# SIS AutoDesorb™ Installation Manual

## **AutoDesorb Model 2000**

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Publication No. 786000M-I

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Patents covering the design, operation, techniques, and unique features of the Short Path Thermal Desorption System and Micro Cryotrap include:

> U.S. Patent #5,065,614, November 19, 1991 U.S. Patent #5,123,276, June 23, 1992 UK Patent #GB 2 253 161 B, February 1, 1995 U.S. Patent #5,596,876, January 28, 1997

> > Other Patents are pending.

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# AutoDesorb<sup>™</sup> Manual

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## **GENERAL INFORMATION**

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AutoDesorb<sup>™</sup> Manual

## This Manual

This manual covers the installation of the AutoDesorb<sup>™</sup> system from Scientific Instrument Services, Inc., onto the Agilent Technologies 6890 and 5890 (with EPC) gas chromatographs. Information pertaining to operation and software can be found in the manuals dedicated to those subjects. Detailed information about system accessories can be found in the appendices. As new equipment is developed, additional appendices will be provided.

The information in this manual is provided with the assumption that the user is familiar with general gas chromatography concepts and the operation of the instrument on which the AutoDesorb system is installed. Refer to the manuals supplied by the manufacturer of your GC for specific information about using the instrument and its data system.

Thermal Desorption Application Notes can be found in the AutoDesorb Software Library. The library can be accessed from the Help menu of the main AutoDesorb program window or from the AutoDesorb CD. New application notes are also available from the SIS web site:

#### http://www.sisweb.com

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> Scientific Instrument Services, Inc. 1027 Old York Rd. Ringoes, NJ 08551



## Warranty and Service

#### Warranty

The AutoDesorb system is warranted against defects in material and workmanship for a period of one year commencing from the date of shipment from the warehouses of Scientific Instrument Services (SIS), Inc. in Ringoes, NJ. SIS's liability for the AutoDesorb System and accessories is limited to the cost of correcting the defect in the product. In no case shall SIS be liable for consequential or special damages. SIS is not obligated to correct defects caused by the buyer's or user's negligence. Tampering with any part of the system or servicing by anyone not specifically authorized by SIS to undertake such service will void this warranty. This includes installation of hardware or software by unauthorized parties. If you decline to have an authorized SIS representative install your AutoDesorb system, this warranty is void, and the purchase of an extended warranty may come under certain restrictions. SIS does not guarantee or warrant the product for any particular purpose. All warranty repairs will be made at the SIS facilities in Ringoes, NJ or other location as designated by SIS. Damage incurred during shipment is excluded from this warranty if original packaging from SIS is not used. SIS's warranty shall end one year after shipment.

#### **Extended Warranty**

An extended one year warranty for parts and labor is available if purchased within 12 months of shipment of the unit. The one year extended warranty will cover parts and labor to repair the thermal desorption unit and controller.

Service and Repair

Any equipment to be serviced under warranty or otherwise should be sent to repair facilities designated by Scientific Instrument Services, Inc. No on-site service is available. A return authorization number (RA#) must be obtained from the offices of Scientific Instrument Services before any equipment is sent for service. To obtain an RA number and the address to which you should return the equipment, please call or write:

Scientific Instrument Services, Inc. 1027 Old York Road Ringoes, NJ 08551 Attn: Repair Department

Phone: (908) 788-5550

#### Keep all Boxes and Packaging.

When returning systems for repair, they must be sent in original system boxes and packaging to avoid damage. If we do not receive your original packaging there will be an added fee for new packaging when the system is returned to you. SIS is not liable for damages incurred in shipping if a system is returned in other than original packaging material. Replacement packaging is available; contact SIS for details.



## **Safety Information**

Improper use of this equipment may result in system damage or personal injury. Please read this manual thoroughly before using the AutoDesorb system.

The following warning symbols are used in this manual to call attention to procedures which, if not adhered to or correctly performed, are most likely to result in equipment damage or personal injury:





Warning: Electrical Hazard



Warning: Burn Hazard

#### The following safety precautions must be observed for the safe operation of the AutoDesorb System.



The controller must be connected to a power source with a protective earth contact. Connecting the AutoDesorb System to a power source that is not equipped with a protective earth ground contact creates a shock and fire hazard and can damage the instrument.



Use only fuses with the required current rating and of the specified type for replacement. The use of incorrect or makeshift fuses, or the short-circuiting of fuse holders, creates a shock and fire hazard and can damage the instrument.





The cases for the both the controller and the desorption unit should only be opened and serviced by a trained and qualified service technician. The power to the controller must be disconnected before any part of the instrument is serviced.



Temperatures up to 450°C are present inside the desorption unit under normal operating conditions. KEEP HANDS AND FINGERS OUTSIDE OF THE DESORPTION UNIT.



The desorption tube and needle will remain HOT for several minutes after desorption takes place. Allow at least 5 to 10 minutes of air cooling before handling these parts.



Do NOT use HYDROGEN Gas in the AutoDesorb System. The device is not explosion proof. Explosive mixtures may develop near high temperature areas or ignition sources.



Do not desorb samples above 450<sup>0</sup> C. Exceeding this temperature may damage heater blocks and surrounding parts.



Do not operate the equipment when moving parts are exposed.



## INSTALLATION

Unpacking Site Preparation Cryotrap Installation Desorption Unit Installation Controller Installation Software Installation



AutoDesorb<sup>™</sup> Manual

## Unpacking

Remove the desorption unit from its packing, and inspect it for signs of shipping damage. Set the unit on a benchtop or other level surface. Remove the electronics module and inspect it as well. Verify that the contents of all cartons agree with the inventory list included in each. Do not proceed with the installation unless all items are accounted for and in good condition. Also confirm that any accessories and supplies that may have been ordered are included.



