

# Agilent 7667A Mini Thermal Desorber

Installation and Operation



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# **Getting Started**

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This section will help the user to understand the basic use and working principle of the 7667A Mini Thermal Desorber as well as the use range of two different types of instruments.



# 7667A Mini Thermal Desorber Introduction

The 7667A Mini Thermal Desorber (herein-after referred to as "Mini TD") is a One-Stage thermal desorber that is characterized by the small size and low power consumption.

The 7667A includes two versions: **The Entry version**(G4370A) and **the Enhanced version** (G4370M).

The entry version (G4370A) does not include the sampling pump and is suitable for offline sampling. It is mainly used in conjunction with the 7820A GC.

The Enhanced version (G4370M) comes with a sampling pump. It supports online automatic acquisition of gas samples and is mainly used in conjuction with the 5975T LTM GC/MSD.

The above versions can also be used in conjuction with other Agilent Gas-phase products through a remote control interface.

# Be familiar with the Mini Thermal Desorber



**Figure 1** Front View of the Entry version Mini Thermal Desorber (G4370A)





# Working Principle of the Mini Thermal Desorber (Mini TD)

The Mini TD has three operation modes: online sampling mode, desorption mode and conditioning mode.

**Online Sampling Mode: Available on the G4370M only.** It integrates all of processes from sampling to post run process after desorbing, which consists leak detection, online sampling, dry purging, desorbing, injection and cleaning process. In sampling process, the sampling pump included in the desorber extracts the sample gas into the internal tubing of the Mini TD (G4370M), and some components of the sample are absorbed on the desorption tube. For the entry version G4370A, the sampling is indicated status "offline".

**Desorption Mode:** In the desorbing mode, the Desorber will only execute desorbing related process, which consists leak detection, dry purging, desorbing, injection and cleaning process. The online sampling process will not be included.

**Conditioning Mode:** Conditioning is not a part of the sample desorbing process. After a long time running or multiple runs, the performance of the sorbent tube might be degraded. The moisture and residual contaminants in the desorber and the desorption tube need to be removed by a long time baking, so that they restore to a better state.





**Leak detection.** For the entry version G4370A, it occurs before dry purging.

Figure 4 Schematic Diagram of Leak Detection, G4370A

**Dry purging stage:** The dry purging stage starts after the completion of leak detection (if the leak detection is turned on). In this process, the carrier gas will be purged into the tube, then the air and the moisture in the tube will be purged out, so that the desorption tube is in a relatively clean state before the desorption. Please specify the parameters as needed.



Figure 5 Schematic Diagram of Dry purging, G4370A

**Desorbing & Injection stage;** The user can select to separate or combine the desorbing process and injection process by enabling/disabling Start Injection Time through software interface or local control panel. When these two processes are separated: the Desorber will heat the desorption tube when the carrier flow is shut-off. Under this situation, the target components will be desorbed and sealed within the tube and wait for injection. In the following Injection process, the carrier flow from GC can take the desorbed components into the GC inlet more quickly and easily. As one of the important benefits, the peak width of components with lower boiling point will be increased. When these two processes are combined: the tube will start to be heated according to the predefined ramping rate, at the same time, the injection phase is started. The target components will be purged into GC inlet immediately as soon as they are desorbed. This desorbing method will have a positive influence on reducing carry-over for target components with higher boiling point.



Figure 6 Schematic Diagram of Desorbing, G4370A



Figure 7 Schematic Diagram of Injection, G4370A

**Cleaning stage:** After the desorption stage, there may still be some residual sample remaining in the desorption tube. To ensure that the residue does not affect the subsequent desorption, the desorption tube needs to be cleaned with high temperature. Compared with the desorption stage, the cleaning stage should last longer (two to four minutes) and should be carried out with greater flow (150 to 200 mL/min) and at a very high temperature (320 °C or so, not exceeding the maximum temperature that can be used for the desorption tube. See the insturctions of the desorption tube for specific information).



Figure 8 Schematic Diagram of Cleaning, G4370A

Idle Stage.



Figure 9 Schematic Diagram of Idle Stage, G4370A



#### Work Flow of G4370M



**Leak detection.** For the Enhanced version G4370M, it occurs before sampling.



Figure 11 Schematic Diagram of Leak Detection, G4370M

**Sampling stage:** In sampling, the integrated pump will pump the gas sample into the tube and the target compounds will be absorbed by the tube. The sampling parameters are used to control the sampling duration and the speed of sampling. These parameters should be set based on the properties and quantity of the sample to be processed.



Figure 12 Schematic Diagram of Sampling Stage, G4370M

**Dry purging stage:** It starts after the completion of sampling and leak detection (if the leak detection is turned on). In this process, the carrier gas will be purged into the tube, then the air and the moisture in the tube will be purged out, so that the desorption tube is in a relatively clean state before the desorption. Please specify the parameters as needed.



Figure 13 Schematic Diagram of Dry purging, G4370M

Desorbing & Injection stage: The user can select to separate or combine the desorbing process and injection process by enabling/disabling Start Injection Time through software interface or local control panel. When these two processes are separated: the Desorber will heat the desorption tube when the carrier flow is shut-off. Under this situation, the target components will be desorbed and sealed within the tube and wait for injection. In the following Injection process, the carrier flow from GC can take the desorbed components into the GC inlet more quickly and easily. As one of the important benefits, the peak width of components with lower boiling point will be dramatically decreased, that means the peak height & sensitivity will be increased. When these two processes are combined: the tube will start to be heated according to the predefined ramping rate, at the same time, the injection phase is started. The target components will be purged into GC inlet immediately as soon as they are desorbed. This desorbing method will have a positive influence on reducing carry-over for target components with higher boiling point.



Figure 14 Schematic Diagram of desorbing, G4370M



Figure 15 Schematic Diagram of Injection, G4370M

**Installation and Operation** 

**Cleaning stage:** After the desorption stage, there may still be some residual sample remaining in the desorption tube. To ensure that the residue does not affect the subsequent desorption, the desorption tube needs to be cleaned with high temperature. Compared with the desorption stage, the cleaning stage should last longer (two to four minutes) and should be carried out with greater flow (150 to 200 mL/min) and at a very high temperature (320 °C or so, not exceeding the maximum temperature that can be used for the desorption tube. See more specific information in the instruction of the desorption tube).



Figure 16 Schematic Diagram of Cleaning, G4370M



Figure 17 Schematic Diagram of Idle Stage, G4370M

**Sample overlap mode: The G4370M** supports **the sequence run.** Under normal circumstances, the sequence is executed in order, i.e. the next desorption does not start until the present run is completely finished and both the Mini TD and the GC have been restored to the ready and idle states. In the sample overlap mode, the Mini TD begins to prepare for the next sample desorption while the current GC analysisis still in progress. *When the GC cycle time is longer than that of the Mini TD, the sample overlap mode can greatly improve the efficiency in the use of the desorber*.



# **Installation and Operation**

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The installation process of the 7667A depends on the purchased system components and the type of instrument used in conjunction with the Mini TD. Follow the steps related to the GC and the Mini TD in this section.



# **Preparation Before Installation**

# Parts Required for Installation

#### Table 1Shipping Kit for G4370A

Description	Part Number	Quantity
Stainless Steel Tube Assembly	G4370-60000	1 EA
Xfer Line Support Assembly	G3504-60620	1 EA
O-Ring 0.364-in-ID	0905-1819	1 PK
PTFE Filter Disks 6.3mm Marks TD PK 10	MKI-U-DISK3	1 PK
Syringe 10uL FN bevel Tip	5190-1483	1 EA
Torx Screwdriver T10 Size	51820-3466	1 EA
Wrench-Double open-end 3/16*1/4 –in-Size	8710-2697	1 EA
Tubing Cutter	G4350-20120	1 EA
Union SS 1/16 inch Tubing	0100-0124	1 EA
Cap,1/16 inch Stainless Steel	0100-0050	1 EA
Cap Carrier Tube	G1544-20150	1 EA
Stainless Nut 1/16 inch	0100-0053	1 EA
1/16inch Frrule set SST	0100-1490	1 EA
Tube fixer Tool	G4372-20048	1 EA
Catch Ferrule	G4372-20049	1 EA
O-Ring 0.239-in-ID	0905-1820	2 PK
Septum Retainer Nut for Transfer Line	G3452-60835	1 EA

