SHORT PATH THERMAL DESORPTION

Model TD-4

Manual

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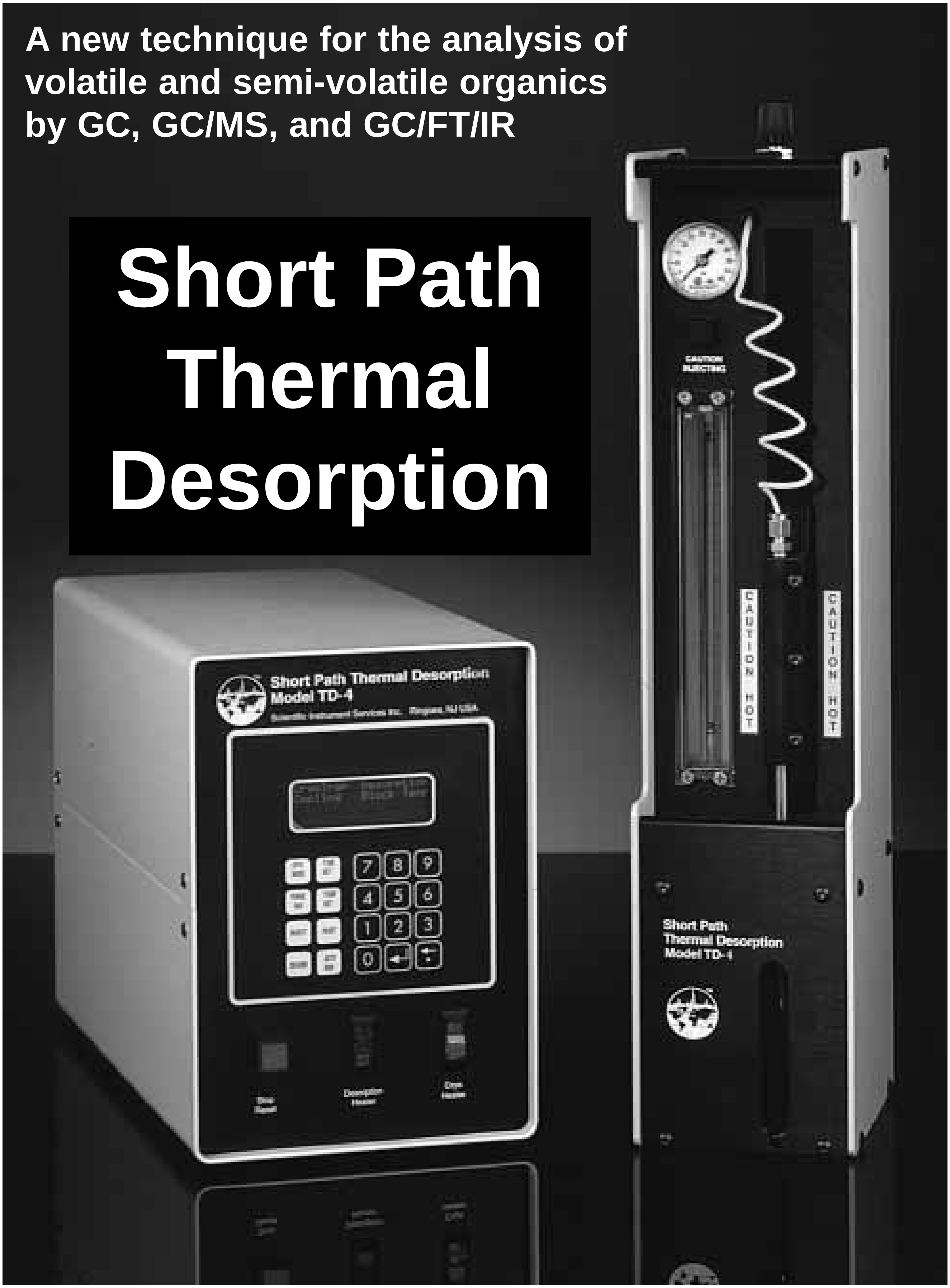
A new technique for the analysis of volatile and semi-volatile organics by GC, GC/MS, and GC/FT/IR

Short Path Thermal Desorption



NOTICE

The information contained in this document is subject to change without notice.

Scientific Instrument Services (S.I.S.) makes no warranty of any kind with regard to the material contained in this manual, including, but not limited to, the implied warranties of merchantability and fitness of the equipment and techniques therein described for a particular purpose.

S.I.S. shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, operation, performance or use of the Short Path Thermal Desorption System described in this manual.

S.I.S. assumes no responsibility for the use or reliability of its equipment that is not furnished by S.I.S.

Patents covering the design, operation, techniques, and unique features of the Short Path Thermal Desorption System are pending.

U.S. Patent #5,065,614, Nov. 19, 1991

Patent Applications:	U.S. 560,440
	U.S. 696,875
	U.S. 696,872
	U.S. 696,869

Safety Information

WARNING Connecting the Thermal Desorption System to a power source which is not equipped with a protective earth ground contact creates a shock hazard for the operator and can damage the instrument.

WARNING Make sure that only fuses with the required current rating and of the specified type are used for replacement. The use of incorrect or makeshift fuses or the short-circuiting of fuse holders creates a shock hazard for the operator and can damage the instrument.

WARNING Any adjustment, maintenance or repair of the opened instrument while it is connected to a power source should be avoided if possible and, if required, should be carried out only by trained persons who are aware of the hazards involved.

WARNING High Temperatures - Keep hands and fingers from inside the cabinet. The Short Path Thermal Desorption Unit contains high temperature moving parts that will seriously burn hands or fingers.

WARNING After samples have been heated and desorbed and the desorption tube is withdrawn from the injection port, the desorption tube and needle will remain hot until they are permitted to air cool to room temperature. It will usually take from 5 to 10 minutes until this assembly has cooled to where it is touchable. In no case should this tube be touched or removed until it has cooled for a minimum of 5 minutes.

WARNING Do NOT use HYDROGEN GAS in the Short Path Thermal Desorption System. The rapid rise of gases to high temperatures does not permit the use of explosive gas mixtures.

WARNING Do not leave Desorption unit heaters in the heated ON position unattended overnight. The Desorption unit heaters rapidly heat and cool to their final operating temperatures and therefore, in order to prolong their life, should be turned off when not actively being used to heat samples for analysis.

WARNING Do not desorb samples above 350^{o} C. Exceeding this temperature may damage thermal desorption blocks.

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I. General Information

Introduction - Theory of Operation

The technique of Short Path Thermal Desorption has been developed to permit the analysis of organic compounds present in air or compounds which can be easily purged from solid and liquid matrices. Samples such as volatile and semi-volatile organics in air, flavors and fragrances in foods and cosmetics, manufacturing chemical residues in pharmaceuticals, volatiles in packaging materials and building products, and aromatic residues in forensic arson samples are just a few of the applications to which this technique has been adapted. Samples to be analyzed are collected on GLT desorption tubes containing an adsorbent resin such as Tenax™ TA or activated carbon. When ready for analysis the GLT desorption tubes are fitted with a syringe needle and attached to the Desorption Unit **(Figure #1-1)**.

The desorption tube containing the sample is then injected into the GC injection port **(Figure #1-2)** and the desorption tube heater blocks are closed around the tube **(Figure #1-3)**. This permits the samples to be heated by the desorption tube heater blocks, desorbed from the adsorbent resin and injected directly into the injection port of a Gas Chromatograph, GC/MS or GC/FT/IR by the shortest path possible, i.e. direct injection into the GC much like a syringe **(Figure #1-4)**. The GC column (either capillary or packed) is normally maintained at subambient temperatures (or at a suitable temperature low enough to retain any volatiles at the front of the GC column) during the initial desorbing of the sample into the GC. This is accomplished by cooling the entire GC oven, or preferably by using the optional SIS Micro Cryo-Trap. This enables the desired components to be collected in a narrow band on the front of the GC column over a long period of time (5 to 15 minutes). When the sample has been fully desorbed into the GC column, temperature programming is commenced to volatilize the organics and to elute and separate them. The new Short Path Thermal Desorption System permits the analysis of samples by desorbing the samples directly into the gas chromatograph injection port for subsequent analysis by conventional GC detectors or by mass spectrometers thereby permitting the maximum delivery of samples to the GC which will result in the optimum sensitivity of analysis.

Short Path Thermal Desorption

The technique of Short Path Thermal Desorption (SPTD) is also commonly known as Purge and Trap Thermal Desorption (P&T-TD) and is widely used in US-EPA methodology including EPA methods 524 and 624 for water analysis. In this technique, volatiles and semi-volatiles are trapped on adsorbent resins inside the desorption tube and then are subsequently thermally desorbed into the gas chromatograph.

Direct Thermal Extraction

An alternative method of analysis using the Short Path Thermal Desorption System is called Direct Thermal Extraction (DTE). This technique permits the analysis of low moisture content samples which have been placed directly in the GLT desorption tubes. Samples such as spices, paint chips, pine needles and fibers can be analyzed using this technique. Water vapor must be minimized since it will condense at the front of the GC column (if kept at subambient temperatures) and possibly plug the GC capillary column.

Description of System

The Short Path Thermal Desorption consists of two modules: a microprocessor controlled Electronics Console and the Desorption Unit. The Desorption Unit is placed directly on top of the injection port of most GC's, where it is used for the direct desorption of samples into the GC injection port and column, providing for the optimum delivery of samples (maximum sensitivity) to the GC by the shortest path possible. **(Figure #1-1)**

The Thermal Desorption Unit sits directly over the GC injection port where the septum nut on the GC slips into a groove in the bottom plate of the Desorption Unit to correctly align the system for injection. No mounting hardware, screws or bolts are required to install the Desorption System. On some systems it may be necessary to add an accessory plate around the injection port to provide a stable base on which the Desorption Unit can sit **(see page 3-1)**. The septum nut groove and the weight of the unit hold the Desorption Unit in place during injection and analysis of samples. The Desorption Unit can be easily lifted off the injection port of the GC for conventional injection of samples by syringe or autosamplers, and can then be easily slipped back onto the injection port nut for desorbing samples into the injection system.

The microprocessor controlled Electronics Console **(Figure 1-5)** connects to the Desorption Unit by a single electronics cable. It contains the microprocessor and keypad to control heating and cooling of the desorption tube. It controls the injection of the desorption tube into the GC as well as the carrier gas flow through the desorption tube and remotely starts the GC. It also controls the operation of the Micro Cryo-Trap accessory.

Short Path Thermal Desorption

Fig. 1-1

Injecting into
Gas Chromatograph

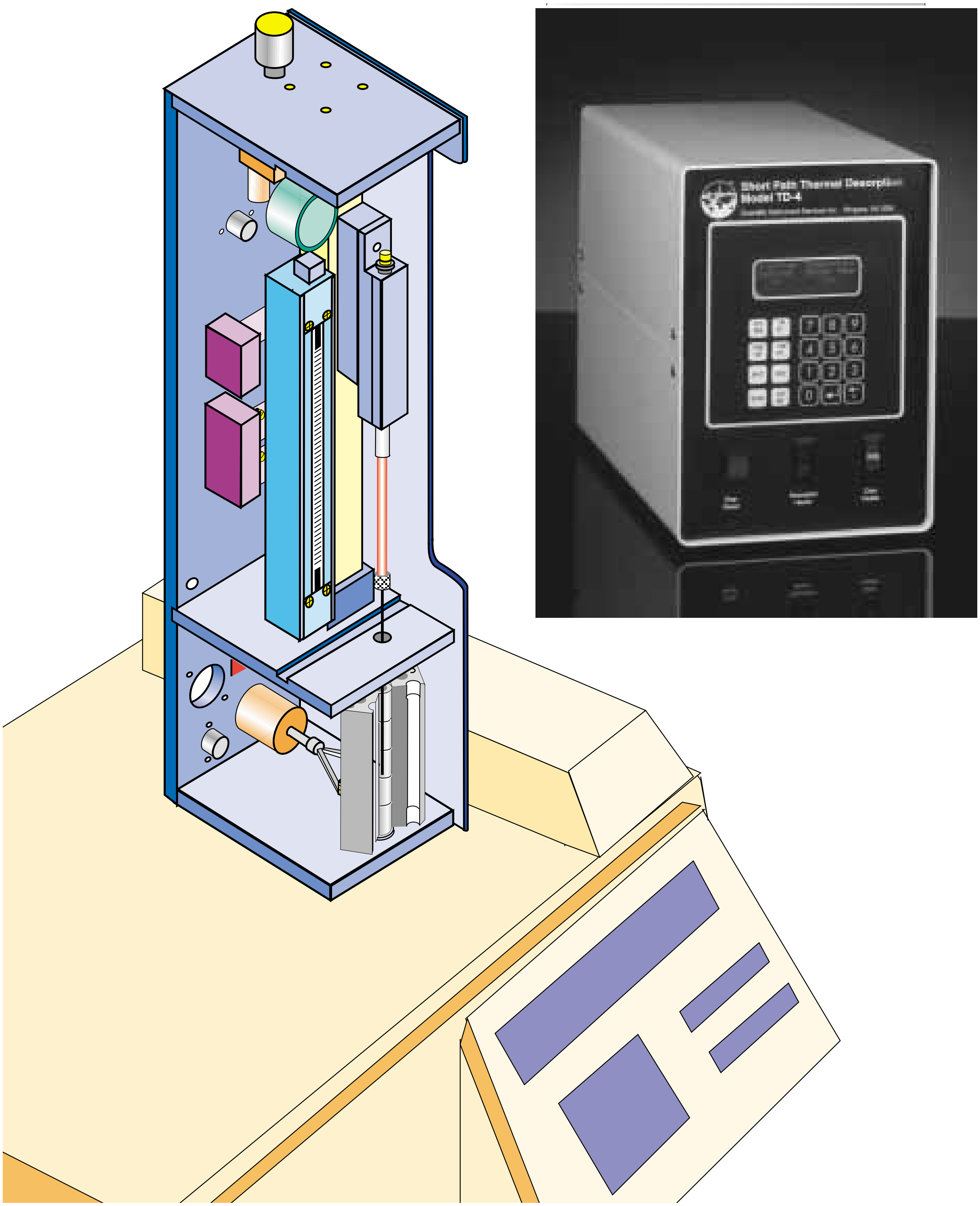


Fig. 1-2

Desorbing Sample into Gas Chromatograph

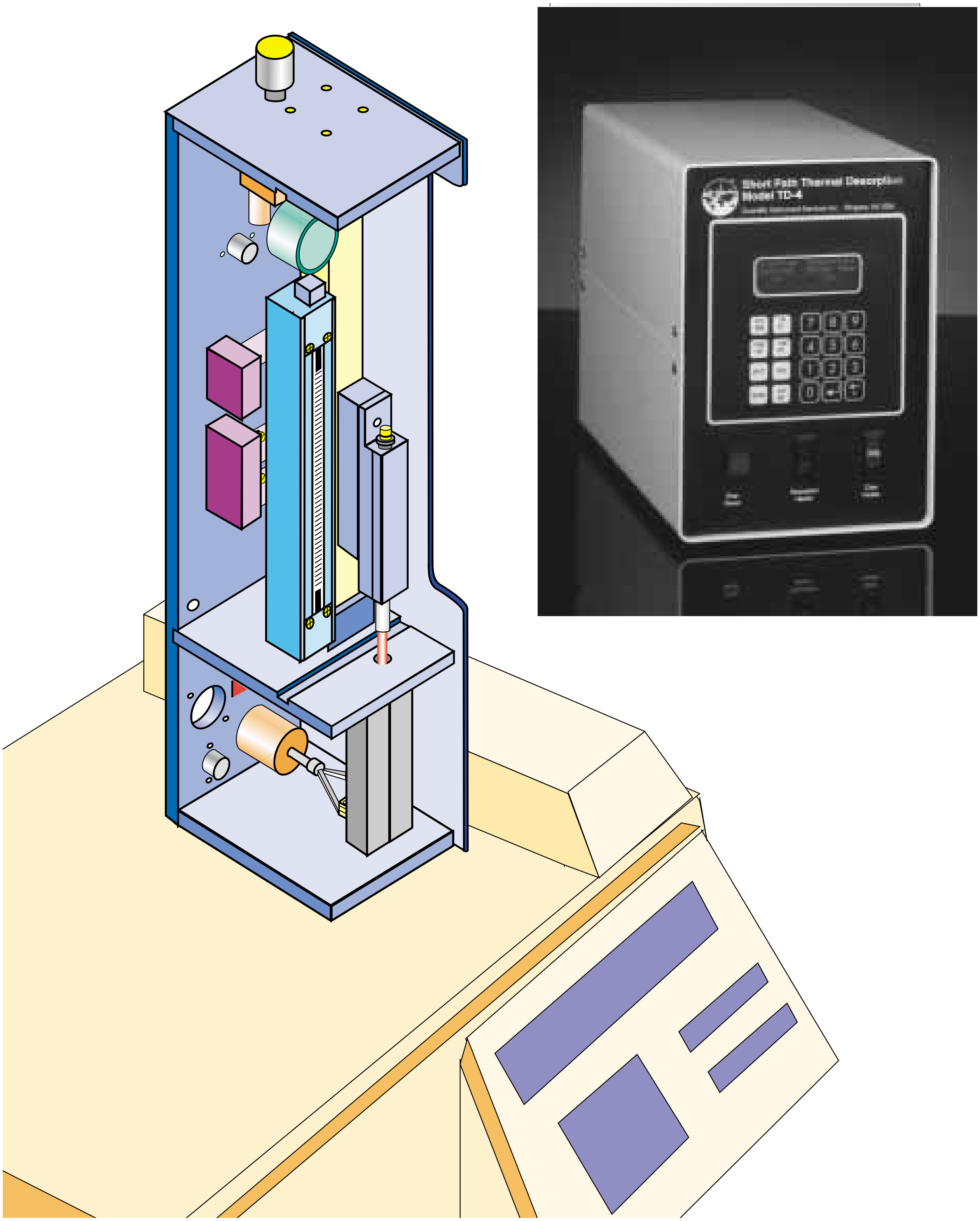


Fig.1-3

Short Path Thermal Desorption Theory of Operation

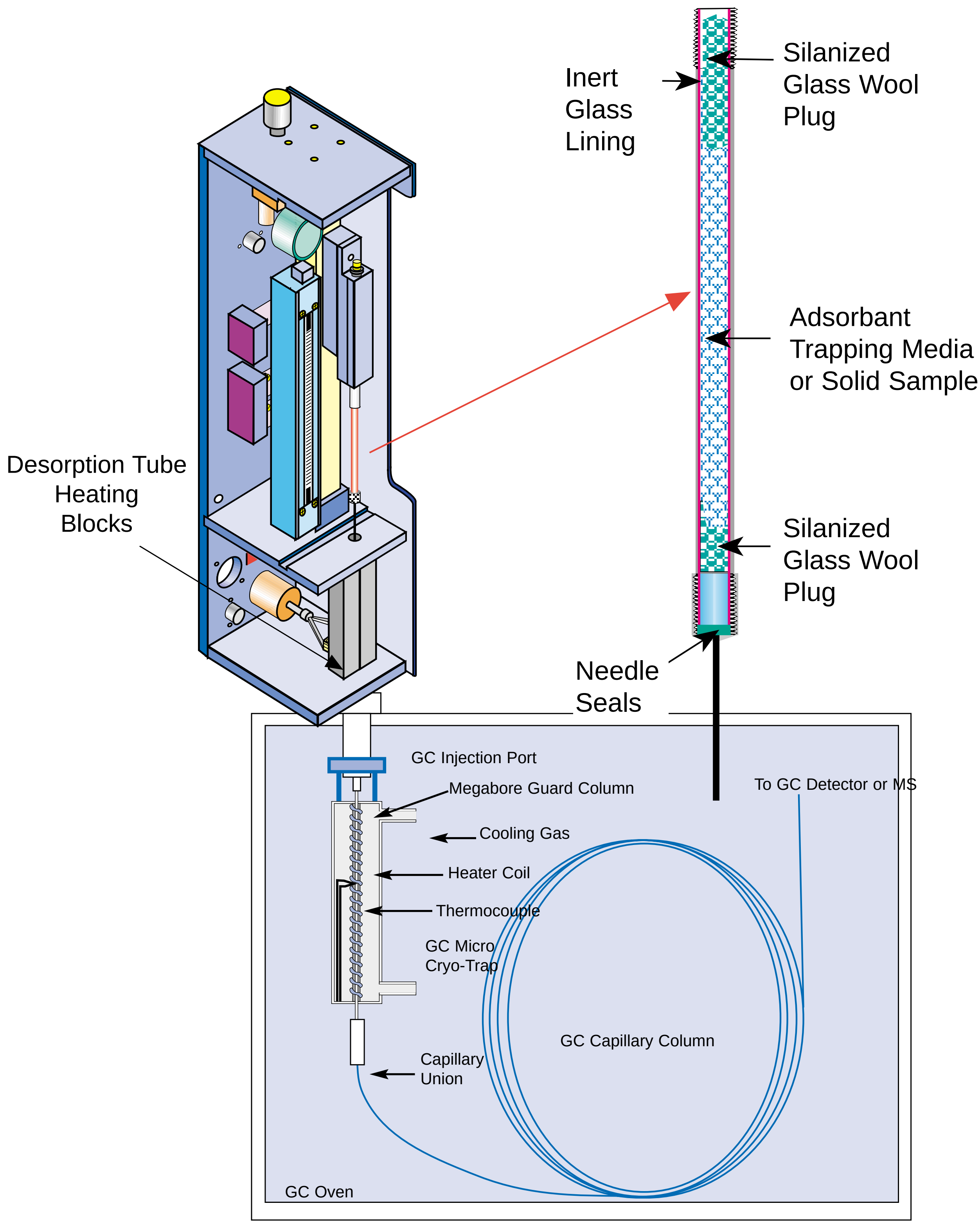


Fig 1-4

Desorption Unit
Electronics Console

Fig. 1-5

Short Path Thermal Desorption System Features

- High sensitivity thermal desorption and direct thermal extraction system
- Short path from sample tube to GC injection port
- Mounts overtop GC injection port, easy installation
- Eliminate tedious sample cleanup by other techniques such as solvent extraction
- No memory effects-individual flow path for each sample preventing contamination of transfer lines
- Compact and portable-easily removable and transferable
- Usable with a wide variety of techniques on capillary and packed GC columns, including direct injection and split/splitless.
- Automatic injection of sample into GC.
- Desorb samples at temperatures from 20 to 350°C either isothermal or temperature program ramp at rates up to 40°/min.
- Rapid heating of samples at rates up to 200°/min.
- User programmable gas purge, injection, desorption and GC delay start times.
- Usable for qualitative and quantitative analysis of samples
- Glass lined stainless steel (GLT) sample tubes are both inert to samples and strong for sample handling and transporting
- Microprocessor controlled electronics system.
- Programmable desorption time from 1 second to 100 minutes
- Automatic programming and control of Micro Cryo-Trap accessory.
- GC remote start feature

Specifications

Power Requirements
115 Volt +/- 10% AC
10 amp max

Gas Requirements
Carrier Gas (He or Nitrogen) 40-60 psi

Compressed Air
Laboratory supply or cylinder 40 psi
Alternative, tee into carrier gas supply

Temperature Control of Desorption Heater Blocks via Microprocessor
Heater Output - 115 volt, 400 watt
Accuracy +/- 0.1%
Input - Platinum Resistance Thermometer (PRT)
Digital Readout for Set Temperature & Actual Temperature 3 digit
Temperature Range 20 to 350°C
Temp. ramp: 0°to 40°/min.

Temperature Control of GC Micro Cryo-Trap Accessory
Heating - Maximum Temperature 400°C
Cooling - Minimum Temperature
CO_2 -70°C
LN_2 -180°C

Microprocessor Control
Remote Access Panel and Keypad
Display:4 lines x 20 character yellow fluorescent display
Serial interface to microprocessor
Operating Voltage: 24 Volt DC
20 Push button Keypad

Microprocessor
16 Kbyte RAM or EPROM
Operating Voltage: 24 Volt DC
Battery Backup
32 timers and 20 Switches

Weight & Dimensions

Desorption Unit
Weight - 17 lbs
Size - 5.5” Wide x 6.5” Deep x 22” High

Electronics Console
Weight - 10 lbs
Size - 7” Wide x 11” High x 14.5” Deep

Warranty

The Thermal Desorption Unit and Electronics Console are warrantied against defects in material and workmanship for a period of one year commencing from the date of shipment from the warehouse of Scientific Instrument Services in Ringoes, NJ, hereafter referred to as the company. The company's liability on the Short Path Thermal Desorption System and accessories is limited to the cost of correcting the defect in the product. In no case shall the company be liable for consequential or special damages. The company will not correct defects caused by the buyers negligence. The company does not guarantee or warrantee the product for any particular purpose. The company'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 Electronics Console within the facilities of Scientific Instrument Services. Service at customers facilities is not available.

Service and Repair

Any equipment to be serviced under warranty or otherwise should be sent to the repair facilities of Scientific Instrument Services in Ringoes, NJ. 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 returned.

Scientific Instrument Services, Inc.

1027 Old York Road

Ringoes, NJ 08551

Attn: Repair Department

RA# ____________________

Phone: (908) 788-5550

KEEP ALL BOXES AND PACKAGING When returning systems for repair they must be sent in original system boxes and packaging. If we do not receive your original packaging,we will charge an extra fee for new packaging when we return the system to you.

II. SITE PREPARATION

Space Requirements

The Short Path Thermal Desorption System is a compact, self contained injection system and desorption system that requires a minimum amount of space. The system is designed only for top injecting GC systems. The Thermal Desorption Unit sits directly over the injection port of the gas chromatograph. The base of the Thermal Desorption Unit is only 5.5" wide x 6.5" deep and the overall height is 22" **See Figure 2.1.**

The Electronics Console is 7" wide x 14.5" deep x 11" high, and normally can sit on top of the GC oven cabinet providing it does not interfere with the operation of the GC. Alternatively, the Electronics Console can be placed on the lab bench next to the GC or on any other suitable shelf or supporting medium within 4' of the GC injection port.

Fig 2.1 Short Path Thermal Desorption System

Line Voltage & Current

The Short Path Thermal Desorption System requires a single 115 volt, 10 amp, 60 Hz, grounded outlet. The system is fuse protected with a main power fuse and a second fuse for the cartridge heater circuit. In no case should a fuse larger than the size recommended in this manual be used.

Power requirements are 115 Volts +/- 10% AC and 10 amp max.

Gas Considerations - Desorbing Gas

Standard GC carrier gas is used as the desorbing gas for the Short Path Thermal Desorption 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 40-60 psi.

The same carrier gas used for the capillary or packed column carrier gas should be used as the desorbing gas in the Short Path Thermal Desorption System to avoid mixing gases in the GC injection port; which could cause an unstable baseline, especially with FID detectors. 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 DESORPTION SYSTEM

NOTE: The carrier gas for the desorption system can easily be plumbed in by adding a cross or tee into the carrier gas line before it enters the gas chromatograph. The instructions below describe how to do this.

The installation of a cross or tee in the carrier gas line requires a tubing cutter, a 7/16" wrench and a Swagelok™ brass cross (B-200-4) or brass tee (B-200-3).

Turn off the carrier gas at its source.

Installation of Cross in Varian 3400 GC Carrier Gas Line

1. **Figure 2.2** shows the standard gas hookup on a Varian 3400 GC.