Call SIS immediately if any items were not received with your system, and especially if ANY ITEMS APPEAR TO HAVE BEEN DAMAGED IN SHIPPING. Save all packaging material and boxes. They will be required if components ever need service.

#### AutoDesorb System, Model 2000





Figure 1 The AutoDesorb System and Components

Part #	Description	Quantity
(1) 786000-D	Desorbtion Unit	1
(2) 786000-C	Controller	1
(3) 786000-M	AutoDesorb Manual	1
(4) 786000-S	AutoDesorb Software CD-ROM	1
(5) 17600	Power Cable	1
(6) 786020	Heater Cable	1
(7) 786010	6 Foot Interconnect Cable	1
(8) CBLMNT9	RS232 Cable	1
(9) 786009	Connecting Tubes	12
(10) 786900	GC Septum Nut	1
(11) 786910	Seal removal tool	1



## Accessory Kit

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	0	
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24 Se	0	

The accessory kit (Part # 786030) contains a set of 12 desorption tubes and needles. We recommend the purchase of at least 1 kit with each AutoDesorb system. Additional tubes and needles can also be purchased separately.



Figure 2 The Accessory Kit contains 12 Desorption Tubes, Needles and Seals and 24 Sealing Caps and PTFE Seals

Part #	Description	Quantity
786005	GLT Desorption Tubes, 3 mm, empty	12
786035	AutoDesorb needle	12
786018	Vespel seal	12
781006	Stainless Steel Tube Cap	24
781007	PTFE Seals for Cap	24

## **Installation Kits**

You should have ordered and received one of the two installation kits that are required to install the AutoDesorb system. Each kit is designed for a specific chromatograph. One installation kit is needed for each GC onto which the AutoDesorb system will be installed.

## Agilent 6890 Installation Kit



#### Figure 3 Agilent 6890 Installation Kit: (Part # 786200)

Part #	Description	Quantity
(1) T125062	PTFE tubing, 1/8"OD x 10'	1
(2) B2003	Тее	2
(3) CBLMNT9	GC Remote Start Cable (RS232 Cable)	1
(4) 7819993	GC Gas Valve Assembly	1



### Agilent 5890 Series II Installation Kit



Figure 4 Agilent 5890 Installation Kit and Components (Part # 786210)

Part #	Description	Quantity
(1) T125062	PTFE tubing, 1/8"OD x 10'	1
(2) B2003	Тее	2
(3) 782012A	GC Remote Start Cable	1
(4) 7819993	GC Gas Valve Assembly	1
(5) 2800672	GC Injection Port Mounting Plate	1
(6) 2800671	GC front cover	1
(7) 2800673	GC front cover mounting bracket	1

## Items Required but Not Included

The following are not included with the AutoDesorb<sup>™</sup> system but are required for installation.

#### Tools

- 1 #20 Torx screwdriver
- 1 <sup>1</sup>/<sub>4</sub> inch wrench for inlet and detector/trasfter line
- 2 5/16 inch wrench for 1/16" Swagelok connections
- 1 3/8 inch wrench for tee fittings
- 2 7/16 inch wrench for 1/8" Swagelok connections
- 1 9/16 inch wrench for 1/4" Swagelok connections
- 1 small flat screwdriver
- 1 3 mm Allen key
- 1 1/8" Allen key
- 1 small file and/or tubing cutter

#### Other materials

-Helium leak detector (thermal conductivity or bubble fluid).

- -Clean 1/8" diameter flexible copper tubing for making carrier gas connections and for CO<sub>2</sub> Cryotrap, if appropriate.
- -Clean ¼" diameter flexible copper tubing and insulation for liquid Nitrogen Cryotrap, if appropriate.
- -A supply of dry, particulate-free compressed air for pneumatics regulated to 60-80 psi.

## **Site Preparation**



Figure 5 - AutoDesorb mounted on Agilent 6890 GC and 5973 MSD

The AutoDesorb System is a compact, self contained injection and desorption system that requires a minimum amount of space. The system is only designed for top injecting GC systems. The AutoDesorb Unit sits directly over the injection port of the gas chromatograph. The base of the Thermal Desorption Unit is 6.5" wide x 13" deep and the overall height is 17". It is designed to mount on top of the injection port plate on top injecting GC's such as the HP 6890 GC as shown above.

Prior to installing the AutoDesorb, verify that all infrastructure requirements have been met. The following tasks should be complete at the time of installation:

1. The GC (or GC/MS) should be installed and operational, including all carrier and detector gases, and computer.

2. A supply of particulate-free compressed air, regulated to 60-80 psi should be available in the vicinity of the GC.

3. If a Cryotrap is to be installed, a supply of liquid nitrogen (LN2) or liquid  $CO_2$  should be available, and the appropriate transfer lines should be installed.

## **Compressed Air**

A supply of clean compressed air or gas is required for the operation of the air solenoids which power the injector and desorption blocks. Compressed air from a laboratory supply or cylinder of at least 60 psi is satisfactory. In no case should you exceed 100 psi otherwise internal components will fail. The compressed air is plumbed into the AutoDesorb Tower via a 1/8" Swargelok fitting on the back of the AutoDesorb Tower.

## **Cryogenic Cooling Gases**

The SIS 2" Cryo-Trap was specifically designed and is recommended for use with the AutoDesorb System. The SIS CryoTrap will produce optimum results with a minimal use of cooling gas. The cooling and heating times and temperatures are set up on the AutoDesorb method screen on the PC and controlled by the AutoDesorb<sup>TM</sup> Controller. The Cryo-Trap can be used with either Liquid Nitrogen or Liquid CO<sub>2</sub> depending on the Cryo Valve that was purchased with the system. GC cryo-cooling capabilities using liquid nitrogen will cool down to -180° C or carbon dioxide will cool down to -70° C.