### Table 2 Shipping Kit for G4370M

Description	Part Number	Quantity
O-Ring 0.239-in-ID	0905-1820	2 PK
O-Ring 0.364-in-ID	0905-1819	1 PK
PTFE Filter Disks 6.3mm Marks TD PK 10	MKI-U-DISK3	1 PK
Syringe 10uL FN bevel Tip	5190-1483	1 EA
Torx Screwdriver T10 Size	51820-3466	1 EA
Wrench-Double open-end 3/16*1/4 –in-Size	8710-2697	1 EA
SCREW-MACHINE ASSEMBLY M4*12MM	0515-0382	2 EA
Injection Needle ASS	G4372-60015	1 EA
Installation Pole Assy	G4370-60010	2 EA
Tube fixer Tool	G4372-20048	1 EA
Catch Ferrule	G4372-20049	1 EA

Description	Part Number	Quantity		
1/8" ODx250cm Cu Tubing Coil Assembly	G1530-61100	1 EA		
Ethyl Acetate 10 ml Amber Ampule	G4370-85002	1 EA		
7667 power Adaptor	0950-5534	1 EA		
RS-232 Cable for ALS	G4370-61207	1 EA		
APG Remote Cable	G4370-61208	1 EA		
Utility DVD	G4600-64006	1 EA		

 Table 3
 Other supplied accessories



7667A Power Adaptor (P/N 0950-5534)



RS-232 Cable for ALS (P/N G4370-61207)



APG Remote Cable (P/N G4370-61208)





PTFE Filter Disks (MKI-U-DISK3)

#### Installation parts for G4370A



Xfer Line Support Assembly (P/N G3504-60620)

#### Installation parts for G4370M

Installation Pole (G4370-20456) In the accessory of 7667A Mini TD Base Assembly for 5975T (G4368-64000)

Installation Pole Assy (G4370-60020) In the accessory of 7667A Mini TD Base Assembly for 5975T (G4368-64000)





Foot (G4370-20600) In the accessory of 7667A Mini TD Base Assembly for 5975T (G4368-64000)



75t Base Assy (G4370-60650) In the accessory of 7667A Mini TD Base Assembly for 5975T (G4368-64000)



Injection Needle ASS (G4372-60015)

### **Tools Required for Installation**

- T-10 Screw driver (P/N 5182-3466)
- 1/4-3/16 Open-end wrench (P/N 8710-2679)
- Tubing Cutter (P/N G4350-20120)
- Precision tubing cutter (for G4370A, P/N 5190-1442, prepared by CE)
- T-20 Screw driver (for G4370M, P/N 5182-3465, prepared by customers or FSEs)
- Column cutting wafer (for G4370A, P/N 5181-7487, prepared by customers or FSEs)
- 7/16 wrench (Prepared by customers or FSEs)
- 5/16 wrench (for G4370A, prepared by customers or FSEs)

### Site Preparation and Confirmation

Verify and ensure that the installation site has been ready in accordance with the requirements of the Site Preparation manual. The appropriate benches, power supply and sockets, gas supply, traps and the necessary tools have been ready.

# **Upgrade GC Firmware (Optional)**

The Mini TD requires that the revision of the 7820A GC firmware is A.01.12.004 or later, and the revision of the 5975T LTM GC/MSD firmware is A.03.04.004 or later. If the firmware revision the user used is earlier than the above revisions, please update the firmware to the latest version (the user may download the latest firmware version from Agilent website (www.agilent.com/chem) or contact your local Agilent sale representitives).

# Install the G4370A

# **Prepare the Instrument**

This procedure describes how to prepare the Agilent **7820A GC** for the installation of the G4370A.

# WARNING The 7820A GC inlet may be hot and cause burns. Cool the inlet to the ambient temperature before starting work.

- 1. Set the temperature of the GC's Inlet, the Detector and the Oven to the **room temperature**.
- 2. Close all gas supplies.
- 3. Remove the front mounting post of the autosampler and injector.
- 4. Locate the inlet carrier gas line. The inlet carrier gas line is a 1/16inch stainless steel tube that runs from the inlet EPC module to the inlet, usually beside the inlet carrier cover on the oven top. For split/splitless inlet, there is also a stainless steel septum purge vent line. The septum purge vent line is larger in diameter, and runs to the septum purge vent on the EPC module.



5. Using the precision tubing cutter (5190-1442), cut the carrier gas line leading into the inlet at approximately 3 to 5 cm from the septum head.

# **CAUTION** Cut the tubing as square as possible. Be careful to avoid kinks in the tubing. Avoid deforming the tubing. The cut should not be beveled or angled.

6. Plug the inlet carrier line at the septum head using the cap assembly (P/N 0100-0050) provided in the ship kit.



7. Install the male half of a 1/16- inch union (P/N 0100-0124) onto the open end of the carrier gas tubing coming from the inlet EPC module.

# NOTEWhen completed, you should be able to reassemble the original<br/>carrier flow path by removing the cap from the plug, then<br/>connecting the two ends of the tubing.

- 8. Use the other half of the 1/16- inch Swagelok union to connect the carrier gas tubing from the EPC module to the end of Cap Carrier Tube (G1544-20150).
- 9. Use the 1/16 inch Swagelok nut (P/N 0100-0053) and ferrule (P/N 0100-1490) to connect the other end of Cap Carrier Tube (G1544-20150) to the 1/16 inch carrier gas interface of the G4370A.
- 10. Check for leaks in the front inlet. Please refer to the **«7820A GC Troubleshooting ».**

# Install the Stainless Steel Tube Seamless in the Transfer Line

1. Cool the G4370A heating zone to a room temperature. Remove three screws as shown in the figure and remove the valve box cover and insulation cotton.



2. Unlock an approximately 1-meter long Stainless Steel Tube Seamless (G4370-20039). Gently pass the pipeline from the end close to the six-port valve through the transfer line till it comes out 6 cm or so from near the other end.



3. Use the 1/4 open-ended wrench to remove the Fitting-internal Reducer 1/16 to 1/32-in (PN 0100-2594) on valve port 1 (see below picture), save it for future use.



Valve port 1 with the Fitting-internal Reducer plugged 4. Assemble the Stainless Steel Tube Seamless according to the figure.



5. Install the Stainless Steel Tube Seamless to valve port 1. Tighten it with hand and then with the 1/4-3/16 open-ended wrench.



- 6. Pull out the Stainless Steel Tube Seamless from the inlet end of the transfer line and make the Stainless Steel Tube Seamless retain an appropriate length at the valve port end.
- 7. Use a column cutter (P/N 5181-7487) to trim the Stainless Steel Tube Seamless at GC inlet end, and make it 60-65 mm out the transfer line.



## Connect the Transfer Line to the 7820A GC Split/Splitless

#### Inlet

## CAUTION

# Wear clean lint-free gloves to prevent the parts from being stained by dust and skin secretions.

- 1. Put the G4370A on the left side of the 7820A GC.
- 2. Replace Septum Retainer Nut for Transfer Line (G3452-60835).
- 3. Put the bracket in the transfer line assembly on the front inlet carrier gas cover. The two positioning holes in the bracket align to the mounting positions of the front injector and calibrating pin respectively. See the figure below.



**Figure 18** Installation position of the bracket. The figure shows the inlet carrier gas cover of the 7820A

- 4. Locate the knurled thumbscrew (in the transfer line support assembly) provided in the Mini TD's Shipping kit.
- 5. Insert the knurled thumbscrew into the mounting position of the the front injector and tighten with hand.
- 6. Insert the tip of the transfer line into the inlet.
- 7. The transfer line should be fixed after installed.

8. Install the Latch/clamp onto the bracket. Adjust the position of the clip so that it supports the transfer line at the highest possible position above the GC, as shown in Figure 20.



Figure 19 Transfer line installed onto the inlet of the 7820A

## **Connect the Cables and Gas Lines**

- 1. Conncet 7667A power adapter cable (P/N 0950-5534) into the power socket of the **G4370A**.
- Connect the RS-232 cable for ALS (G4370-61207) to the Serial Port of the G4370A and the ALS control module of the 7820A GC (Front port).
- 3. Connect the Gas Line according to Figure 20.



Figure 20 Cable and Gas Line interface of G4370A



For G4370A, the carrier gas supply presure of the Mini TD must be larger than 60 psi, less than 85 psi.

# **Restore the 7820A GC to the Operable Conditions**

- 1. Turn on the gas supply.
- 2. Restore the inlet and the oven temperature to the operable conditions.

# Install the G4370M

# **Prepare the Instrument**

This procedure describes how to prepare the Agilent **5975T LTM GC/MSD** for the installation of the **G4370M**.

WARNING	The 5975T LTM GC/MSD inlet may be hot and cause burns.
	Cool the inlet to the ambient temperature before starting
	work.