2. Remove the 1/8" Swagelok™ tee and replace it with a 1/8" Swagelok™ cross. **See Figure 2.3**.

3. Use the top fitting to hook up the carrier gas line to the SPTD unit.

4. Be sure the tubing is completely seated in the fitting and tighten 3/4 turns past finger tight.

Installation of Tee into Carrier Gas Line on an HP5890 and HP6890 GC

1. **Figure 2.4** shows the use of a tubing cutter to cut the carrier gas line just before it enters the GC.

2. Install a 1/8" Swagelok™ Tee (from the SPTD installation kit) as shown with the new 1/8" line coming around the side or top of the GC. See **Figure 2.5.**

3. Be sure the tubing is completely seated in the fitting and tighten 3/4 turns past finger tight.

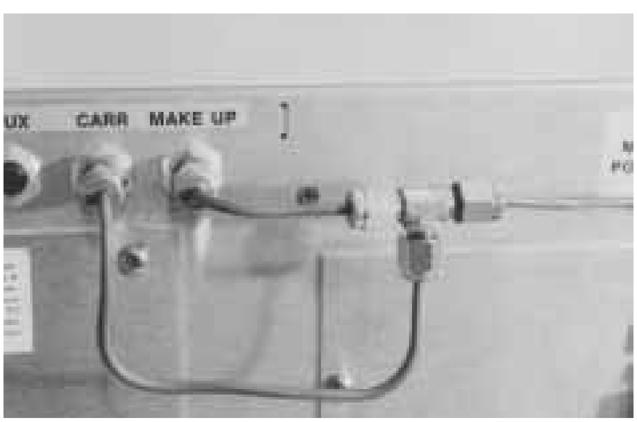


Fig 2.2 Standard carrier gas hookup on Varian 3400 GC.

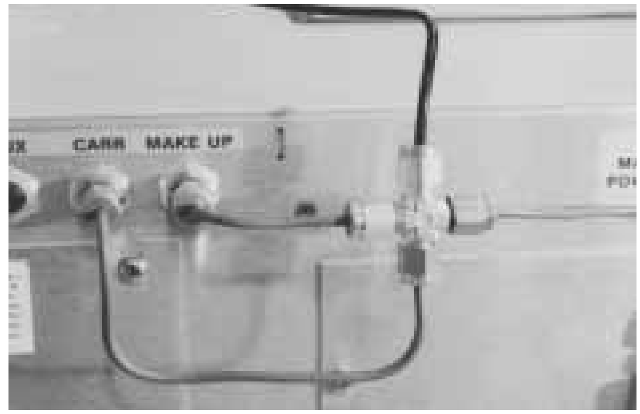


Fig 2.3 Cross installed allowing carrier gas hookup to S.P.T.D. system.

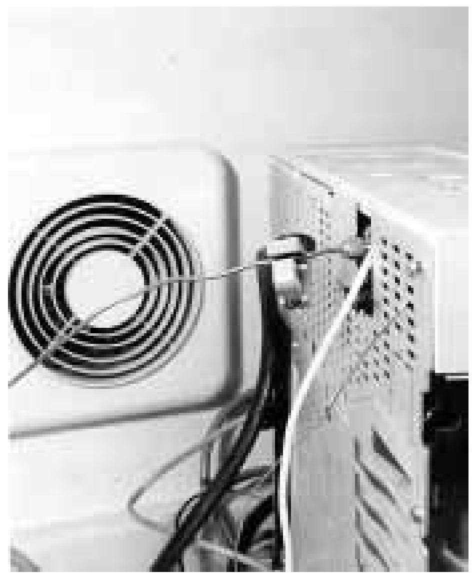


Fig 2.4 Install tee at back of GC.

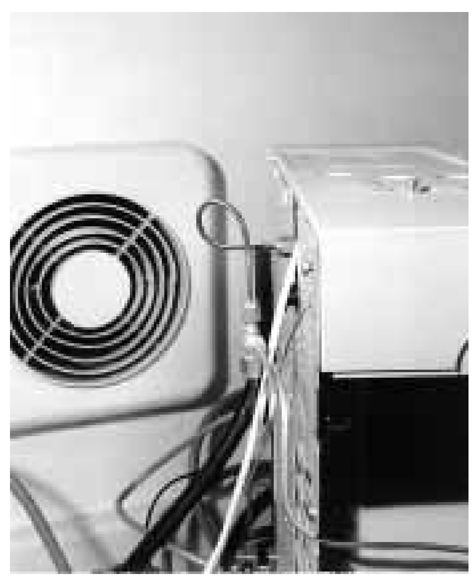


Fig 2.5 Tee installed allowing carrier gas hookup to S.P.T.D. system.

Compressed Air for Solenoids

A supply of clean compressed air or nitrogen is required. compressed air from laboratory supply or cylinder at 40 psi is required. The fitting required for installation to the S.P.T.D. system is a 1/8" Swagelok fitting.

Cryo-Cooling

GC cryo-cooling capabilities using liquid nitrogen or carbon dioxide are recommended. Normally samples are desorbed from the desorption tube and trapped on the front of the GC column. In some cases these volatiles can be trapped on thick film megabore columns such as the J&W DB-624 column. However for microbore capillary columns and thin film columns it is preferable to cool the column below 0°C in order to trap the volatiles. Cryo-cooling is available on most gas chromatographs. Usually this accessory is ordered when the GC is first purchased, however most gas chromatographs can be upgraded to include cryo-cooling.

For optimum results and minimum use of cooling gas, use the Micro Cryo-Trap which can be purchased as an accessory for the SPTD System. The Micro Cryo-Trap can be controlled by the SPTD electronics to regulate both cooling and heating temperatures as well as the switching between the two states.

III. INSTALLATION

Instructions follow for installation of the Short Path Thermal Desorption System (S.P.T.D.) on various gas chromatographs. The following sections are included:

Careful adherence to these installation instructions should allow the user to install the S.P.T.D. system on the gas chromatograph. Be certain that you read through the instructions completely and acquire all the tools required before proceeding. The section on site preparation found in this manual should also be read through completely to be sure that you are ready for installation.

For further help in installation call S.I.S. customer support at (908) 788-5550.

The entire S.P.T.D. system can be installed by the end user. However, Scientific Instrument Services also provides installation by S.I.S. qualified staff. This includes installation of the Short Path Thermal Desorption on your GC or GC/MS system and training to get you up and running with your new system. The part number and price are below. Price includes all travel and lodging and expenses for 1.5 days at your location. Please call S.I.S. for more details.

Part No.	Description	Price
782100	S.P.T.D. System Installation and Training	$2500.00

Installation of the Short Path Thermal Desorption System on the Varian 3400 Gas Chromatograph

Unpacking

1. The following items should have been received with your shipment, and are required for installation of the Short Path Thermal Desorption (S.P.T.D.) System on the Varian 3400 GC. Be sure you have all the items before proceeding. If any items were not received with your system call S.I.S. immediately. See **Figure 3.1** and **Figure 3.2** to aid in determining that all items were received. Save all packaging material and boxes in case future factory service is required.

Part #	Description	Qty.
Short Path Thermal Desorption Model TD4 #784000		
784000D	S.P.T.D. Unit	1
784000C	S.P.T.D. Controller	1
784000M	S.P.T.D. Model TD4 Manual	1
784000N	S.P.T.D. Application Notes	1
782010	Interconnect Cable, 6 Ft.	1
783500	Power Cable for Controller	1
Installation Kit #782200		
783999-24	GC Carrier Gas Solenoid Valve	1
782011	Remote Cable for Varian 3400	1
781002	GLT Tube, 4mm ID	1
781006	SS Solid Caps for GLT Tube	2
781016	Graphite Needle Seal	1
781018	Graphitized Vespel Needle Seal	1
781053	Needle Assy., 35mm Side Hole	1
CL424	Pliers	1
T125062	PTFE Tubing, 10'.	1
782013	Remote Cable 2	1
B2003	Tee	1

2. The following are not included with the system, but are required for installation.

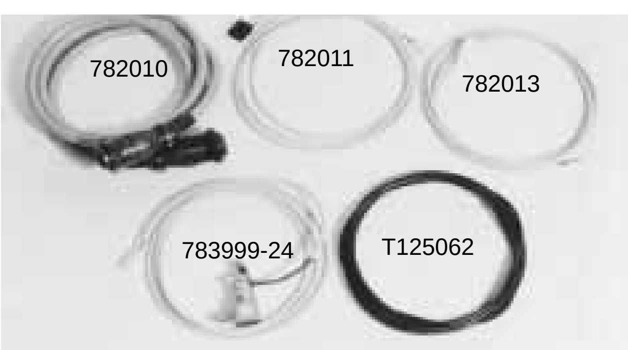
a. Tubing cutter or small file for cutting 1/16" S.S. carrier gas line.

b. 5/16" wrench to tighten 1/16" Swagelok fittings.

c. 7/16" wrench to tighten 1/8" Swagelok fittings.

d. Snoop leak detection fluid or other Helium detector.

Fig 3.1 The Short Path Thermal Desorption System for the Varian 3400 gas chromatographs. The system includes all items shown.



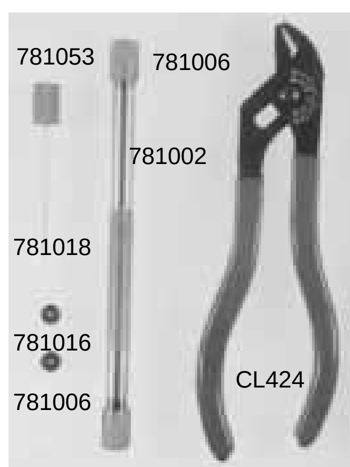


Fig. 3.2 Shows items included in SPTD Installation Kit #782200 for the Varian GC

NOTE: The S.P.T.D. must be used on the rear injection port of the GC if more than one injection port is present.

CAUTION: BE SURE ALL POWER TO THE GAS CHROMATOGRAPH IS OFF AND UNPLUG GC BEFORE PROCEEDING.

3. Remove the covers of the gas chromatograph as shown in **Figure 3.2** to gain access to the injection port.

GC Carrier Gas Solenoid Valve Installation

NOTE: Installation of the GC carrier gas solenoid (783999-24) is optional **but is recommended**. Please see “GC Carrier Flow” on page 7-1 of this manual for a discussion on the use of this valve. If you decide not to install this solenoid valve proceed to step 10.

4. There is a single 1/16" stainless steel gas line which enters the front of the injection port which provides the carrier gas. **See Figure 3.3**. Verify that this is the carrier gas line by tracing the line back from the injection port to its source. The carrier gas line will eventually terminate at the carrier gas inlet for the gas chromatograph.

5. Turn off the carrier gas flow at its source.

6. Using the tubing cutter, cut the carrier gas line approximately 1-2 inches from the injection port. **See Figure 3.4**. Be sure that both ends of the carrier gas line where you have made the cut are open and clean. If not, clean with a small needle file. **Figure 3.5** shows the carrier gas line cut.

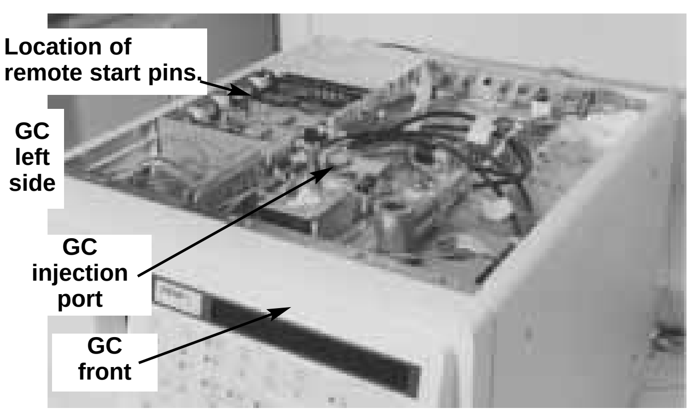


Fig 3.2 Varian 3400 GC with covers removed

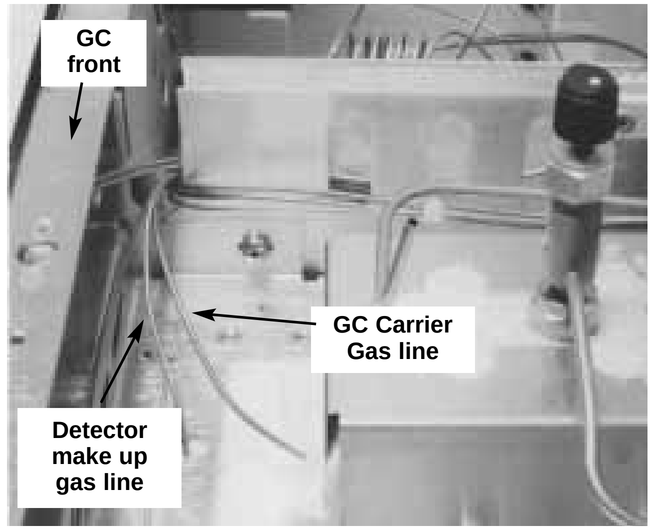


Fig 3.3 Close-up of the injection port area.

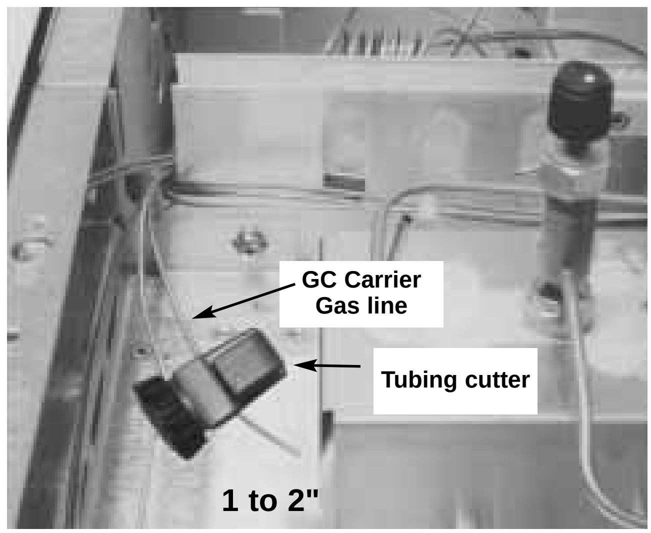


Fig 3.4 Cutting the GC carrier gas line for installation of the carrier gas solenoid valve.

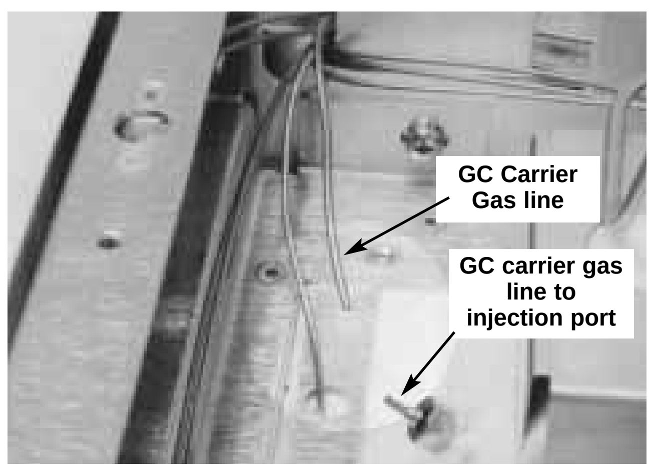


Fig 3.5 The GC carrier gas line has been cut and is ready for installation of the carrier gas solenoid valve.

7. Determine the flow direction of the GC carrier gas solenoid valve (783999-24). There is an arrow on the side of the valve which notes the flow direction of the valve. Install the valve where you have just made the cut in the carrier gas line such that the outlet of the valve is toward the injection port of the GC. Install the valve in such a position that the valve will not interfere with the covers of the GC when they are replaced later. **See Figure 3.6**. Be sure the 1/16" SS line is completely seated in the Swagelok™ fittings on the solenoid valve. Tighten the fittings finger tight and then tighten 3/4 turn further with a 5/16" wrench. **See Figure 3.7**

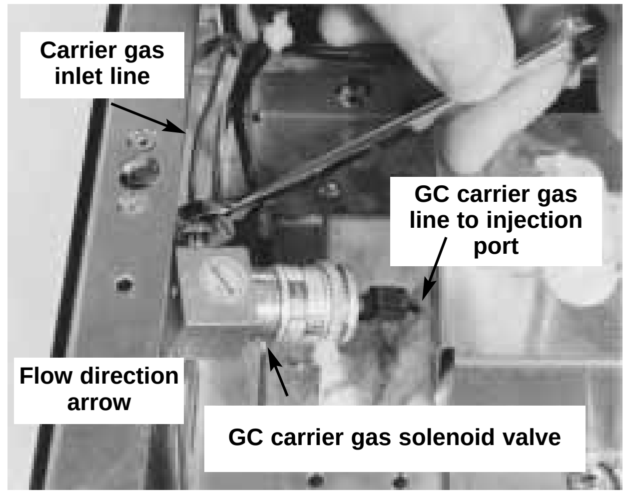


Fig 3.7 The Swagelok fittings should be tightened 3/4 turns past finger tight using a 5/16" wrench.

8. Turn on the carrier gas flow again and check for leaks in these connections using Snoop. **See Figure 3.8**. If leaks occur, re-tighten the fittings and leak check again.

9. Feed the GC carrier gas solenoid valve electrical lead from the injection port to the rear of the GC. Keep the lead away from any hot surfaces. This line must exit through an opening in the back panel of the GC so that it can be plugged into the S.P.T.D. controller later. **See Figure 3.9.**

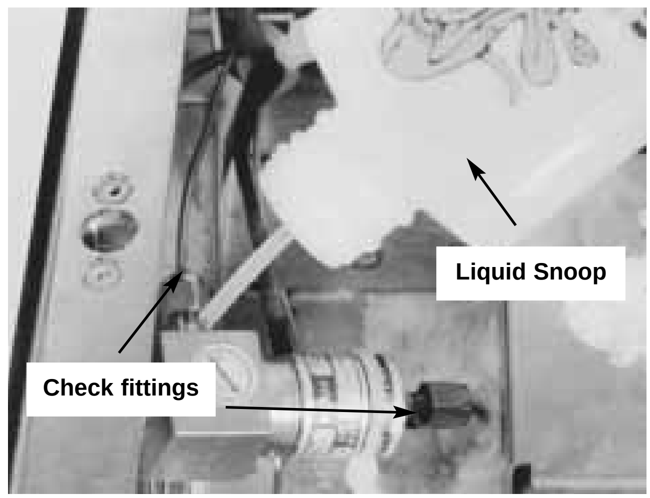


Fig 3.8 Check for leaks at these fittings using SNOOP.

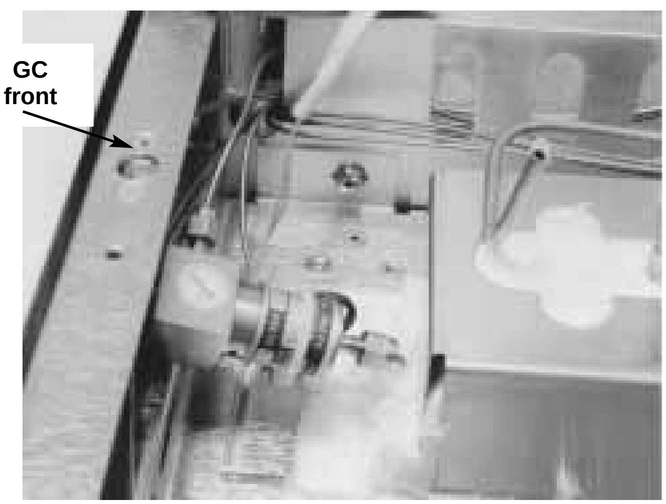


Fig 3.6 Installing the GC carrier gas solenoid valve.

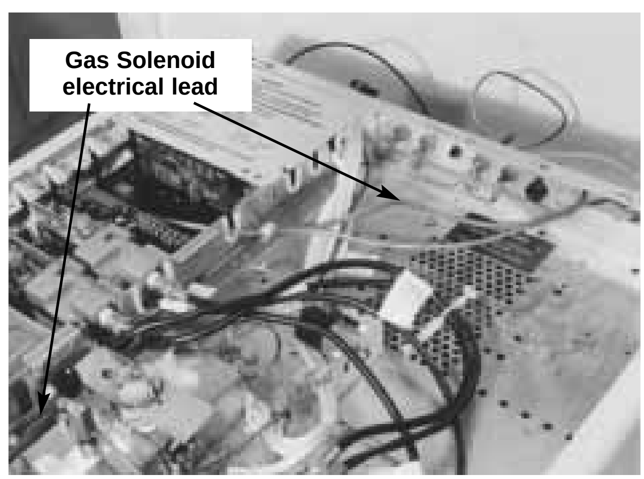


Fig 3.9 The electrical lead from the GC carrier gas solenoid valve should be directed out through the back of the GC.

Installation of GC Remote Cable

10. With the covers still removed locate the pins for the remote cable installation on the GC control board. **See Figure 3.10** and **Figure 3.2.** See information found in the Varian 3400 manual concerning installation of GC remote input cable.

11. Plug the appropriate end of the remote cable (782011) onto the pins on the GC control board. **See Figure 3.11.** Feed the cable out to the rear of the GC so that it can be later plugged into the rear of the S.P.T.D. controller.

12. Replace all GC covers.

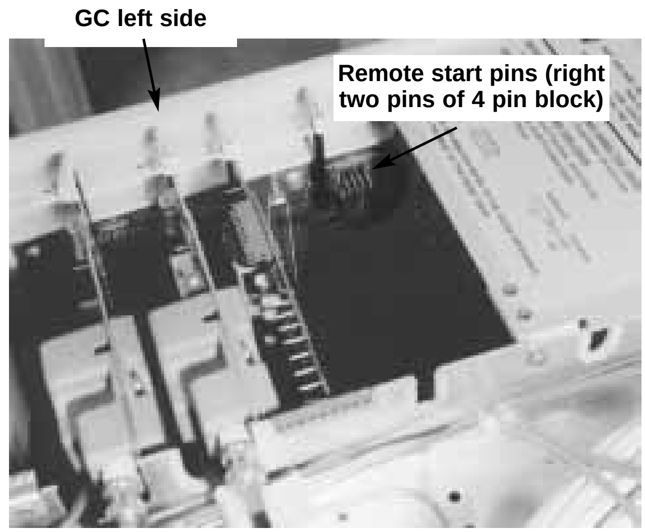


Fig 3.10 The pins for the remote start on the Varian 3400 are located on the left side of the interior of the GC.

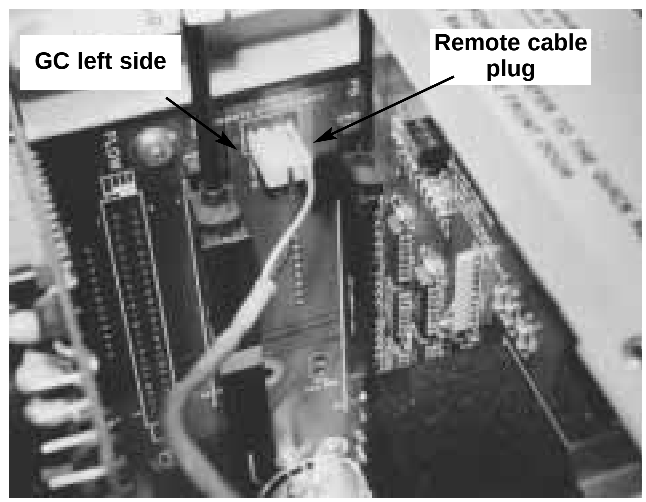


Fig 3.11 The remote start cable has been installed. The lead should be directed out through the rear of the GC.

Installation of S.P.T.D. Unit

13. The S.P.T.D. unit is manufactured with a counterbore in the bottom of the unit which fits snugly over the GC septum nut on the Varian GC. **See Figure 3.12**. Place the unit over the septum nut so that the nut is fitted in this counterbore. **See Figures 3.13 and 3.14**. There are no clamps or other fittings required to mount the system to the GC.

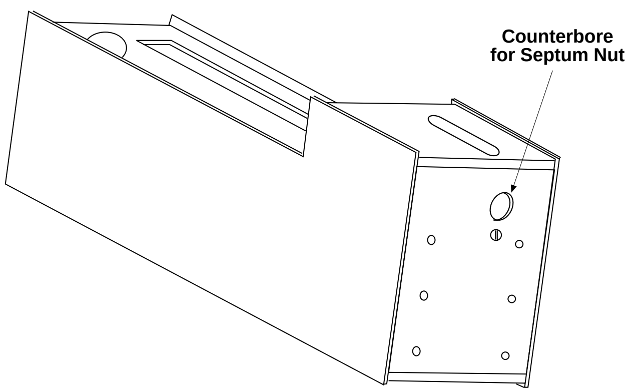


Fig 3.12 Schematic shows the counterbore which fits over the GC septum nut on the Varian™ 3400.

Fig 3.13 Align the S.P.T.D unit over the septum nut.

Fig 3.14 The S.P.T.D. fits snugly over the septum nut.

14. The back of the S.P.T.D. unit has two 1/8" Swagelok™ fittings for connection of the carrier gas and the air which is used to operate the S.P.T.D. system. They are labeled “GAS” for the carrier gas and “AIR” for the compressed air. **See Figure 3.15**.

NOTE: For more discussion on the gas line setup and installation see the “Site Preparation” section of this manual.

15. Using the 1/8" PTFE tubing supplied with the S.P.T.D. system cut a length of tubing long enough to reach from the tee or cross which was installed in the GC carrier gas line during site preparation to the fitting labeled GAS”. Fully seat the tubing in the fittings and then tighten the fittings finger tight. Using a wrench tighten the fittings 3/4 turns further.

WARNING: Do not use hydrogen as a carrier gas in this system. Keep PTFE tubing lines clear of GC exhaust fan and other heated areas.

16. As before, cut an appropriate length of the 1/8" PTFE tubing supplied with the S.P.T.D. to connect from the compressed air source to the fitting labeled “AIR”. Be sure the tubing is fully seated in the fittings and then tighten the fittings finger tight. Using a 7/16" wrench tighten the fittings 3/4 turns further. **See Figure 3.16**.

17. Using Snoop, check for leaks at these connections.

18. Plug the interconnect cable (782010) into the appropriate plug on the back of the desorption unit. The cable is reversible so either end can be plugged into the unit. **See Figure 3.17.**

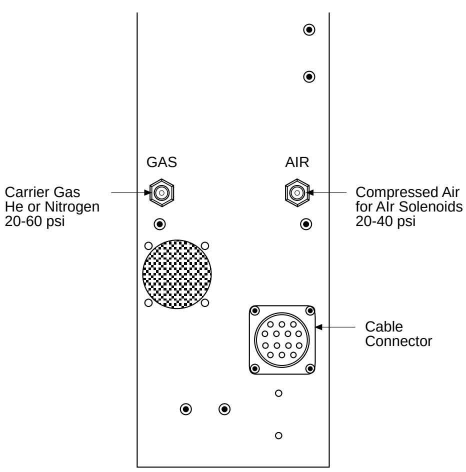


Fig 3.15 Schematic shows the location of the gas inlets on the rear of the S.P.T.D. unit.