**Liquid Nitrogen**. Liquid Nitrogen should be supplied in a large dewar, and liquid coolant at low pressure (30 psi) is recommended for optimum performance of the Cryo-Trap. When ordering Liquid Nitrogen specify that it is for Liquid use at Low pressure.

For the installation of the Liquid Nitrogen Cryo Valve, ¼" copper lines are used for all connections. The 1/4 inch lines should be connected from the Liquid Nitrogen Tank directly to the Cryo Valve. A Second ¼" copper line will connect the Cryo Valve to the Cryo-Trap inside the GC oven. These connections should always be made with 1/4 inch Swagelok<sup>™</sup> fittings. For optimum results the Liquid Nitrogen lines should be insulated and the gas source should be located as close as possible to Cryo valve system. In addition the Cryo valve should be located as close as possible to the GC injection port. By keeping all lines as short as possible and insulated as much as possible, cooling cycle times will be minimized and cryogen gas usage will be minimized. LN2 must be delivered as a liquid under low (<50 psi) pressure, and the line from the supply should be insulated ¼" metal tubing. Special evacuated or insulated coolant lines work well.



**Liquid CO**<sub>2</sub>. Liquid CO<sub>2</sub> should be supplied in an A size gas tank. When ordering be sure to specify that it is to deliver Liquid and make sure the tank has a DIP tube.

For installation of the CO<sub>2</sub> Cryo-Trap, a 1/8" stainless steel line provides the cooling gas from the tank to the Cryo Valve. This line does not need to be insulated, but for optimum performance should be kept as short as possible.Cryogen supplies should be located within 6-7 meters of the Cryotrap for best results. CO<sub>2</sub> should be delivered with 1/8" metal tubing, and must be in liquid form. Insulation is not necessary for liquid CO<sub>2</sub> supply lines. Tanks should be ordered specifically with an eductor or "dip" tube. Connect the tank to the supply line with a CGA #320 fitting (US) and a  $\frac{1}{4}$ " NPT (female) to 1/8" swagelok (male) adapter. A Regulator is



NOT used with a  $CO_2$  tank. The connection from the Cryo Valve to the Cryo-Trap is made with a 1.0 meter length of 1/16" stainless steel tubing with an I.D. of 0.040" for optimum performance. It is recommended that this line not be shortened or changed to achieve optimum performance of the Cryo Trap cooling.

Figure 6 - Liquid CO2 Gas tank fitting CGA320 Nut (left), Nipple (center), 1/8"Swagelok Adaptor (right) and Sealing Ring (foreground)



**Cryogenic Valves** are manufactured for specific gases and gas pressures. A  $CO_2$  Valve should only be used with  $CO_2$  and a LN2 Valve should only be used with LN2. Using the wrong gas type and pressure will destroy the Cryogenic Valve.

### **GC Carrier and Desorption Gases**

The same GC carrier gas that is used as your GC carrier gas is also used as the desorbing gas for the AutoDesorb<sup>™</sup> System. High purity gases such as nitrogen or helium are recommended. The carrier gas should have a purity of at least 99.995% and must be delivered at 40 to 60 psi. The use of a hydrocarbon, moisture and oxygen filters are recommended. The Triple gas filter available for SIS is recommended for use with the AutoDesorb system. It contains all three filters in one with visual indicators when it has expired.

The same carrier gas used for the chromatography column should be used as the desorbing gas in the AutoDesorb<sup>™</sup> System. In no case should two different gases be used. This avoids the mixing gases in the GC injection port which could cause unstable baselines. Due to The high temperatures and rapid heating of the components in the desorption system, the use of hydrogen could create an explosive condition.



CAUTION - DO NOT USE HYDROGEN GAS IN THE THERMAL DESORPTION SYSTEM

The next step in the AutoDesorb installation process involves preparing the gas chromatograph. Follow the steps outlined below:

1. Cool all heated zones, turn off all detectors, and set all inlet pressures and auxiliary flows to 0.



PARTS OF THE CHROMATOGRAPH MAY BE AT HIGH TEMPERATURE. TO AVOID BURNS BE SURE THAT THE INJECTION PORT AND DETECTORS/TRANSFER LINES HAVE COOLED BEFORE PROCEEDING WITH THIS SECTION.

2. Turn off all power and gas supplies to the gas chromatograph before proceeding with this section.

3. The AutoDesorb System is designed to use the GC's front inlet. If no inlet is present in the front position, one must be installed or moved there. Refer to Agilent Technologies technical support if you need assistance installing or moving an inlet.

4. Remove the septum nut and septum from the inlet. Open the inlet weldment, remove the inlet liner, and inspect the inlet for dirt, debris, etc. Replace the inlet liner, liner seal, bottom seal and septum as needed.

5. Remove all covers from the top of the instrument. For the 6890 these include the inlet and inlet fan covers, the hinged cover above the oven, and rear flow module cover. For the 5890 these include the hinged cover above the oven, the front panel on top of the oven, and the right-hand top cover

6. If the system is equipped with a Mass Selective Detector (MSD), move it away from the left side of the GC. Venting and shutting down the MSD is recommended before moving the instrument.

7. Remove left side cover from the GC.



## **CryoTrap Installation**

If a Cryotrap is to be used with the system, it should be installed first. The Cryotrap may be installed on either the front or rear inlet; however, the AutoDesorb system is designed to operate with the front inlet only. When installing any SIS Cryotrap, pay particular attention to the region where the heater and thermocouple cables enter the Cryotrap body. There is a springtype strain relief mechanism at this point. It is important that the cable is not twisted or kinked in the region of the strain relief spring, as this may result in irreparable damage to the instrument.

- 1. Locate and unpack the Cryotrap and installation kit.
- 2. Remove the column from the inlet and cap it with a septum.

3. Remove insulation cup from the bottom of the injection port, exposing the reducing nut. Completely remove the two screws that hold the insulation cup.(Figure 7)



**NOTE-** These screws are subject to frequent temperature changes and have been known to bind in their holes. When extracting them, use care not to strip the threads.