# CAUTION Do not install the G4370M on the mounting post of the autosampler, otherwise, the Mini TD will be damaged. Remove the mounting post of the autosampler.

- 1. Cool the inlet and the oven to room temperature.
- 2. Close all gas supplies.
- 3. If the 5975T LTM GC/MSD is equipped with an autosampler, remove the autosampler and its mounting post.

## Install the G4370M on the 5975T LTM GC/MSD

1. Put the Foot (P/N: G4370-20600) on the bottom of the G4370M, and fix 4 screws with red marked by using T20 screw driver.



2. Insert the Installation Pole Assy into the injector (ALS) miounting position and tighten it with hand.



 Open the side cover, put the bottom bracket assembly (PN: G4370-60650) on the inlet of the 5975T and follow the steps 1-2 as shown below.



**Figure 21** Positions of fixed screws This figure should be replaced by that with G4370M installed

4. Put the **G4370M** on the bracket and tighten the four fixing screws (P/N 0515-1269) on both sides with T20 screw driver.



Figure 22 Fixed screws on the Mini TD

- 2. Insert Installation Pole into the Installation Pole Assy position and tighten it with hand.
- 1. Fix the bracket assembly with M4\*12\*2 screws.

# **Connect the Cables and Gas Lines**

- 1. Connect the 7667A power adapter cable (P/N 0950-5534) into the power socket of the **G4370M**.
- Connect the RS232 Auto Injector Cables to the RS 232 interface of the G4370M and the 5975T ALS control module respectively.
- 3. Connect the Gas Line according to Figure 23.



Figure 23 Cable and Gas Line interface of G4370M



For G4370m, the carrier gas supply presure of the Mini TD must be larger than 20 psi above Inlet pressure used in GC method. The dirver gas pressure is about 50 Psi.
CAUTION	When connecting the Desorber with sample gas for
	sampling, the length and inner diameter of the sampling
	tubing will affect the maximal sampling flow rate. Agilent
	will recommend use tubing with $1/8"$ I.D. and length shorter
	than 10 meters. Otherwise, the sampling flow rate may not
	meet Agilent published specification.
CAUTION	If the sample gas is pressurized, please make sure the sample gas
	is pressure regulated at a stable pressure. Otherwise, it will have
	negative influence on repeatability of sampling

#### Restore the 5975T LTM GC/MSD System to the Operable

#### Conditions

- 1. Open the right door of the **G4370M**.
- 2. Rotate the locking handle to the vertical position, unlock the injection slider, and push the injection slider to low injection position.



- 3. Rotate the locking handle back to the locked position.
- 4. Close the side door. The installation of the G4370M is completed.
- 5. Turn on the gas supply.
- 6. Restore the 5975T LTM GC/MSD inlet and the oven temperature to the operable conditions.

# **Replace the Desorption Tube**

### Install the Desorption Tube

1. Open the top cover of the desorber, loosen the triangular nut counter-clockwise, and pull out the desorption tube assembly.



- 2. Loosen the desorption tube locking nut about 30 degree.
- 3. Take out the desorption tube sealing O-ring (0905-1014) and Filter ( MKI-U-DISK3) from the shipping kit.



- 4. Install the desorption tube according to the order in the figure;
  - Check whether the Filter is already in tube handle and then rotate the desorption tube locking nut but not completely tighten it.
  - Set the sealing O-ring around the front end of the desorption tube.
  - Insert the front end of the desorption tube into the locking nut. Tighten the locking nut with hands or Tube Fixer Tool ( P/N G4372-20048).



1



2



3



5. Insert the tube handle with the desorption tube into the desorber and tighten the triangle nut with hand.

#### **Installation and Operation**

### **Remove the Desorption Tube**

WARNING Be careful! Tube temperature may be hot enough to cause burns. Please cool its temperature to room temperature, and then wear heat-resistant gloves during operating.

- 1. Open the top cover of the desorber, rotate the triangular nut counter-clockwise to loosen, and pull the desorption tube assembly.
- 2. Loosen the desorption tube locking nut about 30 degree and pull out the desorption tube.



# **Connection with Other Agilent Gas-phase Products**



7667A Back Panel



Agilent GC Back Panel



APG Remote Cable (P/N G4370-61208)

# **7667A Control Panel Operation**

# Power Switch

#### **Power Switch and Status Indicators**

Figure 24 Schematic Diagram of the Control Panel

The current state of the Mini TD is indicated with the status indicators;

- Green indicates running.
- Yellow indicates that the Mini TD is in the Not Ready state (see the figure).
- Red indicates there are errors or potential dangers occurred in the Mini TD.
- Status indicators off indicates the ready state.

#### **Run Key**

This key is used for the pre-preparation before the sampling desorption, to run the method and stop running.



**Installation and Operation** 

Bron Dun	Press this key to start the ppreparation before				
Prep Run	Injection or Desorption/Injection .				
Stort	Press this key to start Injection or Desorption				
Start	/Injection process.				
Stor	Press this key to terminate the running				
Stop	method.				

## Menu Key, Cancel Key and Dial



Menu	Press to the Main Menu.			
Cancel	Press to return to a previous menu, or cancel a function during a method.			
Dial/Enter	Rotate Dial to select or change settings. Press Enter to confirm selections or settings.			

#### Status



There are two different status pages on Control Panel: one is 'flow diagram'; and another is 'status table'.

Flow Diagram

🔆 Mini TD Status - Idle					
ltem	Actual	Status/Setpoint			
Tube temp	0.0	0.0			
Valve box	0.0	0.0			
Transfer line	0.0	0.0			
Sample flow	0.0	0.0			
Carrier flow	0.0	0.0			

**Status Table** 

#### To creat a method

This section lists method parameters, along with a brief description of each

	one.
Table 3	List of Mini TD Setting parameters

Parameters	Range	Description
Select Operation Mode	Online sampling	Select the operation mode as
	mode	required
	Desorbing mode	
Sampling	Conditioning Mode	
Enable Pump	Ø/D	Enable/disable Pump
	ت)/ LJ	Specify the gas flow in the
Sampling Flow	5-150 mL/min*	sampling
Sampling Time	0-100 min	Specity the duration of the sampling operation
PreSampling Time	0-100 min	Specify the preparation time of the desorber before sampling
Dry Purging		
Enable Dry Purge		Enable/disable this function
Dry Purging Flow	5-200 mL/min	Specify the gas flow in the dry purging
Dry Purging Time	0-100 min	Specify the duration of the dry purging process
Desorbing		
	$\square$	On/Off
Tube Temp	<b>≤325</b> ℃	Specify the initial temperature of the desorption tube
Carrier Flow	5-200 mL/min	Specify the carrier gas flow
Injection Start Time		Turn On/Off the injection function
	0-100 min	Specify the duration of injection (unit: minute)
Ramp	0-500℃/min	Spcify the rate of temperature Ramp of the first order
Cleaning		
Enable Clearning	⊻/⊔	Enable/disable this function
Clearning Flow	5-200 mL/min	of the Mini TD
Clearning Time	0-100 min	Specify the duration of the cleaning operation
Clearning Temp	<b>≤325</b> ℃	Specify the temperature in the cleaning of the Mini TD
General		
Enable Leak Detection		Enable/disable this function
TransferLine Temp	<b>≤200</b> ℃	Specify the transfer line temperature
ValveBox Temp	≤175℃	Specify the valve box temperature
Conditioning Parameters		
Condition Flow	5-200 mL/min	Specify the gas flow in the Conditioning
Condition Time	0-100 min	Specify the duration of the Conditioning process
Condition Temp	<b>≤325</b> ℃	Specify the temperature in the Conditioning

\* The sampling flow range will be affected by ambient pressure. The Maximal sampling flow rate will degrade in high altitude region.

Installation and Operation

To create a basic method, you will access to method parameters using [Menu]. For a list of method parameters, see Table 3.

