Pharmaceutical

Residual Solvents - Headspace

DPS Instruments, Inc.

www.dps-instruments.com

There is no way around it, residual process solvents are commonly detected in pharmaceutical products. Consequently, many government agencies have made it mandatory to measure the residual solvents for the release testing of all active pharmaceutical ingredients. Analyses are also routinely performed on process intermediates used during the drug synthesis. The help with these regulations the DPS Residual Solvents GC Analyzers use a built-in Headspace Concentrator to fully automate the sampling and analysis and a sensitive FID detector for low level detection of these residual solvents. Liquid samples can also be analyzed in these GC Analyzers by direct injection. The Series 600 GC is for analyses in the lab, or use the Portable Companion 2 GC Systems for analyses right where the samples are taken. The fully integrated Residual Solvents GC Analyzer Systems are small and lightweight and all DPS systems are modular for expandability, upgrades, and easy service.

Series 600 GC

Available Configurations Include:

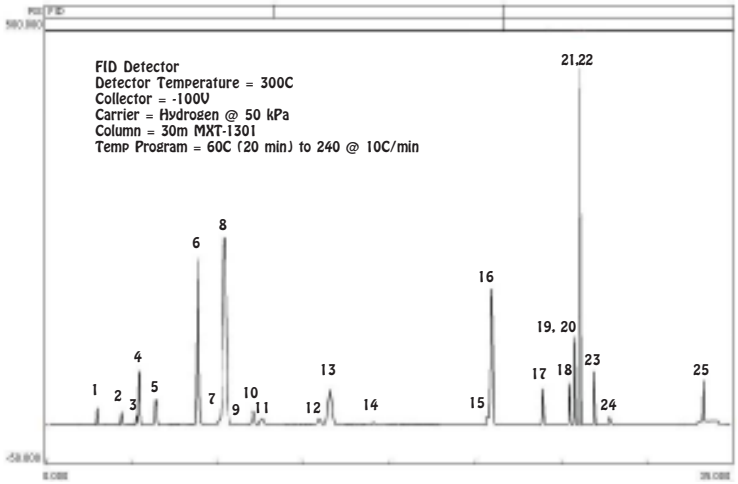
600-C-145 - Series 600 Residual Solvents GC Analyzer (FID, Headspace Concentrator, 30m Column)

500-C2-145 - Companion 2 Portable Residual Solvents GC Analyzer (FID, Headspace Concentrator, 30m Column)

Residual Solvents Analysis

Companion 2 Portable GC
(with Headspace Concentrator)

Peak	Component
1	Methanol
2	1,1-Dichloroethane
3	Acetonitrile
4	Methylene Chloride
5	Hexane
6	cis-1,2,-Dichloroethane
7	Chloroform
8	1,1,1-Trichloroethane
9	Carbon Tetrachloride
10	Benzene
11	1,1-Dichloroethane
12	1,1,2-Trichloroethene
13	Methylcyclohexane
14	1,4-Dioxane
15	Pyridine
16	Toluene
17	2-Hexanone
18	Chlorobenzene
19	DMF
20	Ethylbenzene
21	m-Xylene
22	p-Xylene
23	O-Xylene
24	N,N-Dimethylacetamide
25	1,2,3,4-Tetrahydronapt

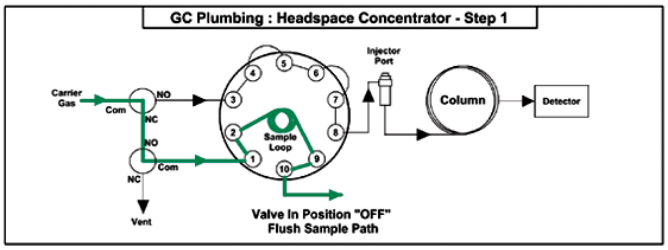


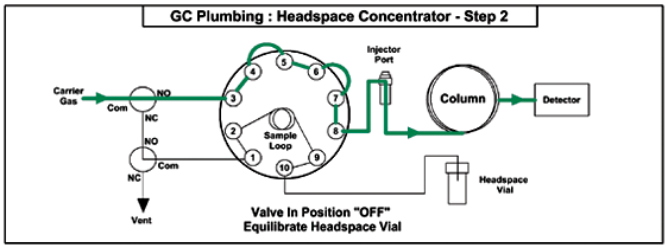
DPS Companion 2 Residual Solvents Layout