Figure 7- Remove Insulation Cup from Inlet

4. Remove the Cryotrap from the mounting assembly by sliding the trap off of the end of the guide rods.

5. Place a portion of the ring-shaped insulation from the original cup in the insulation cup of the Cryotrap mounting assembly (Figure 8). Loosely mount the assembly to the top of the GC oven in place of the original insulating cup using the two screws (use of a graphite-based thread lubricant such as Neolube is highly recommended). Use a spare column nut in the injection port to center the mounting assembly. Rotate the cup so that the slide portion of the assembly is closest to the left wall of the GC oven, and align the hole in the insulating cup with the bottom of the reducing nut. Tighten the two screws to hold the Cryotrap mounting bracket firmly in place.

## AutoDesorb<sup>™</sup> Manual





Figure 8 - Install Insulation in Cryotrap and mount in GC oven

6. Run the heater/thermocouple cable outside of the oven to the rear of the GC. A spare injection port can be used for this purpose, although one of the holes in the left-side oven wall is recommended (Figure 9). In order to pass the cable through one of these holes, the connectors may need to be removed. Pay particular attention to the wiring of the connectors if they are removed, and immediately reattach them to the cable once it is through the oven wall. Leave 6-8 inches of slack in the cable inside the oven to allow the Cryotrap to slide up and down the guide rods freely.



Figure 9 - Run wires through hole in side of the GC (Note LN2 Lines also exit the side of the GC Oven)

7. Install the Cryotrap onto the mounting assembly by inserting the guide rods into the holes in the Cryotrap body and sliding the unit up onto the rods. Orient the trap so that the heater and thermocouple leads are at the top of the unit (Figure 9). Install the Cryotrap clamp under the Cryotrap. Position the top of the Cryotrap approximately 1 to 2 cm from the bottom of the insulation cup and tighten the clamp (Figure 10).



TO AVOID PERSONAL INJURY, THE CRYOGEN EXIT MUST BE DIRECTED TO THE REAR OF THE GC OVEN!





8. For installation on the Agilent 5890 GC, the small stop collar may be installed at the level of the MS transfer line to prevent the Cryotrap from accidentally breaking the column by sliding down too far.

Figure 10 - Stop Colar an Clamp for Cryo-Trap

#### LN2 Lines Exit GC Oven through spare Injection Port

9. Plumb cryogen supply and exhaust lines from the Cryotrap outward. A spare injection port may be used to plump the LN2 lines (Figure 11); however, use of the holes in the left oven wall makes for a much neater installation (see Figure 9). Cryogen should be supplied to the Cryotrap through the ¼" swagelok opening that points toward the right side of the GC. The other ¼" swagelok fitting (pointing toward the rear of the GC) should be used for venting the trap, if necessary. Installing the Cryotrap in another orientation may result in failure of the Cryotrap, or inability for the GC to control oven temperature.

Figure 11 - Cryotrap installed in oven (Note LN2 lines exit through the top of GC Oven in this alternate installation)

\*NOTE - Liquid N2 (LN2) coolant must be supplied by 1/4" tubing (flexible copper is best) and must be vented from the Cryotrap outlet outside the oven. A short piece of tubing run through the same hole as the coolant supply is sufficient.



DIRECT THE LN2 VENT LINE AWAY FROM AREAS WHERE PERSONAL CONTACT WITH THE VENTED COOLANT IS POSSIBLE. INSURE ADEQUATE VENTILATION IN AREAS WHERE LN2 COOLANT IS USED.

Cryo Gas

IN

**Cryo Gas** 

**Exhaust** 



\*NOTE - Liquid CO<sub>2</sub> coolant is supplied from the valve through 1/16" stainless steel tubing. It is generally not necessary to vent the Cryotrap when using CO2 unless difficulty controlling oven temperature is encountered.



AVOID CONTACT WITH LIQUID CO2. NEVER OPEN THE GC OVEN WHILE CRYOTRAP IS IN COOLING MODE. INSURE ADEQUATE VENTILATION IN AREAS WHERE LIQUID CO $_2$  COOLANT IS USED

10. Run cryogen supply and exhaust lines as well as the heater/thermocouple cable along the side of the GC toward the rear of the instrument. Cutouts in the back plate may be removed to make room for the lines to exit the back of the GC.



11. Connect the cryogen supply tube to the outlet of the appropriate valve. When using LN2, insulate the valve body and the length of tubing that runs from the valve to the oven wall. Plumb the valve to the cryogen supply.

12. Lower the Cryotrap a few inches by loosening the clamp and sliding the unit down the guide rods. Reinstall the column, remembering to run it through the Cryotrap before assembling the nut and ferrule on the inlet end. When the column has been installed, raise the Cryotrap so the top of the unit is 1-2 cm from the bottom of the insulating cup. Position the clamp to hold the Cryotrap in place and tighten.

Figure 12 - Column installation through cryotrap



## **Carrier Divert Valve Installation**

On the 6890 and most 5890 GCs the desorption purge and carrier divert valves are mounted to the left side of the GC under the plastic cover. On some 5890 models with non-EPC controls, there is not enough room on the left side to mount the valves. In this case, they should be mounted on top of the GC oven where space permits, as close as possible to the front inlet (see Figure 28).



Figure 13 - Attaching mounting bracket to valve set

Locate and unpack the carrier valve assembly (Part # 7819993) in the installation kit. Attach the mounting bracket as shown in Figure 13.

#### Installation of Carrier Gas Valve in 6890 GC

Locate the drilled and tapped hole in the left side of the GC chassis alongside the rear inlet and about11/2 inches down from the top. Mount the valve assembly as described above with the 3 mm Allen screw and washer supplied (Figure 14 and 15). Run the valve assembly cable out the rear of the GC along with the Cryotrap cable and coolant lines.