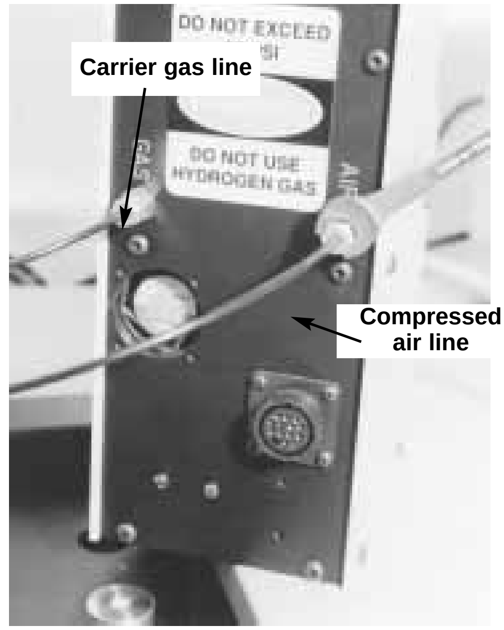


Fig 3.16 1/8" PTFE tubing is used for the carrier gas and compressed air lines on the S.P.T.D unit.

Fig 3.17 Installation of the interconnect cable (782010) on the rear of the S.P.T.D unit.

Installation of the S.P.T.D. Controller

19. The S.P.T.D. controller (784000C) can be positioned next to the unit as shown in **Figure 3.18** or can be positioned on the bench next to the GC. **Figure 3.19** shows the back of the controller.

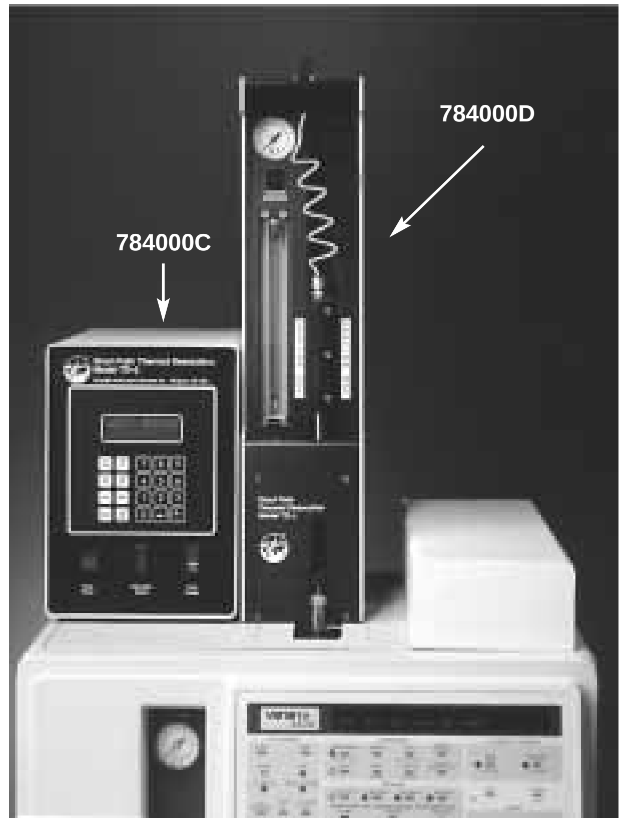


Fig 3.18 The S.P.T.D. unit installed on the Varian 3400. The S.P.T.D. controller fits well next to the unit.

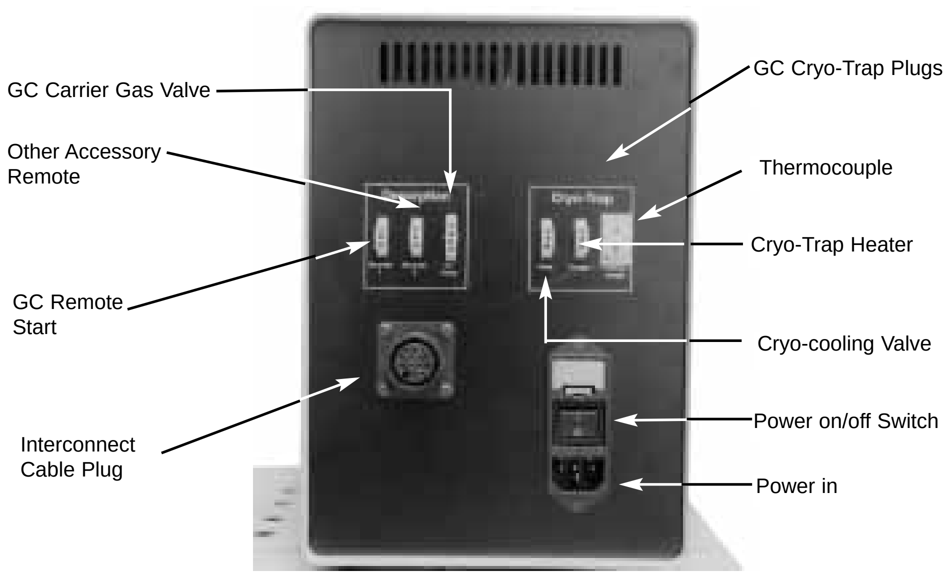


Fig 3.19 Rear view of the S.P.T.D. controller (784000C).

20. Plug the interconnect cable (782010) into the appropriate connection on the back of the controller. See **Figure 3.20.** The other end of the cable should already be plugged into the rear of the desorption unit.

21. Plug the cable from the GC carrier gas solenoid valve (781999-24), if it was installed earlier, into the fitting labeled “GC Valve” on the back of the S.P.T.D. controller. **See Figure 3.21.**

22. Plug the remote start cable (782011) into the fitting labeled “Remote 1” on the back of the S.P.T.D. controller. **See Figure 3.21**.

NOTE: The plug labeled “Remote 2” is available for starting recorders, integrators or other equipment. An extra cable (782013) is included if the operator chooses to use this option.

In addition, the plug labeled “Cryo-Trap” is available if this accessory is being used.

23. Plug in the power cord from the back of the S.P.T.D. controller into a grounded 115V, 10 Amp outlet.

24. Installation is now complete. Refer to the operator section of this manual for proper use and operation of the S.P.T.D. system.

25. If the Micro Cryo-Trap accessory was purchased with the SPTD system the GC cooling valve, cryo heater and thermocouple are plugged in the appropriate receptacle in the back of the controller box. See Section IX (Accessories) for details on the Micro Cryo-Trap installation.

Fig 3.20 Installing the interconnect cable (782010).

Fig 3.21 Plug the remote start cable into the plug labeled "Remote 1" on the rear of the S.P.T.D. controller.

Installation of the Short Path Thermal Desorption System on the Hewlett-Packard 5890 Gas Chromatographs

B. Unpacking

1. The following items should have been received with your shipment, and are required for installation of the Short Path Thermal Desorption (S.P.T.D.) System on the Hewlett-Packard 5890. Be sure you have all the items before proceeding. If any items were not received with your system call us immediately. See **Figures 3.22 - 3.25** and the parts list included with your installation kit to aid in determining that all items were received. Save all packaging material and boxes if future factory service is required.

a. Short Path Thermal Desorption System - #784000

Part #	Description	Qty.
784000D	S.P.T.D. Unit	1
784000C	S.P.T.D. Controller	1
784000M	S.P.T.D. Model TD4 Manual	1
784000N	S.P.T.D. Application Notes	1
782010	Interconnect Cable, 6'.	1
783500	Power Cable for Controller	1

Fig 3.22 The Short Path Thermal Desorption System for the HP5890 Series gas chromatographs. The system includes all items shown

b. H.P. Installation Kits

HP5890 Series I Installation Kit - #782210

Part #	Description	Qty.
CL424	Pliers	1
T125062	PTFE tubing, 1/8"OD x 10’	
B2003	Tee	2
781002	GLT tube, 4mm ID	1
781006	SS solid caps for GLT tube	2
781016	Graphite needle seal	1
781018	Graphitized Vespel needle seal	1
781053	Needle assy., 35mm side hole	1
781106	Septum adaptor	1
781123	Mounting plate, HP5890 Series I	1
782012	Remote Cable for HP5890	1
782013	Remote cable, remote 2	1
78399924	GC gas valve assembly	1

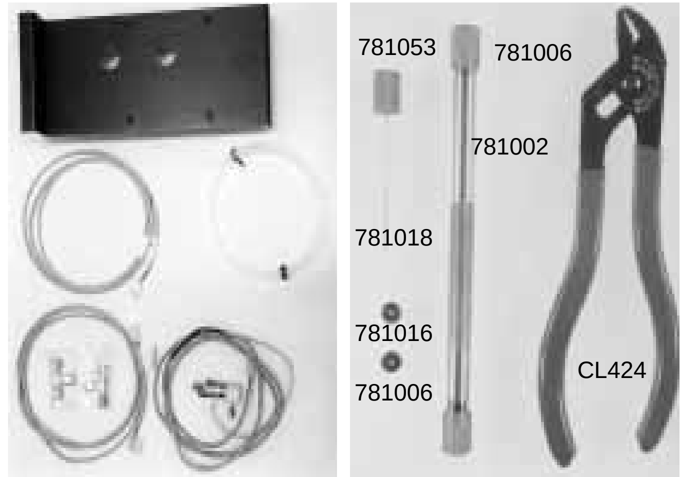


Fig. 3.23 Shows items included in SPTD Installation Kit #782210 for the HP5890 Series I GC.

H.P. 5890 Series II Installation Kit - # 782230

Part #	Description	Qty.
CL424	Pliers	1
T125062	PTFE tubing, 1/8" OD x 10'	1
B2003	Tee	2
781002	GLT tube, 4mm ID	1
781006	SS solid caps for GLT tube	2
781016	Graphite needle seal	1
781018	Graphitized Vespel needle seal	1
781053	Needle assy., 35mm side hole	1
781106	Septum adaptor	1
781121	Mounting plate, HP5890 Series II	1
782012	Remote Cable for HP5890	1
782013	Remote cable, remote 2	1
7839993	GC gas valve assembly	1

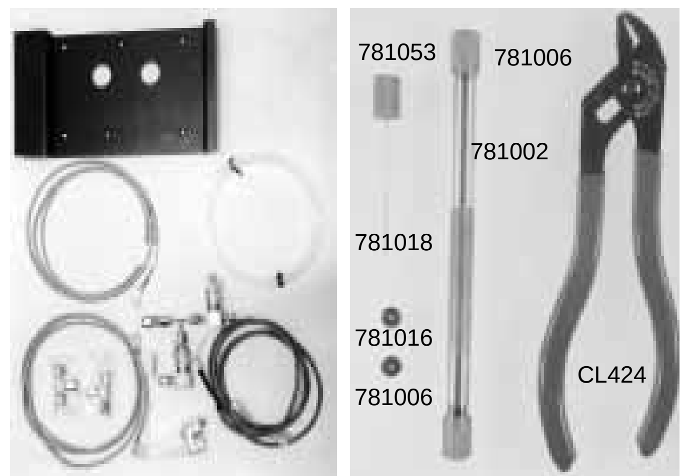


Fig. 3.24 Shows items included in SPTD Installation Kit #782230 for the HP5890 Series II GC with EPC.

H.P. 5890 Series II (No EPC) Installation Kit - # 782220

Part #	Description	Qty.
CL424	Pliers	1
T125062	PTFE tubing, 1/8" OD x 10'	
B2003	Tee	2
781002	GLT tube, 4mm ID	1
781006	SS solid caps for GLT tube	2
781016	Graphite needle seal	1
781018	Graphitized Vespel needle seal	1
781053	Needle assy., 35mm side hole	1
781106	Septum adaptor	1
781121	Mounting plate, HP5890 Series II	1
782012	Remote Cable for HP5890	1
782013	Remote cable, remote 2	1
78399924	GC gas valve assembly	1

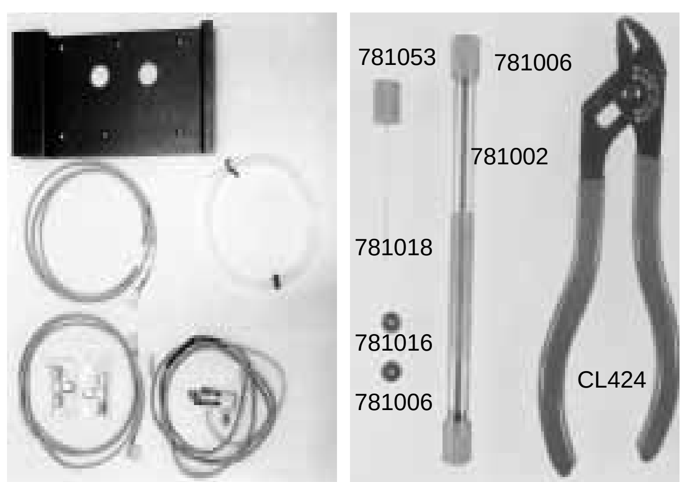


Fig. 3.25 Shows items included in SPTD Installation Kit #782220 for the HP5890 Series II GC without EPC.

2. The following are not included with the system but are required for installation.

a. Tubing cutter or small file for cutting 1/16" S.S. carrier gas line.

b. 5/16" wrench to tighten 1/16" Swagelok™ fittings.

c. 7/16" wrench to tighten 1/8" Swagelok fittings.

d. Snoop leak detection fluid or other helium leak detector.

e. For the HP5890 with EPC, approximately 18" of 1/8" flexible copper tubing.

NOTE: The S.P.T.D. must be used on the front injection port of the GC if more than one injection port is present. If injection port is installed on rear position it will need to be moved to the front position.

While the SPTD may be installed on various HP inlets, the split/splitless injection port is recommended

CAUTION: BE SURE ALL POWER TO THE GAS CHROMATOGRAPH IS OFF AND THE GC IS UNPLUGGED BEFORE PROCEEDING.

3. Remove the injection port cover plate and the right top cover plate of the gas chromatograph as shown in **Figure 3.26** to gain access to the injection port.

GC Carrier Gas Solenoid Valve Installation

NOTE: Installation of the GC carrier gas solenoid (78399924) is optional but **is recommended**. Please see "GC Carrier Flow" on page 7-1 of this manual for a discussion on the use of this valve. If you decide not to install this solenoid valve proceed to step 10. If you have EPC on your GC, please see page 3-23 of this manual.

4. There are two 1/16" stainless steel gas lines which enter the front of the injection port, one is for the septum purge and the other is for the carrier gas. **See Figure 3.27. Determine which line is for the carrier gas by tracing the line back from the injection port.** The carrier gas line will pass through an internal gas filter on the inside back cover of the GC and eventually terminate at the back of the GC where the carrier gas line enters the GC from its source. Be sure that you have correctly determined which line is the carrier gas line.

5. Turn off the carrier gas flow at its source.

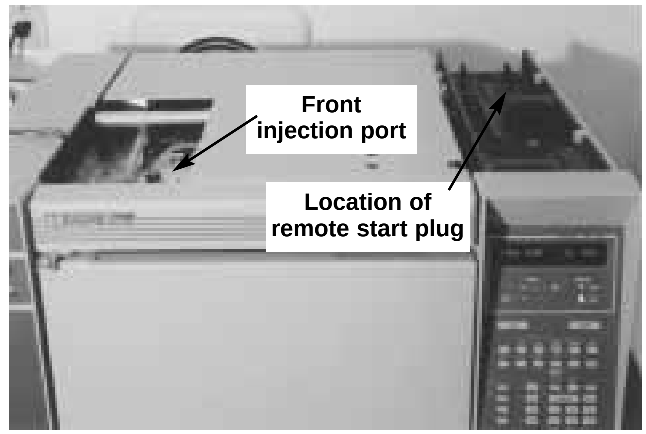


Fig 3.26 The HP 5890 gas chromatograph with the covers, removed

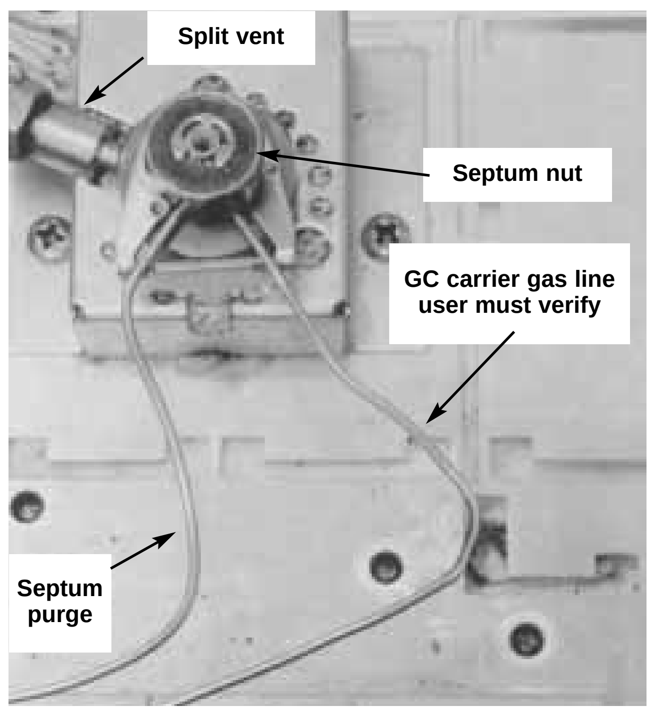


Fig 3.27 Close-up of the injection port of the HP5890, showing septum purge and carrier gas lines.

6. Using the tubing cutter, cut the carrier gas line approximately 1-2" from the injection port. **See Figure 3.28**. Be sure that both ends of the carrier gas line where you have made the cut are open and clean. If not, clean with a small needle file. **See Figure 3.29**

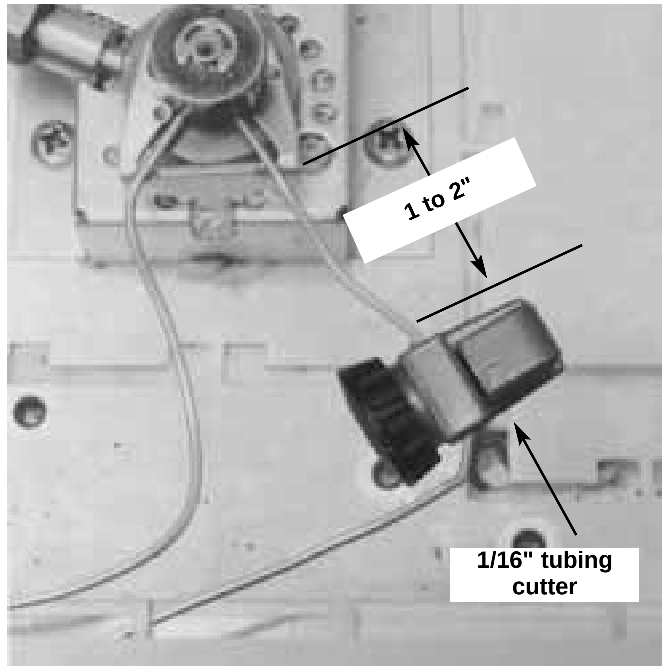


Fig 3.28 Cutting the GC carrier gas line.

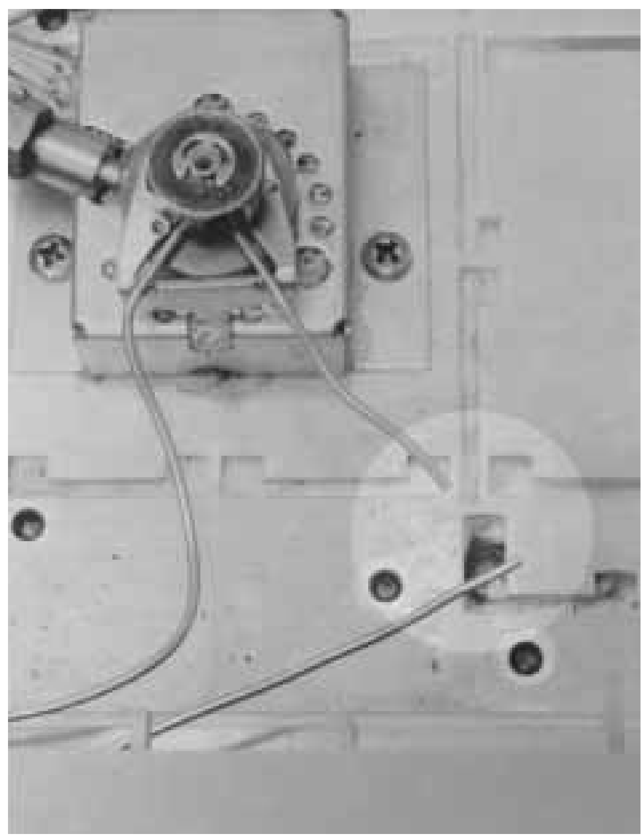


Fig 3.29 Be sure that both ends of the carrier gas line are open and clean where the line was cut.

7. Determine the flow direction of the GC carrier gas solenoid valve (78399924). There is an arrow on the side of the valve which notes the flow direction of the valve. Install the valve where you have just made the cut in the carrier gas line such that the outlet of the valve is toward the injection port of the GC. Install the valve in such a position that the valve will not interfere with the covers of the GC when they are replaced later. **See Figure 3.30**. Be sure the 1/16" SS line is completely seated in the Swagelok™ fittings on the solenoid valve. **See Figure 3.31**. Tighten the fittings finger tight and then tighten 3/4 turn further with the 5/16" wrench. **See Figure 3.32**

Fig 3.31 The solenoid valve must be installed in such a manner that it does not interfere with the GC injection port cover.

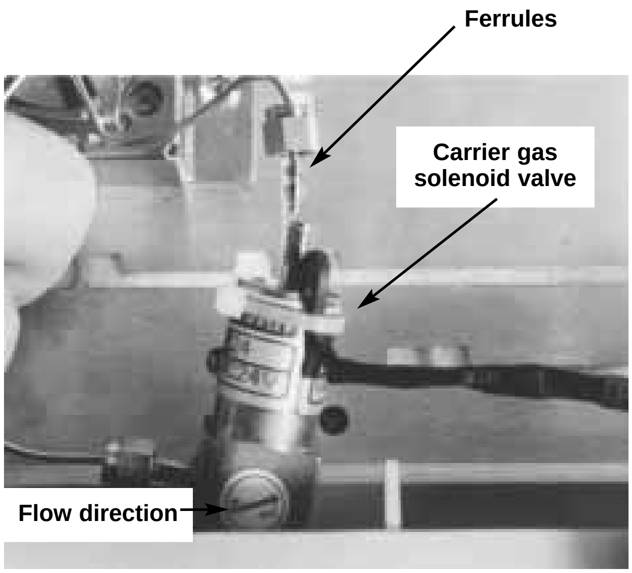


Fig 3.30 Installing the GC carrier gas solenoid valve. Notice the back ferrule and front ferrule used in the Swagelok fittings. Be sure these are properly installed.

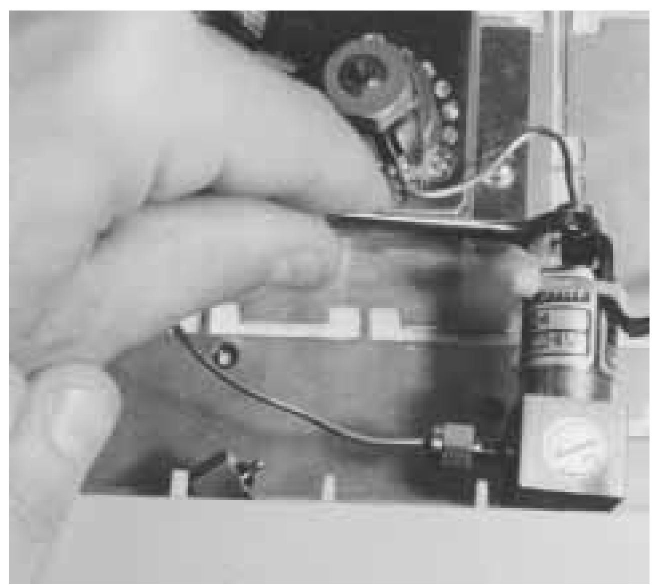


Fig 3.32 Tighten the Swagelok fittings 3/4 turn past finger tight with a 5/16" wrench.

8. Turn on the carrier gas flow again and check for leaks in these connections using Snoop. **See Figure 3.33**. If leaks occur re-tighten the fittings and leak check again.

9. Feed the GC carrier gas solenoid valve electrical lead from the injection port to the rear of the GC. Keep the lead away from any hot surfaces. This line must exit through an opening in the back panel of the GC so that it can be plugged into the S.P.T.D. controller later. **See Figure 3.34**

NOTE: For GC's with EPC a two valve system is used. See page 3-23 for details.

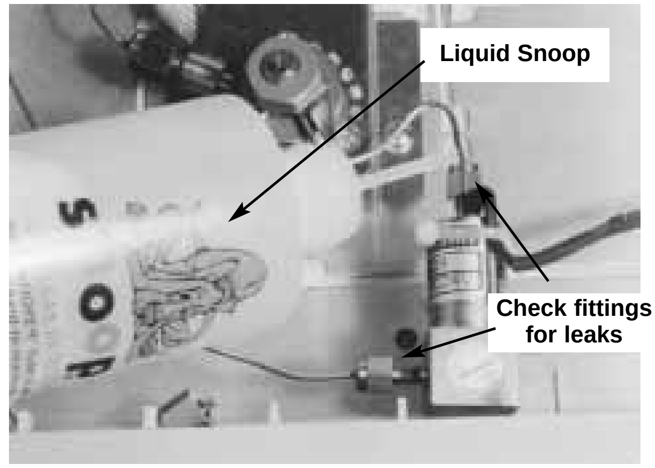


Fig 3.33 Using SNOOP to leak check fittings on the GC carrier gas solenoid valve.

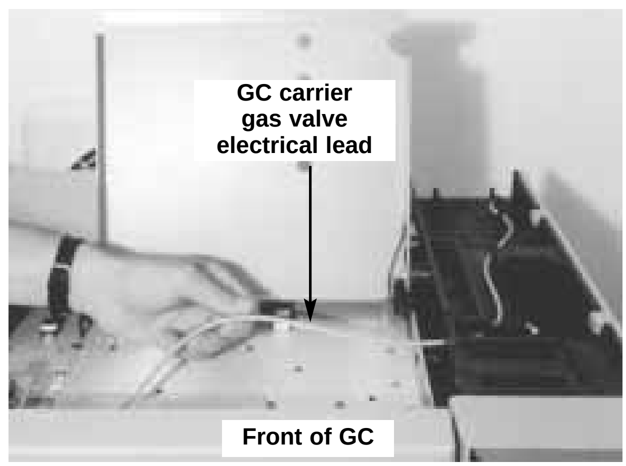


Fig 3.34 Feed the electrical lead from the solenoid valve through the rear of the GC.

Installation of GC Remote Cable

10. With the covers still removed locate the plug for the remote cable installation on the GC control board. **See Figures 3.35 and 3.26.**

11. Plug the appropriate end of the remote cable (782012) onto the plug on the GC control board. **See Figure 3.33.** Feed the cable out to the rear of the GC so that it can be later plugged into the rear of the S.P.T.D. controller.

NOTE: If you have a MSD or autosampler attached to your GC you will need to use the HP Parallel/Remote PCA (HP# 05990-60320) and the HP Parallel/Remote Cable (HP#05990-60019) which you should have received from HP with your GC/MS. The parallel/remote cable plugs into the receptacle on the GC labeled "Remote". The HP parallel/remote PCA plugs into the HP parallel/remote cable allowing hookup of the HP remote cable (HP# 35900-60700) to the MSD or autosampler and hookup of the desorption remote cable (SIS# 782012) to the desorption system. **See Figure 3.37**. Plug the appropriate end of the desorption remote cable (782012) into the parallel/remote PCA and then feed the other end of the desorption cable out the back of the GC so that it can be later plugged into the rear of the S.P.T.D. controller.