1. Press [Menu]. The display shows the current operation mode.

📲 Mini TD Method & Configuration						
Select Op	eration Mode	e:	Online	Sampling M	ode 🔽	Save/Load
Sampling	Dry Purging	Des	orbing	Cleaning	General	System
☑ Enal San San	ble Pump ppling Flow   ppling Time	0.3 0.3		]mL/min ]min		
PreS	ampling Time	1		min		
Press (Dial)	to select mode				2	012-11-08 12:59:59

3. Rotate Dial to operation mode options, then press Enter.

🔆 Mini TD Method & Configuration						
Select Op	peration Mode	e:	Online	Sampling Mode		Save/Load
Sampling	Dry Purging	Des	<mark>Online</mark> Desorb	<mark>Sampling Mode</mark> ing Mode		System
🗹 Enable Pump 🛛 🖸		Condito	oning Mode			
Sampling Flow 0.3			mL/min			
Sampling Time 0.3 min						
PreSampling Time 1 min						
Press (Dial) to select mode. 2012-11-08 12:59:59						

- 4. Rotate Dial to select the desired Operation Mode, then press Enter.
- 5. Rotate Dial to switch between different tabs, then press Enter to enter the desired tab.
- 6. Rotate Dial to switch between different parameters.
- 7. When checkbox is highlighted, press Enter to on/off it.
- 8. When textbox is highlighted,
  - a. Press Enter to edit the value.
  - b. Rotate Dial to change the settings.
  - c. Press Enter to confirm the new settings.

- 9. Press [Cancel] to return to the selection;
- 10. Then press [Cancel] to return to Mode Selection and Load/Save button;
- 11. Setting up a basic method is complete. Save the method.(see 'to Load/Save the method')

Select Or	eration Mod	e: Online	Sampling M	lode 🔽	Save/	nad
Sampling Dry Purging Desorbi			Cleaning	General	System	
⊠ Enal San San PreS	ble Pump npling Flow   npling Time   ampling Time	0.3	mL/min min min			
ress (Dial)	to select mode			2	2012-11-08	12:5

# To Configure the Carrier Gas Type

<b>Wa</b> 1.	Mini TD Carrier Gas type must be consistence with the GC Carrier Gas type. 如
	t
	To configure the carrier gas type, you will access to the system page.
	1. Press [Menu] twice.
	2. Rotate Dial to select 'System' page.
	3. Press Dial to enter Carrier Gas Type selection table.
	4. Rotate Dial to select the desired Carrier Gas Type.
	5. Press Dial to confirm the selection.
	Mini TD Method & Configuration
	Select Operation Mode: Online Sampling Mode 💌 Save/Load
	Sampling Dry Purging Desorbing Cleaning General System
	Carrier Gas Type C N2 He
	Maintenance Limit 0.0
	[Rotate] to switch between tab pages. 2012-11-08 12:59:59

## To Load/Save a Method

To Load/Save a method:

- 1. Press [Menu].
- 2. Rotate Dial to Load/Save button.
- 3. Press Enter. When prompted, rotate Dial to select Save or Load, and then press Enter to open the Load/Save list window.
- 4. Rotate Dial to select the slot number to which or from which you want to save or load method.
- 5. Press Enter to confirm your choice.

🖄 Mini TD Met	🖄 Mini TD Method & Configuration				
Select Operati	on Mode: Online Semaline Mode	Save/Load			
Sampling Dry	To ave ui Ludu	System			
Enable P	Do you want to save or load method?				
Samnling	Save				
amhuni					
Sampling	Time   0.3 min				
Press (Dial) to sel	ect mode.	2012-11-08 12:59:59			
Mini TD Mot	had & Configuration				
	*Save Method				
Select Operat	Choose a location to SAVE method	Save/Load			
Sampling Dry	to.	System			
🗹 Enable P	NO.				
Samplin	M_00				
	M_01				
Samplin	M_02				
	M 04				
		0010 11 00 10 50 50			
Press (Dial) to sel	ect mode.	2012-11-08 12:59:58			
🖄 Mini TD Met	had & Configuration				
Select Operat	Chasse a location to LOAD method	Save/Load			
Sampling Dry	from.	System			
		· · · · · · · · · · · · · · · · · · ·			
🖾 Enable P	NU. M. 00				
Samplin	M 01				
Samplin	M_02				
2.4114.00	M_03				
	M_04				
Press (Dial) to sel	ect mode.	2012-11-08 12:59:59			

#### Log

1. To access log, press Enter twice on flow diagram page.



2. The display will indicate the number of entries the log contains.

🔻 System Log	
Log details # 1: 2012-11-27 15:00:01	Service Counter: 0 (0)
System power on	
Total log: 45	

- 3. Rotate Dial to select different log information.
- 4. Press Enter to return to flow diagram page.



**First Start** 

Materials Needed 51 Condition the Desorption Tube 5 Set the Checkout Method 44 Prepare the Checkout Sample 45 Run the Checkout Method 46 Compare the Checkout Results 47

This section describes how to start the Agilent 7667A Mini Thermal Desorber for the first time and check out the performance of the **G4370A/G4370M** in the system.

Perform only the procedure consistent with your instrument configuration.

#### **Materials Needed**

- 10 uL Syringes P/N 5190-1483
- Checkout samples (Ethyl Acetate) P/N G4370-85002
- Desorption tube P/N C-TBP1TC ( Details see Appendix )

## **Conditioning the Desorption Tube**

In order to ensure that the desorption tube works normally, the desorption tube need to be conditioned before the first run of the sample desorption.

- 1. Install the Desorption Tube. (Please refer to the **"Replace the Desorption Tube"** section in this manual).
- Set the conditioning parameters on the panel (see List 4; for specific operation, refer to the "7667A Control Panel Operation" in this manual). Or load the Condition Method already saved in the system.
- 3. Press "**Start**" key on the control panel to start running the conditioning method.

Parameters	G4370A	G4370M
Conditioning flow	50 ml/min	50 ml/min
Conditioning time	20 min	20 min
Conditioning temperature	320 °C	320 °C
Transfer line temperature	150 °C	150 °C
Valve box temperature	150 °C	150 °C
Type of carrier gas	Nitrogen	Nitrogen/Helium

**Table 4** Recommended conditioning methods for the Mini TD

## Set the Checkout Method

Use the **Agilent data system (OpenLAB ChemStation/EZChrom, MassHunter)** to set the checkout method. The **ChemStation** is taken as an example below (refer to the **"Software Operation Guide"** in this manual for specific operation)

- 1. Run the OpenLAB ChemStation.
- On the "Method and Run Control" interface, choose Instrument
   > Edit Agilent 7820A parameters to go to the "Setting method" interface.
- 3. Choose "**Configuration**" **module** to go to the gas type selection operation interface. Select the connected carrier gas type.
- 4. Edit the checkout method according to List 5 and List 6.
- 5. Apply the method to the instrument.
- 6. Save the method with a new name.

Table 5 Checkou	ut method paramet	ters of the Mini TD
Parameters	G4370A	G4370M
Sampling	Off	Off
Leak Detection	On	On
Enable Dry Purge	On	On
Dry Purging Flow	100 mL/min	100 mL/min
Dry Purging Time	1 min	1 min
Start Injection	On	On
Start Injection	1 min	1 min
Time		
Tube initial temp	40°C	40°C
Tube initial time	0	0
Tube ramp temp	500°C	500°C
Tube final temp	310 °C	310 °C
Tube final time	1 min	1 min
Cleaning	On	On
<b>Cleaning Flow</b>	100 mL/min	100 mL/min
Cleaning Time	5 min	5 min
Cleaning temp	320 °C	320 °C
Xferline Temp	150 °C	150 °C
ValveBox Temp	150 °C	150 °C
Carrier Flow	60 ml/min	60 ml/min

	able 5	Checkout metho	d parameters of	the Mini TD
--	--------	----------------	-----------------	-------------

Table 6	The Method parameters of the 7820A GC/5975	T LTM
	GC/MSD	

Oven	
Equilibration Time	0.5 min
Initial temperature	40 °C
Initial time	0.5 min
Rate1	20 °C
Final temperature1	200 °C
Final time1	0
Inlet (split/splitless)	
Mode	Split
Heater	250 °C (On)
Pressure	3.8791 psi
Split ratio	200:1
Split flow	160 mL/min
Total flow	101.59 mL/min
Carrier gas saver	Off

GC/MSD (continued)	
Column 1(19091J-413)	
Mode	Constant Flow mode
Flow	0.8 mL/min
Pressure	3.8791 psi
Average line speed	15.361 cm/s
Detector – FID	
Temperature	<b>300</b> ℃
Hydrogen flow	30 mL/min
Air flow	400 mL/min
Mode	Constant makeup flow
makeup flow	25 mL/min
MS or MSD	
Solvent delay	0 min
Running time	8.5 min
Scan range	45 to 300
Method used	Atune File
Inlet pressure	82.8 kPa (12 psi)
Column flow	1.1 mL/min

 Table 6
 The Method parameters of the 7820A GC/5975T LTM GC/MSD (continued)

NOTE

The table lists the settings of the 7820A GC/5975T LTM GC/MSD. For other GC types, use the similar settings.