Figure 14 - Locating Hole for mounting Valve Assembly on Side of GC





Figure 15 - Fastening Valves to side of GC (Carrier Gas Line is extended to its maximum length)

Locate the carrier gas supply line running from the EPC module to the front inlet. Verify the identity of the line by checking its origin and outer diameter (carrier supply is 1/16" OD tubing, septum purge is 2 mm OD tubing). The carrier supply and septum purge tubing form a loop that hangs down into an opening in the GC chassis (Figure 14) at a point even with the front injector. Pull the carrier loop out, leaving the septum purge as it is. Using the extra tubing supplied by the loop, stretch the carrier line and cut it (Figure 15) so that the end coming from the EPC control will fit into the inlet of the three-way valve. Insert the weldment end of the tubing through the hole in the chassis where the septum purge loop is, and attach it to the outlet of the three-way valve (Figure 16).



Figure 16 - Plumbing Valve Assembly in carrier gas line





Carrier Gas IN

Locate the main carrier gas supply at the back of the GC. Cut the 1/8" tubing just before it attaches to the inlet GC module, and insert one of the supplied brass Swagelok Tees. Tighten the connections <sup>3</sup>/<sub>4</sub> turns past finger tight with a 7/16" wrench (Figure 17).



DO NOT USE HYDROGEN GAS. The AutoDesorb system is not explosion proof. Instrument damage and personal injury can occur.

Cut a piece of 1/8" flexible copper tubing to about 24" long, and connect one end to the tee installed in the main GC carrier supply line. Pass the other end of the tubing through an opening in the rear plate of the GC, and along the left side. Connect the tubing to the inlet of the Desorption Purge valve with the Swagelok fitting provided, and tighten both connections with a 7/16" wrench.

Carrier Gas to Desorption -System

Carrier Gas from T-valve Assembly



Figure 18 - Plumbing purge supply to valve assembly 6890







**Carrier Gas to** Desorption System



Locate the length of 1/8" PTFE tubing included in the installation kit. Find the middle of the tubing, and cut it in half. Take care to leave a square end on the cut. Attach an end of one of the pieces to the last remaining 1/8" connection on the carrier valve assembly, and tighten (Figure 18). Route the tubing toward the back of the GC, running it under other wires



and tubing present. Run the tubing up through an opening in the GC chassis behind the injection port cooling fan (Figure 19). Avoid routing the tubing where it will interfere with the instrument covers when they are replaced. Place the desorption unit on the bench or other convenient location within 3 feet of the GC inlet and attach the end of the tubing to the port labeled GAS on the rear of the desorption unit (Figure 20).

**Desorption** System from Valve Assembly

Figure 20 - Attaching Carrier Gas Line to back of AutoDesorb Tower



Turn on the carrier gas to the instrument, pressurize the front inlet and check all plumbing connections for leaks.



Figure 21 - AutoDesorb Installed on the 6890 GC with 5973 MSD

Replace all instrument panels and covers. Attach AutoDesorb tower onto the 6890 GC injection port plate (Figure 21).

The figures on the next page (Figure 22) show the relative positions of the various components of the Carrier Divert Valve Assembly into the GC injection flow system. The system is designed such that in the normal operation, carrier gas flow is flowing into the GC injection port and GC column in a normal mode of operation. When the valve are switched, the carrier gas flow is diverted through the desorption tube and then into the GC injection port.













#### Installation of Carrier Gas Valve in 5890 GC with EPC

Locate the hole in the plastic chassis on the left side of the GC as indicated in Figure 23. Pressure controls for non-EPC inlets may be present in this location, in which case an alternate location for the valve assembly must be found. On some 5890 models with non-EPC controls, there is not enough room on the left side to mount the valves. In this case, they should be mounted on top of the GC oven where space permits, as close as possible to the front inlet (see Figure 25). Any open space on top of the GC oven close to the injection port may be used . Run the valve assembly cable to the rear of the GC, avoiding contact with heated zones.



Figure 23 - Left side of 5890 GC - Locating Carrier Gas Line

Locate the carrier gas supply line running from the EPC module to the front inlet. Verify the identity of the line by checking its origin and outer diameter (carrier supply is 1/16" OD tubing, septum purge is 2 mm OD tubing). Cut the supply line (Figure 23) so that the three-way valve in the valve assembly may be inserted (Figure 24). Verify that the EPC supply is connected to the inlet of the valve, and that the injection port is connected to the valve outlet.





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Tighten the connections to the three-way value  $\frac{3}{4}$  turn past finger tight using a 5/16 inch wrench.

Locate the main carrier gas supply to the GC. Cut the tubing just before it enters the GC, and insert one of the supplied brass Swagelok Tees. Tighten the connections <sup>3</sup>/<sub>4</sub> turns past finger tight with a 7/16" wrench (Figure 24).



Figure 25 - Valve Assembly Mounted on side of 5890 GC



**DO NOT USE HYDROGEN GAS**. The AutoDesorb system is not explosion proof. Instrument damage and personal injury can occur.

Cut a piece of 1/8" flexible copper tubing to about 24" long, and connect one end to the tee installed in the main GC carrier supply line (Figure 24). Pass the other end of the tubing through an opening in the rear plate of the GC, and along the left side or along the top, depending on where the carrier valve assembly has been installed. Connect the tubing to the inlet of the desorption purge valve with the Swagelok fitting provided, and tighten both connections with a 7/16" wrench (Figure 25).



Locate the length of 1/8" PTFE tubing included in the installation kit. Find the middle of the tubing, and cut it in half. Take care to leave a square end on the cut. Attach an end of one of the pieces to the last remaining 1/8" connection on the carrier valve assembly, and tighten (Figure 25). Route the tubing out of the back or side of the GC so that it will not interfere with the instrument covers or panels when they are replaced. The free end of the tubing should be able to reach the area around the front inlet (Figure 26).