12. Replace the GC cover which is located over the GC remote input. Do not replace the cover over the injection port.

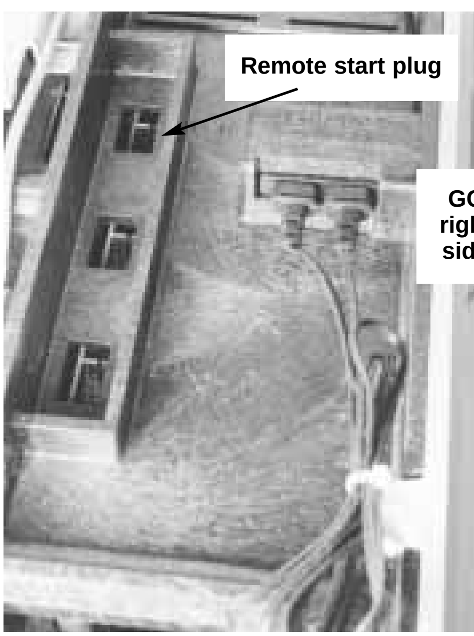


Fig 3.35 The GC remote start plug location.

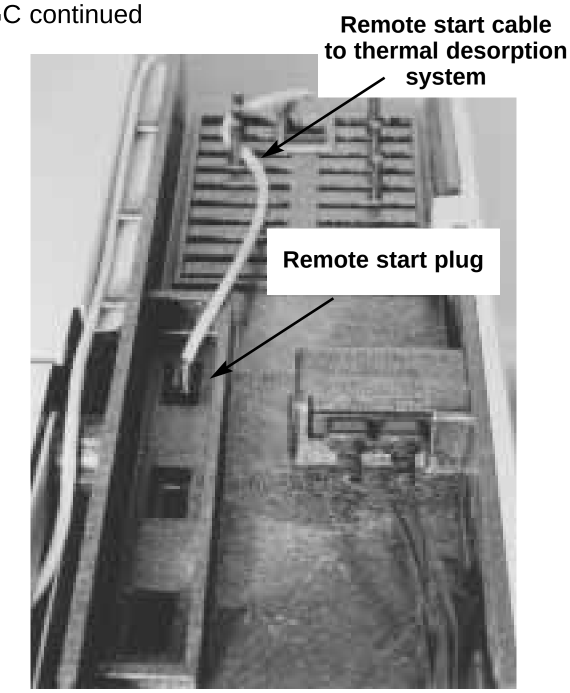


Fig 3.36 Plug the remote cable (782012) into the GC remote start plug. The cable is keyed so that it fits in only one direction.

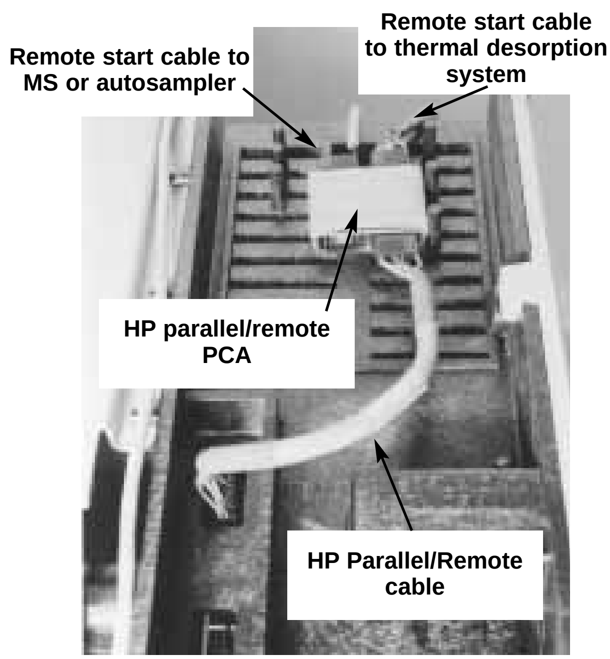


Fig 3.37 Installation of the remote cable (782012) if an autosampler or MSD is present.

Installation of S.P.T.D. Unit

13. Place the mounting plate (781121 or 781123) where the original HP injection port cover would be. The standoffs should support the mounting plate at the correct height. The standoffs on the mounting plate must sit firmly on the injection port mounting surface. If any gas lines or the gas solenoid valve previously installed are in the way gently move them to provide clearance for the mounting plate standoffs. **See Figures 3.38 and 3.39.**

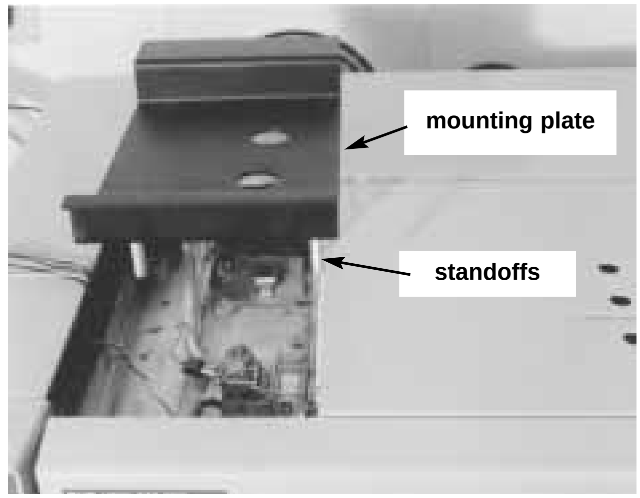


Fig 3.38 Installation of the desorption unit mounting plate.

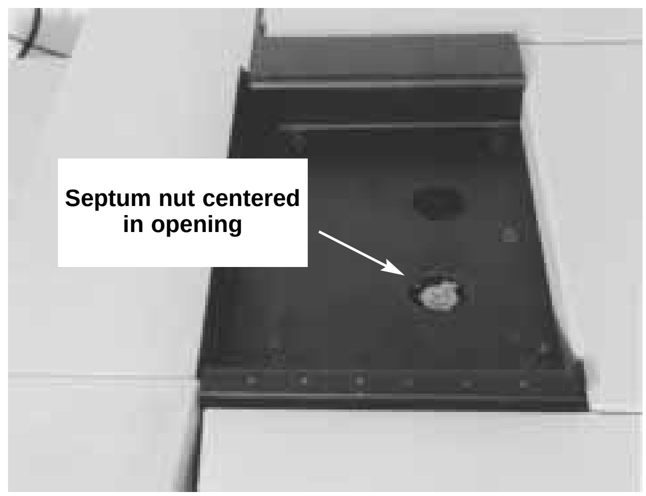


Fig 3.39 The mounting plate should sit firmly and be level, with the septum nut centered in the opening in the mounting plate.

14. Place the aluminum septum nut adapter (781106) over the front septum nut. The bottom of the adapter will protrude through the hole in the mounting plate, while the remainder will rest evenly on the mounting plate. **See Figures 3.40 and 3.41.**

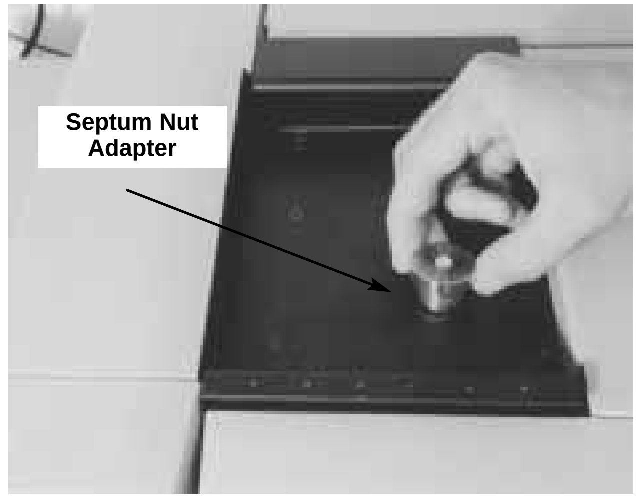


Fig 3.40 Installing the septum nut adapter (781106).

Fig 3.41 The septum nut adapter should sit firmly and evenly on the mounting plate.

15. The S.P.T.D. unit is manufactured with a counterbore in the bottom of the unit which fits snugly over the septum nut adapter just installed. **See Figure 3.42**. Place the unit over the septum nut adapter so that the adapter is fitted in this counterbore. **See Figures 3.43 and 3.44**. There are no clamps or other fittings required to mount the system to the GC.

Fig 3.43 Align the S.P.T.D. unit over the septum nut adapter.

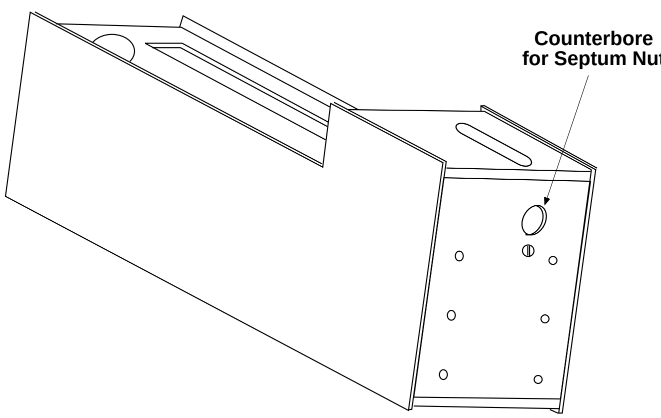


Fig 3.42 Schematic showing counterbore for septum nut adapter.

Fig 3.44 The S.P.T.D. unit installed on the HP 5890.

16. The back of the S.P.T.D. unit has two 1/8" Swagelok™ fittings for connection of the carrier gas and the air which is used to operate the S.P.T.D. system. They are labeled “GAS” for the carrier gas and “AIR” for the compressed air. **See Figure 3.45.**

NOTE: For more discussion on the gas line setup and installation see the “Site Preparation” section of this manual.

17. Using the 1/8" PTFE tubing supplied with the S.P.T.D. system cut a length of tubing long enough to reach from the tee which was installed in the GC carrier gas line earlier to the fitting labeled “GAS”. Be sure the tubing is fully seated in the fittings and then tighten the fittings finger tight. Using the 7/16” wrench tighten the fittings 3/4 turns further.

WARNING: Do not use hydrogen as a carrier gas in this system. Keep PTFE tubing lines clear of GC exhaust and other heater areas.

18. As before, cut an appropriate length of the 1/8" PTFE tubing supplied with the S.P.T.D. to connect from the compressed air source to the fitting labeled “AIR”. Be sure the tubing is fully seated in the fittings and then tighten the fittings finger tight. Using the 7/16" wrench tighten the fittings 3/4 turns further. **See Figure 3.46**.

19. Using Snoop check for leaks at these connections.

20. Plug the interconnect cable (Part #782010) into the appropriate plug on the back of the desorption unit. The cable is reversible so either end can be plugged into the unit. **See Figure 3.47.**

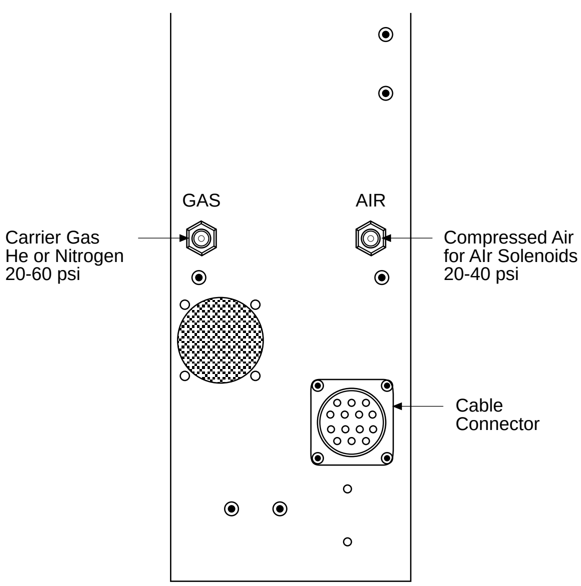


Fig 3.45 Schematic showing the location of the gas inlets on the rear of the S.P.T.D. unit.

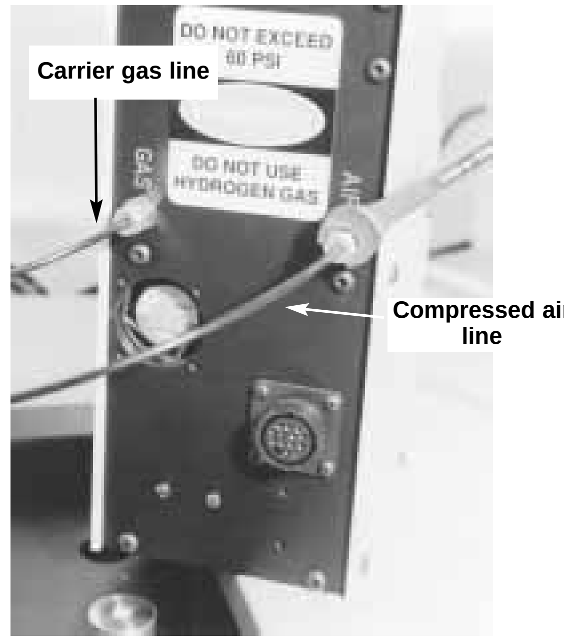


Fig 3.46 1/8" PTFE tubing is used for the carrier gas line and the compressed air line on the S.P.T.D. unit.

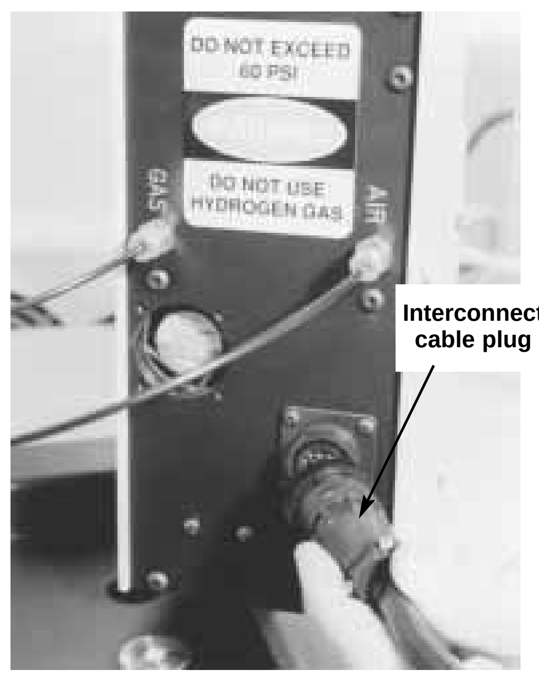


Fig 3.47 Installation of the interconnect cable (782010)

Installation of the S.P.T.D. Controller

21. The S.P.T.D. controller can be positioned next to the unit as shown in **Figure 3.48** or can be positioned on the bench next to the GC. **Figure 3.49** shows the back of the controller.

Fig 3.48 The S.P.T.D. unit installed on the HP5890.

Fig 3.49 Rear view of the S.P.T.D. controller.

22. Plug the interconnect cable (782010) into the appropriate connection on the back of the controller. **See Figure 3.50.**

23. Plug the cable from the GC carrier gas solenoid valve (78199924), if it was installed earlier, into the fitting labeled “GC Valve” on the back of the S.P.T.D. controller. **See Figure 3.51**.

24. Plug the remote cable (782012) into the fitting labeled “Remote 1” on the back of the S.P.T.D. controller. **See Figure 3.51**.

NOTE: The plug labeled “Remote 2” is available for starting recorders, integrators or other equipment.

An extra cable(782013) is included if the user wishes to use this option.

NOTE: The fitting labeled Cryo-trap is available if the user wishes to use the GC Cryo-Trap accessory.

25. Plug in the power cord from the back of the S.P.T.D. controller into a grounded 115V, 10 Amp outlet.

26. Return power to the gas chromatograph.

27. If you purchased a Micro Cryo-Trap for use with the SPTD please refer to the Micro Cryo-Trap Manual for installation information or to Section IX (Accessories) in this manual.

Refer to the “Setup” and “Standard Operating Parameters” sections of this manual for further instructions and operating procedures.

Fig 3.50 Installing the interconnect cable (782010).

Fig 3.51 Plug the remote start cable into the plug labeled "Remote 1" on the rear of the S.P.T.D. controller.

Installation of the Short Path Thermal Desorption System on the H.P. 5890 Series II GC with Electronic Pressure Control Injection Port

When the electronic pressure control injection port is used, a two-valve assembly must be installed into the GC injection port carrier gas lines. This valve assembly is included in the HP5890 Series II with EPC installation kit (part # 782230). This assembly consists of two 24 volt normally open valves wired in parallel. A single cable from the valves plugs into the back of the desorption system controller. This two-valve assembly is also used on the HP6890. Please refer to the following pages related to installation of this valve assembly. Refer to **Figure 7.1** in **Section 7** of this manual for a schematic diagram of the proper installation and operation of the EPC valve assembly.

Follow the directions for HP 5890 installation (pages 3-12 and 3-13) up to and including Step #6 (cutting the carrier line). After the carrier line has been cut, locate the valve assembly (P/N 7819993) and attach the 3-way valve with 1/16" fittings to the carrier line as shown in **Figure 3.52**. Be sure to verify the correct flow direction by looking at the label on the base of the valve.

Resume the regular HP 5890 installation at Step #9 (page 3-15) and proceed up to and including Step #16 (page 3-20). Use 1/8" flexible copper tubing to connect the tee fitting installed in the carrier gas line **(see "Site Preparation" page 2-2)** with the inlet of the remaining valve on the valve assembly. The tubing can be directed through the upper portion of the back panel of the 5890 for this purpose. Connect the outlet of the valve to the fitting labeled "GAS" on the back of the Thermal Desorption Unit with a length of 1/8" PTFE tubing (supplied with the installation kit).

Tighten all connections of the valve assembly, tee, and desorption unit. Turn on the carrier gas, and leak check the connections with liquid Snoop or another appropriate method. Complete the installation, resuming at Step #18.

The GC can be operated in both the split mode and the splitless mode. To operate the splitless mode, the purge valve on the GC should be set to the "OFF" position. In this mode the only paths for carrier gas are through the GC column and out the septum purge. No flow is permitted out of the split vent. Keep in mind, however, that 3.0 ml/min does escape out the septum purge which act as a split. To operate the system in the split mode, the purge valve should be set to the ON position. In this mode, carrier gas passes through the column, out the septum purge and also out the split vent. This purge valve ON or OFF is set on the GC keypad or with the Chem Station software for the GC operation.

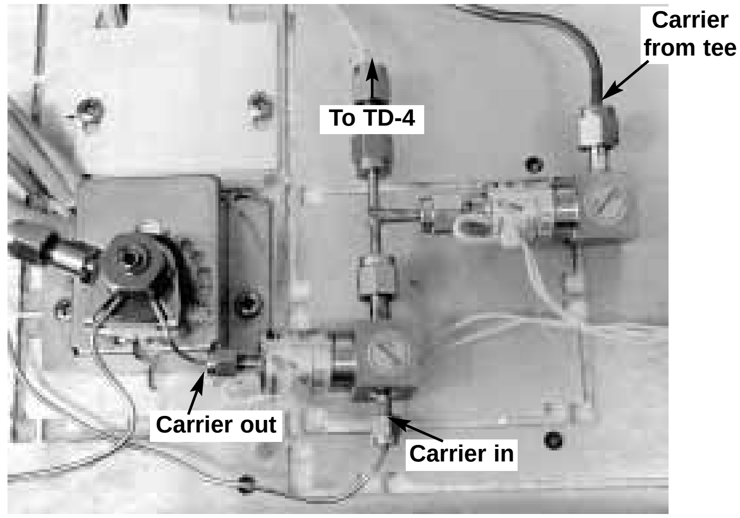


Fig. 3.52 Two gas valve assembly.

*Please order installation kit #782230 which includes all items necessary for installation of the SPTD on an HP GC with EPC

Installation of the Short Path Thermal Desorption System on the Hewlett-Packard 6890 Gas Chromatographs

Unpacking

1. The following items should have been received with your shipment, and are required for installation of the Short Path Thermal Desorption (S.P.T.D.) System on the Hewlett-Packard 6890. Be sure you have all the items before proceeding. If any items were not received with your system call us immediately. See **Figure 3.53 and 3.54** and the parts list included with your installation kit to aid in determining that all items were received. Save all packaging material and boxes if future factory service is required.

Part #	Description	Qty.
Short Path Thermal Desorption System #784000		
784000D	S.P.T.D. Unit	1
784000C	S.P.T.D. Controller	1
784000M	S.P.T.D. Model TD4 Manual	1
784000N	S.P.T.D. Application Notes	1
782010	Interconnect Cable, 6 Ft.	1
783500	Power Cable for Controller	1
HP6890 Installation Kit - #782231		
CL424	Pliers	1
T125062	PTFE tubing, 1/8" OD x 10'	1
B2003	Tee	2
78225	Riser plate	1
781002	GLT tube, 4mm ID	1
781006	SS solid caps for GLT tube	2
781016	Graphite needle seal	1
781018	Graphitized vespel needle seal	1
781053	Needle assy., 35mm side hole	1
781106	Septum adaptor	1
781123	Mounting plate, HP5890 Series I	1
782016	Remote cable for HP6890	1
782013	Remote cable, remote 2	1
7839993	GC gas valve assembly	1

Fig 3.53 The Short Path Thermal Desorption System for the HP6890 gas chromatographs. The system includes all items shown.

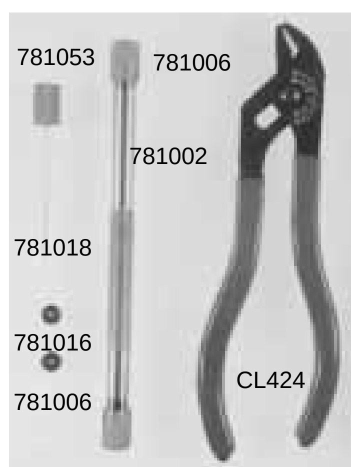


Fig. 3.54 Shows items included in SPTD installation kit #782231 for the HP6890

2. The following are not included with the system but are required for installation.

a. Tubing cutter for cutting $^{1}/_{16}$" S.S. carrier gas line.

b. $^{5}/_{16}$" wrench to tighten $^{1}/_{16}$" Swagelok fittings.

c. $^{7}/_{16}$" wrench to tighten $^{1}/_{8}$" Swagelok fittings

d. Snoop leak detection fluid.

e. Approximately 18" of $^{1}/_{8}$" flexible copper tubing.

NOTE: The S.P.T.D. must be used on the front injection port of the GC if more than one injection port is present. If injection port is installed on rear position it will need to be moved to the front position.

While the SPTD may be installed on various HP inlets, the split/splitless injection port is recommended

CAUTION: BE SURE ALL POWER TO THE GAS CHROMATOGRAPH IS OFF AND THE GC IS UNPLUGGED BEFORE PROCEEDING.

3. Shut off carrier gas at the tank and vent the supply line to the GC. Install the $^{1}/_{8}$" Swagelok™ tee fitting supplied with the installation kit as shown in **Section 2** of this manual **(page 2.2)**.

4. Remove the injection port cover plate and the left side cover plate of the gas chromatograph as shown in **Figure 3.55** to gain access to the injection port. This will require movement of the MSD if installed.

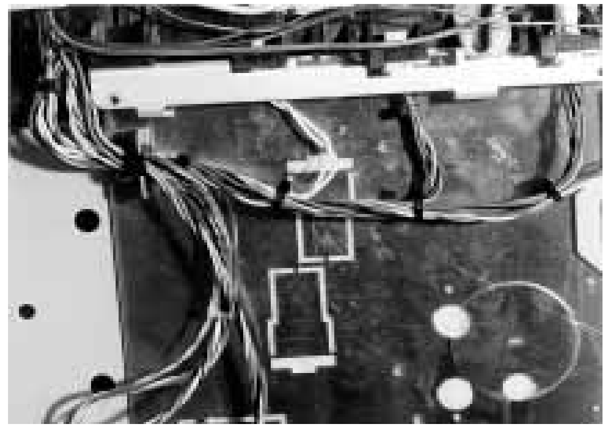


Fig 3.55 View of left side of GC with side cover removed.

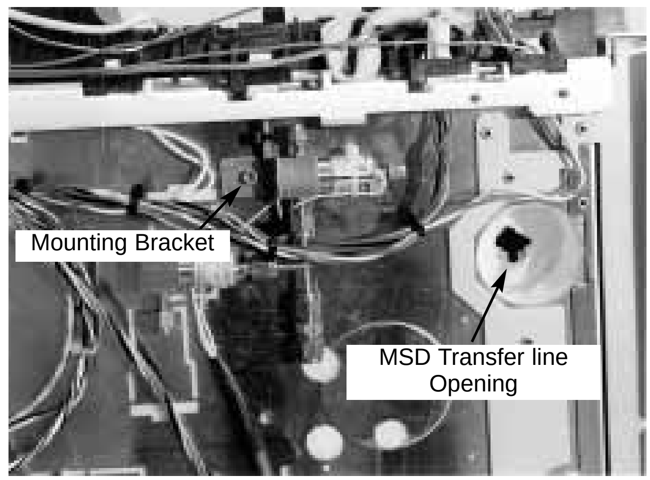


Fig 3.56 Valve Assembly Installed

GC Carrier Gas Solenoid Valve Installation

5. Install the carrier gas solenoid valve assembly (7819993) using the bracket enclosed with the valve assembly. This valve can be mounted on the left side of the GC with the bracket as shown in **Figures 3.55 and 3.56.**

14. Place the septum nut adapter (Part #7811060) which is included in the installation kit over the GC septum nut.

15. Place the tower onto the GC injection port. Align the tower so that the counterbore in the mounting plate on the bottom of the tower fits onto the septum nut adapter.

16. The back of the SPTD Desorption Unit has two 1/8" Swagelok™ fittings for connection of the carrier gas and the air that is used to operate the SPTD system. They are labeled AIR and GAS. See **Figure 3.62.** 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. Be sure that the tubing is fully seated in the fitting and then tighten 3/4 turn past finger tight.

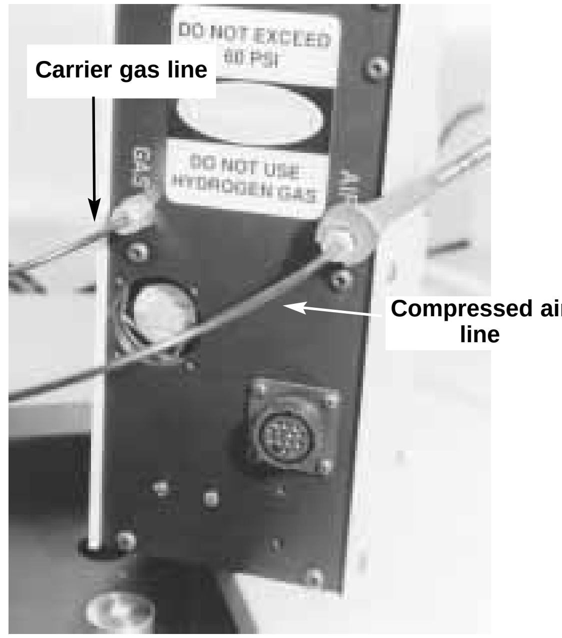


Fig 3.62 1/8" PTFE tubing is used for the carrier gas line and the compressed air line on the S.P.T.D. unit.