### Prepare the Checkout Sample

- 1. Take out the Syringe and checkout samples from the shipping kit.
- 2. Open the top cover of the desorber, loosen the triangular nut, and pull out the desorption tube assembly.
- 3. Disconnect the desorption tube from tube handle.
- 4. Use the Syringe to draw 0.4ul checkout sample.
- 5. Inject the checkout sample into the desorption tube from the grooved end.
- 6. Insert the desorption tube into the desorber and tighten the triangle nut with your hands.
- 7. Close the top cover.

#### **Run the Checkout Method**

- 1. Select **Run Control > Sample Information** to go to the "**Sample Information**" interface.
- 2. Select the save path of the output signal, the output file name and the vial location (Vial 1 is the default injection location of the Mini TD, no need to change it).
- 3. Press the "**Run the Method**" button to start running the checkout method.
- 4. Or press "**Confirm**" button to close the "**Sample Information**" box, and then select **Run Control > Run the Method** to run the checkout method.

#### **Prepare the Checkout Results**

When the run completes, compare the chromatogram against the example below. If correctly installed and operated properly, the output chromatogram should be similar to the example shown below.



Check out chromatogram



# **Software Operation Guide**

**Preparation Before Start** 56 ChemStation/Ezchrom Operation 56 Start Online Instrument 56 Gas Type Configuration 57 **Parameter Settings** 58 Create the Method 61 Save the Method 62 Run the Method 63 Create the Sequence 64 Save the Sequence 65 Run the Sequence 65 MassHunter Operation 66 Start Online Instrument 66 Gas Type Configuration 66 **Parameter Settings** 67 Create/Save the Method 67 Run the Method 67 Create the Sequence 68 Save the Sequence 69 Run the Sequence 69 **Online Help Information** 70

This guide describes how to use the Agilent data system to control the Mini TD.

# **Preparation Before Start**

This guide assumes that:

- Agilent data system (**OpenLab ChemStation C.01.05 or above**/ **OpenLab EzChrom A.04.05 /Mass Hunter B.07.00 or above**) has been installed.
- The 7820A GC/5975T LTM GC/MSD is configured as online instrument.
- The 7820 GC/5975T LTM GC/MSD and the Mini TD are both ready.

# **OpenLAB ChemStation C.01.05/Ezchrom Operation**

## A.04.05

#### **Start Online Instrument**

1. In the "Start" menu, click the "OpenLAB" icon.





# 2. In the "**OpenLAB Control Panel**" box, press the "**Launch**" button to open the **ChemStation**.

Management	Edit	Lock Create Configure
Management	Notifications	Instrument
Control	Properties	Actions
Navigation Instruments 146.208.245.1 146.208.246.207 146.208.246.218 246.61	≪ 146.208. Start ⊙ S <sup>L</sup> Instrur ⊙ Det	.246.218         Instrument         Launch       Launch Offline         .aunch an online instrument session         ment does not have status information         tails

#### **Gas Type Configuration**

1. Select "Instrument > Edit Agilent 7820 Parameters..." to go to the "Setting Method" interface.



2. In the "Setting Method" interface, select "Configuration" module to go to the gas type selection interface. Nitrogen or helium is available.

ALS	Mini TD	- Inlets	Columns	Oven	Detectors	( Events	Signals	Configuration	1,2, Counters	Readiness	GC Calculators
Miscellar	neous Colum	ns Module	s ALS						10		
			Front Inlet SSZ Inlet	N2	•						
		В	ack Detector FID								
		Catl	Makeup	N2	▼						
		Jen	Mini TD		oard.						
		Ca	mier Gas Type	N2 He N2							

WARNING

The carrier gas type of the Mini TD must be consistent with the that of the GC.

#### **Parameter Settings**

1. Select "Instrument > Edit Agilent 7820 Parameters..." to go to the "Setting Method" interface.

- 2. Click the Mini TD icon to go to the Mini TD parameter setting interface.
- 3. The 7667A Mini Thermal Desorber has three operation modes and the user may choose the proper operation mode based on their application.





#### Software Operation Guide

5 Mini TD J	nlets Colum	ns Oven Det	ectors Events Signal	s Configurati	1,2, on Counters	Readiness GC	Calculators			
Dne Stage Thern Select Operation Mo Enable Sample O Sampling	mal Desorber de: Online Overlap	[ Model Number: Sampling Mode	G4370M ] Detection Dry Purging			Desorbing				
Sampling Flow: Sampling Time:	Setpoint 50 mL/min 1 min	Actual 0 mL/min	Enable Dry Purge Dry Purge Flow: Dry Purge Flow:	Setpoint 100 mL/min	Actual 0 mL/min	<ul> <li>✓ Tube Temp:</li> <li>✓ Carrier Flow:</li> <li>✓ Injection Start Ti</li> </ul>	Setpoint 55 °C 30 mL/min ime: 1 min	Actua 55 °C 30 ml	l ./min	
PreSampling Time	0 min		Dry Purge Time:	1 min		▶ (Initial) Ramp 1	Rate °C/min 600	Value ℃ 55 310	Hold Time min 0	Run Time min 1.42
Cleaning Cleaning Flow: Cleaning Flow:	Setpoint 100 mL/min	Actual 0 mL/min	General TransferLine Temp: ValveBox Temp:	Setpoint 150 °C 150 °C	Actual 149.9 °C 155.4 °C					
Cleaning Time: Cleaning Temp:	4 min 320 °C	0 °C								

Figure 26 Online sampling mode interface

) ble Clean Setpoint Ac 1 Flow: 0 mL/min 0	Cleaning					) Detection	Enable Leak D		
ble Clean Setpoint Ac 1 Flow: 0 mL/min 0	Cleaning								
ble Clean Setpoint Ar ) Flow: 0 mL/min 0	Enable Clean			Actual	Setpoint	Desorbing			Dry Purging
; Flow: 0 mL/min 0				40 °C	40 °C	🔽 Tube Temp:	Actual	Setpoint	Enable Dry Purge
	Cleaning Flow:		min	0 mL/mi	35.063 mL/m	[√] Carrier Flow:	0 mL/min	0 mL/min	Dry Purge Flow:
g Time: 0 min	Cleaning Time:	Run Time	Hold Time	Value	ne: 0 min Rate	Injection Start Ti		0 min	Dry P <mark>urge</mark> Time:
g Temp: 0 °C 0	Cleaning Temp:	min	min	*C 40	°C/min	(Initial)			
		2.2333	2	320	1200	Ramp 1			
									General
							Actual	Setpoint	
;Temp: 0	Cleaning Temp:	0 2.2333	0	40 320	1200	▶ (Initial) Ramp 1	Actual 24.9 °C	Setpoint	General



			🎢 藰	hu	×	1,2,	ំរុំ	
S Mini TD In	ets Columns	Oven D	etectors Events S	ignals	Configuration	Counters	Readiness	GC Calcu
One Stage Therm	al Docorbor ( <b>k</b>	adal Numba	C (1270A 1					
One Stage Therm		nodel Numbe	. G4370A j					
Select Operation Mode	e: Conditioni	ing Mode	•					
-			_					
Conditioning			General					
Conditioning	Setpoint	Actual	General	Set	point	Actual		
	Setpoint	Actual	General ☑ TransferLine To	Setj	point	Actual 24.8 °C		
Conditioning Condition Flow:	Setpoint 50 mL/min	Actual 0	General ☑ TransferLine Te	Set; emp: 0 °	point C	Actual 24.8 °C		
Conditioning Condition Flow: Condition Time:	Setpoint 50 mL/min 30 min	Actual 0	General ☑ TransferLine Te ☑ ValveBox Temp	Sety emp: 0 ° o: 0 °	point C	Actual 24.8 °C 36.8 °C		
Conditioning Condition Flow: Condition Time:	Setpoint 50 mL/min 30 min	Actual 0	General ☑ TransferLine Te ☑ ValveBox Temp	Set emp: 0° o: 0°	point C	Actual 24.8 °C 36.8 °C		

Figure 28 Conditioning Mode interface

4. Table 7 lists all of the editable parameters and the parameter range.

		runge.
Table 7	List of Mini TD	Setting parameters

Parameters	Range	Description
Select Operation Mode	Online sampling mode Desorption mode Conditioning Mode	Select the operation mode as required
Enable sample overlap	$\square$	Enable/disable this function
<ul> <li>After the previous injection is completed</li> <li>Prepare sample</li> <li>Imin</li> <li>before end of GC run</li> </ul>	<ul><li>⊙/○</li><li>⊙/○</li><li>0-100 min</li></ul>	<ol> <li>Start the next sampling after the completion of the present injection (or after the completion of the cleaning if cleaning is needed).</li> <li>Begin to prepare the sample # minutes before the completion of the GC running.</li> </ol>
Enable Leak Detection	$\square$	Enable/disable this function
Sampling		
Enable Pump	$\square / \square$	Enable/disable Pump
Sampling Flow	5-150 mL/min*	Specify the gas flow during sampling
Sampling Time	0-100 min	Specify the duration of the sampling
PreSampling Time	0-100 min	Specify the preparation time before sampling
Dry Purge		
Enable Dry Purge	$\square$	Enable/disable this function
Dry Purging Flow	0-200 mL/min	Specify the gas flow during dry purging
Dry Purging Time	0-100 min	Specify the duration of the dry purging process

\* The sampling flow range will be affected by ambient pressure. The Maximal sampling flow rate will degrade in high altitude region.