Carrier Gas Line for AutoDesorb System



Figure 26 - Routing Gas Line for AutoDesorb System

Place the desorption unit on the bench or other convenient location within 3 feet of the GC inlet and attach the end of the tubing to the port labeled GAS on the rear of the desorption unit (Figure 27) .



Turn on the carrier gas to the instrument, pressurize the front inlet, and check all plumbing connections for leaks.

Figure 27 - Attaching Carrier Gas Line to back of AutoDesorb Tower

On some 5890 models with non-EPC controls, there is not enough room on the left side to mount the valves. In this case, they should be mounted on top of the GC oven where space permits, as close as possible to the front inlet (Figure 28). Any open space on top of the GC oven close to the injection port may be used . Run the valve assembly cable to the rear of the GC, avoiding contact with heated zones.



Figure 28 - Alternate Installation of valve Assembly on Top of 5890 GC

Replace all instrument panels and covers. In place of the front panel along the top of the oven, use the replacement fromt Plate from the installation kit. When replacing the hinged cover above the oven, install the cover support and AutoDesorb baseplate in place of the original tubular hinge. First install the cover support by removing the screw from the right side of the injection port fan cover, and sliding the square end of the support between the fan cover and the top of the oven (Figure 29).

Injection Port Cooling Fan



Figure 29 - Installing Hinge Assembly on 5890 GC





Replace the screw to hold the fan cover and oven cover support in place. Install the AutoDesorb baseplate by fitting the left-hand hinge over the molded plastic pivot, and securing the right-hand hinge to the cover support and hinged oven cover using the screw provided (Figure 30).



Figure 30 - Installing the Baseplate on the 5890 GC

Secure the remaining hinge on the oven cover using the original screw and washer. Remember to attach the grounding wire between the oven cover and the GC chassis.



Figure 31 - Baseplate installed on 5890 GC





Figure 32 - Baseplate and new front plate installed on 5890 GC



Figure 33 - AutoDesorb system mounted on top of 5890 GC





## **Desorption Unit Installation**

Original

## Septum Nut Replacement

Install the replacement septum nut (Part # 786900) on the GC inlet. Place the desorption unit onto the GC injection port. Align the desorption unit so that the counterbore in the mounting plate on the bottom fits onto the septum nut adapter (Figure 34).



Septum Nut for AutoDesorb System

Figure 34 - Installing New Septum Nut on GC Injection Port

#### Gas Lines



The back of the desorption unit is shown in Figure 35. It has two 1/8" Swagelok™ fittings for connection of the carrier gas and the compressed air that is used to operate the mechanical motion of the injection system. They are labeled AIR and GAS. As illustrated, connect the free end of the PTFE tubing from the carrier gas solenoid valve assembly to the "GAS" fitting on the back of the desorption unit if not already conncted as described previously. Be sure that the tubing is fully seated in the fitting and then tighten 3/4 turn past finger tight.

Cut an appropriate length of the PTFE tubing to connect the fitting labeled "AIR" on the desorption unit to the compressed air supply. Be sure the fitting is tight and leak free.

Figure 35 - Back of AutoDesorb Tower



## **Connection to Controller**

Plug the interconnect cable (Part #782010) into the back of the desorption unit. The cable is reversible so either end can be plugged into the desorption unit. See Figure 36. In the same manner, plug one end of the heater cable into the desorption unit.



Figure 36 - Interconnect Cable Plug

**Controller Installation** 

The controller can be positioned next to the desorption unit as shown in or can be placed on the bench next to the GC (as shown in Figure 21).

Figure 37 shows the back of the controller for the connection of the various cables.



Figure 37 - Back of AutoDesorb Electronics Controller

#### **Computer Connections**

Confirm that all power to the computer and gas chromatograph is off. Attached the RS232 cable supplied (P/N CBLMNT9) to a COM port on the rear of the computer. A 24 to 9 pin adapter (P/N GENDER259) may be used, if the only available COM port on the computer is a 24 pin connection. Attach the other end of the RS232 cable to the rear of the controller as shown in Figure 37. Note which of the computer's COM ports the cable was attached to (e.g. COM1:, COM2:, etc.). This information will be helpful during software installation.

### **Controller Connections**

Plug the interconnect cable from the AutoDesorb tower into the appropriate connection on the back of the controller as shown in Figure 37.

Plug the cable from the GC carrier gas divert solenoid valve into the fitting labeled "GC Valve" on the back of the controller.

Plug the remote cable into the fitting labeled "6890 Remote ".



NOTE: Due to an engineering change, the plug labeled "5890 Remote " is not used. Use the plug labled "6890 Remote" for the 5890 GC too.

Attach the Cryotrap cable to the plug labeled "Cryo\_Trap".

Plug in the power cord from the back of the controller into a grounded 115V or 230V, 10 Amp outlet.



VERIFY VOLTAGE. Before connecting power cord be sure to check and verify the voltage of the power and adjust the imput module on the AutoDesorb Electronics control as appropriate for the power being utilized.

Return power to the gas chromatograph and computer.



## **Software Installation**

#### Introduction

The AutoDesorb Software controls almost all aspects of the system operations. It supports a graphical user interface that allows the user to input methods and sequences in a simple and efficient fashion. The Software also fully integrates with the Agilent ChemStation system. This permits coordination of methods, synchronization between the programs, and logging of analyses.

The AutoDesorb Software supports releases of MS ChemStation after version G1701AA (including G1701CA) and versions of GC ChemStation after version G2070. Every attempt is made to support new versions as they are released by Agilent. For the latest information see the readme.txt file.

The AutoDesorb Software can be Uninstalled using the Windows Remove Programs feature. See the readme.txt file for further information.

In addition to the AutoDesorb Control Software, the installation CD includes a large array of reference material related to thermal desorption techniques. These include application notes, data on adsorbent resins, and an electronic copy of the Operation Manual.