WARNING: Do not use hydrogen as a carrier gas in this system. Keep PTFE tubing lines clear of GC exhaust and other areas which may be hot.

17. Cut an appropriate length of the PTFE tubing to connect the fitting labeled “AIR” on the tower to the compressed air supply. Be sure the fitting is tight and leak free.

18. 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 3.63.**

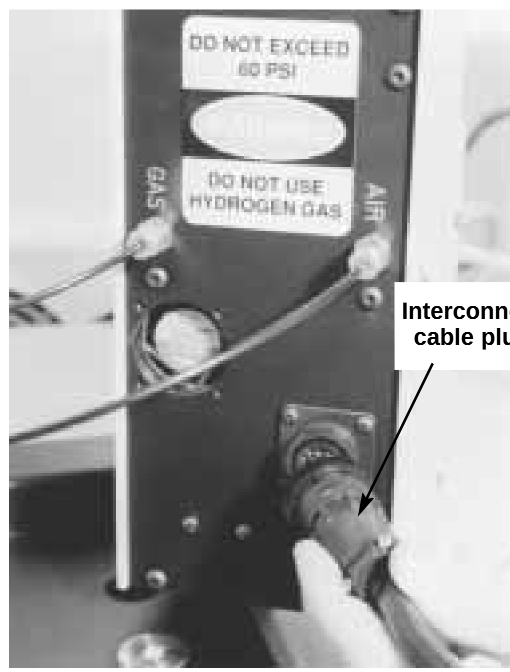


Fig 3.63 Installation of the interconnect cable (782010)

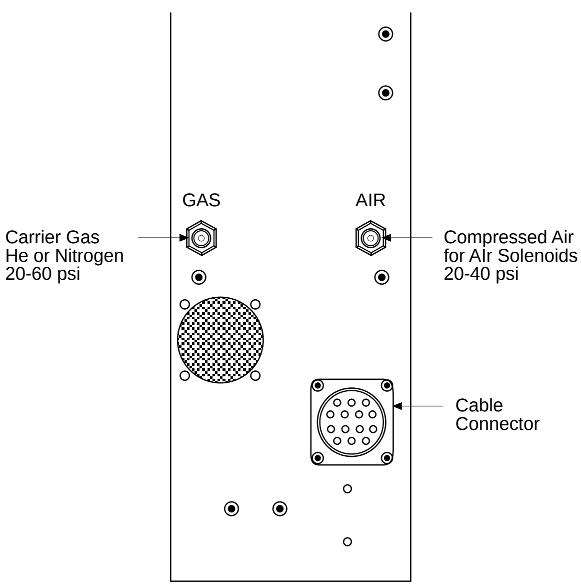


Fig 3.61 Schematic showing the location of the gas inlets on the rear of the S.P.T.D. unit.

13. Attach the mounting plate (Part #78225) which is included with the desorption unit installation kit to the bottom of the desorption tower. The screws for securing this plate to the tower are included with the plate. See **Figures 3.59 and 3.60**.

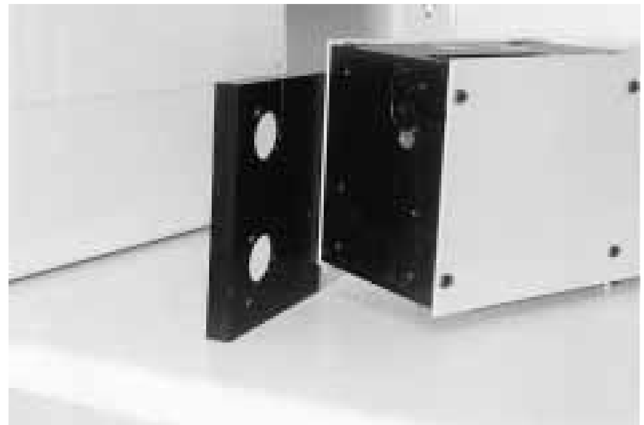


Fig 3.59

Fig 3.60

14. Place the septum nut adapter (Part #7811060) which is included in the installation kit over the GC septum nut.

15. Place the tower onto the GC injection port. Align the tower so that the counterbore in the mounting plate on the bottom of the tower fits onto the septum nut adapter.

16. The back of the SPTD Desorption Unit has two 1/8" Swagelok™ fittings for connection of the carrier gas and the air that is used to operate the SPTD system. They are labeled AIR and GAS. See **Figure 3.62.** Connect the free end of the T tubing from the carrier gas solenoid valve assembly to the "GAS" fitting on the back of the Desorption Unit. Be sure that the tubing is fully seated in the fitting and then tighten 3/4 turn past finger tight.

WARNING: Do not use hydrogen as a carrier gas in this system. Keep PTFE tubing lines clear of GC exhaust and other areas which may be hot.

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

18. 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 3.63.**

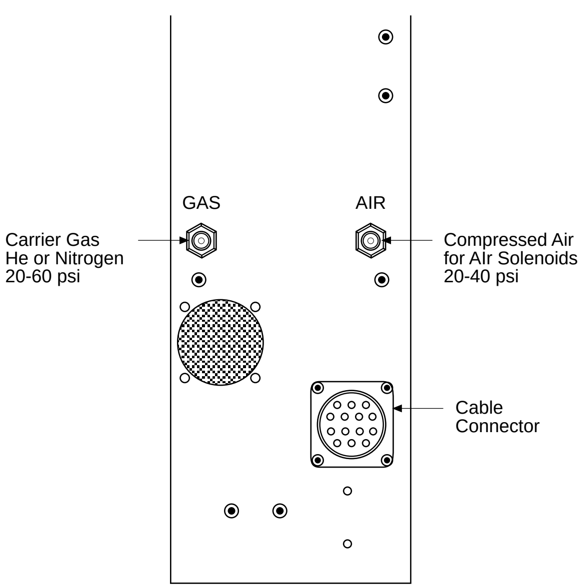


Fig 3.61 Schematic showing the location of the gas inlets on the rear of the S.P.T.D. unit.

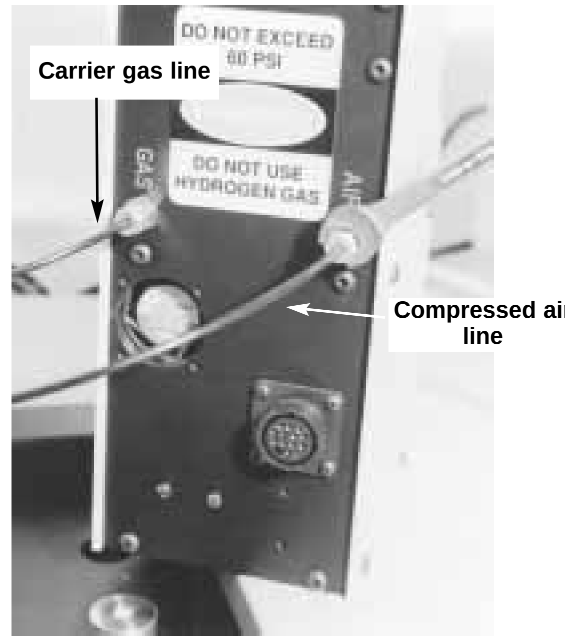


Fig 3.62 1/8" PTFE tubing is used for the carrier gas line and the compressed air line on the S.P.T.D. unit.

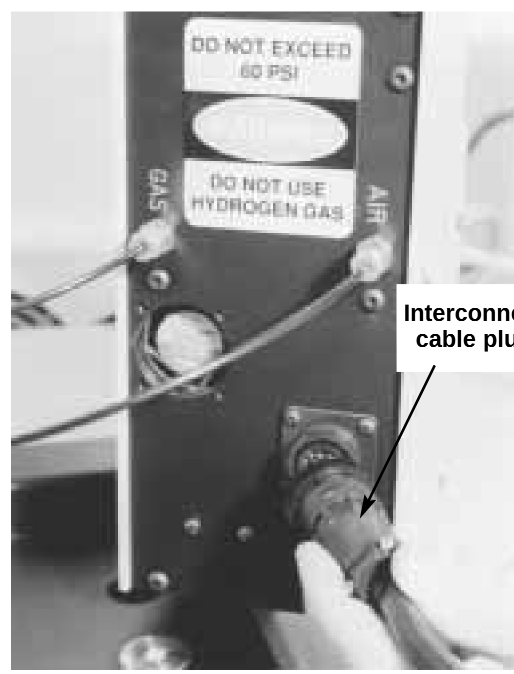


Fig 3.63 Installation of the interconnect cable (782010)

Installation of the S.P.T.D. Controller

20. The S.P.T.D. controller can be positioned next to the unit as shown in **Figure 3.64** or can be positioned on the bench next to the GC. **Figure 3.65** shows the back of the controller.

Fig 3.64 The S.P.T.D. unit installed on the HP6890.

Fig 3.65 Rear view of the S.P.T.D. controller.

21. Plug the interconnect cable (782010) into the appropriate connection on the back of the controller. **See Figure 3.66.**

22. Plug the cable from the GC carrier gas solenoid valve (7819993) into the fitting labeled “GC Valve” on the back of the S.P.T.D. controller. **See Figure 3.67**.

23. Plug the remote cable (782016) into the fitting labeled “Remote 1” on the back of the S.P.T.D. controller. **See Figure 3.67**.

NOTE: The plug labeled “Remote 2” is available for starting recorders, integrators or other equipment.

An extra cable **(782013)** is included if the user wishes to use this option.

NOTE: The fitting labeled Cryo-trap is available if the user wishes to use the Micro Cryo-Trap accessory.

24. Plug in the power cord from the back of the S.P.T.D. controller into a grounded 115V, 10 Amp outlet.

Refer to the “Setup” and “Standard Operating Procedures” sections of this manual for further instructions and operating procedures.

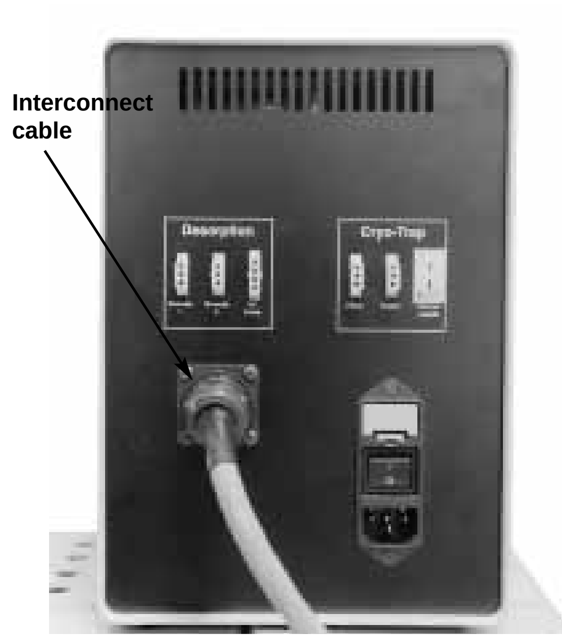


Fig 3.66 Installing the interconnect cable (782010).

Fig 3.67 Plug the remote start cable into the plug labeled "Remote 1" on the rear of the S.P.T.D. controller.

Installation on other top loading GCs

The S.P.T.D. system can be installed on most top loading gas chromatographs. For example the system has been installed on the Varian 3700 and the Perkin-Elmer AutoSystem GC. Please call our customer support personnel for help in installing the desorption system on these gas chromatographs.

The S.P.T.D. system is manufactured to provide a variety of options for mounting and installation. The bottom plate of the Desorption Unit has 6 drilled and tapped holes into which spacers or standoffs can be attached to provide legs for the desorption unit if required **(Figure 3.68)**. These tapped holes enable the user to adapt the thermal desorption unit to most models of GCs. By attaching #10-32 machine bolts with washers, standoffs or other suitable spacers, the height of the Desorption Unit from the top of the GC injection port can be adjusted. These drilled and tapped holes also permit the user to permanently attach the Desorption Unit to the GC cover if so desired. However this is not normally necessary, and is not recommended. Gravity firmly holds the Desorption Unit in place. It will not move or tip over even when injecting through hard 3-layer type septa.

Custom septum nuts, adapter fittings, and spacers can be custom manufactured by S.I.S. to adapt various models of GCs to the desorption system. Custom mounting plates can be designed and fabricated to fit a particular instrument. Please call SIS customer support personnel for further information.

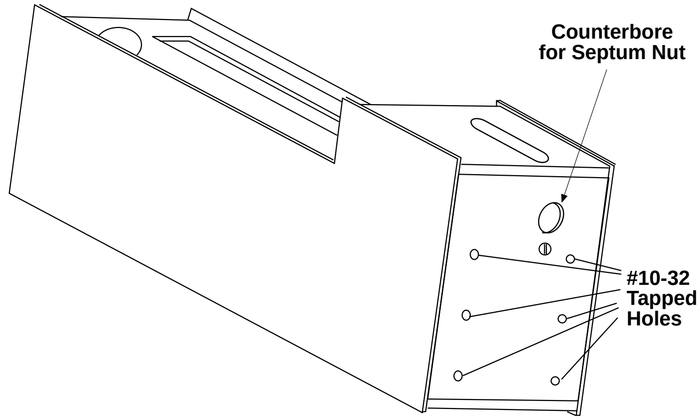


Fig 3.68 Tapped holes on bottom of S.P.T.D unit aid in installation.

IV. DESORPTION UNIT

Description of Desorption Unit

The Thermal Desorption Unit is designed as a compact self contained injection and desorption system that mounts directly over the GC injection port. The autoinjector permits the user to inject the desorption tube with needle attached into the GC injection port with a push button on the Electronics Console or with the built in automatic control software. The desorption tube and needle are attached to the autoinjector assembly and carrier gas flows through the tube and needle continuously when activated. The carrier gas flow through the desorption tube is regulated by a flow controller valve mounted on the top of the Desorption Unit. The flow can be monitored by either a two ball rotameter or a pressure gauge, both of which are mounted on the front of the Desorption Unit. The rotameter enables the measurement of carrier gas flows between 1 and 120 ml/min. The pressure gauge permits the measurement of carrier gas pressures at the top of the desorption tube between 0 and 60 psi. The front viewport at the bottom of the desorption unit permits the easy viewing of the injection port and desorption tube when injected and also provides for the cooling of the desorption tube when the desorption heater block is not activated. It can be easily observed that the needle is proceeding properly into the GC injection port and that the Desorption Unit is properly aligned with the GC injection port.

The Desorption Unit is designed with a wide variety of components built into the case **(Figure #4-1)**. The carrier gas flow system inside the desorption cabinet consists of a valve, a flow controller, a pressure gauge, and a 2-ball rotameter. The autoinjecting system consists of a valve, an auto-injector air slide column, and assembly block. The desorbing system consists of a solenoid and the heater block assembly. In addition, a cooling fan maintains the temperature inside the cabinet and a heat overload thermostat provides protection from system overheating.

The drawings in **Figure #4-2** show the Desorption Unit with the sides, rotameter, pressure gauge and the top and front plates removed to provide a visual representation of its operation. Samples to be analyzed are collected inside the GLT desorption tubes described later. When ready to be analyzed, a needle is attached to the desorption tube and then the tube is attached to the connecting tube on the autoinjector assembly on the Desorption Unit (LOAD POSITION). The carrier gas through the Desorption Unit is turned on via the Electronics Console and the flow through the desorption tube is adjusted with the flow controller and monitored by the rotameter and/or the pressure gauge. The desorption tube and needle are injected into the GC inlet (INJECTING).

When injection is complete (INJECTION COMPLETE), the flows are readjusted as required by the method of analysis, (i.e. split/splitless, etc.). In this position the sample is not being desorbed into the GC since the heating blocks have not yet closed around the desorption tube. The temperature of the tube remains close to room temperature due to the action of the cooling fan. Carrier gas flows, desorption temperatures, and GC parameters can be adjusted as required.

The microprocessor control actuates a valve which moves the hinged heating blocks from the open to the closed position around the desorption tube (HEAT & DESORB). The tube ballistically heats up to the set temperature, or the temperature program ramp for the heater blocks begins. The combination of the heat applied and the carrier gas flow through the desorption tube purges the desired components into the GC injection port and onto the front of the GC column.

The various parameters are set and used according to the application requirements. Normally desorption temperatures between 70°C and 250°C are suitable for most applications. The maximum desorption temperature permissible with the system is 350°C. A heat overload thermostat located under the fan has been included to prevent overheating and subsequent failure of components **(Fig. #4-1)**. The “Reset” buttons for this thermostat is located in its center. In order to reset the thermostat, the left side of the desorption cabinet must be removed to access the reset button. In the event of repeated tripping of the thermostat, both the desorber and electronics console should be returned to the factory for service.

The heater blocks can be ballistically heated or temperature programmed at ramp rates up to 40°C/min. Normal desorption times vary from 3 minutes to 15 minutes, however longer desorption times up to 100 minutes are permitted. Carrier flow through the desorption tube can be accurately adjusted from 1.0 ml/min to 110 ml/min using the two-ball rotameter and flow controller (i.e. 1.0 ml/min for direct splitless analysis and 100 ml/min for split methods permitting split ratios of 1 to 100). Since the column is normally maintained at subambient temperatures, the desorbed compounds of interest are trapped on the front of the GC column in a narrow band. Despite the long desorption times, the peaks eluted from the column are extremely sharp and well resolved.

The Micro Cryo-Trap can be purchased as an accessory for the SPTD System. The Micro Cryo-Trap consists of a small heating/cooling chamber which is ¾" in diameter and 1" long. In the center of the chamber is a small stainless steel capillary through which the fused silica capillary column freely passes. Capillary columns up to megabore (0.53mm I.D.) diameters can be used. Around the stainless steel capillary tube a heating coil is wound to provide for the rapid heating of the capillary tube. A thermocouple provides accurate measurement of both the cooling and heating temperatures. Either liquid CO_2 or liquid nitrogen for cooling is introduced into the Micro Cryo-Trap, and is exhausted through the outlet which can either be vented either into the GC or outside the oven. Control of the Micro Cryo-Trap is provided by output signals from the SPTD Model TD-4 controller unit.

Autoinjector Description

Use of the autoinjector, which is controlled by the electronics console, permits the injection and removal of the needle assembly from the GC injection port without physically handling the desorption tube during the injection process. This is quite important, since the tube is often at 250°C or higher after the sample has been desorbed. After injection and desorption of the sample into the GC, it is important not to touch the desorption tube immediately after its removal from the injection port. Allow it to cool a minimum of 5 minutes after the end of the heating cycle.

The desorption tube and needle assembly is self aligning when the Desorption Unit is set in place over the GC injection port nut. The end of the needle should line up with the approximate center of the passage hole in the middle plate of the desorption unit base. The straight injection motion of the autoinjector and the cone shaped design of the GC septum nut provide for accurate needle penetration through the GC septum. The weight of the Desorption Unit is sufficient to maintain the position of the unit during injection, even through hard rubber septa. Compressed air from laboratory system supplies or from commercially available tanks may be used for the Desorption Unit. If compressed air is used for the flame ionization detector on the GC, this supply may also be used to provide the air for the Desorption Unit. The SPTD system however will use relatively large quantities of compressed air. Gas pressures between 40 and 60 psi are required to activate the autoinjector. For prolonged life, the gas supplied to the autoinjector should be filtered to contain no particles larger than 50 microns. A standard laboratory air filter will normally provide for this purity. Water traps are also recommended.

NOTE - Adjustments to the autoinjector should be performed by a technician knowledgeable in these systems. Extreme caution must be exercised when adjusting the system. Moving parts of the autoinjector that are under high pressure can be dangerous. It is **RECOMMENDED THAT THE DESORPTION UNIT BE SERVICED BY A TRAINED TECHNICIAN AT S.I.S.** when adjustment is required. S.I.S. is not responsible for damage done by untrained individuals making system adjustments.

Heater Block Operation

Activating the 'Desorb' switch on the Electronics Console will close the desorption block assembly around the desorption tube and the tube will be heated to release the sample volatile

Short Path Thermal Desorption Interior View

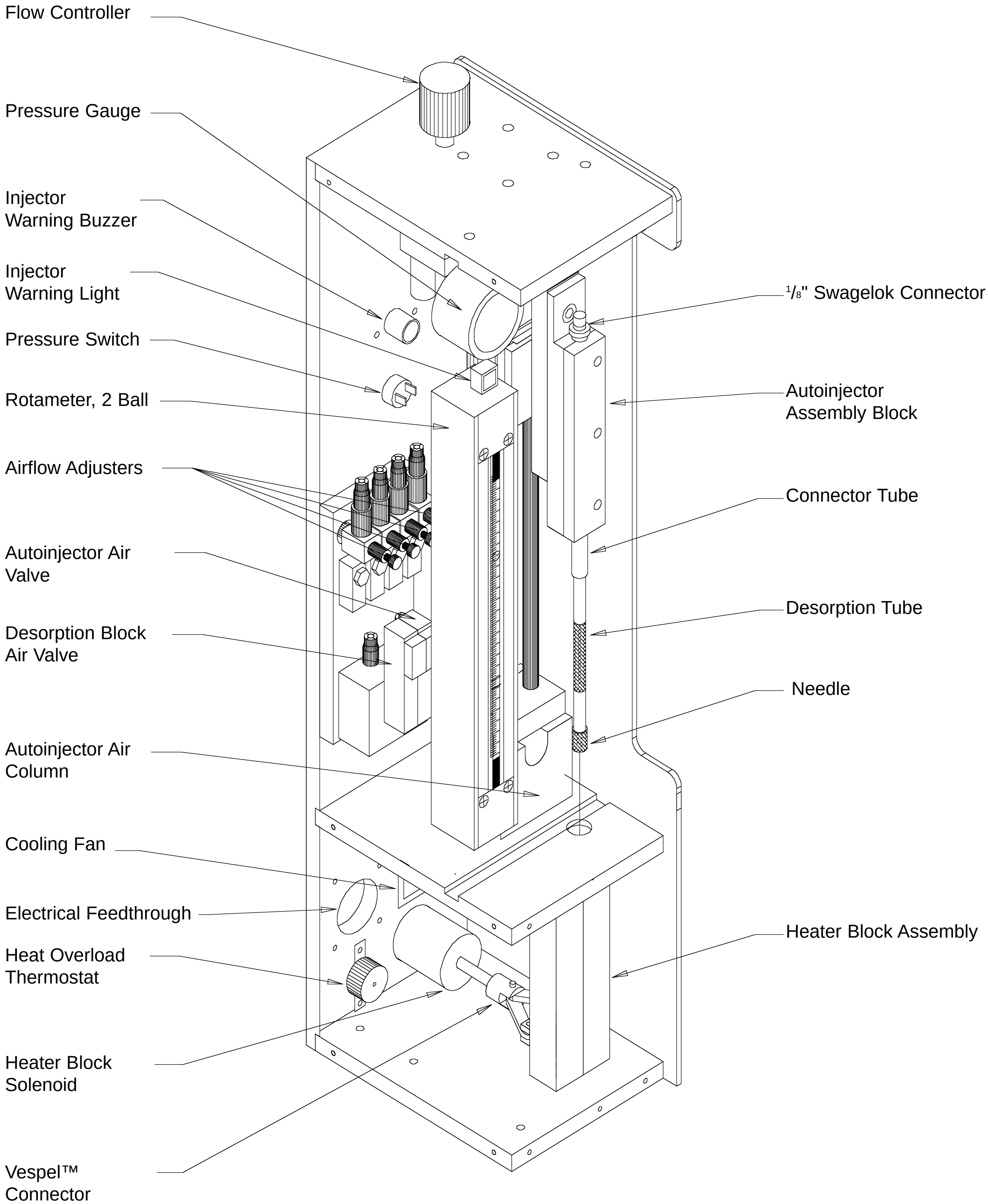


Fig.4-1

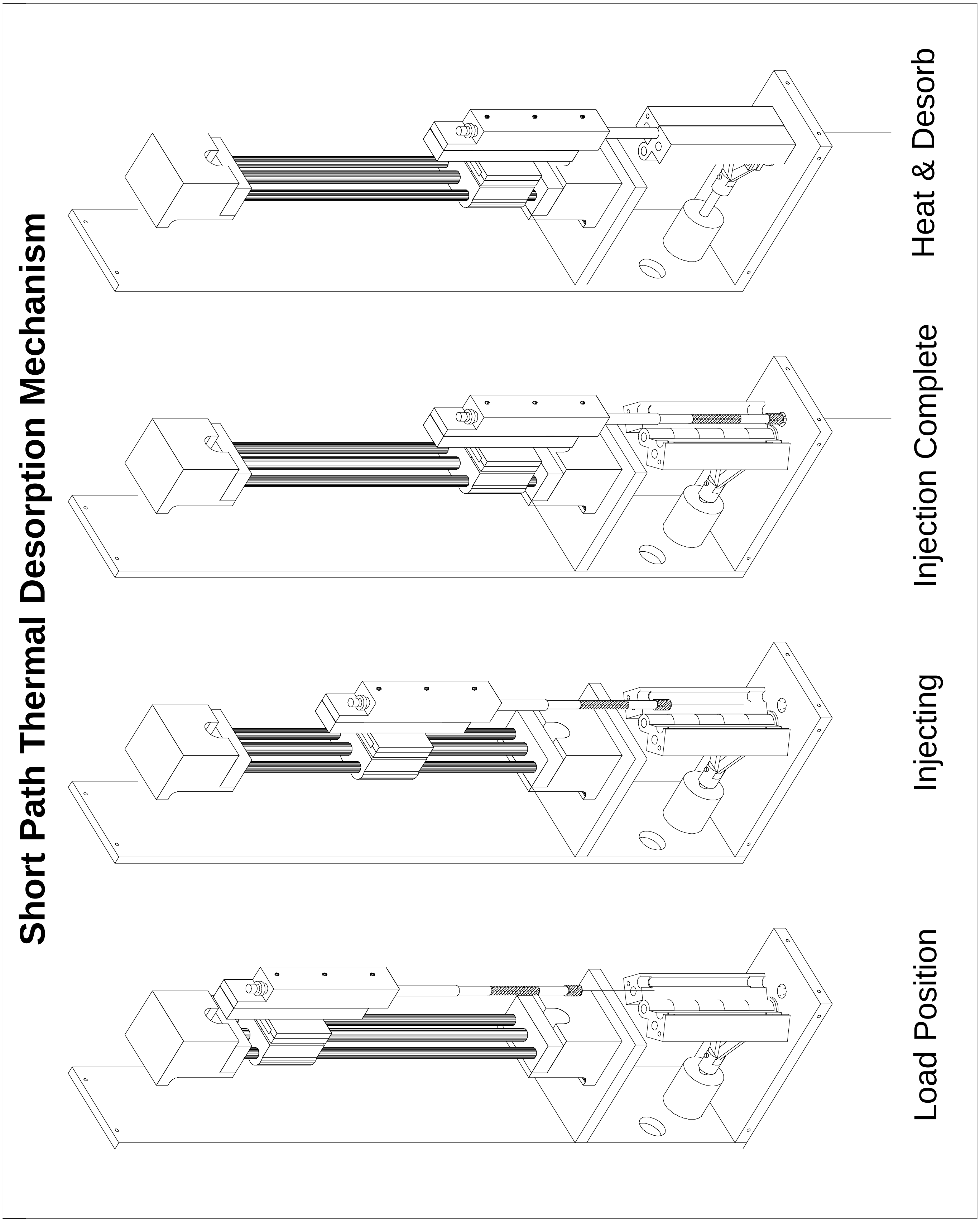

Short Path Thermal Desorption Mechanism
Load Position
Injecting
Injection Complete
Heat & Desorb

Fig.4-2

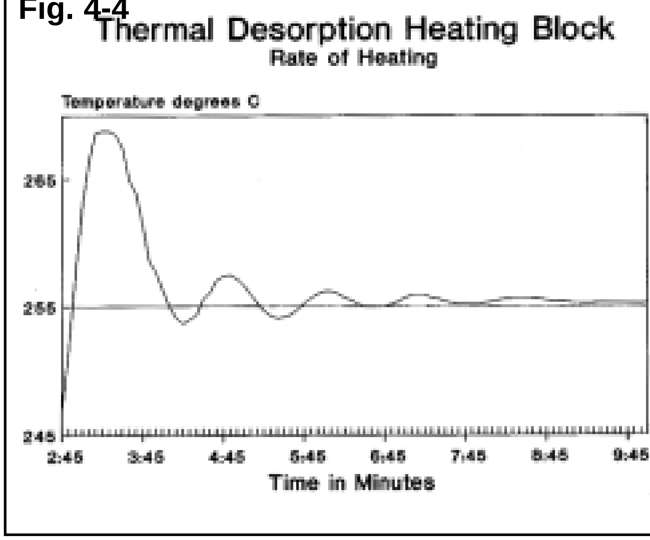
components into the carrier gas as it passes through **(Fig. 4-2)**.

