

## Agilent 8697 Headspace Samplers



### Overview

Agilent 8697-XL tray and 8697 are the next generation of best-in-class headspace samplers from Agilent. Based on the industry-leading 8890 GC and 7697A headspace sampler, these headspace samplers have been designed to deliver unprecedented precision and performance.

Key to their performance is the use of advanced electronic pneumatic control (EPC), inert flow path, and high-performance temperature control of the oven, valve and loop, and transfer line. This leads to extremely precise area reproducibility, the basis for all chromatographic measurement.

As the first Agilent headspace samplers to feature integrated communication with the GC, and remote accessibility through the browser Interface, the 8697 headspace samplers give the freedom to manage workflows from a single interface and simplify productivity.

## Chromatographic performance

- Typical area repeatability <0.7% RSD
- Typical carryover <0.0001%

**Note:** Using either 8697-XL tray or 8697, 8890 GC with EPC (split), and Agilent data system for analysis of ethanol (RSD) and DMSO in water (carryover). Results may vary with other samplers and conditions. Conditions and parameters are listed on pages 5 and 6.

## Sample handling

- 8697-XL tray 120-vial capacity
  - Five removable 24-vial racks for in-rack vial capping (racks are resistant to common solvents used in gas chromatography)
  - Racks exchangeable during sequence for continuous operation
- 8697 48-vial capacity
  - Two removable 24-vial racks for in-rack vial capping (racks are resistant to common solvents used in gas chromatography)
  - Racks exchangeable during sequence for continuous operation
- Twelve-position air-bath oven for precise temperature control of every sample throughout its equilibration time
- Adaptive algorithmic sample overlapping to maximize throughput
- Vial shaker with adjustable frequency and acceleration parameters provides faster sample equilibration
- Integrated barcode reader available

## Sampling method

- Robust valve and loop headspace sampling system with standard full electronic pneumatics provides complete control of the sampling process (allows independent vial pressurization and GC column head pressures)
- Unrestricted GC column selection from 50 to 530  $\mu\text{m}$  regardless of sampling conditions
- Chemically inert sample flow path
- Fully automated purging of sample and vent paths between each analysis

## Sample vials

- Adaptor-free compatibility with headspace vials of 10 mL, 20 mL, and 22 mL sizes that meet the following specifications:
  - Screw or crimp top closure
  - Flat or rounded bottom style
  - Dimensions:

Size	Dimensions
10 mL	47.0 mm minimum height with closure
20 mL and 22 mL	79.0 mm maximum height with closure
All Sizes	22.40 to 23.10 mm width
- Unrestricted use of different vial sizes within a single sequence

## Modes of operation

- Single extraction mode with overlapping of up to 12 vials for maximized sample throughput while maintaining constant heating time for each vial
- Multiple headspace extraction (MHE) mode with up to 100 extractions per vial
- Multiple headspace concentration (MHC) mode with up to 100 extractions from a single vial followed by one GC start to maximize sensitivity

- Method development mode used to optimize headspace extraction by incrementing one of the following parameters: equilibration time, oven temperature, or vial shaking

## System control

- LED indicators for Ready, Not Ready, Error, Rack Present, and Tray Park
- Instrument control via GC touchscreen (8890, 8860, or Intuvo 9000 GCs)
- Control software interfaced via external LVDS connection from GC and available for integrated control via Agilent GC and MSD data systems (OpenLAB CDS 2.x, OpenLab CDS ChemStation, OpenLab CDS EZChrom, and MassHunter)
  - Headspace parameters are controlled via configuration and method dialogs
  - System actuals are displayed in conjunction with GC and GC/MS status
  - Headspace sequence status window displays individual sample information in graphical and detailed layouts
  - Event logging captures each headspace action and makes data available for reporting
- Enhanced control of instrument scheduling parameters
  - Tray diagrams for graphical display of sample status (available in select data systems)
  - "Wizards" for headspace method generation from:
    - Existing methods of either valve and loop or pressure transfer headspace sampling techniques
    - Sample specific information (solvent, boiling point)

## Thermal control

All temperature zones (oven, valve and loop, transfer line) have setpoint increments in 1 °C with 0.1 °C resolution for actual temperatures and can be set to off (uncontrolled).