Note that Agilent ChemStation software must be installed before installing the AutoDesorb Control Software. If a new version of ChemStation is installed, the AutoDesorb Control Software must be reinstalled.

#### **Computer Requirements**

Pentium Processor or greater Operating System - Windows NT or 95 3 MB available on Hard Disk Spare RS232 serial Port Required

#### Setup



The installation CD is located at the back of this manual. The disk includes a readme.txt file in its root directory. This file contains the latest information about the software.

Place the disk in a CD-ROM drive of the computer you on which you have installed ChemStation. The CD should automatically display the CD Browser shown in Figure 38. If this does not occur within 15 seconds, launch the browser by clicking on the Windows Start button and selecting "Run". Browse to the file"Start.exe" in the root directory of the CD-ROM and select it for execution.





The CD-Browser offers three primary options. The first is installation of Software components from the AutoDesorb<sup>™</sup> Software Library. The second is to access SIS application notes. Most of these cover topics related to thermal desorption. The third option is to view information regarding the use and characteristics of absorbent resins. Options two and three will display HTML pages. They will open more quickly if your browser is opened first.

Before beginning software installation it is recommended that you close any other programs that may be running.



Figure 39 - AutoDesorb Initialization

Clicking on the "Install Software" text will launch the library installation program. The first two steps in the installation process are shown in Figures 39 and 40. The window shown in Figure 40 requests an acknowledgement that you agree to the SIS License Agreement shown. This agreement is also printed on pages 4-6 of this manual. By clicking "yes" you are subject to the terms of this agreement.

51	S License Agreement
	The following license agreement covers use and distribution of this software.
	Scientific Instrument Services, Inc. Software License Agreement
	Be sure to fill out and send in the enclosed registration card. This is required for technical
	The information below includes important limitations to your right to copy or distribute any of the software included on this disk or installed by this software. It informs you that the Software is protected under U.S. Copyright Law. Licensee may copy it only to make a backup copy. If any component of this software is installed on more than one computer you must license a separate copy for each user. If Licensee's computer system is a local area network or wide area network, Licensee must license those respective versions of the Software. If you do not agree with the terms and conditions of this agreement, close this installation and return the complete package to Scientific Instrument Services, Inc.
	Do you agree to use this software under the terms of this agreement?
	< <u>B</u> ack <u>Y</u> es <u>N</u> o

Figure 40 - AutoDesorb Software License



The next step in the installation process is selecting the path to your Agilent ChemStation data system. The installation program will search your computer for possible paths and display the first one that it finds. See Figure 41. Note that a data system is only required by the main AutoDesorb<sup>™</sup> Control Program. If you are not installing this component of the AutoDesorb Library, you can click on "Next" regardless of the path shown. If you have multiple copies of Agilent ChemStation installed on your system and the one shown is not the one that will be associated with the AutoDesorb<sup>™</sup> system, then browse to the correct directory. Once the proper directory is selected, click "Next".



Figure 41 - AutoDesorb Software Setup Screen

If no data system is found on your computer or if you enter a path to an unsupported data system, the screen shown in Figure 42 will be displayed. It lists versions of Agilent ChemStation that are specifically supported by the AutoDesorb<sup>™</sup> Control Program. None of the versions released by Agilent before those shown on this screen will function with the AutoDesorb<sup>™</sup> systems. Please contact SIS for information about upgrading your version of Agilent ChemStation.



Figure 42 - AutoDesorb Software Warning Screen



If you have a more recent version of Agilent ChemStation than those shown, then this version of the AutoDesorb<sup>™</sup> control program may still be compatible. In this case, read the section below regarding installation on an unsupported data system.

#### Installation on an unsupported data system

When Agilent releases a new beta or production version of their ChemStation data system SIS immediately begins testing for compatibility with our software. In many cases on changes or only minor changes are required.

When Installing the AutoDesorb<sup>™</sup> control program on an unsupported data system the following points may be helpful.



Contact SIS. We may have new information regarding your version of ChemStation. You will have to specifically click on the check box for the AutoDesorb Control Program in the components selection window (Figure 44)

If the AutoDesorb Control Program does not work with your version of ChemStation you will have to manually restore the ChemStation macros.

The next step in the installation process is the selection of a destination directory for the SIS software shown in Figure 43. The default is C:\SIS\. If another directory is preferred use the "Browse" button to select it. It is recommended that all components of the AutoDesorb<sup>™</sup> Software Library be stored in the same directory.



Figure 43 - AutoDesorb Screen for Selection of Destination

The next screen will allow you to select various components form the AutoDesorb<sup>™</sup> Software Library. If a supported data system has been found the AutoDesorb Control Program will be checked by default. The space requirements on your system will be different than those shown in Figure 44. Each of the available components is summarized below.





Figure 44 - AutoDesorb Components Selection Window

## AutoDesorb<sup>™</sup> Control Program

The main program for controlling the automated thermal desorption hardware. This program will integrate closely into Agilent ChemStation.

-Thermal Desorption Application Notes

Approximately 80 application notes. These may be left on the CD by removing the checkmark, and later examined using the CD-browser. They are also available at the SIS web site at www.sisweb.com

-On-line Help Files Help information that can be accessed from within the AutoDesorb<sup>™</sup> Control Program.

-Resins Data Information on the use of Adsorbent Resins

-Manual

A copy of the Operation Manual in electronic format.



Figure 45 - AutoDesorb Verify Information Screen



Before beginning file transfer the installation program will display the selected setting as illustrated in Figure 45. Click "Next" to begin File transfer. The progress will be displayed on the screen.

After the File transfer the installation program will attempt to make changes to the Agilent ChemStation Macros. If any errors occur in this process, notices will be displayed on the screen. If this occurs, contact SIS for technical support.