The heating blocks have a high coefficient of heat transfer so the desorption block assembly can be quickly heated and cooled during operation. Due to this ability to rapidly heat and cool it is not necessary to leave the desorption heaters on continuously. The heater cartridges should be turned off when not in use, especially when unattended overnight. This will prolong the life of the heaters and related circuitry. The blocks will return to room temperature between samples. **DO NOT** leave heaters on overnight.

Each of the heating blocks is provided with a 200 watt cartridge heater. The desorption block assembly also includes a 100 ohm platinum resistance thermometer (PRT) mounted in one of the aluminum blocks. The PRT provides feedback to enable the Electronics Console to regulate the temperature as well as provide an accurate temperature indication for the desorption block assembly. The PRT enables temperature control of the heating blocks to within +/-1°C, and over a range from room temperature to 350°C.

The chart below shows the rate of heating of the desorption block assembly **(Fig. #4-3).** When the system is first turned on and the desorption block temperature is set via the digital temperature controller on the Electronics Console, the final temperature can be achieved in 5 minutes or less, indicating that the system is ready for injection and desorption of samples. The auto tuning feature of the temperature controller quickly adjusts the block temperature to its set position with minimal cycling. The enlargement of the top portion of the heating cycle curve **(Fig. #4-4)**, indicates an initial overshoot of the required temperature and then gradually decreasing amplitude of the temperature cycling.

Fig. 4-4



The next chart **(Fig. #4-5)** indicates the rate of cooling for the desorption blocks. Beginning at an initial temperature of 250°C, the desorption heaters were turned off, and allowed to cool via air circulation provided by the internal cooling fan. The desorption blocks cooled down close to room

Fig. 4-3

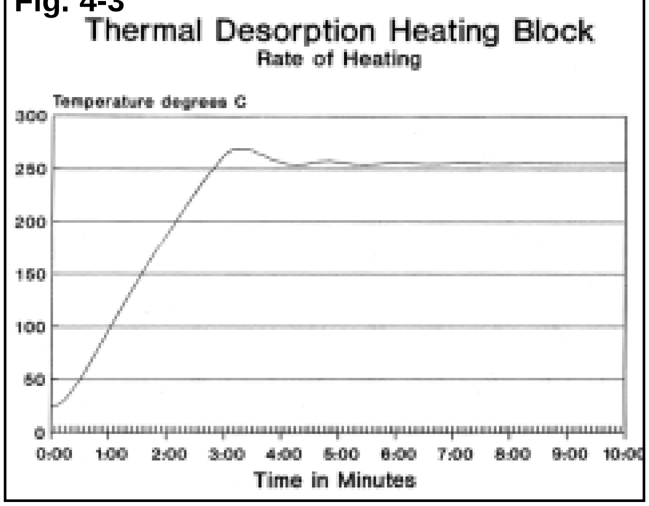
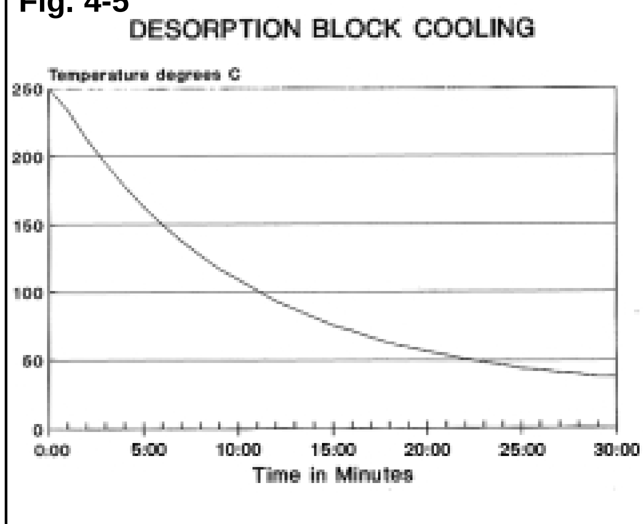
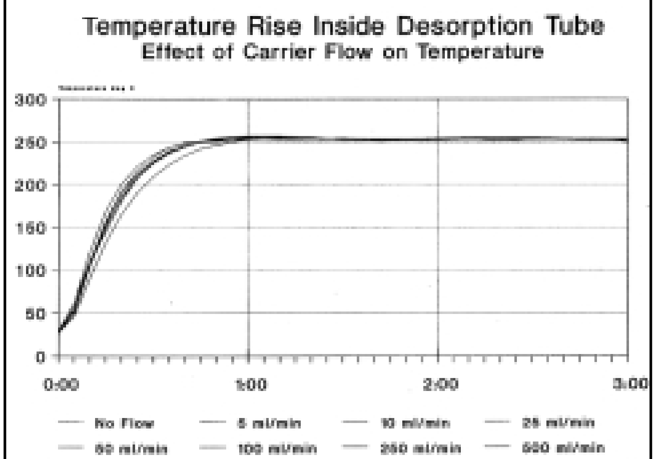


Fig. 4-5



temperature after about 30 minutes. This is the recommended time to allow the system to cool before the main power switch on the Electronics Console is turned off. By leaving the main power switch on, the cooling fan will continue to circulate and cool the Desorption Unit after the heating blocks are turned off. This routine cooling sequence will provide for maximum life and performance of the Short Path Thermal Desorption System.

Fig. 4-6



The next chart indicates the rate of temperature rise within the interior of the desorption tube **(Fig. #4-6)**. Temperatures were measured in the center of the desorption tube with a 0.010” diameter Type J thermocouple. Studies were performed at a wide variety of carrier gas flows through the desorption tube to test the effect of the gas flow on the rate of heating. The lowest line on the curve between 20 and 50 seconds is the rate of temperature with no carrier flow through the desorption tube . The remaining curves are superimposable. The rate of carrier gas flow through the desorption tubes appears to have no effect on the rate of heating or on the final temperature of the interior of the desorption tubes over the flow range of 5 ml/min to 500 ml/min tested. The desorption tube interior heats up rapidly, at a rate greater than 200°/min., and the final temperature is reached in less than 1 minute. Samples need to be desorbed for at least 1.0 minute to reach a temperature of 250°C. Additional desorbing time will purge the required components from the adsorbent medium during this time. Samples should be desorbed a minimum of 5 minutes at an appropriate temperature to achieve maximum recovery of the higher boiling components.

Fig. 4-7

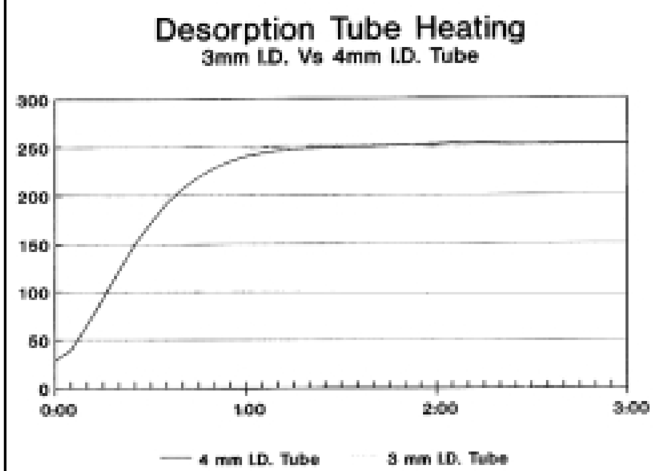


Figure #4-7 compares the rates of heating of the 3.0mm I.D. desorption tube and the 4.0mm I.D. desorption tube. The curves of the interior temperatures are near identical for both sizes, with the final temperature being achieved in less than 1.0 minute.

In no case should the desorption blocks be heated to temperatures exceeding 350°C. A manual heat overload thermostat is located on the back plate of the desorption unit base to protect the desorption unit from overheating. This thermostat is wired in series with the cartridge heaters. In the event the temperature at the thermostat exceeds 60°C, the circuit will be opened and the heater blocks will no longer be heated until the temperature inside the desorption unit falls below this temperature level. In order to restore the heating capabilities of the desorption system, the left side of the desorption cabinet must be removed and the thermostat reset button pushed IN to reset the desorption system heaters. In the event of repeated tripping of the heat overload thermostat, both the Desorption Unit and Electronics console should be returned to the factory for servicing.

The heater blocks for the SPTD System can be programmed to heat up between two temperatures at ramp rates of up to 40°C/ min. It is often desirable, especially in direct thermal extraction (DTE methods, to ramp the desorption temperature and avoid exposing lighter volatiles to extreme heat. After desorption is complete, the heaters are automatically shut down to prevent the buildup of excess heat inside the unit. It is necessary to push the 'Reset' button on the Electronics Console to return the blocks to the initial temperature for the next sample.

Carrier Gas Flow & Regulation

The carrier gas supplied to your GC is used in the SPTD to purge samples into the GC injection port. The SPTD was designed for systems that use helium or nitrogen carrier. **In no case should hydrogen be used in the SPTD due to the possibility of explosion.**

A solenoid valve that can be activated from the Electronics Console controls carrier flow in the Desorption Unit. Pushing the 'Purge Gas' button on the console manually toggles gas flow on and off in the unit. The solenoid valve is activated automatically in the 'Auto Run' mode.

The carrier gas flow regulator (located on top of the Desorption Unit) is a mass flow control device capable of accurately delivering a set gas flow regardless of changes in downstream pressure. The controller is normally configured to deliver flow rates between 1 and 110 ml/min, however other configurations are available. Contact SIS technical support for more information.

Rotameter

A two ball rotameter is mounted on the front panel of the Thermal Desorption Unit **(Fig. #4-1)** and permits the visual indication of the carrier gas flow. The 150 mm flow tube contains a glass ball for flow ranges of 0 to 30 ml/min of air and a second carboloy ball for flow ranges of 0 to 130 ml/min of air **(See Figure 4-8)**. If required other flow range tubes can be factory installed in the desorption unit. Contact SIS for additional details. No special maintenance is normally required for the rotameter. Dirt or contamination may create problems within the flow tube by causing a restriction in the flow passage. Such conditions can be easily diagnosed by examining the flow tube.

Flowmeter Calibration Data

Fig.4-8

Standard conditions	1 atmos @ 70 deg. F
Metering Temperature	70 deg. F
Metering Pressure	14.70 PSI (1 atm)
Metered Fluid	Air

Scale Readings	Glass Ball Flow (Black)	Carboloy Ball Flow (Silver)
150.0	29.3 ml/min	130 ml/min
140.0	24.9	112
130.0	22.2	101
120.0	20.8	91
110.0	17.7	78
100.0	14.1	67
90.0	12.2	58
80.0	10.9	50
70.0	8.8	40
60.0	7.0	34
50.0	5.7	30
40.0	4.9	25
30.0	4.0	20
20.0	3.1	15
10.0	1.9	9

The most obvious indication of obstruction is if the ball is stuck in the flow tube. If the existence of contamination is determined it will be necessary to remove the flowtube from the frame and disassemble the float and top and bottom retaining plugs from the flowtube. Use tweezers to handle the floats and the plugs and store them in a container with a lint free surface. Note the order of removal of the two balls so that they can later be reinserted in the same order. Using an ultrasonic cleaner, clean all parts including the flowtube, rinse and thoroughly dry. Reinsert the parts and test for free motion before reinstalling in the system.

In addition to permitting the visual regulation of the carrier gas flow through the desorption unit, the rotameter is used to sense when problems are occurring in the operation of the desorption unit. For example in the splitless mode of operation at low flows, the ball in the rotameter normally falls down to zero upon the initial injection of the desorption tube syringe into the GC injection port, but will eventually rise back up to its set value. This is due to the initial surge of backpressure from the gas pressure in the GC injection port. If the rotameter continues to read zero, it indicates that the needle is probably clogged. If after desorption has begun, the flowmeter ball continues to slowly fall to zero, it indicates that the column is beginning to plug. This is most likely due to the formation of an ice plug at the front of the column if cryo-cooling is used. See the troubleshooting section for details of how to eliminate this problem.

Pressure Gauge

A 0-60 psi pressure gauge is mounted on the front of the Desorption Unit **(Fig. #4-1)**. This gauge can be used in conjunction with the pressure gauge on the GC injection port to regulate the operation of the system as well as troubleshoot when problems are occurring such as leaking seals, plugged needles and bad septa in the GC. The pressure gauge on the Desorption Unit measures the carrier gas pressure at the top of the desorption tube. The pressure gauge on the GC measures the pressure in the injection port and upon injection the pressure at the bottom the desorption tube. With experience, the user should develop the ability to monitor system performance using the pressure gauge and rotameter. See troubleshooting section for more information.

Flexible Connecting Line

A $^1/_8$" diameter coiled PTFE gas line provides the flow path of carrier gas from the pressure gauge inside the Desorption Unit to the top of the desorption connector tube. It permits the autoinjector to move up and down while still providing carrier gas through the desorption tube.

Connecting Tube

The connecting tube is machined from stainless steel, and provides the fitting into which the desorption tubes are attached, as well as an $^1/_8$" Swagelok™ fitting at the top for attachment to the flexible connecting line. Both a graphite seal with a metal insert <u>and</u> a graphitized Vespel™ seal must be used to provide a leak free connection to the desorption tube. As always, the seals must be inserted so that the end of the desorption tube contacts the stronger Vespel™ seal, with the graphite material between the connecting tube and the Vespel™ piece. Reversing these seals will result in improper sealing and disintegration of the soft graphite piece. See **Figure 4-9**. The harder Vespel™ material provides a more durable sealing surface, however more torque is required to produce an adequate seal. It may be necessary to use a small pair of pliers to turn the tube an additional $^1/_{16}$ to $^1/_8$ turn beyond finger tight to obtain a leak-free seal. **Overtightening of the desorption tube should be avoided**, however, because it leads to excessive wear and cracking of the seals.

The connecting tube can be removed and packed with a suitable adsorbent material to provide a final conditioning step for the carrier gas. This is recommended particularly for those who use the direct thermal extraction technique on powdery samples. Once packed, the connecting tube can be periodically cleaned in the SIS Conditioning Oven by replacing one of the tube conditioning handles with the connecting tube, and inserting it in the oven. The best adsorbent materials for this purpose are Carbosieve™ SIII and Carboxen™ 569. These

Properties of Various Materials Used in Sealing Washers

	Graphite	Graphitized Vespel™
Abrasive Resistance	P	Ex
Heat Resistance	Ex+	Ex
Maximum Temperature	450°C	350°C
Sealing Properties	EX	G
Ease of Removal	G	EX

P - Poor
EX - Excellent
G - Good

Fig. 4-9

materials are spherical in shape, and present less backpressure when packed in the connecting tube. They are also both quite aggressive, and will provide many hours of continuous use between conditionings. When reattaching the Connecting Tube, take care to leave a 3/16" space between the bottom of the Swagelok fitting and the mounting block.

Pressure Switch

A pressure switch is located inside the desorption unit in the carrier gas line. The purpose of this switch is to detect carrier gas leaks when the system is used in the automatic mode of operation. After the desorption tube and needle have been injected into the GC, the back pressure on the desorption tube pressure gauge should exceed 3 pounds positive pressure within 30 seconds. If it does not, it is an indication that a gas leak exists either in the injection port of the GC or, more likely at one of the seals of the desorption tube. In the automatic mode of operation the desorption block heaters will not close unless the backpressure in the system exceeds 3 pounds of pressure. This permits the user to retrieve samples before the heater blocks begin desorbing the volatiles into a leaking system. The user can stop the desorption process from continuing by pushing the 'Reset' button on the electronics console.

The pressure switch can be adjusted to change the pressure at which the system will indicate a pressure leak. This is not normally necessary, but a description of the adjustment is printed below. Call SIS Technical Support before adjusting this switch.

1. Looking at the rear of the SPTD unit the adjustment screw access hole is 6.0" from the top of the unit on the left side.

2. Use a 5/64" allen wrench for adjustment through the access hole.

3. Looking from the rear, rotating the wrench counter clockwise will decrease pressure and clockwise will increase the pressure.

NOTE: The pressure switch is only active in the automatic mode of operation.

Warning Indicators

Both a warning buzzer and light **(Figure # 4-1)** are mounted on the desorption unit. The purpose of these warning devices is to notify the user before the injection process begins. The buzzer will begin to sound and the warning light will turn on five seconds before the injection process begins and both devices will shut off at the end of the injection cycle. Both of these devices must be operative at all times. In no case should they be deactivated by the user. In the event of the failure of either of these warning devices, the system should be returned to the factory for service.

WARNING - When the warning buzzer and light are ON, remove hands and other objects from the path of the injection needle.

Accessories for Thermal Desorption System

Part #	Description
781005	GLT Desorption Tube, empty, 3.0mm I.D.
781002	GLT Desorption Tube, empty, 4.0mm I.D.
781006	Cap, Desorption Tube, Solid
781007	Cap, Desorption Tube, Drilled 0.040" Hole
781052	Needle on Cap, 50mm L x 0.63 O.D. x 0.32 I.D., side hole
781053	Needle on Cap, 35mm L x 0.63 O.D. x 0.32 I.D., side hole
781054	Needle on Cap, 25mm L x 0.63 O.D. x 0.32 I.D., side hole
781023	Needle on Cap, 50mm L x 0.63 O.D. x 0.32 I.D.,20° point
781025	Needle on Cap, 35mm L x 0.63 O.D. x 0.32 I.D.,20° point
781024	Needle on Cap, 25mm L x 0.63 O.D. x 0.32 I.D.,20° point
781003	Viton Seals for Caps and Needles, pkg of 10 .210" O.D. x .080" I.D. x .0625" thick
781004	PTFE Seals for Caps and Needles, pkg of 10 .210" O.D. x .120" I.D. x .0625" thick
781015	Graphite Top Sealing Washer w/Metal Insert .210" O.D. x .060" I.D. x .0625" thick
781016	Graphite Needle Sealing Washer .210" O.D. x .040" I.D. x .0625" thick
781017	Graphitized Vespel™ Top Sealing Washer .210" O.D. x .060" I.D. x .0625" thick
781018	Graphitized Vespel™ Needle Sealing Washer .210" O.D. x .040" I.D. x .0625" thick
781070	High Temp. Green Septa for Caps, pkg of 50
781040	Aluminum Tags for Desorption Tubes, pkg of 10
781010	Desorption Tube Hose Connector
781011	Desorption Tube to 1/4" Tube Connector
781012	Desorption Tube to 1/8" Swagelok Connector
781019	Desorption Tube to 1/4" Swagelok Connector

Desorption Tube Cleaning Systems

781051	Desorption Tube Conditioning Oven & Controller, for 6 tubes simultaneously, 6 ball rotameters, 6 desorption tube handles and 2 needle handles, Temperature Programmable Controller
781013	Desorption Tube Handle
781014	Desorption Needle Handle

V. ELECTRONICS CONSOLE

Description of Microprocessor Controlled Electronics Console

The Electronics Console **(Fig. #5-1)** consists of a user adjustable microprocessor which is interfaced with a 20 push button keypad and a yellow fluorescent display to control the operation of the desorption system. The main power switch is located on the back of the Electronics Console and controls the power to the entire desorption system. The heater switch on the front of the controller turns on the power to the heater cartridges in the desorption tube heater blocks. The Cryo-Trap heater switch controls the power to the heater circuit for the Micro Cryo-Trap accessory. The keypad permits the user to interface with the microprocessor to control or modify its operation. The yellow fluorescent display indicates the system status or prompts the user for input.

The desorption system can be operated in either a manual or an automatic mode. The desorption system functions can be activated manually using the function switches on the keypad **(Fig. #5-1)** or an automatic program can be initiated to process a series of timing and switching functions to operate the system unattended. From the main screen and keypad the operator has the choice of beginning the automatic program sequence, modifying the automatic program timers and temperature settings, or manually operating the desorption system (**Fig. #5-2).**

Electronics Circuit

A 115 VAC 10 amp power source provides the total electric power required by the Electronics Console, Desorption Unit and Micro Cryo-Trap accessory. The main fuse circuit breaker controls the incoming power to restrict the total input to less than 10 amps of current.

A single 6' cable assembly provides the connection between the Electronics Console and the Desorption Unit. This cable has two screw on connectors on each end which mate to the fittings located on each of the two assemblies. The cable can be used reversibly since the connectors on both ends are identical. This cable provides the signals to control the operation of all the systems within the Desorption Unit including the heating blocks, cooling fan, gas valve, injector, desorber, buzzer and warning lamp.

Three additional remote terminals are located on the back of the Electronics Console under the word ‘Desorption’. The terminal on the right is the GC carrier gas remote. The plug from the GC carrier gas valve is plugged into this receptacle for the automatic operation of the GC carrier gas valve (see chapter 3 for information on installing this valve). The two other remote receptacles, labeled ‘Remote 1’ and ‘Remote 2’, are for remote start of external devices such as a GC, mass spectrometer, or recorder. At the end of the desorption cycle, the electronics console momentarily closes a relay across the two outputs of the remote plug. This contact closure is the standard method of activating other instruments through the appropriate connections in those instruments. There are also connectors for plugging in the Micro Cryo-Trap if this accessory has been purchased.

A ‘Stop/Reset’ button is provided on the front of the Electronics Console. When this button is pressed, it will light up and all controls will be returned to their ground state. That is: the desorption blocks will open, the injector will return to the up position and the purge gas will be turned off. When the ‘Stop/Reset’ button is pushed once again, the light will turn off and the system will reset the program to the beginning status screen to permit system operation. This switch is designed to be first an emergency switch to cancel the operation of the desorption system at any step of its operation. It will also enable the operator to return the system to the first step of operation in case of system leaks, bent needle or other operation failure.

Desorption Unit
Electronics Console

Fig.5-1

Microprocessor Program Control

Figure 5-2 indicates the microprocessor program logic for the operation of the desorption system. When the system is first turned on, the operator has three choices of program direction: Program Modification, Automatic Operation and Manual Operation. The fluorescent display defaults to the System Status screen which provides information about the current state of the desorption system, including the Cryo-Trap (if present) mode and temperature, the desorption block temperature, and which step of a desorption run is being executed.

Program Modification Mode

The ‘Temperature Set’ and ‘TimeSet’ switches permit the user to adjust the various timers and temperature-controlled zones in the TD-4 and Micro Cryo-Trap.

Time Set

Enter the Time Set menu by pressing the ‘Time Set’ button on the Electronics Console. You will be prompted for input for the various system timers. Values are entered as minutes:seconds. After entering a value for minutes, press the ‘Return’ (↵) key, then enter a value for seconds, and press the ‘Return’ key again. If you make a mistake before pressing the ‘Return’ key, you may use the backspace key (←) to correct it. After pressing the ‘Return’ key, the value cannot be changed, and you will need to step all the way through the ‘Time Set’ menu and start again. Pressing the ‘Return’ key without entering anything accepts the previously set value. Set values will remain in memory even when the unit is turned off or unplugged. **NOTE: All timers must have a non-zero value for automatic mode operation.** If you wish to set a timer for as short a time as possible, use a value of 00:01.

Purge Gas Time describes the amount of time that a sample will be purged prior to injection. It serves to remove oxygen and water from samples, and may be extended to aid the removal of solvents, etc. Allowable times are 00:01 to 99:59 for automatic operation.

Inject Time is the period between injection of the needle into the GC inlet and the closure of the heating blocks around the sample tube. During this time the pressure in the inlet is allowed to equilibrate and inlet pressure and split flow may be monitored and adjusted if necessary. Larger values will allow more time for adjustments, and are usually not detrimental to samples because no heat has been applied to the tube, and materials purged off of the sample in this step enter the GC inlet and are trapped on the column. In the automatic mode of operation, this timer activates the carrier gas cutoff/divert valve at 00:20. Therefore, allowable times for automatic operation are 00:21 to 99:59.

Desorb Time is the amount of time the heater blocks stay closed around the desorption tube. Times of at least five minutes are suggested for quantitative removal of material from most adsorbents, however requirements will vary depending on the application. When using the Direct Thermal Extraction technique, longer desorption times result in larger quantities of higher-boiling material being transferred to the GC inlet. When using a temperature program, remember to allow enough Desorb Time for the program. For example, ramping from 80°C to 180°C at 10°C/minute requires at least 10 minutes of Desorb Time. Allowable values are 00:01 to 99:59 for automatic operation.

Start Delay is an interval at the end of the Desorb Time that allows for reequilibration of the GC pressure and flow before a run is started. The Micro Cryo-Trap, if present, remains in COOL mode for the duration of the Start Delay. Allowable values are 00:01 to 99:59.

Temp Set

Enter the Temp Set menu by pressing the ‘Temp Set’ button on the Electronics Console.