<b>Table 7</b> List of Mini TD Setting parameters (Continued	Setting parameters (Continued)
--	--------------------------------

Clearning		
Enable Clearning	$\Box / \Box$	Enable/disable this function
Clearning Flow	5-200 mL/min	Specify the flow in the
		cleaning of the Mini TD
Clearning Time	0-100 min	Specify the duration of the
		cleaning operation
Clearning Temp	<b>≤325</b> ℃	Specify the temperature in
		the cleaning of the Mini TD
General		
Carrier Flow	5-200 mL/min	Specify the carrier gas flow
Xferline Temp	<b>≤200</b> °C	Specify the transfer line
		temperature
ValveBox Temp	<b>≤175</b> ℃	Specify the valve box
		temperature
Desorbing		
Tube Temp	$\square$	On/Off
	<b>≤325</b> ℃	Specify the initial
		temperature of the
		desorption tube
Injection Start Time	$\Box / \Box$	Turn On/Off the injection
		function
	0-100 min	Specify the duration of
		injection (unit: minute)
Ramp	0-500 °C/min	Spcify the rate of
		temperature Ramp of the first
		order
Conditioning Parameters		
Condition Flow	5-200 mL/min	Specify the gas flow in the
		Conditioning
Condition Time	0-100 min	Specify the duration of the
		Conditioning process
Condition Temp	<b>≤ 325</b> ℃	Specify the temperature in
		the Conditioning

NOTE

The 7667A Mini Thermal Desorber only supports first-order ramp and the highest ramp rate is 500  $^\circ\!\!C$  /min.

#### **Create the Method**

- 1. Select "Instrument > Edit Agilent 7820 Parameters..." to go to the "Setting Method" interface.
  - \*\*\*\*\*
- 2. Click the Mini TD icon to go to the Mini TD parameter setting interface.
- 3. Select the operation mode as required.
- 4. Input/change other parameters as required. Refer to "Parameter Setting" in the above.
- 5. Press the "**Apply**" button to apply the method to the Mini TD.
- 6. Edit the 7820 GC parameters (refer to the 7820A GC Manual).

 Select Instrument > Select Injection Source to open the "Select Injection Source" dialog box; select "Mini TD" as the injection source.

Select Injection Source:	
Manual	UK
GC Valve	Court
Mini TD	Lancel
S <u>e</u> lect Injection Location:	

#### Save the Method

Select "**Method** > **Save Method As...**" to open the "**Save Method As**" diaglog box. Enter a new file name and press "**OK**" button to save the current method. Meanwhile, the method is applied to the GC-Mini TD system you have connected.

🥶 78	20-TD (online	): Method and	d Run Control
File	RunControl	Instrument	Method Sequence View Abort Help
			Run Time Checklist
			Method Information
			Edit Entire Method
			Custom Fields Setup
			Method Audit Trail
			Enable Audit Trail
			New Method
			Load Method
			Save Method
			Save Method As
			Print Method
			Export eMethod
			Import eMethod
			1 CONDITIONB.M
			2 DESORB.M
			3 0910.M
			4 CHECKOUT.M

Save Method as: 7820-TD Name: CONDITIONB.M	Folders: c:\chem32\1\methods
0831.M 0910.M 1.M 100FID.M 905.M CARRIER GAS.M CARRIER GAS.M CET.M CHECKOUT.M CONDITIONB.M DEF.GC.M DESORB M ESTD_EX.M ISTD_EX.M ISTD_EX.M NPD_ECD.M	Image: Constraint of the second sec
Types:	Drives:
Method(".M)	C: Vetwork

#### **Run the Method**

1. Select "**Run Control > Sample Information**" to open the "**Sample Information**" box. Then select the save path of the output signal, the output file name and the vial position (Vial1 is the default position of the Mini TD).

)perator name:	SYSTEM			4			
Data file	26						
Path:	C:\Chem;	32\1\DATA\		•]	Subdirectory: 0	91001	•
	Name Pat	tern					
Signal 1:	test091104	4			>	( )	
	test09110	04.D					
		2					
Vial/Location	n:	Vial 1 test	(blank run if no entr	у)	Sample amount:	0	
Vial/Location Sample nam Multiplier:	n:	Vial 1 test 1	(blank run if no entr Dilution: 1	y)	Sample amount: ISTD amount:	0	
Vial/Location Sample nam Multiplier: Comment:	n:	Vial 1 test 1	(blank run if no entr Dilution: 1	y)	Sample amount: ISTD amount:	0	*

2. Press the "**Run Method**" button to start running the method, or press the "**OK**" button to close the "**Sample Information**" box. Then select "**Run Control > Run Method**" to start running the method.

#### **Create the Sequence**

1. Select "Sequence > Sequence Table" to open the sequence table.

File	RunControl	Instrument	Method	Sequence View Abort Help
				Sequence Parameters
			(	Sequence Table
				Custom Fields Values >
				New Sequence Template
				Load Sequence Template
				Save Sequence Template
				Save Sequence Template As
				Import Sequence Template (Front)
				Import Sequence Template (Back)
				Print Sequence Template
				Partial Sequence
				1 DEF_GC.S
				2 1018.S
				3 1012.5
				4 0831.S

In the sequence table, enter the vial position (Vial1), the method name and the data file name.

Currently Ru Line:	Method:						Viał	[]	Inj:	Injector Front	Back	
ample Info fo	or Vial 1:							* *				
Line 1 2	Vial Sample Name Vial 1 sample1 Vial 1 sample1		Method Name 11275 11275	Inj/Vial 1	Sample Type Sample Sample	Cal Level	Update RF	Update RT	Interval	Sample Amount	ISTD Amount	Multiplie
		•										
Insert nsert/FillDo	Cut (	Copy ]	Paste Custom Fields	Append	Line Un	do All			Run Se	quence	lelp	
er of inject	tions of this sample to be mad	le									Configure Table	

Sau

×

#### Save the Sequence

3. Select "Sequence > Save Sequence Table As..." to save the sequence.

File RunControl Instrument Method Sequence View Abort Help



#### **Run the Sequence**

4. Select "**Run Control > Run the Sequence**" to start running the current sequence.



## **MassHunter Operation**

#### **Start Online Instrument**

1. On the "Desktop", click the online instrument icon to enter the



"MassHunter" main interface.

## **Gas Type Configuration**

1. Select "Instrument > GC Parameters..." to go to the "Edit GC Parameters" interface.

Method	Instrument	Sequence	View	Abort	Checkout	Window	Graphics	Help
	Inlet/Inj	ection Types						
	Edit Mo	nitors						
	GC Para	meters						
	GC Real	-Time Plots	13					
	GC Con	figuration						
	GC Sho	w/Hide Statu	s					
	MS Tun	e File		5-				
	MS Edit	Parameters						
	MS Ten	nperatures						
	MS Vac	uum Control	<b></b>					
	Tune M	SD						
	Edit Tur	ne Parameter	s					
	CI Cont	rol						
	EMF Uti	lities		-				

- 2. In the "Edit GC Parameters" interface, select "Configuration" module to go to the gas type selection interface.
- 3. OnlyHelium available.

#### **Parameter Settings**

- Select "Instrument > GC Parameters" to go to the "Edit GC Parameters" interface, and enter the GC parameters. (The specific operating steps are the same as those of "ChemStation").
- Select "Instrument > MS Parameters" to go to the "Edit MS Parameters" interface, and enter the MS parameters.

#### **Create/Save the Method**

The operating steps of Create/Save Method is the same as those of "ChemStation".

#### Run the Method

Select "Method > Run the Method"	to start running the current
method.	