- Oven: Off, Ambient +5 °C to 300 °C
- Valve and loop: Off, Ambient +5 °C to 300 °C
- Transfer line: Off, Ambient +5 °C to 300 °C

Optional cooling tray for 8697-XL tray

- Regulates sample temperature to set points between 4 °C to 60 °C
- Includes aluminum trays and temperature sensor
- Requires external recirculating chiller

## Pneumatic control

- Electronic pneumatic control (EPC) with the following specifications:
  - Compensation for barometric pressure and ambient temperature changes is standard
  - Pressure setpoints may be adjusted by increments of 0.001 psi, with typical control  $\pm 0.001$  for the range 0.000 to 75.000 psi
  - Flow setpoints may be adjusted by increments of 0.01 mL/min, with typical control  $\pm 0.01$  for the range 0.0 to 200 mL/min
  - User may select pressure units as psi, kPa, or bar
  - Pressure sensors:

Parameter	Value
Accuracy	< $\pm 2\%$ full scale
Repeatability	< $\pm 0.05$ psi
Temperature Coefficient	< $\pm 0.01$ psi/°C
Drift	< $\pm 0.1$ psi/6 months

- Flow sensors:

Parameter	Value
Accuracy	< $\pm 5\%$ depending on gas
Repeatability	< $\pm 0.35\%$ of setpoint
Temperature Coefficient	< $\pm 0.20$ mL/min (NTP*) per °C for He; < $\pm 0.05$ mL/min (NTP*) per °C for N <sub>2</sub>

- Vial pressurization is fully controlled by the included onboard EPC module
  - Gas settings selectable for helium and nitrogen
  - The following modes are available:

Mode	Description
Default	with user-settable vial pressure and the vial fill is algorithmically computed
Flow to Pressure	with user-settable vial fill flow and pressure allows gentle vial pressurization to minimize sample disturbance
Pressure	with user-settable vial pressure
Constant Volume	with user-settable volume of pressurization gas to add to the vial

- Loop fill is fully controlled by the included EPC module. The following modes are available:
  - Default where the loop fill is automatically computed
  - Custom where the fill rate (0.1 to 200.00 psi/min in 0.01 psi/min increments), final pressure (75.00 psi max), and equilibration time (0 to 999.99 min in 0.01 min increments) are user settable

- Carrier control options

- External source controlled by 8890, 8860, or Intuvo 9000 gas chromatograph (Compatible gas types: helium, nitrogen, hydrogen, and argon/methane (95%/5% mix))

## Timing control

- Vial equilibration time from 0 to 999.99 min in 0.01 min increments
- Injection duration from 0 to 999.99 min in 0.01 min increments
- GC cycle time from 0 to 999.99 min in 0.01 min increments
- Sample probe purge time from 0 to 999.99 min in 0.01 min increments

## Sample pathway

- Sampling probe is Agilent UltiMetal Plus deactivated stainless steel
- The standard 1 mL sample loop is Agilent UltiMetal Plus deactivated stainless steel; optional sample loops are available in 0.025 mL, 0.050 mL,

## Interfacing with the GC

GC Inlet Type	Connection Type	Comments
Split/Splitless (S/SL) Multimode (MMI) Volatiles Interface (VI)	Transfer line through GC inlet top	Standard configuration
Cool On-Column (CoC) Purged Packed (PP)	Transfer line through GC inlet septum	Optional configuration
S/SL or MMI with 8890, 8860, or Intuvo 9000 Transfer Line Interface Accessory	Direct connection to carrier gas stream via unique heated CFT assembly	Enables ALS tower and headspace sampler to be connected to a single GC inlet
None	GC column connected directly to headspace sampling valve	Optional configuration; Bypasses GC inlet completely; requires carrier gas supply from a GC

0.100 mL, 0.500 mL, 2 mL, 3 mL, and 5 mL sizes with Agilent UltiMetal Plus deactivation

- The transfer line heater assembly is 1 m in length and accommodates fused silica or metal capillary with 0.2 to 0.6 mm ID and 0.8 mm maximum OD

## Sample integrity

- Automatic vial leak checking ensures vials have been sealed correctly before sampling and requires no calibration or setup
- Post-injection sample probe purge with user-settable flow (0 to 200 mL/min) and time (0 to 999.99 min)
- Logging of movements, events, and errors for each vial
- Sequence actions give the user complete system control via logical operators (continue, skip, pause, wait, and abort) when any of the following occur: missing vials, wrong vial size, vial leak detected, and system not ready
- Optional barcode reader with support for checksums and the following formats:
  - 128
  - 3 of 9
  - matrix 2 of 5
  - standard 2 of 5
  - interleaved 2 of 5
  - UPC-A
  - EAN/JAN 13
  - EAN/JAN 8
  - UPC-E

## System integrity

- System leak check diagnostics for the complete flow path
- Counters, alarms, and log for tracking of routine maintenance items
- Maintenance procedures, diagnostics, and help available through Browser Interface
- Detailed power-on self-test with error reporting

## Environmental, health, and safety

- Resource conservation settings allow the user to reduce environmental impact
  - Instrument scheduling allows sleep and wake settings of time and instrument parameters
  - Gas saver settings
    - Between samples the sample probe purge is adjustable for both flow and time
    - Between sequences vial pressurization gas flow can be reduced
- Excess vial gases are safely depressurized via vent fitting on instrument and can be plumbed to traps or hoods as appropriate

## Communication

- External LVDS port for communication with Agilent GCs

## Environmental conditions

Parameter	Value
Operation	10 °C to 40 °C
Storage	-40 °C to 70 °C
Humidity	5% to 95% (noncondensing)
Power Requirements	
Line Voltage	100 to 240 VAC
Frequency	50/60 Hz
Power	852 VA maximum