Short Path Thermal Desorption Program Flow Schematic

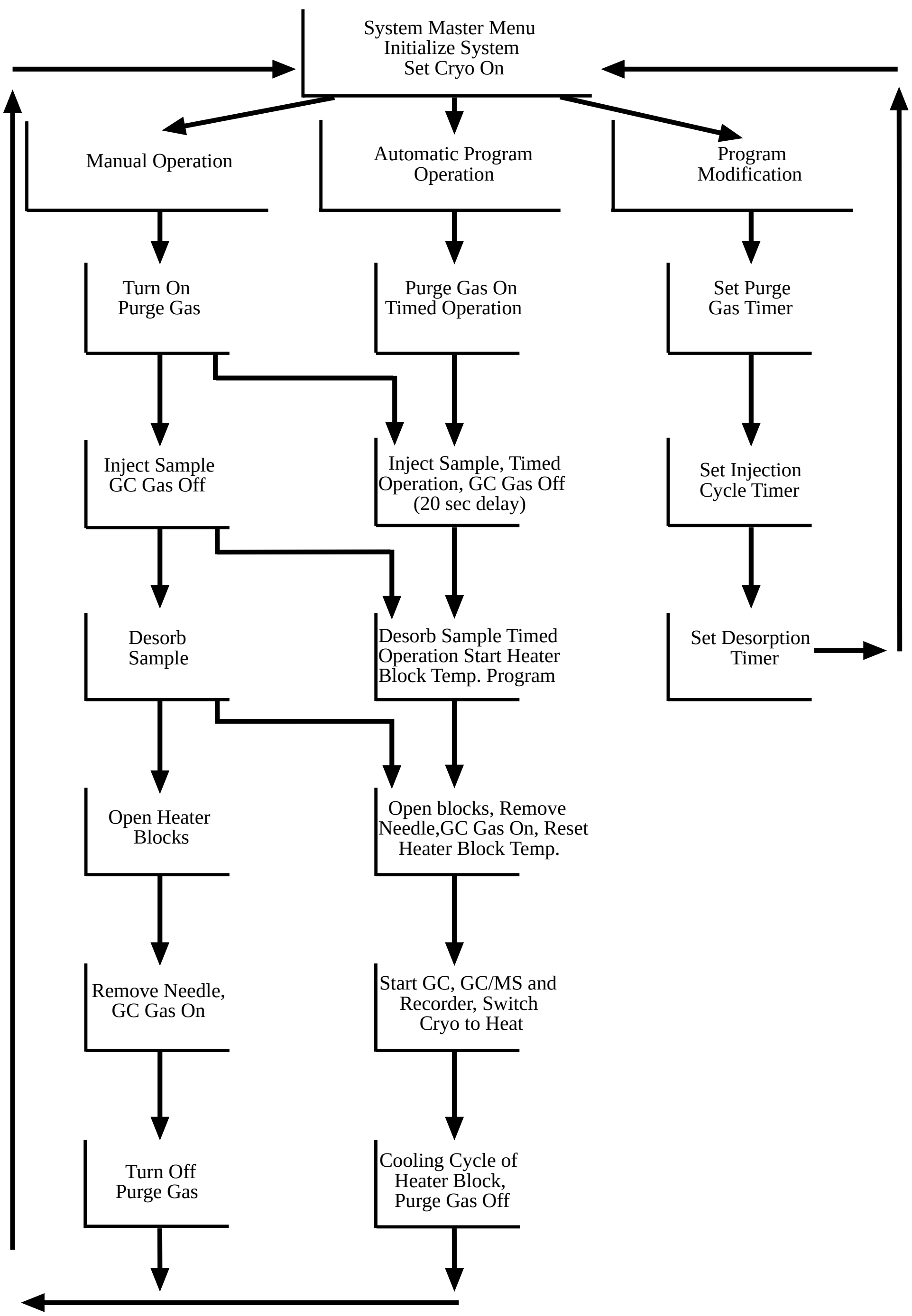


Fig.5-2

As with the Time Set values, press the 'Return' key after each value entered. Mistakes may be corrected in the same way, and entered values cannot be changed once the 'Return' key is pressed, except by stepping through the menu and starting again. Previously set values can be accepted by pressing the 'Return' key without any input.

Modify Desorb Temp is the first step in the Temp Set menu. You will be prompted to enter a starting temperature (Begin) in °C, a ramp rate in °C/minute, and a final temperature (End). To operate without a temperature program, enter the same value for 'Begin' and 'End'; any value for 'Rate' will be ignored. The maximum temperature for the TD-4 is 350°C.

Modify Cryo Temp is the second and final step in the Temp Set menu. The 'Heating' value determines the temperature to which the Micro Cryo-Trap will rise after the desorption and Start Delay are complete. This value should be slightly higher than the GC inlet temperature. The maximum Micro Cryo-Trap temperature is 400°C. The Cooling value sets the temperature at which volatile material is trapped on the GC column head or guard column. The minimum temperature for the Micro-Cryo-Trap is -180°C for liquid nitrogen coolant, and -70°C for liquid CO_2. **NOTE: Liquid nitrogen and liquid CO_2 systems differ significantly. Use only the recommended coolant for your system.** Your system can be converted to use a different coolant; contact SIS technical support for details.

Automatic Mode of Operation

The automatic mode of operation steps the system through the sequence required to inject and desorb the sample into the GC. Press the 'Reset' button on the Electronics Console to assure that the heater blocks are on and at their set temperature. If the Micro Cryo-Trap accessory is used, the Cryo-Trap button should be pushed once to start cooling the trap. When the Cryo-Trap has cooled to near its setpoint, automatic operation may be started by pressing the 'Auto Run' button at the lower left of the keypad. The following sequence of steps will be initiated:

1) The sample will be purged for a set time with carrier gas (Purge Gas Time).

2) The desorption tube (with needle attached) will be driven by the injector into the GC inlet. The tube and needle will be held in this position for a preset time (Injection Time). The GC inlet will now be supplied with two carrier flows, one from the normal inlet, and another from the thermal desorption system.

3) In the case of EPC systems, 20 seconds after injection begins the purge flow will be cut off and the normal carrier flow will be diverted through the TD-4. If your system does not have EPC, the normal carrier will be cut off at this time, but the purge flow will continue and will be used as the carrier through the desorption process. The time between the valve action and the end of the Injection Time can be used to adjust split flows for non-EPC systems, and to verify adequate flow through the Desorbtion Unit for EPC systems. Both of these tasks are accomplished using the flow control knob at the top of the Desorption Unit, and the Injection Time may be extended for this purpose.

4) When the Injection Time expires, the heater blocks will close around the tube and the Desorption Time begins. If a temperature ramp was set up in the program, it will begin at this time. In the event of a leak in the GC inlet or at the desorption tube seals the heater blocks will not close, and the message "Pressure Leak" will appear on the Electronics Console display. If this occurs, press the 'Reset' button, check the inlet pressure and verify that the desorption tube and needle are securely in place, then initiate automatic operation again by pressing 'Auto Run'. Remember to toggle the Micro Cryo-Trap to the COOL position before restarting the run. Refer to the troubleshooting section of this manual if the problem persists.

5) At the end of the preset Desorption Time, the heater blocks will open, and the injector will remove the tube and needle from the GC inlet. A preset Start Delay will begin at this time, and the purge flow through the desorption tube will be maintained for five minutes to cool the tube. Power to the heater blocks is shut off at the end of each run, and it is necessary to press the 'Reset' button on the Electronics Console to return them to their preset initial temperature.

6) When the Start Delay expires, the Micro Cryo-Trap automatically switches from cooling to heating, and the remote start signals are activated to begin the GC run and/ or data acquisition.

Manual Mode of Operation

The manual mode of operation allows the user to move through the series of desorption steps one by one, by pressing the appropriate function key on the Electronics Console. Pressing the same function key again will reverse the selected action. Operation can be changed to the automatic mode at any point in the process by pressing the 'Auto Run' key. In that case, the program would pick up from the current stage of the desorption process and complete the run using the preset time and temperature setpoints in memory. Additionally, a sequential safeguarding function is built into the system to prevent damage from oversight or misuse. This feature prevents the steps from occurring out of sequence. For example, if a user were to press the 'Inject' key before turning on the purge gas, the purge would automatically turn on before the injection was made. **NOTE: The carrier cutoff/divert valve is not activated in the manual operation mode.** Be aware that this creates the potential for excess sample splitting. The temperature ramping capability is also not functional in manual mode. The heater blocks will only maintain the initial temperature setpoint. For these reasons, the manual mode of operation is not recommended for routine analysis, but rather has its greatest utility in troubleshooting, cleaning the system, and method development. The function keys for manual operation are:

Cryo Mode toggles the Micro Cryo-Trap between cooling, heating, and off modes.

Purge Gas activates a solenoid valve in the Desorption Unit that allows carrier to purge the sample tube.

Inject initiates the pneumatically powered injection of the needle into the GC inlet.

Desorb closes the heater blocks around the desorption tube and begins transferring volatile and semi-volatile material from the sample to the inlet.

Note that there is no minimum time requirement for operation in the manual mode, unless the operator switches to the automatic mode prior to desorbing the sample. Also, timer activity is disabled in the manual mode. Each step that is executed in manual mode must also be manually terminated by pressing the same function key again, pressing the 'Reset' key, or activating the next step. That is, once a step is initiated, the system will stay in that step indefinitely.

VI. DESORPTION TUBES

The glass lined stainless steel desorption tubes are available in two inside diameters, 3 mm and 4 mm. Each tube is 4.0" long by 1/4" outside diameter and is threaded on both ends. After conditioning and sample loading, the ends of the tubes are fitted with stainless steel caps with PTFE seals to maintain the integrity of the medium and sample. The threads on the desorption tube also provide the means of attaching the desorption tubes to the connecting tube and the needle.

Samples to be analyzed are collected on the glass lined stainless steel (GLT) desorption tubes that have been previously packed with a porous polymer such as Tenax™ (TA) or activated charcoal and conditioned. Solid samples can also be placed directly in the GLT sample tubes and thermally extracted without the use of adsorbents. The glass lining provides an inert surface for samples and can be silylated if so desired. After sample collection the GLT desorption tubes are capped with stainless steel caps with PTFE liners to maintain sample integrity during storage and transportation. When ready for analysis, the caps are removed and a stainless steel needle is attached to the desorption tube. The collected sample can then be desorbed directly into the injection port of the gas chromatograph.

<u>Features</u>

- Strong stainless steel (S.S.) outer shell
- Inert glass lining
- S.S. caps with seals to prevent contamination
- Available in two inside diameters, 3mm and 4mm I.D.
- Two methods of analysis
- Thermal Desorption from adsorbent resins
- Direct Thermal Extraction for solid samples

Needles for Desorption Tubes

Needles for desorption tubes are available in a wide variety of sizes and shapes. Needles are permanently silver soldered to the caps to provide a strong leak-free assembly. Depending on the application and on the type of injection system of the GC, a suitable length of needle will need to be used. Needle lengths between 25 mm and 60 mm are available. The shorter the needle, the longer the expected life of the needle. Four styles of needles are available, including a special needle for the Jade™ injection system.

NOTE: Side hole needles are recommended for most applications due to the elimination of coring of the septa when these needles are used. The 50 mm side hole needle is recommended for most applications. Refer to your current SIS catalog for the various styles of needles that are available for use with the Short Path Thermal Desorption System.

Cleaning of Desorption Tubes

For a complete detailed description of the conditioning oven and cleaning of desorption tube see the conditioning oven section of this manual.

Desorption Tube preparation is usually a multi step process including:
(1) washing, (2) silylating, (3) rinsing, (4) drying,
(5) packing, (6) conditioning, (7) sealing,
(8) storage

All desorption tubes should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C. Cleaning by additional solvents may be required depending on the applications of the user. It is important to remove all solvent residues by thoroughly baking out the tubes.

Treatment with a suitable silylation reagent such as Dimethyldichlorosilane (DMDCS) is optional depending on the nature of the components being analyzed and the requirements of the user.

The procedures of the manufacturer should be followed including thorough rinsing and baking out of the prepared tubes.

Note: Suitable safety precautions must be taken when working with solvents.

Packing Desorption Tubes

Samples are collected on glass lined stainless steel (GLT) sample tubes packed with a porous polymer resin such as, 2, 6-diphenyl-p-phenyleneoxide sold under the trademark TENAX™ TA or carbon molecular sieve material sold under the trademark CARBOXEN™. There are many other adsorbent materials which function well and are available from many manufacturers including S.I.S. For help in determining the proper adsorbent for your application, visit the S.I.S. website at www.sisweb.com.

The desorption sample tubes are usually packed with approximately 20-200 mgs of adsorbent. The amount of adsorbent used depends on the users' requirements. The ends of the tubes are plugged with approximately 1cm of silanized glass wool on each end to hold the adsorbent in place.

As an alternative, the GLT tubes can be packed with the actual materials to be analyzed for controlled direct thermal analysis of residual components such as packaging materials, construction materials, fibers, paint chips, etc.

Conditioning Thermal Desorption Tubes

In order to prepare the adsorbent packed desorption tubes for the collection of samples, the tubes must be conditioned to remove all foreign materials including water vapor. The following procedure or a suitable version of this method should be used to condition the desorption tubes. The maximum temperature will be determined by the properties of the adsorbent material used in the desorption tubes.

The GLT sample tubes containing the adsorbent are heated from ambient temperature to 300°C at a rate of 4°/min while purging with nitrogen or helium at a flow rate of 2 to 20 ml/min. The tubes are held at the upper temperature limit for not less than four hours for optimum conditioning under continuous flow. After conditioning and cooling under constant flow, the tubes are immediately capped on both ends with stainless steel caps with appropriate liners that have also been conditioned. The GLT sample tubes are then fitted with identification tags. Tubes prepared in this manner exhibit excellent adsorptive capacity and contain no organic background when analyzed by GC/MS.

The stainless steel caps with liners can easily be conditioned by baking out in a GC oven.

Desorption Tube Conditioning Oven

For the conditioning of the desorption tubes, a Desorption Tube Conditioning system is available from S.I.S. which includes a Conditioning Oven and Controller for conditioning 6 tubes simultaneously. The self contained system includes 6 rotameters with flow ranges of 0 to 50 ml/min, 6 desorption tube handles,two needle handles and a digital temperature programmable controller. **(See Section 10)** The Controller system with program memory permits the temperature program ramp described above to proceed unattended. A procedure of up to 6 steps with various ramp cycles and hold times can be programmed into the controller.

The desorption tube Conditioning Oven can also be used to clean the desorption tube needles. Two needle handles are included with each unit . The needles are normally conditioned at 300°C with gas flow.

VII. STANDARD OPERATING PARAMETERS

General Methodology

The SPTD can be installed on instruments from a variety of manufacturers. These instruments exhibit a wide spectrum of flow system designs which when combined with the variety of injection techniques, such as direct injection, split, splitless, and cool-on-column result in many different ways to operate the Short Path Thermal Desorption System. Considerable variation in the configuration of setup is anticipated from one instrument to another. Also, depending on the type of analysis and its unique problems methodology must be developed to analyze these samples using the Short Path Thermal Desorption System. As a result no one methodology can be outlined to suit all situations. The following methodology is intended to be a general guide which can be modified to suit the analyst's needs.

GC Carrier Flow

When using the SPTD system, GC carrier flow through the column is redirected through the desorption system. This is discussed in detail in Chapters 2 and 3 and following. An understanding of your particular GC design is important in determining optimal parameters.

Care must be used in operating the thermal desorption system on instruments such as the Hewlett Packard 5890 series GC's, which operate with back pressure regulation on the injection port. In these instruments, any gas flow which exceeds the capacity of the column at the preset pressure will be diverted out of the split vent. During desorption the only flow into the GC injection port is through the desorption tube. The flow can be accurately regulated to provide either split or splitless injection. **Figure 7-1** shows the flow schematic for the desorption system when using the carrier gas solenoid valve assembly part #7819993. The deactivated mode represents standard GC carrier operation. In the activated mode, during injection and desorption, the carrier flow is directed through the desorption system.

GC Requirements

The GC column should be capable of being cooled to subambient temperatures of between 0°C and -180°C. This is required to permit the desorbed samples to be concentrated and collected in a narrow band at the front of the GC column. This can be accomplished by using a GC equipped with subambient cooling capability (liquid nitrogen or CO_2) or with the Micro Cryo-Trap Accessory. Temperature programming is required to enable the system to be heated for the subsequent analysis by the GC and detector system. Some analysis may be accomplished by collecting samples at room temperature using thick film macrobore capillary columns (such as J&W's DB-624 column) or packed GC columns.

The specific GC column and temperature program employed will be dependent on the specific compounds being analyzed. Generally, a nonpolar stationary phase (e.g. DB1, DB-5, SE-30, OV-1) temperature programmed from -30°C to 250°C at 4-10°/minute will be suitable. Capillary column dimensions of 0.20mm to 0.53 I.D. and 25 to 60 meters long are generally appropriate although other I.D.'s and lengths may be sufficient in many cases. The system can also be used with packed columns, however the Micro Cryo-trap can only be used with capillary columns.

Often a deactivated fused silica precolumn of approximately one meter in length by 0.53 mm i.d. is used at the injection port end of the GC column. The precolumn will help prevent the plugging of the system by water, which is present in most samples and which is desorbed into the GC. The larger I.D. of this precolumn permits a larger surface area for the sample to collect with less chance of plugging by water vapor condensing on its surface. Samples with high water content should be avoided when possible.

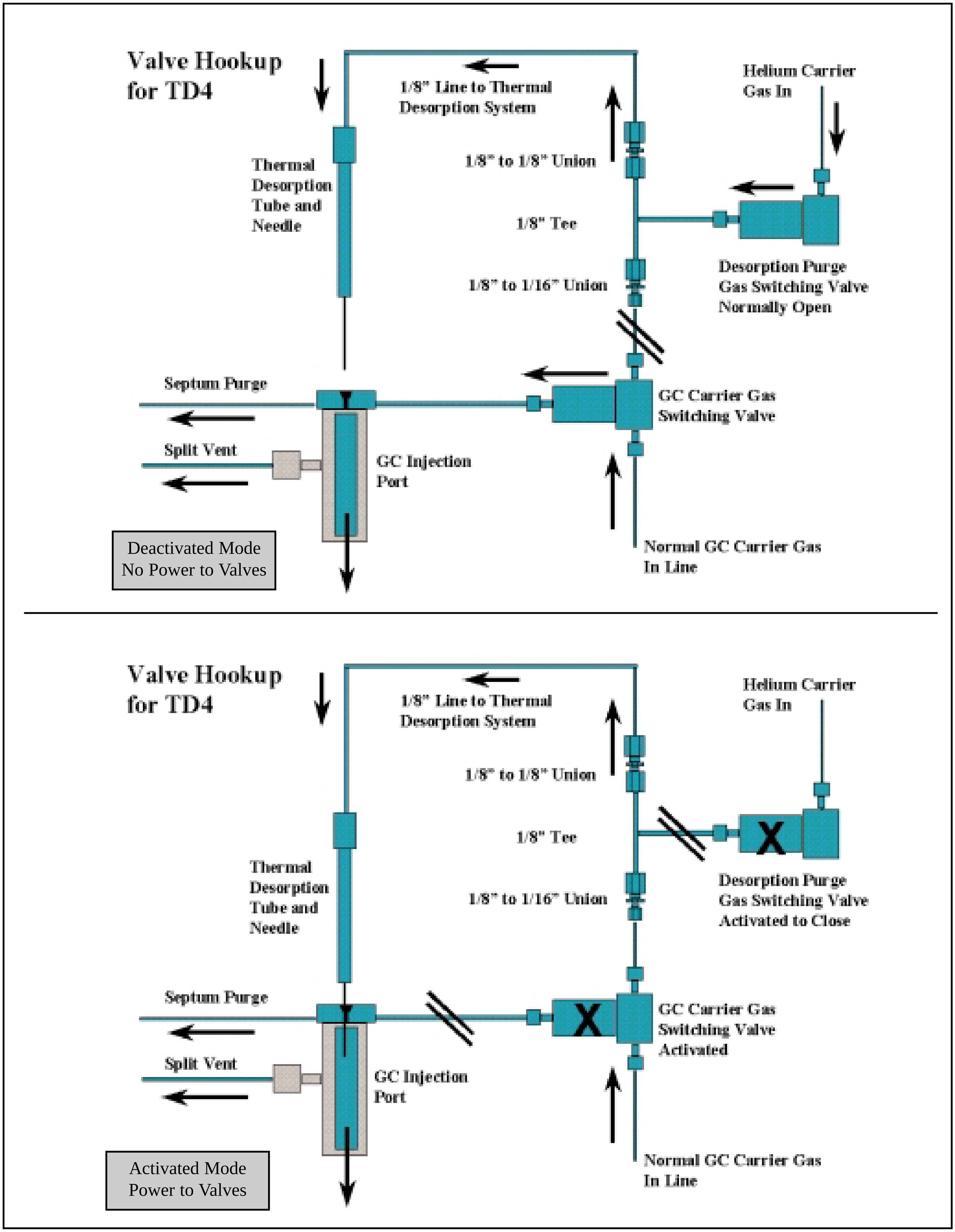

Valve Hookup for TD4
1/8" Line to Thermal Desorption System
Helium Carrier Gas In
Thermal Desorption Tube and Needle
1/8" to 1/8" Union
1/8" Tee
1/8" to 1/16" Union
Desorption Purge Gas Switching Valve Normally Open
Septum Purge
GC Carrier Gas Switching Valve
Split Vent
GC Injection Port
Normal GC Carrier Gas In Line
Deactivated Mode No Power to Valves
Valve Hookup for TD4
1/8" Line to Thermal Desorption System
Helium Carrier Gas In
1/8" to 1/8" Union
1/8" Tee
Thermal Desorption Tube and Needle
1/8" to 1/16" Union
Desorption Purge Gas Switching Valve Activated to Close
Septum Purge
GC Carrier Gas Switching Valve Activated
Split Vent
GC Injection Port
Normal GC Carrier Gas In Line
Activated Mode Power to Valves

Fig. 7-1

Techniques and methods will vary from user to user and depending on the set-up, but listed below are several which may be used as guidelines in thermal desorption analysis.

Splitless Operation with the Hewlett Packard Split/Splitless Injection System.

The correct carrier gas valves should be in place before proceeding. With the tube and needle assembly withdrawn from the GC injection port, the GC carrier gas flow is set by adjusting the injection port head pressure to provide the required column flow. The total flow is adjusted to the level where no flow is evident at the split outlet. This will provide for splitless injection and running of the system. The steps outlined below can be run either manually or automatically as described in Section V of this manual.

When the desorption tube and needle are installed, the carrier flow through the desorption tube is adjusted to 1 to 3 ml/min. with the flow controller on top of the desorption unit. The desorption tube and needle are injected into the GC injection port. Upon initial injection a drop in flow through the desorption tube is seen on the rotameter and the head pressure observed at the pressure gauge on the GC injection port will drop. This is caused by the injection port momentarily depressurizing.

After a short time (5-10 seconds) the flow will return to its normal level. If the head pressure at the front of the GC column falls to zero and remains there, it is an indication of a leak at the seal of the injection needle or a leaking septum. The column head pressure should normally return to some positive value. At this point the normal carrier gas flow through the GC is turned off, and the only flow through the GC column is provided by the desorption system. The flow is adjusted to the point where no flow is detected at the split vent but where sufficient flow is provided to elute the desorbed material from the desorption tube.

After the flow is adjusted to its proper level the sample can be desorbed into the GC. Desorption times of 5.0 to 15 minutes are sufficient for most samples. Samples are collected at the front of the GC column which has been lowered to subambient temperatures.

After desorption is complete, the GC carrier gas is turned back on, and the desorption tube and associated needle are removed from the injection port. The GC is returned to normal operation with the preset GC carrier flow and head pressure. GC temperature programming can begin for the analysis of the desorbed components which have been trapped on the front of the column.

Split Operation with the Hewlett Packard 5890 Series GC's

The operation of the H.P. 5890 series GC's for split operation is conducted much the same as described above, except that the total flow through the desorption tube is increased to the level where the desired split is achieved at the GC split vent. The desorption blocks are not closed until all the flows are stabilized and the desired split flow is measured at the split vent.

Split or Splitless Mode Operation with the Varian 3400 GC with Split/Splitless Injector

When the desorption tube and needle are installed, the carrier flow through the desorption tube is adjusted to 1 to 10 ml/min. (depending on the diameter and flow capacity of the GC column) with the flow controller on top of the desorption unit. The desorption tube and needle are injected into the GC injection port. Upon initial injection a drop in flow through the desorption tube is seen on the flowmeter on the desorption unit and the head pressure observed at pressure gauge on the GC injection port may drop. This is caused by the injection port momentarily depressurizing upon injection. After a short time (5-10 seconds) the flow will return to its normal level. If the head pressure at the front of the GC column falls to zero and remains there, it is an indication of a leak at the seal of the injection needle or a leaking septum. The column head pressure should normally return to some positive value. For the split mode of operation, the flow would be adjusted for the proper split ratio. The desorption cycle is initiated. Desorption times of 5.0 to 15 minutes are sufficient for most samples. Samples are collected at the front of the GC column which has been lowered to subambient temperatures.

After desorption is complete, the desorption tube and associated needle are removed from the injection port. The GC is returned to normal operation with the preset GC carrier flow and head pressure. GC temperature programming can begin for the analysis of the desorbed components which have been trapped on the front of the column.

Direct Thermal Extraction

With the Direct Thermal Extraction (DTE) technique, the sample to be analyzed is placed directly in the desorption tube for subsequent analysis of the volatile and semi-volatile organics present. This technique proves useful for materials such as spices, paints, fibers and plastics. It is important to avoid samples with high water content since the water vapor will be desorbed into the GC injection port and will tend to form a plug at the front of the column, restricting carrier flow. This problem can be reduced by:

(1) using small samples

(2) using a larger diameter precolumn (megabore column)

(3) desorbing at low temperatures (below 70°C)

(4) analyzing dry compounds

(5) using a megabore or packed GC column

Any of the techniques normally used in GC analysis including split, splitless, direct injection, etc. can be used for DTE analysis. A wide range of temperatures can be used to drive off the compounds of interest. It is possible to desorb the same sample several times, each time at a higher temperature, to enable a fractionation of the sample components for subsequent analysis. The use of temperature ramping is recommended when using the DTE techniques.(See application note #60 at www.sisweb.com).

Suggested Standard SPLIT/SPLITLESS INJECTION Protocol

<u>Temperatures, times, and other parameters given are only guidelines and should be optimized for each analytical method.</u>

Setting Up the Desorption Unit

- Turn on Main Power on the Desorption System

- Adjust Desorption Heater Block to 200 degrees C on the Controller, then allow to equilibrate

- Attach GLT sample tube to Injector assembly (graphite washer with metal insert and graphitized vespel™ seals required in Injector assembly)

- Insert graphite & graphitized vespel™ seals into needle cap

- Attach needle to GLT sample tube

- Turn carrier gas on (push 'GAS' button on Electronics Console)

- Adjust flow to 2ml/min with flow controller valve located on top of Desorption Unit

- Flow is monitored by the rotameter

- Flush/Purge system for 2-3 minutes to eliminate oxygen

Setting Typical GC/MS Parameters

- Set Scan mass from 35 to 350 at 2 scans/sec

- Set Injection Port Temperature = 260° C

- Set Detector/GC transfer line = 280° C

- Set GC column temperature to -40°C or use the Micro Cryo-trap

- Allow parameters to equilibrate

Thermal Desorption and Sample Analysis
Manual Mode

- Inject Sample (INJECT button on Console)

- Check and adjust flow to desorption tube at 2 ml/min

- Desorb Sample at 200 degrees for 10 minutes. (‘DESORB’ button on Console)

- Check column head pressure and readjust flow if necessary

- After desorption is complete, turn GC flow back on

- Press ‘Desorb’ key (Heating blocks will open)

- Press ‘Inject’ key to raise injector

- Leave flow on through the Desorption tube for 5-10 minutes allowing the tube to cool (WARNING-VERY HOT!)

- After Desorption is complete, program GC run from -40°C to 40 degrees C at 10 degrees/min, then 4°C/min from 40°C to 280°C; Total GC run = 68 minutes

NOTE: This method is for reference only and will need to be modified for your particular analysis.

Thermal Desorption and Sample Analysis
Automatic Mode

Set time and temperature parameters for the TD-4 as described in section 5.