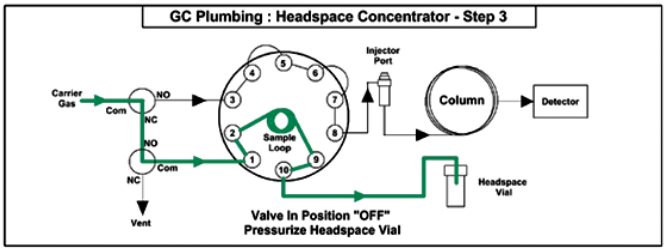
Plumbing Diagram

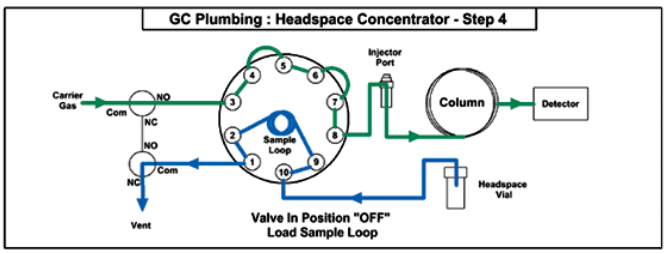
Headspace Concentrator - The Headspace Concentrator for Series 600's and Companion GC's are built right in to provide the shortest possible sample path. The Sample Vial is heated and then consistently Pressurized before loading the Sample Loop. A fixed Sample Loop ensures reproducible sampling and the sample lines are Flushed between analyses to limit any cross over contamination. The entire sequence of the Headspace Concentrator is automated through the Timeline sequence of the DPS GC Control Software for the analysis of one sample at a time, while two other samples are heated and allowed to equilibrate.

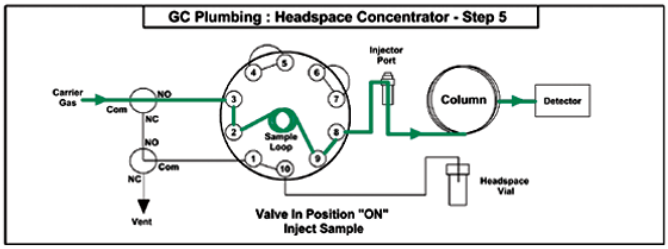
Plumbing Diagram - In the 1st Step the carrier gas is diverted to Flush out the Sample Lines between runs. During the 2nd Step the carrier gas flows to the analytical column and the Headspace Vial is heated with the Vial Heater and allowed to equilibrate. The Sample Probe is then inserted into the Headspace Vial. During the 3rd Step the Headspace Vial is pressurized for a few seconds. In the 4th Step the sample is loaded onto the Sample Loop by releasing the pressure in the headspace vial. In the 5th Step the Sample Valve is rotated to the ON position and the carrier gas sweeps the components from the Sample Loop onto the analytical column.











Headspace Plumbing Diagram

Results, Data & Conncetivity

Results: In this Headspace plumbing configuration the sample is placed inside a vial and then heated. The sample can be raw materials, tablets, pellets, or packaging material. The detector will respond with the same peak areas for the same concentration no matter which source the sample comes from.

Data and Connectivity: The built-in computer is used to collect and store the data. Data can also be copied to a USB Stick to transfer to another computer. Data can be transferred from the built-in computer to another computer on the LAN through the Ethernet port using standard Windows protocols. Or, we can use a USB cable to connect the GC to the remote computer where the data can be collected and stored on that hard drive.

GC Control Software

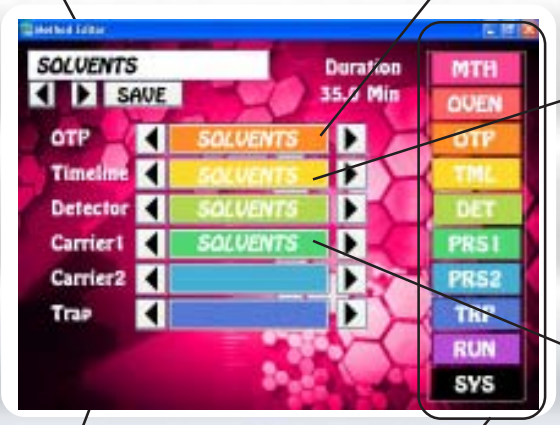
Easy to learn and master using a Graphical User Interface (GUI) and Color Touch Screen.

