

# Agilent 7697A GC Headspace Sampler



#### **Overview**

The Agilent 7697A headspace sampler is part of Agilent's line of gas chromatography products. Based on the architecture of the market-leading GC and 7693A ALS line of products, the 7697A has been designed to exceed the expectations of the most demanding laboratories.

The 7697A headspace sampler is designed to conserve electricity, gas, and other valuable resources, with features such as automatic shutdown and wake-up.

What's more, the headspace control software simplifies the process of reducing gas flow, so you can more efficiently manage your supply of essential gas.

#### Models

- Agilent 7697A headspace sampler: 12 vials with single position oven for sequential sample heating
- Agilent 7697A headspace sampler with tray: 111 vials with 12-position oven for optimized sample overlapping

# **Chromatographic performance\***

#### Typical area repeatability

- 7697A <1.5% RSD
- 7697A with tray <1% RSD

\*Using 7697A, 7890 GC with EPC (split), and Agilent data system for analysis of ethanol. Results may vary with other samples and conditions. Conditions and parameters are listed on page 7.

# Sample handling

#### Agilent 7697A

- 12-vial capacity
- Single-position, solid aluminum vial oven

#### Agilent 7697A with tray

- 111-vial total capacity
  - 108 vials in three removable 36-vial racks suitable for inrack vial capping (racks are resistant to common solvents used in gas chromatography)
  - Three vials in priority sample positions
  - Racks exchangeable during sequence for continuous operation
- Available 108-vial cooling plate
- Twelve-position air-bath vial 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
- Available vial cooling plate (5 °C to ambient range, depending on environmental conditions, as described in Table 4) with temperature sensor allows critical samples to remain cold until the time of analysis (requires recirculating chiller)

#### 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  $\mu$ 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:
  - 10 mL size (47.0 mm minimum height with closure)
  - 20 mL and 22 mL sizes (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

- Standalone operation
  - Control and monitoring by full-function, chemical-resistant keypad
  - Multiline display with English and Chinese language settings
  - LED indicators for Not Ready, Run, Sleep, Service Due, and Tray Park
  - Setpoints and actual monitoring for all parameters
  - Store up to 32 user-defined headspace methods (plus five preset methods)
  - Store up to 9 user-defined sequences
- Control software interfaced via LAN connection and available for integrated control via Agilent GC and MSD data systems (OpenLab CDS ChemStation, OpenLab CDS EZChrom, GC ChemStation, and MSD ChemStation)
  - 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) (Table 1).

Table 1. Valid setpoints

	Agilent 7697A	Agilent 7697A with Tray	
Oven	Off, 35 °C to 210 °C	Off, Ambient +5 °C to 300 °C	
Valve and loop	Off, 35 °C to 210 °C	Off, Ambient +5 °C to 300 °C	
Transfer line	Off, 35 °C to 250 °C	Off, Ambient +5 °C to 300 °C	

#### 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:
  - Accuracy: < ± 2% full scale
  - Repeatability: < ± 0.05 psi
  - Temperature coefficient: < ± 0.01 psi/°C
  - Drift: < ± 0.1 psi/6 months

- · Flow sensors:
- Accuracy: < ± 5% depending on gas
- Repeatability: < ± 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:
  - *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 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 such as a gas chromatograph
  - Compatible gas types: nitrogen, helium, hydrogen, and argon/methane (95%/5% mix)
  - Onboard carrier EPC module (optional)
  - Compatible gas types: nitrogen, helium, hydrogen, and argon/methane (95%/5% mix)

- Operation modes: Constant Pressure, Constant Flow, Ramp Pressure, and Ramp Flow
- Configuration modes: Direct Control and Additive Flow
- Supports a maximum of 10 GC oven ramps and 5 pneumatic ramps

### **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 UltiMetal Plus deactivated stainless steel
- The standard 1 mL sample loop is UltiMetal Plus deactivated stainless steel; optional sample loops are available in 0.025 mL, 0.050 mL, 0.100 mL, 0.500 mL, 2 mL, 3 mL, and 5 mL sizes with UltiMetal Plus deactivation
- The transfer line heater assembly is 1 m in length and accommodates the following tubing types:
  - Fused silica capillary of 0.25 mm, 0.32 mm, and 0.53 mm ID (maximum OD of 0.67 mm)
  - Metal capillary of 0.53 mm ID (such as Agilent UltiMetal or ProSteel) with maximum OD of 0.67 mm

## Interfacing with GC

See Table 2.

Table 2. Interfacing with 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 7890 or 8890 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	Bypasses GC inlet completely; requires carrier gas supply from either the optional carrier EPC module or a GC	

## 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 (0−200 mL/min) and time (0−999.99 min)
- Logging of movements, events, and errors for each vial
- Sequence actions gives the user complete system control via logical operators (continue, skip, pause, 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 fonts:

• 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
- Instrument utilities software included, which enables firmware updates and diagnostics as well as providing all instrument manuals via LAN connection
- 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 both vial pressurization gas and optional carrier supply gas flows 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

- LAN

- Remote start/stop

#### **Environmental conditions**

- Operation: 10 °C to 40 °C

- Storage: -40 °C to 70 °C

Humidity: 5% to 95% (noncondensing)

Power requirements

 Line voltage: 120/200/220/230/240 ±10% supported by configurable transformer

• Frequency: 50/60 Hz

• Power: 850 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

# Other specifications

**Table 3.** Agilent 7697A dimensions

	Height	Width	Depth	Weight (avg)	
Agilent 7697A					
Footprint	606 mm (23.9")	509 mm (20.0")	636 mm (25.0")	84 lb	
Maximum		629 mm (24.8")	680 mm (26.8")		
Agilent 7697A with tray					
Footprint	800 mm (31.5")	509 mm (20.0")	636 mm (25.0")	101 lb	
Maximum		665 mm (26.2")	689 mm (27.1")		

**Table 4.** 7697A cooling plate specs

Vial cooling to 10 °C within the following conditions		Vial cooling to 5 °C within the following conditions		
Ambient Temp (°C)	Maximum Relative Humidity (%)	Ambient Temp (°C)	Maximum Relative Humidity (%)	
37	55	37	35	
30	5	30	50	
23	75	23	65	
19	80	19	75	

# **Conditions and parameters**

Order of vial analysis

Vial numberVial contents1500 μL water2Ethanol in water3Ethanol in water4Ethanol in water5Ethanol in water6500 μL water

#### Headspace parameters

Oven menu

Oven temperature: 60 °C Loop temperature: 60 °C Transfer line temp: 100 °C

Time menu

GC cycle time: 5.00 min
Vial equilibration time: 15.00 min
Press equib time: 0.25 min
Inject time: 0.50 min

Vial menu

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 extraction: No
Vial size: 20 mL

Carrier menu

Carrier: External Supply

Sequence

Method: Current method

Vials: Vial range (for example, 1–6)

Injections per vial: 1

Gas chromatograph parameters

Inlet: Split/splitless

(Agilent p/n: G3452-64000)

Liner: Agilent p/n 5190-2295 (without glass wool)

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

Oven

Equilibration: 1 min
Initial temperature: 35 °C
Hold time: 3.0 min

Detector: FID (Agilent p/n G3462-64000),

250 °C

Signals: FID, 50 Hz (0.004 min)

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