## Safety and regulatory certification

- Canadian Standards Association (CSA) C22.2 No. 61010-1
- CSA/Nationally Recognized Test Laboratory (NRTL): UL 61010-1
- International Electrotechnical Commission (IEC): 61010-1, 61010-2-010, 61010-2-081
- EuroNorm (EN): 61010-1
- CISPR 11/EN 55011: Group 1 Class A
- IEC/EN 61326
- Designed and manufactured under a quality system registered to ISO 9001
- Declaration of Conformity available

## Dimensions

- 8697

Parameter	Value
Height	800 mm
Width	Footprint: 393 mm Maximum: 436 mm
Depth	Footprint: 561 mm Maximum: 642 mm
Weight	35 kg

- 8697-XL tray

Parameter	Value
Height	810 mm
Width	Footprint: 394 mm Maximum: 591 mm
Depth	Footprint: 565 mm Maximum: 641 mm
Weight	Std tray: 39 kg Cooler tray: 47 kg

## Conditions and parameters for RSD measurement

Sequence	
Vial Number	Vial Contents
1	Water (500 µL)
2	Ethanol in water (500 µL)
3	Ethanol in water (500 µL)
4	Ethanol in water (500 µL)
5	Ethanol in water (500 µL)
6	Water (500 µL)

Headspace Parameters	
<b>Oven Menu</b>	
Oven Temperature	60 °C
Loop Temperature	60 °C
Transfer Line Temperature	100 °C
<b>Time Menu</b>	
GC Cycle Time	5.00 min
Vial Equilibration Time	15.00 min
Press Equilibration Time	0.25 min
Inject Time	0.50 min
<b>Vial Menu</b>	
Fill Mode	Flow to Pressure
Fill Pressure	15.00 psi
Fill Flow	50.00 mL/min
Loop Fill Mode	Custom
Ramp Rate	20.00 psi/min
Final Pressure	10.00 psi
Final Hold Time	0.05 min
Vent After Extract	No
Vial Size	20 mL
<b>Sequence</b>	
Method	Current method
Vials	Vial range (for example, 1 to 6)
Injections Per Vial	1

Gas Chromatograph Parameters	
Inlet	Split/splitless
Liner	Agilent p/n 5181-8818 2 mm straight
Temperature	200 °C
Total Pressure	33.505 psi
Total Flow	75 mL/min
Septum Purge	3 mL/min
Split Mode	5:1
Gas Saver	20 mL/min at 3 min
Column	Agilent J&W DB-ALC2, 260 °C, 30 m × 320 µm, 1.2 µm (Agilent p/n 123-9234)
Constant Flow	12 mL/min
Average Velocity	111.39 cm/s
<b>Oven</b>	
Equilibration	1 min
Initial Temperature	35 °C
Hold Time	3.0 min
Detector	FID, 250 °C
Signals	FID, 50 Hz (0.004 min)

## Conditions and parameters for carryover measurement

Sequence	
Vial Number	Vial Contents
1	Blank vial
2	50% DMSO in water (1 $\mu$ L)
3	Blank vial

Headspace Parameters	
<b>Oven Menu</b>	
Oven Temperature	120 °C
Loop Temperature	160 °C
Transfer Line Temperature	170 °C
<b>Time Menu</b>	
GC Cycle Time	30.00 min
Vial Equilibration Time	20.00 min
Press Equilibration Time	0.10 min
Inject Time	0.50 min
<b>Vial Menu</b>	
Fill Mode	Flow to Pressure
Fill Pressure	15.00 psi
Fill Flow	50.00 mL/min
Loop Fill Mode	Custom
Ramp Rate	20.00 psi/min
Final Pressure	10.00 psi
Final Hold Time	0.05 min
Vent After Extract	No
Vial Size	20 mL
<b>Sequence</b>	
Method	Current method
Vials	Vial range (for example, 1 to 3)
Injections Per Vial	1

Gas Chromatograph Parameters	
Inlet	Split/splitless
Liner	Agilent p/n 5181-8818 2 mm straight
Temperature	250 °C
Total Pressure	34.101 psi
Total Flow	75 mL/min
Septum Purge	3 mL/min
Split Mode	5:1
Gas Saver	20 mL/min at 3 min
Column	Agilent J&W DB-ALC2, 260 °C, 30 m $\times$ 320 $\mu$ m, 1.2 $\mu$ m (Agilent p/n 123-9234)
Constant Flow	12 mL/min
Average Velocity	111.39 cm/s
<b>Oven</b>	
Equilibration	1 min
Initial Temperature	40 °C, hold for 2 min
Final Temperature	160 °C, hold for 2 min
Rate	20 °C/min
Detector	FID, 250 °C
Signals	FID, 50 Hz

[www.agilent.com/chem/8697](http://www.agilent.com/chem/8697)

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