697 Headspace Samplers: single, multiple, and concentrated. Vent After Extraction provides the option to vent the residual pressure from the used sample vial after the extraction is performed. Between sample vials, the headspace sampler will purge the sample probe, sample loop, and vent. If experiencing carryover, the user could increase the purge flow or purge time by editing the post injection purge settings to sweep any residual sample vapors from the system. Using the onboard electronic pneumatic control, the instrument is able to detect leaks. The user can select the default value for Acceptable Leak Check or adjust according to their preference. These features replace the G1888A Advanced Functions – Multi. HS Extr., V2 Sequence Purge, and Leak Test, respectively.

The 7697A and 8697 Headspace Samplers have optional cooling tray and barcode reader accessories. The user can monitor the cooling tray temperature, and set the Barcode Symbology and Barcode Checksum. Reference the 7697A Headspace Sampler Advanced Operation Manual (G4556-90016EN) and the 8697 Headspace Samplers Operation Guide (G4511-90004EN) for more information on these advanced settings.

Sequence actions

The 7697A and 8697 Headspace Samplers give much more control over sequence actions in response to certain types of Headspace Sampler or GC errors (for example, Vial Missing, Wrong Vial Size, Leak Detected, or System Not Ready) that can occur when handling sample vials for a run or a sequence of runs. If one of these errors are detected, the user can control the headspace by setting the following actions: Wait (8697 only), Continue, Skip, Pause, or Abort. These settings replace and expand on the G1888A Advanced Function 9 – Check for Ready settings of No Check, Wait, and Abort.

For example, System Not Ready: when the Headspace Sampler becomes Ready, it checks if the GC is Ready. If the GC is not ready for a new injection, the Headspace Sampler follows the specified action. The default setting is Abort. For example, System Not Ready: when the Headspace Sampler becomes Ready, it checks if the GC is Ready. If the GC is not ready for a new injection, the Headspace Sampler follows the specified action. The default setting is Abort.

Guided troubleshooting

The G1888A's limited self-diagnostics using the Advanced Functions – Diagnostic and Leak Test features have been significantly improved and expanded in the 7697A and 8697 Headspace Samplers. The 7697A Headspace Sampler includes sensors for monitoring performance and troubleshooting errors. A restriction test, cross-port leak test, and pressure decay test are available through Service Mode, as well as error monitoring through the sequence log, event log, and status displays.

The integrated 8697 Headspace Sampler can run tests and display errors through the GC touchpad. Upon encountering an error, the GC gives a guided maintenance walkthrough for both the GC and Headspace based on a decision tree developed by Agilent's chromatography experts. Step by step visual guides for the following tasks are included:

- Install/remove a transfer line to the GC inlet
- Install/remove fused silica from the transfer line
- Replace the sample probe
- Replace the sample loop
- Replace the six port valve
- Replace the rotor
- Clean the six port valve and rotor
- Clean the oven
- Clean the sample tray assembly

Early Maintenance Feedback (EMF)

The 7697A Headspace Sampler allows users to set Early Maintenance Feedback (EMF) counters using Service Mode and log the maintenance events. These counters include:

- Transfer line
- Probe
- Vent valve
- 6-Port rotor
- 6-Port valve
- Sample loop
- Vent tubing
- Gripper pads
- Tray calibration

The 8697 Headspace Sampler uses the GC touchpad to set Early Maintenance Feedback (EMF) counters, as well as to view and download the logged maintenance events. These counters include:

- Gripper pads
- Headspace "On" time
- Headspace run count
- Probe
- Sample loop
- Six-port rotor
- Six-port valve
- Transfer line
- Tray calibration
- Vent tubing
- Vent valve
- Custom settings

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