Editors let you customize the files associated with the GC Method.

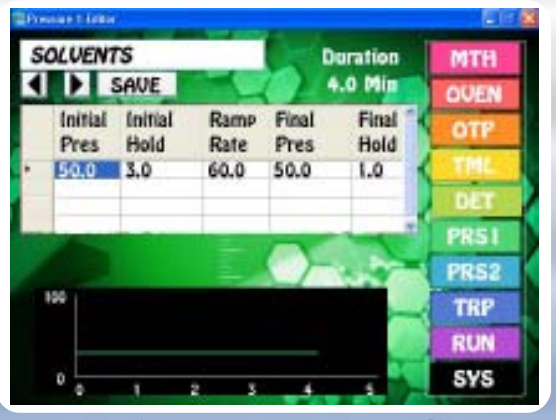
Oven Temp Program Editor

Timeline Editor

Method Name



File Selection Arrows



Carrier Pressure 1 Editor

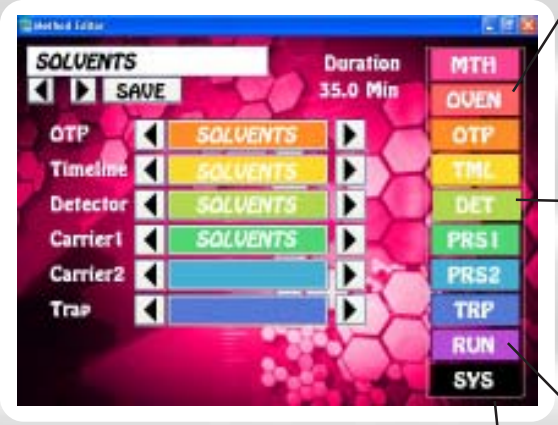
Navigation Buttons to Quickly jump from one screen to another. Most pages are one button away!

Keyboard to Enter Filenames

Number Pad for entering Values

GC Status pages display the parameters in the method, both graphically and as text and values.