The original macros will be saved to a folder labeled ads\_backup in the main ChemStation executable directory (hpchem/CORE/, hpchem/MSEXE/ or msdchem/MSEXE/). Details of the files effected can be found in the readme.txt file.



IMPORTANT-BEFORE REINSTALLING THE ADS SOFTWARE, RENAME THE /ADS\_BACKUP/ FOLDER .

The next screen, Figure 46, allows the user to select the Window Program Folder. "AutoDesorb Software Library" is recommended.



Figure 46 - Select Program Folder

The final Setup window, Figure 47 gives the user the option of viewing the readme.txt file and executing the config.exe program. Both of these are highly recommended. Execution of the config.exe program is discussed below.





Figure 47 - View Readme text file

### Configuration

We recommend that a separate online ChemStation instrument be configured for use with the AutoDesorb system. Below are procedures to follow for configuring instruments with the GPIB and LAN ChemStations.

#### **GPIB ChemStation**

- 1. Perform an Autotune if you are running an MS instrument.
- 2. Close ChemStation.

📲 System Configuration				
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			▼ Execute	
		Current System Configurat	tion	
Inst#	Name	Mass Spectrometer	Gas Chromatograph	Data Analysis
	Instrument #1	5973 (HP.IB = 20)	6890 (HDJR = 15)	Enhanced
1		poro (n -10 - 20)	)	
2	MS Probe	5973 (HP-IB = 22)	<none></none>	Enhanced
3	Instrument #3	5973 (HP-IB = 20)	6890 (HP-IB = 15)	Enhanced
4	Instrument #4	5973N (LAN = 0.0.0.0)	6890 (LAN = 1.0.0.0)	Enhanced
	1			
				<b>•</b>
4				



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Instrument	GC/MS Instrument #1	
<u>Offline Instr</u>	rument	
<u>Mass Spectrome</u>	neter <u>G</u> as Chromatograph	
5973	▼ ● 6890	
	O 5890	
H <u>P</u> -IB Addres	ss 20 O A/D 35900 C 🗹	
MS Options	O Other	
Electron I	Ionization O None	
🗌 🗖 <u>C</u> hemical	I Ionization HP- <u>I</u> B Address 15	
	Autosampler	
	O 7673 + Tray	
<u>D</u> ata Analysis M	Mode Other or None	
Enhanced Que	HP-IB Address 8	
OK	Cancel Help	

4. View configuration of current instrument(s), make note of HPIB addresses, CI options, etc.

5. Configure an additional online instrument with identical HPIB information except autosampler, if present.

6. Save the instrument configuration, and enter DC polarity information if requested (MS systems). Get polarity information from the tune report.

7. Top and Data Analysis icons for the new instrument may be moved to desktop. Exit ChemStation configuration editor.

- 8. Start SIS/ADS configuration editor.
- 9. Select the new instrument for use with ADS and assign the appropriate COM port.
- 10. Save settings and exit the ADS configuration editor.
- 11. Start the original instrument and verify that ADS software DOES NOT start.
- 12. Close ChemStation
- 13. Start new instrument and verify that ADS software starts automatically.

AutoDesorb Configuration				
Select the GC instrument that should use the AutoDesorb system:	Select the serial (COM) port on which the SIS controller is connected:			
<ul> <li>Instrument #1</li> <li>Instrument #2</li> <li>Instrument #3</li> <li>Instrument #4</li> </ul>	COM1			
Save Changes Cancel				



For help with the installation or if you encounter problems, call SIS tech support at (908) 788-5550.

### LAN ChemStation (5973N)

The LAN-enabled MSD ChemStation (G1701CA) allows for the configuration of a maximum of two LAN and two GPIB instruments. Configurations that already use two instruments with the same interface will require that one of them be shared with the ADS. Switching between the ADS and another operational mode may require reconfiguring through the ChemStation configuration editor each time if an element such as a 7673 ALS is to be used in the other mode. Alternatively, if there are no conflicts in the configuration (autosampler, etc.) the ADS configuration editor may be used to enable and disable the ADS without reconfiguring the ChemStation instrument. ChemStation will need to be restarted each time the change is made. The following steps should be followed with ChemStation version 1701CA to configure a new instrument for use with the ADS.



1. Perform an Autotune if you are using an MS Instrument.

2. Close ChemStation.

3. Open the ChemStation configuration editor from the Productivity Tools section of the Windows Start menu.

4. View configuration of the current instrument(s) and make a note of HPIB and LAN addresses, CI options, etc.

5. Configure another online HPIB or LAN instrument using addresses for GC, MS, etc. identical those used for the existing hardware. Do not configure an autosampler.

6. Save the instrument configuration and enter DC polarity information if requested (MS systems; get polarity info. from the tune report).

7. The Top and Data Analysis icons for the new instrument may be moved to desktop. Exit ChemStation configuration editor.

8. Start the SIS/ADS configuration editor.

9. Select the new instrument number for use with the ADS and assign the appropriate COM port.

10. Save settings and exit the ADS configuration editor.

11. Start the original instrument and verify that the ADS control software DOES NOT start.

- 12. Close ChemStation.
- 13. Start the new instrument, verify that the ADS software starts automatically.

For help with the installation or if you encounter problems, call SIS tech support at (908) 788-5550.

#### **Multiple Installations**

To support several AutoDesorb<sup>TM</sup> systems it is necessary to install several copies of the AutoDesorb<sup>TM</sup> Control Program. Install each in a different destination directory. Be sure that the correct instrument numbers and COM ports are selected in the configuration.

#### Uninstallation

To remove the ADS software from your computer, use the Add/Remove Programs feature in the Control Panel window. The Control Panel window is accessible from the Windows start menu. Some program elements may need to be removed manually. After removing the program, changes made to ChemStation macro files will still be in place. Refer to the Readme.txt file on the installation CD for information about restoring the original macros.