Method	Instrument	Sequence	View	Abort	Checkout	Window	Graphics	Help
Load	l Method							
Save	Method							
Save	Method As							
Run	Method							
Print	Method							
Edit	Entire Method.							
٨ddi	tional Method	Information						

#### **Create the Sequence**

Method	Instrument	Sequence	View	Abort	Checkout	Window	Graphics	Help
		Load S	equence			2		
		Save Se	equence					
		Save Se	equence	As				
		Run Se	quence.					
		Restart	Paused	Sequenc	e			
		Edit Se	quence					
		Simula	te Seque	ence 🛛				
		Position and Run Sequence						
		1						
		2						
		3						
		4						
		View S	equence	Log				
		Print S	equence	Log				
		Additio	onal Sequ	uence Op	tions			

1. Select "Sequence > Edit Sequence" to open the "Sequence Table".

2. In "**sequence table**", enter the vial position (Vial1), the method name and the data file name.

	Name	Vial	Method File	Data File	Туре		Level	Dil.	Comment
1	Sample 1	1	default.m	)	Sample	-		1	
2	Sample 2	1	default.m	)	Sample	•		1	
3	Sample 3	1	default.m		Sample	-		1	

#### Save the Sequence

3. Select "Sequence > Save Sequence Table As..." to save the current sequence.

Method Instrument	Sequence View Abort Checkout	Window	Graphics	Helj
	Load Sequence			
	Save Sequence	and the second		
(	Save Sequence As			
	Run Sequence			
	Restart Paused Sequence	-		
	Edit Sequence			
	Simulate Sequence			
	Position and Run Sequence			
	1			
	2			
	3			
	4			
	View Sequence Log			
	Print Sequence Log			
	Additional Sequence Options			

## **Run the Sequence**

4. Select "Sequence > Run the Sequence" to run the current sequence.

Method Instrument	Sequence View Abort Checkout	Window	Graphics	Help
	Load Sequence Save Sequence Save Sequence As			
	Run Sequence			
	Restart Paused Sequence			
	Edit Sequence Simulate Sequence Position and Run Sequence			
	1 2			
	3			
	4			
	View Sequence Log Print Sequence Log			
	Additional Sequence Options			

# **Online Help Information**

The Agilent Integrated Mini TD Software includes an extensive online help system with detailed information and common tasks on how to use the software.

3 78xx Series GC Help								
Hide Back Forward Print								
Contents Search	Mini Thermal Desorb	er (Mini T	<b>)</b> )			<u>^</u>		
What's New?           Loading Methods           Method Editor           ? Setting Method Parameters           ? Graphical Plots           Arrow Status Panel           Atl S (rejectors)           Mini Thermal Desorber           ? Mini Thermal Desorber Concepts           Valves           Valves           Columns           Oven           ? Oven           ? UTM Series II Temperature Contric	The Mini TD icon in the Method Editor is available if you have a Mini Thermal Desorber installed in your system. See Mini Thermal Desorber Concepts for more information. You can specify the following six group parameters: Sampling Parameters, Dry Purging Parameters, Desorbing Parameters, Cleaning Parameters, Conditioning Parameters, and General Parameters. You can also turn on/off Overlap Mode and Leak Detection. Note You will need to specify the Carrier Gas Type before setting the method parameters. See Method Editor > Configuration > Modules for more information. Mode Selection The available modes depend on the type of Mini TD installed. Each mode has a different combination of parameters. See the following matrix for details. Mode available for different type of Mini TD installed:							
? Temperature Ramp ? Cryo Heaters Detectors Detectors	Modes	Entry	Enhanced					
Events (Run Time Events)	Online Sampling Mode	×	1					
Configuration Backflush Wizard	Desorbing Mode	V	×					
Readiness	Conditioning Mode	V	~					
Instrument Configuration     Maintenance     Sample Prep     Tasks     Tutonal	Combination of parameters	or different Mo	des selected: npling Mode	Desorbing Mode	Conditioning Mode			
	Sampling		√	×	×	-		
	Dry purging		1	1	×			
	Desorbing		4	N	x			
	Cleaning		1	V	×			
	Conditioning		×	×	Ń			
	General		1	1	~			
×	Mouse over the parameters and you	will see a suggest	ed minimum and ma	aximum value limitation				
🚱 🖉 📜 🚺	* 🦻 🔒 🛷					▲ 🖿 🖿 🌵 6:58 PM 6:58 PM 1/25/2013		



# **Routine Maintenance**

Consumables List 72 Update the Mini TD Firmware 72 Replace the Injection Needle Ass 73 Install the Injection Needle Ass 73 Remove the Injection Needle Ass 75 Replace the O-ring and the Filter 76 Replace the Trap (for G4370M) 79 Remove the Stainless Steel Tube Seamless from the Transfer Line 82

The information contained in this section will help you to successfully use the Mini TD and ensure that the 7667A Mini Thermal Desorber system can run with full performance. The maintenance intervals of the G4370A/G4370M vary with the use frequency of the instrumen
#### **Consumables List**

Table 8 lists a number of consumables accompanying the 7667A. For the latest parts and consumables, visit Agilent's website: www.agilent.com/chem.

Table 8         Consumables		
Consumables	Agilent Part Number	
Desorption tube	C-TBP1TC	
O-Ring 0.239-in-ID (O-ring)	0905-1820	
O-Ring 0.364-in-ID (Large O-ring)	0905-1819	
Injection Needle Ass	G4372-60015	
Filter	MKI-U-DISK3	
Trap	G1544-80500	

#### Upgrade the Firmware of Mini TD

Please use **Agilent Instrument Update Utility** (**A.01.01**) to update the Mini TD firmware. Users can download the latest revision of firmware from Agilent's website (**www.agilent.com/chem**) or contact your local Agilent's sale representitives.

### **Replace the Injection Needle Ass**

1.

#### Install the Injection Needle Ass

open the side door.

2. Confirm the injection slider in the locked high level.

Put the G4370M flat on the bench with side door facing up, and



3. Use the T10 screwdriver to remove the screw as shown in the illustration.



Inlet adapter

Lock high level

- 4. Push the inlet adapter out from the inside.
- 5. Use a 1/4 inch wrench to remove the lock nut.



6. Insert the Injection Needle Ass (P/N G4372-60015) into the lock nut and tighten it on the injection slider with your hands and then use the wrench to reinforce it.



7. Rotate the locking handle to the vertical position to unlock the injection slider. Push the injection slider to the injection low level.



Error! Use the Home tab to apply 标题 1 to the text that you want to appear here.



9. Push the injection slider to the locked high level, and then rotate the locking handle back to the locked position.

#### **Remove the Injection Needle Ass**

# Attention! The transfer line may be hot and cause burns. Cool it to room trmperature to avoid burns.

- 1. Cool the transfer line to room temperature.
- 2. Open the side door, and rotate the locking handle to the vertical position to unlock the injection slider.
- 3. Push the injection slider to the locked high position and rotate the locking handle to the locked position.
- 4. Close the side door.
- 5. Turn off the power and gas supply. Remove all the gas lines.
- 6. Remove the G4370M from the 5975T and put it flat on the bench.
- 7. Open the side door and remove the inlet adapter.
- 8. Loose the lock nut and pull out the Injection Needle Ass with hand.

#### **Replace the O-ring and Filter**

- 1. Open the top cover.
- 2. Unscrew the triangle rotation nut counterclockwise and take out the desorption tube assembly.



3. Remove the lock nut, pull out the desorption tube, and remove the O-ring and Filter.



- 4. Replace the O-ring and Filter.
- 5. If a larger O-ring does not need to be replaced, ignore steps 6-9.
- 6. Use a T-10 screwdriver to remove the screw as shown in the illustration.



7. Remove the lock nut and triangle rotation nut.



- 8. Replace with a larger O-ring.
- 9. Restore the desorption tube assembly.
- 10. If the sealing O-ring on the groove side of the desorption tube needs to be replaced, follow the steps below; or ignore the steps below.
- 11. Use a T-10 screwdriver to remove the nut as shown in the illustration and remove the cotton insulation cover and cotton insulation.



12. Loosen and pull out the screw as shown in the illustration.



13. Use a 5/16 inch wrench to unscrew the nut as shown in the illustration.



14. Remove the screws as shown in the illustration.



15. Push backward the heating tube assembly, and replace the

O-ring and Filter as shown in the illustration.

16. Restore the desorption tube heater status.

#### Replace the Trap (for G4370M)

- 1. Turn off all heating zone temperature.
- 2. Turn off the power and unplug it.
- 3. Turn off the gas supply and then remove the gas line on the instrument.
- 4. Open the side door.
- 5. Rotate the locking handle to the vertical position (see the illustration below) and unlock the injection slider.