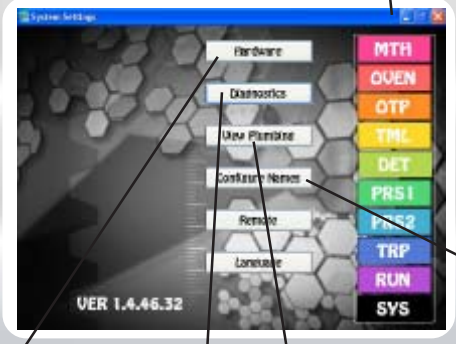
Oven Status

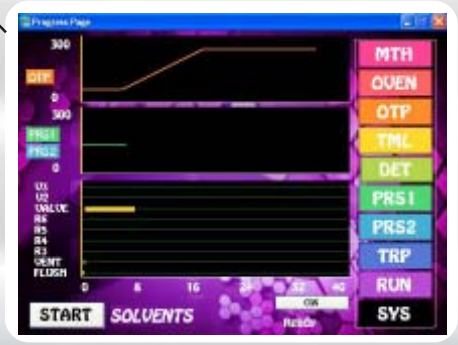


Method Editor

Detector Status

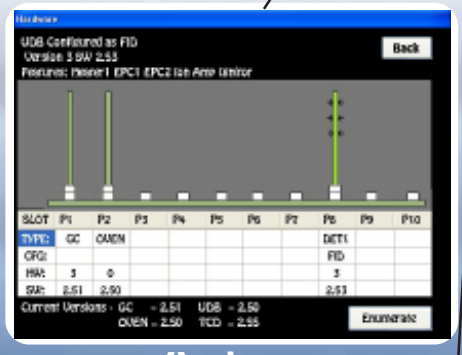
System Status



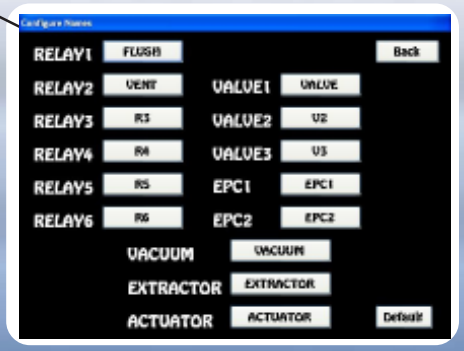


Run Status

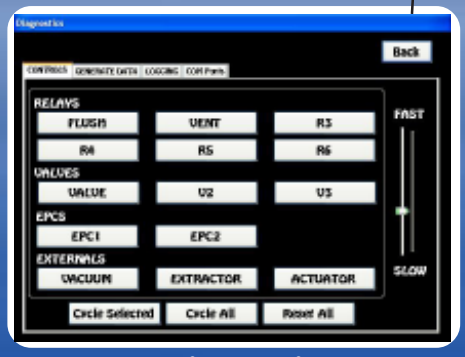
System status pages display the health and viability of the GC instrument.



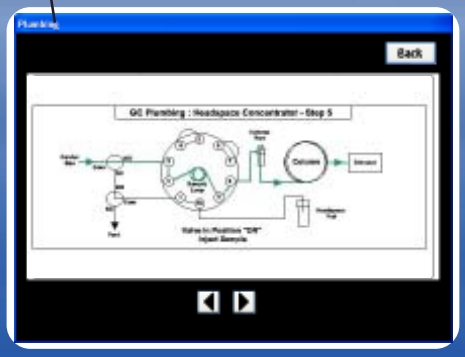
Hardware



Configure Names



Diagnostics



Plumbing

Residual Solvents GC Specifications:

Electronics Module:
- Enter and store GC Methods via Color Touch Screen
- Actual and set-point display of all GC parameters
- Safety Limits on all user entered parameters
- Oven Temperature Programs (OTP) with Multiple Ramps
- Pressure Programs for Carrier Gases with Multiple Ramps
- Timeline for sequencing Relays and Valve
- Detector Control of all Parameters on one page
- Electronic Pressure Controllers (EPC's):
  Atmospheric Pressure & Temperature Compensation
  EPC Pressure Control with 0.1 kPa set-point resolution
- Plug and Play GC Control, Oven, and Detector Board
- Microprocessor Controlled
- Proprietary Digital Signal Processing
- Digital Signal Outputs for each Detector
- Universal voltage input (85 – 240 Vac) with line filter and breaker.

Detector:
FID – Flamelonization Detector (1ng detection limit, dependent on sample loop size)

- 400 °C Temperature Limit with 0.1 °C set-point resolution
- 24-bit Digital Outputs for the detector via USB
- EPC Pressure Control with 0.1 kPa set-point resolution

Columns:
30m Capillary

Results:
Automatically calibration corrected and reported

Series 600 Oven Module:
- Ambient to 400°C Column Oven
- Up to 100 °C per/min Oven Ramp
- Fast Cooldown 300 °C to 50 °C in 3.5 min
- 1000 watt total Heater Elements
- Temperature Ramps with 0.1 °C set-point resolution
- 23 x 23 x 20 cm area for Glass, SS, or Capillary Columns

Companion 2 Oven Module:
- Ambient to 325 °C Column Oven
- Up to 80 °C per/min Oven Ramp
- Fast Cooldown 300 °C to 50 °C < 4 min
- 200 watt Heater Element
- Temperature Ramps with 0.1 °C set-point resolution
- 12.5 x 10.5 x 12.5 cm area for Packed, or Capillary Columns
- 14 amps at 48 Vdc total power consumption

Built-In Accessories:
- Sample Valve - Electronically Actuated
- Heated Valve Oven
- Headspace Concentrator
- Flow Control Solenoids

Injector:
- Heated On-column Injector
- Multiple Pressure Ramps with 0.1 kPa set-point resolution

Data Communications:
- Bi-directional communication with popular Data System

Network Connectivity:
- Enterprise Compatible Network GC running Windows XPe
- Ethernet Connection using Windows Network Protocol
- On Board ETX Computer for GC Control and Data Acquisition
- Remote Control of GC and Data Acquisition over LAN

Lab Quality Analyses in the Field,
"It Goes with you Anywhere!"

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