- 6. Push the injection slider to the locked high level, and then rotate the locking handle to the locked position.
- 7. Close the side door.
- 8. Remove the G4370M from the 5975T.

9. Put the instrument on the bench. See the illustration below.Remove the instrument rear cover.



10. Remove the two screws as shown in the illustration (see the illustration below).



11. Move the trap cover to left



- 12. Remove trap by hand. ....
- insert a new trap (G1544-80500).
   Restore the instrument to the original state.

#### **Remove the Stainless Steel Tube Seamless from the**

## Transfer Line (for G4370A)

CAUTION	Disconnecting the transfer line may interrupt the GC carrier gas flow. Cool the GC Column Oven and inlet as needed to prevent damage to the column.
WARNING	Attention! The column oven and inlet may be hot and cause burns. Please wear protective gloves to avoid burns.
1.	Cool the G4370A heating zone to a safe handling temperature.
2. 3.	Remove the valve box cover and take out the cotton insulation. Remove the Fitting-internal Reducer 1/16 to 1/32-in.
	The first of the six-point value. Save it for future use.

- 4. Loosen the long transfer line Latch/Clamp on the GC.
- 5. Pull out the Stainless Steel Tube Seamless from the inlet.
- 6. Gently grasp the stainliss steel tube on an inlet end and pull the column from the transfer line.

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Symptom 1: Start failure 84 Symptom 2: The carrier gas flow does not reach the set point 84 Symptom 3: The chromatogram is not shown 84 Symptom 4: The chromatograms indicate a lot of impurities 85 Symptom 5: Gas leak 85 Symptom 6: Overloading 85 Error Messages 86

This section only deals with the problems related to the Mini TD. However, many of the symptoms described here may also originate from other problems, especially GC's temperature and the stability of GC's gas supply.

If you are unable to correct the problem, please contact Agilent's support.

## Symptom 1: Start failure

Switch on 7667A and it is not initialized pre-	operly.
--	---------

Possible Cause	Operation
Power cord is not plugged	Check whether the power cord is plugged.
The power adapter plug is not	Check if the power adapter 6-hole connector is plugged
plugged	

# Symptom 2: The carrier gas flow does not reach the set

## point

Possible Cause	Operation
The gas supply is not connected properly	Tighten the carrier gas interface. The carrier gas configuration should be consistent with that of the actual gas
	type.
Insufficient pressure leads to	Check the upstream carrier gas pressure that should be
unregular carrier gas supply	higher than 60 psi (or 0.4 Mpa)
Injection Needle Ass clogged	Replace the Injection Needle Ass

## Symptom 3: The chromatogram is not shown

Possible Cause	Operation
Gas chromatography failure	Recommended to disconnect the thermal desorber, prepare standard checkout sample and observe if the sample will make a peak
The Mini TD's Injection Needle Ass is not inserted into the GC inlet	Insert the Injection Needle Ass completely into the gas chromatography inlet
The desorption tube is not properly configured in the thermal desorption instrument	Check if the desorption tube is installed properly. Check if the desorption tube is with the correct sorbent. Check if the driver gas is connected and pressurized.
Sampling fails; the carrier gas type is not configured properly	If you are using the G4370M version, ensure that the sample enters the inlet. Observe if the sample flow reaches the set piont; if it is very low or is zero, restart the instrument and re-configure the carrier gas type. If you are using the G4370A version, ensure that the sampling process is correct
The desorption tube leaks at the connection point The sampling flow is normal, but the flow in purging does not reach the set point or is zero	Confirm if the triangle nut in the desorption tube assembly is tightened; if it does not work, replace the O-ring
The carrier gas flow does not reach the set point	Check the upstream carrier gas pressure that should be higher than 60 psi (or 0.4 Mpa)

# Symptom 4: The chromatograms indicate a lot of impurities

Possible Cause	Operation
There are some Residual impurities in the column and the desorption tube	Before testing the sample, it is recommended to condition the chromatographic column and the desorption tube and test with a blank run, which may be Nitrogen instead. If the blank sample still has a lot of mixed peaks, confirm if the gas chromatography inlet is contaminated.
The liner and septum are contaminated	Replace the liner and septum

#### Symptom 5: Gas leak

The flow does not reach the set point or is zero in the purging process.

Possible Cause	Operation
The desorption tube is not	It is recommended to tighten the desorption tube or replace
tightened properly into the	the O-ring.
thermal desorber, or the	
desorption tube's O-ring needs	
to be replaced	

## Symptom 6: Overloading

Serious residue is found in the blank run after the injection (Carryover is larger than 1%.).

Possible Cause	Operation
The sample concentration is	It is recommended to extend the cleaning time to 10 min,
too high	heat the desorption tube to the maximum allowable
	temperature, and set the flow to 200 ml/min.
High-boiling-point sample	Confirm if the sample contains high boiling point
enters the thermal desorber	components. This thermal desorber does not support the
	component with boiling point higher than those of the
	n-alkanes C16. If any high-boiling-point component, whose
	melting or boiling point is higher than those of the n-alkanes
	C16, enters, which have an impact on the test, the first
	recommendation is to condition the Mini TD system
	completely. If it doesn't work, replace the valve box or tube
	weldments.
	Please contact Customer Service.

#### **Error Messages**

Table 9 lists the error messages that the Mini TD reports (in the Workstation Log and Control Panel). If a message received is not included in the table below, please note it down and report the error message to Agilent's support.

Error Code	Code Meaning	Possible Cause
0x0001	Transfer line heater shut down	Transfer line is burned
0x0002	Transfer line heater sensor open	Transfer line is burned or the sensor is damaged
0x0004	Valve box heater shut down	Valve box is burned
0x0008	Valve box heater sensor open	Valve box is burned or the sensor is damaged
0x0010	Tube heater shut down	Heating wire is damaged
0x0020	Tube heater sensor open	Heating wire is damaged or the sensor is damaged
0x0040	Sampling flow EPC shut down	Sampling channel is blogged
0x0080	Sampling flow EPC not functional	Sampling channel is blogged
0x0400	Carrier flow EPC shut down	Upstream carrier gas pressure is insufficient or there is no carrier gas; carrier gas configuration is not consistent with the actual gas type
0x0800	Carrier flow EPC not functional	Upstream carrier gas pressure is insufficient or there is no carrier gas; carrier gas configuration is not consistent with the actual gas type
0x1000	Lost communication	Sync cable is not connected
0x2000	Leak detected	Desorption tube is not tightened, or O-ring in the desorption tube is not sealed
0x3000	TD is disconnected form GC	RS232 Cable for ALS interface is not connected

 Table 9
 Error messages

## Appendix

Tube parameters:



Markes International Ltd. Gwaun Elai Medi Science Campus, Llantrisant RCT, CF72 8XL, United Kingdom

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#### How to condition your sorbent tubes

Your tubes are packed with: Tenax TA

They have a maximum desorption temperature of: 350°C

Tubes should be conditioned using clean carrier gas Carrier gas (e.g. Oxygen Free Nitrogen or Helium at 5N grade and ideally with a hydrocarbon filter in the gas line), at a flow of between 50ml/min and 100ml/min.

We recommend the following conditioning method for these freshly packed sorbent tubes:

#### 2 hours at 320°C followed by

#### 30 mins at 335°C

Once cleaned tubes should be capped with  $\frac{1}{4}$  inch brass storage caps fitted with  $\frac{1}{4}$  inch combined PTFE ferrules. These caps should be tightened finger tight and a quarter turn.

Tubes should be stored in a CLEAN environment and the brass storage caps should not be removed until immediately prior to sampling or desorption.

For trace level (low ppb / ppt level) analyses it is advisable to run blank desorptions prior to sampling.

We recommend the following maximum desorption temperature to reduce artefacts and extend sorbent lifetime: 300 - 320°C

For reconditioning tubes we recommend:

#### 15 - 30 mins at 335°C

However if the tubes become severely contaminated or are left for a period of time (1week+) without being capped then it is recommended that the full procedure shown above be repeated.

These tubes have a lifetime of approximately: **100** cycles, which includes both desorption and conditioning cycles. We recommend that the tubes be repacked with fresh sorbent after this.

Further information about selection of sorbents and conditioning criteria can be found in Markes International's Thermal Desorption Technical Support Note TDTS05. Further information about minimising tube artefacts during storage and / or transportation can be found in Markes International's Thermal Desorption Technical Support Note TDTS19.

These and many other technical publications can be obtained by registering on the Markes web site <u>www.markes.com/registration</u> or by contacting Markes International on <u>consumables@markes.com</u>