Example:

	Parameter	Setting	Keystrokes
Time Set:			
	Purge Gas:	1 minute	1↵ 0↵
	Inject Time:	1 minute	1↵ 0↵
	Desorb Time:	5 minutes	5↵ 0↵
	Delay Start:	30 seconds	0↵ 30↵
Temp Set (Desorption blocks):*			
	Begin:	200°C	200↵
	Rate:	accept previous value	↵
	End:	200°C	200↵

***Note: If temperature ramp is desired, enter appropriate Begin, Rate, and End values.**

(Cryo-trap):			
	Cooling:	-40°C	-40↵
	Heating:	260 °C	260↵

- Verify that GC flow settings are correct. For systems with EPC, set split flows normally.
- If the Micro Cryo-Trap accessory is used, press the ‘Cryo’ key on the controller once to start cooling the trap.
- Press the ‘Auto Run’ key on the controller. Gas switching, injection, desorption and GC start will be handled automatically by the TD-4.
- For non-EPC systems, check split flow during Inject time (after 20 seconds) to verify appropriate flow through the desorption unit. If flow is too high or too low, adjust with the flow controller on top of the desorption unit. This adjustment should only have to be done once for a given set of instrument conditions. Extending the injection time for the first run may aid in making the adjustment.

Note: This method is for reference only and will need to be modified for your particular analysis.

VIII. Troubleshooting the Short Path Thermal Desorption System

Introduction

This chapter is concerned with the diagnostic procedures to follow when problems occur with the Short Path Thermal Desorption System. This section is divided into two areas in which problems can occur.

A. Flow & Mechanical System

B. Electronics Console

A. Flow and Mechanical System Problems

(1) Problem - Pressure Gauge on Desorption System reads a higher pressure than normal when desorbing a sample into the GC injection port

Possible causes

(a) Clogged desorption tube needle

(b) Ice plug in column

Solution a - A high pressure is usually an indication of a clog in the system, usually in the desorption tube needle. Compare the pressures displayed on the desorption system and the GC injection port (if so equipped). Normally these two pressures should be the same within 1 or 2 pounds. If the desorption pressure is much higher, then there is a clog in the system. Typically the clog will be found in the syringe needle. If the desorption pressure is much lower then there is a leak in the desorption system. Uninject the desorption tube; the pressure reading of the desorption system gauge should drop quickly - if it does not then the needle is probably clogged. Unscrew the needle to confirm this source of clogging. With use the needle can become clogged with septa or graphite from the sealing ferrules or perhaps a previous sample that was analyzed. The needles can be cleaned by baking out if contaminated with volatile samples or with a cleaning wire if clogged with septum and graphite.

Note: We do not recommend standard 20° point needles for use with the desorption system. When these needles are used, they tend to core the GC septum and the plug removed from the septum stays inside the needle. Therefore we recommend side port needles with the system to eliminate this coring problem.

Solution b - If samples which contain high water content are thermally desorbed into the GC and cryofocused at the front of the GC column, ice plugs can occur. This can be eliminated by one of the following :

1. Use smaller samples

2. Install a 0.5 to 1.0 meter megabore deactivated fused silica guard column in front of the regular GC column to minimize the chance of ice plugs. This also will improve resolution on early eluting peaks.

3. Use a larger diameter capillary column or a packed GC column.

4. Trap volatiles purged from the samples onto a Tenax or other adsorbent resin which has a low affinity for water, and then subsequently desorb the traps into the GC.

(2) Problem - Zero reading or low pressure reading on the Short Path Thermal Desorption Pressure Gauge when desorbing sample into the GC injection port.

Possible Causes

(a) Leaking Seal at needle connection to desorption tube

(b) Leaking seal at top of desorption tube to connecting tube

(c) Bad GC Septum

(d) No carrier gas is being supplied to the desorption system

(e) Broken GC Column at the injection port

Solutions

The most likely cause of low or zero pressure readings on the Short Path Thermal Desorption System is leaks in the desorption tube. The most likely location for such a leak is at the seal between the needle and desorption tube. Leaks can also occur between the desorption tube and the connecting tube. Either tighten these fittings or replace the seals. One can easily check the desorption system by uninjecting the desorption tube and needle and sliding an old GC septum over the needle. With the carrier gas flow On through the desorption system, the pressure should rise fairly quickly and settle out at the carrier gas pressure being used. If the pressure does not rise or rises too slowly there is a leak in the system. This leak should be able to be located with Snoop leak detection fluid with the needle plugged as described above.

If the problem is not located in the desorption system, then it is probably in the GC injection port. Check the GC injection port septum - if it is bad, replace it. Also check the GC column. The column may be broken or the column fitting at the injection port may be loose.

(3) Problem - Ball stuck in Rotameter

Cause

(a) Dirt or contamination has entered the rotameter glass tube

Solution

Remove the rotameter glass tube and clean. Call S.I.S. Tech Support for more information.

(4) Problem - Bending Needles

Cause

(a) Misalignment of needles on injection

(b) Incorrect injection port

(c) Septum nut adapter not in place

Solution

When setting up the system and installing the desorption tube and needle, visually inspect to be sure the needle is close to being centered over the hole in the desorption system middle plate. If misaligned, gently straighten to the approximate center position. Also check that the septum nut adapter is in place and sitting squarely over the septum nut.

If an on column injector is being used, the desorption needle may not fit inside the capillary column (even if a megabore guard column is used in the injection port). Either use a smaller O.D. needle which will fit inside the guard column or use a conventional split/splitless injector.

(5) Problem - Ice Plugs in GC column when cryo trapping components during the desorption process.

Cause - water content of sample being analyzed is too high.

Solution

1. Using smaller samples

2. Install a 0.5 to 1.0 meter megabore deactivated fused silica guard column in front of the regular GC column to minimize the chance of ice plugs. This also will improve resolution on early eluting peaks.

3. Use a larger diameter capillary column or a packed GC column.

4. Trap volatiles purged from the samples onto a Tenax or other adsorbent resin which has a low affinity for water, and then subsequently desorb the traps into the GC.

(6) Problem - No peaks are present on the GC or Mass Spec chromatogram.

Causes - no sample is reaching the detector due to:

(a) Leaks in the desorption system

(b) Bad GC Septum

(c) Broken GC column

(d) Split ratio too high

(e) Sample size too small

(f) Ice plug in column

(g) Clogged desorption tube needle

Solution

(a) Leaks in the flow path of the desorption system are the most common cause for the absence of GC peaks in the chromatogram. These leaks usually occur at the desorption tube needle during injection and desorption. Check the pressure gauges and flow rotameters on the desorption system during the desorption process. The rotameter should read some positive flow and the pressure gauge should be close to the injection port pressure reading. Both of these readings can be used to check for desorption system leaks, clogged needles, broken GC columns or ice plugs as described previously in this section.

(d) Depending on the sample size being used, the split ratio may be too high. Vary the sample size being analyzed and adjust the split ratio or run splitless as required. Measure the split flow at the split vent using a flow meter.

(7) Problem - Desorption system heater blocks will not close around the sample in the Auto Mode of operation. System reads "PRESSURE LEAK"

Cause

(a) Desorption system head pressure is less than 3 pounds of pressure due to:

(1) purge gas flow too low

(2) bad septum

(3) broken or disconnected GC column

(4) insufficient time for injection time cycle

(5) leak in desorption system

(b) Defective pressure switch in the desorption system

Solution

In the automatic mode of operation, a pressure switch is activated which will not permit the desorption system heater blocks to close unless the head pressure in the desorption system is more than 3 pounds. If there are leaks in the system or the GC septa is bad, the desorption system pressure will be less than three pounds of pressure, the message "PRESSURE LEAK" will be displayed on the Electronics Console and the blocks will not close. Find the source of leaks as outlined above to correct the problem. If using megabore columns which require head pressures less than three pounds of pressure, the pressure switch can be adjusted for a lower pressure. See Section 4 for details on the adjustment of this switch. If preferred the system can be operated in the manual mode. When operated in the manual mode, the pressure switch is not active, and the desorption system blocks will close regardless of the system head pressure.

It is also possible that the pressure switch is defective. If the pressure switch cannot be adjusted as outlined in the manual, then it should be replaced. Call S.I.S. Tech Support for more information.

(8) Problem - Desorption system heater blocks will not heat

Causes

(a) Heater fuse on the electronics console is blown

(b) Overheat sensor switch on the desorption system tower has been activated.

(c) Heater cartridge on the heater block is blown

(d) Platinum Resistance Thermometer is defective

(e) Cable between Electronics Console and Desorption Unit is disconnected or defective

Solutions

Check the main power fuses on the desorption system Electronics Console and make sure the connecting cable is installed. If the problem is still apparent, remove the side from the Desorption Unit and reset the over heat sensor circuit breaker on the back plate(red button). If this does not solve the problem, the heater cartridge or PRT is bad and will need to be checked out by a qualified technician at S.I.S..

(9) Problem - GC peaks are broad at beginning of chromatogram

Causes - Cryo Trap temperature not low enough

Solutions

We have found that we can trap and resolve compounds with melting points 0 to 10° below the cryo trap temperature. Therefore if the cryo trap is set at -40° C, then we can routinely trap compounds with melting points down to -50° on an uncoated deactivated fused silica capillary column. In general we have found the best results using a megabore deactivated fused silica precolumn or guard column with all capillary columns. This megabore column provides a greater I.D. and surface area to minimize water plugs. In addition the use of uncoated guard columns as a cryo trapping area provides for better peak shape and resolution versus trapping directly on the liquid phase of the capillary column. If the trapping area of the guard column is coated with a liquid phase the trapping efficiency can be improved provided that the liquid phase is still active at the set temperature (eg. DB-WAX can not be used at cryo temperatures). Using a thick film megabore guard column (5.0 µm film thickness x 0.53 mm I.D. DB-5) we can quantitatively trap compounds with melting points 40° below the trapping temperature.

(10) Problem - Extraneous Peaks in Chromatogram

Cause - Extraneous peaks can occur in the GC chromatogram or the Mass Spec total ion chromatogram. The cause of these peaks can be one or more of the following

(1) Contaminated desorption tube

(2) Contaminated desorption tube needle

(3) Contaminated connecting tube

(4) Contaminated GC Septum

(5) Contaminated injection port liner or injection port

(6) Bleeding GC Septum

(7) Injection port too hot

(8) Bad GC guard column

(9) Guard column or capillary column was over heated, decomposing liquid phase on column

(10) Desorption temperature too hot

(11) Contaminated GC carrier gas

(12) Breakdown of Adsorbent resins in packed thermal desorption tubes

(13) Contaminated Carrier Gas line traps

(14) Injection port and carrier gas lines are contaminated

Solutions

Often three peaks are present in the chromatogram resulting from either septa bleed or GC column coatings. If the analysis is being done on a mass spectrometer, these three peaks will have most abundant ions at 207, 281 and 355 (or 267) consecutively. These correspond to the siloxanes used to either coat the capillary columns or bleed from the silicone septa. These three siloxane compounds are:

a. Hexamethylcyclotrisiloxane (M.W. 222) - major mass spec peaks at 207

b. Octamethylcyclotetrasiloxane (M.W. 296) - major mass spec peaks at 281

c. Decamethylcyclopentasiloxane (M.W. 370) - major mass spec peaks at 355 and 267

In a normal GC run these compounds are the reason for background in the chromatogram. However in the thermal desorption process, samples are injected over 5 to 10 minutes and trapped at the front of the GC column. If these siloxanes originate at the septum or from the guard column itself at the injection port end of the column, they will be trapped in the cryo trap section of the GC system and result in distinctive peaks in the total ion chromatogram. They can be minimized by using low bleed septa (we recommend Supelco's Thermogreen Septa), replacing the septum regularly, keeping the injection port at the minimum temperatures required for the analysis, regular replacement of the guard column and minimizing the upper temperatures to which the guard column and capillary column are subjected. These steps will minimize the decomposition of the septum and column coatings. Also note that the higher the desorption block temperature as well as the injection port temperature, the more pronounced these siloxane peaks appear in the total ion chromatogram. Best results are always obtained at the lowest possible desorption block and injection port temperatures required to perform the analysis on the compounds of interest.

If the contaminant or background peaks are not siloxanes, then the source of contamination should be located by checking or changing each of the problem areas outlined above. Begin by installing a baked out and clean desorption tube and needle on the desorption system. Repeat the analysis.

If the peaks still appear, remove the connecting tube from the desorption system and bake out in the conditioning oven at 300 to 350 degrees C for 1 hour. If powdered samples were analyzed without glass wool plugs being placed on top of the samples before thermal desorption, it is possible for these samples to blow back and up into the connecting tube when the carrier gas flow through the desorption system is turned off. It is recommended that glass wool plugs be inserted on top of all solid powder samples in the desorption tubes to eliminate this blow back from occurring. After the connecting tube has been baked out, reattach it to the desorption system along with a clean and baked out desorption tube and needle. Repeat the analysis.

To verify that no contamination is originating from the desorption system, attach the carrier gas line directly to the connecting tube using a clean stainless steel gas line and a needle valve in line to control the carrier gas flow. This will by-pass the entire desorption with the exception of the connecting tube, desorption tube and needle. Repeat the analysis. If the contaminant peaks disappear, then the problem is contamination in the desorption system. Replace the PTFE coiled connecting tube. Recheck the system. This should correct the problem.

If the extraneous peaks still occur, then the problem is in the GC injection port end of the GC system. Begin by replacing the GC septum, installing a new injection port liner or cleaning the GC injection port and replacing the GC guard column or removing several inches from the front of the GC column. Bake out the injection port before the column is reattached. Repeat the analysis.

If the problem still persists, the only sources left for checking are the GC column itself and the GC carrier gas purity. Replace the GC column. Install a new carrier gas tank and replace the hydrocarbon and oxygen traps of the carrier gas lines. It is also possible that the entire GC injection port may have been contaminated by a previous analysis in which large volumes of solvents were syringe injected in to the GC injection port which backed up into the GC injection port lines, contaminating the entire GC plumbing system lines. If this is the case the GC injection port and accompanying plumbing will need to be broken down, plumbing lines and traps cleaned or replaced, baked out and reassembled. See S.I.S. website at www.sisweb.com for more information.

Users doing Direct Thermal Extraction work or analyzing compounds not well bound to adsorbent media may experience some carryover. This results from the depressurization of the injection port, which causes a momentary net backward flow of carrier after injection. The most common cause of this problem is the use of powdery or friable samples which may be carried back into the desorption unit with the backward gas flow. The use of inappropriate trapping media may also contribute to this type of contamination, as inadequately bound volatiles may also be swept back into the system.

Naturally, the choice of an appropriate adsorbent can rectify the problem in the latter case, and the addition of a small conditioned glass wool plug inserted on top of the sample may help in the case of DTE samples. However if the desorption unit has been substantially contaminated, or if the unit is to be used frequently for samples which pose this type of problem, the connecting tube may be packed with an adsorbent material to provide a final carrier filter that can be regenerated if contamination occurs:

- Remove the connecting tube by disconnecting the PTFE carrier line at the top, then removing the three round-head cap screws from the tube mounting bracket with a 1/8” allen wrench.
- Remove the Vespel™ and Graphite top sealing washers from the connecting tube.
- Insert a plug of clean silanized glass wool into the bottom of the tube and use a long, thin instrument such as a length of 1/16” tubing to seat the plug in the top of the connecting tube.
- Pack the tube with 1-3 grams of adsorbent. Spherical molecular sieve materials such as Carboxen™ or Carbosieve™ are recommended, as they provide less backpressure. Graphitized activated Charcoal (GAC) materials may also be used, provided the mesh size is not too small.
- Insert a plug of silanized glass wool into the tube and seat it with a suitable tool to hold the adsorbent material in place. Replace the graphite and graphitized Vespel™ seals.
- Remove the PTFE tubing from one of the handles on the desorption tube conditioning System and attach it to the packed connecting tube. Place the connecting tube into the conditioning oven and condition for at least two hours at 300 °C with 30-40 ml/minute gas flow.
- Carefully remove the tube from the oven and allow it to cool with gas flowing through it. Reattach the tube to the PTFE carrier line on the desorption unit, and replace the three screws. Before tightening the screws, make sure there is approximately 3/16” between the bottom of the hex nut on the connecting tube and the top of the tube mounting bracket.

(11) Injection or Uninjection too slow or too fast

Causes

(1) Air pressure too high or too low

Solution

When the desorption systems are set up in the factory, they are adjusted to operate optimally at 60 psi of compressed air for all the air solenoids and columns in the system. Check the air pressure to the desorption system tower to be sure that it is delivered at 60 psi. At this point, the system can be adjusted to operate slightly faster or slower by changing the air pressure. An increase in air pressure (use care not to exceed 100 psi) will cause the injection process to speed up. Likewise a lower pressure (do not use less than 10 psi) will cause the system to operate more slowly.

B. Electrical System

(1) Problem - No Power to the System, Electronics console does not light up.

Causes

(a) System not plugged in or power line is dead.

(b) Fuses Blown on back of Desorption System Electronics Console

(c) Electronics Console is in need of service

Solution

Check that the power cord is plugged into the electrical socket. Check and or replace the fuses. If fuses are blown and continue to blow, unit should be serviced by a qualified technician at S.I..S.

(2) Problem - Display does not respond, reads COM INTERACTIVE or TERMINAL MODE

Causes

(a) Loose connections inside Electronics Console

(b) Bad Power Supply

(c) Display Module is Bad

(d) Microprocessor software chip has been damaged

(e) Stop/Reset button is depressed

Solutions

The most common cause of the display not responding and giving erroneous messages is a loose microprocessor printed circuit board or a loose cable connection to the microprocessor. This cable can become loose in shipping. The cover should be removed from the electronics console and the board and cables checked for proper installation. If the system has been exposed to an electrical surge or electrical discharge, the microprocessor software chip may have been damaged or destroyed and will need to be replaced. If this is the problem or if problems persist, call S.I.S. If the Stop/Reset is depressed and the light is on, push the button again and then turn off the power to the system. Turn on the system again and retry.

(3) Problem - Display has missing or Bad characters

Causes - The system received some type of power surge

Solution

Turn off the main power switch on the electronics console, allow to set 30 seconds and then turn back on. This should solve the problem. If not, remove the Electronics console main cover and check the microprocessor board for proper installation. Also check the cable which goes from the microprocessor board to the display for tightness and proper installation.

(4) Problem - Heater temperature does not reach set point.

Possible causes - Heater cartridge burned out or defective.

Solution

Remove interface cable from unit. Using an OHM meter take a resistance reading between pins 1 & 2. The reading should be approximately 36 OHMS. If the reading is approximately 72 OHMS then one of the heater cartridges is burned out or defective. Unit should be returned to SIS for repair.

(5) Problem - System does not heat.

Possible causes - System is set to run a ramping temperature program while being operated in the manual mode of operation.

Solution:

In the manual mode the heater blocks must be operated in the ballistic mode. To ramp the heater blocks use the automatic mode of operation.

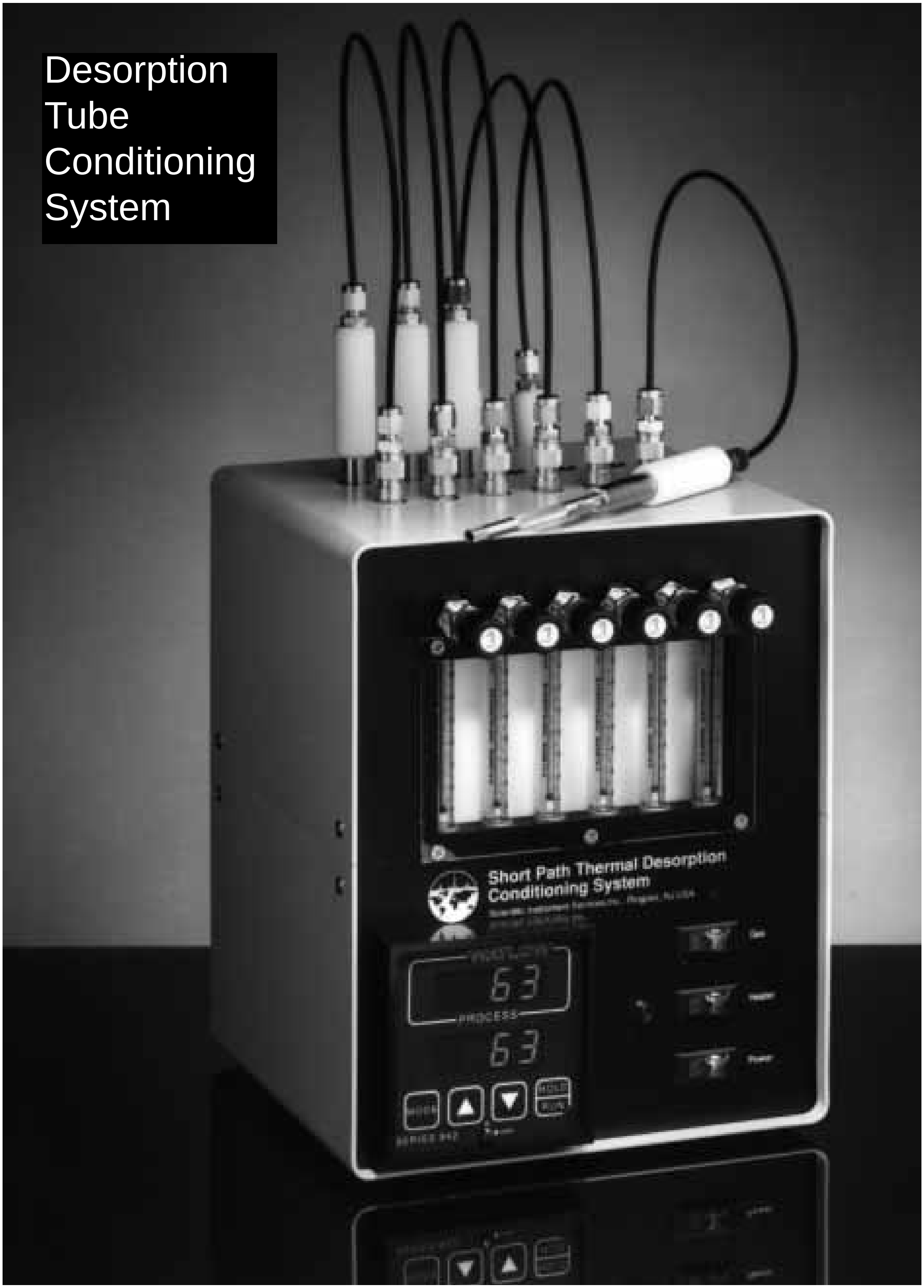
(6) Problem - System stops after injecting.

Possible causes - Inject time is set to zero.

Solution:

All timers must be set to a value greater than zero. Check all timers and correct as required.

Desorption Tube Conditioning System



Conditioning Oven

I. Introduction

A. Theory of Operation

The Desorption Tube Conditioning Oven is recommended for the flow conditioning of packed glass-lined stainless steel (GLT) desorption tubes as well as for the flow conditioning of desorption tube needles **(Fig.1)**. A high purity gas such as helium or nitrogen is recommended for use in this system to purge the packed desorption tubes while baking out at elevated temperatures. By proper conditioning of the desorption tubes and adsorbents, one can be assured that no foreign contaminants will interfere with or contribute to the composition of the samples being analyzed. The system consists of six flow adjustable rotameters and a heater block with six ports (i.d. 0.40" x 4.0" deep) for the cleaning of six desorption tubes or needle caps simultaneously at temperatures up to 350°C. A Watlow precision programmable temperature controller provides the heater circuit to heat the blocks and permits the programming of the temperature at which the tubes are to be conditioned. Temperature programs of up to six steps with various ramp cycles and hold times can be programmed into the controller for the unattended conditioning of the desorption tubes. Programs are stored in the system's memory. Accuracy of the programming temperatures is +/-.1% of the full scale reading. An L.E.D. digital readout displays the set temperature and the actual temperature of the system, and the bubble meters indicate the flow through each desorption tube or needle.

B. Specifications

1. Safety - Warning Messages

WARNING Do not condition tubes or needles above 350°C otherwise damage may occur to heater blocks or internal circuitry.

WARNING Maximum gas pressure is 60 P.S.I.

WARNING Dot Not use Hydrogen Gas in the Conditioning Oven. Use only Helium or Nitrogen.

WARNING Make sure that only fuses with the required current rating and of the specified type are used for replacement. The use of incorrect or makeshift fuses or the short-circuiting of fuse holders creates a shock hazard for the operator and can damage the instrument.

WARNING Do Not increase temperature range of heat overload current.

WARNING If the System overheats, return to manufacturer for all service. Any adjustments, maintenance or repair of the opened instrument while it is connected to a power source must be avoided.

WARNING Do Not leave Conditioning Oven heaters in the heated ON position unattended overnight. The Conditioning Oven heaters rapidly heat and cool to their final operating temperatures and therefore, in order to prolong their life and avoid premature failure of the system they should be turned off when not actively being utilized to condition tubes or needles.

WARNING Use grounded outlet only. Connecting the Conditioning Oven to a power source which is not equipped with a protective earth ground contact creates a shock hazard for the operator and can damage the instrument.

WARNING Hot surface exposed. Do NOT TOUCH Desorption Tubes or needles when removed from heater block. Use PTFE Handles to remove tubes & needles from block. Allow to cool before touching.

WARNING Do Not condition needles on the desorption tubes. Condition desorption tubes and needles separately using appropriate handles.

2. **Electrical Specification**

   Power Requirements

   Voltage - 110 VAC
   Current - 10 amps maximum
   Electrical Cord - 110 v/grounded outlet

   Temperature Controller

   Heater Circuit - Accuracy - +/-.1% of full scale - Range - up to 350°C
   Heater output - 115 volt, 600 watt
   Sensor Input - Platinum Resistance Thermometer
   Heater Cartridges - 4 each - 115 v, 150 watts
   Digital Readout for set & actual temperatures 3 or 4 digit
   Temperature Range - Room Temperature to 350°C
   Programmable - autocalibration temperature controller

3. **Gas / Pressure Specification**

   Type - Nitrogen or Helium (high purity)
   Maximum pressure - 60 P.S.I.
   Flow - maximum 50 ml / min per tube
   - Maximum total 300 ml / min
   Quick disconnects - automatically turn off gas

4. **Weight and Dimensions**

   Weight - 12 pounds
   Size - 9" wide x 9" deep x 13" high

Warranty

The Desorption Tube Conditioning System is warranted against defects in material or workmanship for a period of 90 days commencing from the date of shipment from the warehouse of Scientific Instrument Services in Ringoes, NJ, hereafter referred to as the company. The company's liability on the Desorption Tube Conditioning System and accessories is limited to the cost of correcting the defect in the product. In no case shall the company be liable for consequential or special damages. The system should not be run unattended overnight. The company will not correct defects caused by buyers negligence. The company does not guarantee or warrantee the product for any particular purpose. The companies warranty shall end 90 days after shipment.

Extended Warranty

An extended one year warranty for parts and labor is available if purchased within 30 days of shipment of the unit. The one year extended warranty will cover parts and labor to repair the Desorption Tube Conditioning System within the facilities of Scientific Instrument Services. Service on customers facilities is not available.

Service and Repair

The Condition System should be serviced only by qualified SIS staff. Any equipment to be serviced under warranty or otherwise should be sent to the repair facilities of Scientific Instrument Services in Ringoes, NJ. 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 returned.

Scientific Instrument Services, Inc.
1027 Old York Road
Ringoes, NJ 08551
Attn: Repair Department
RA#________________
Phone: (908) 788-5550

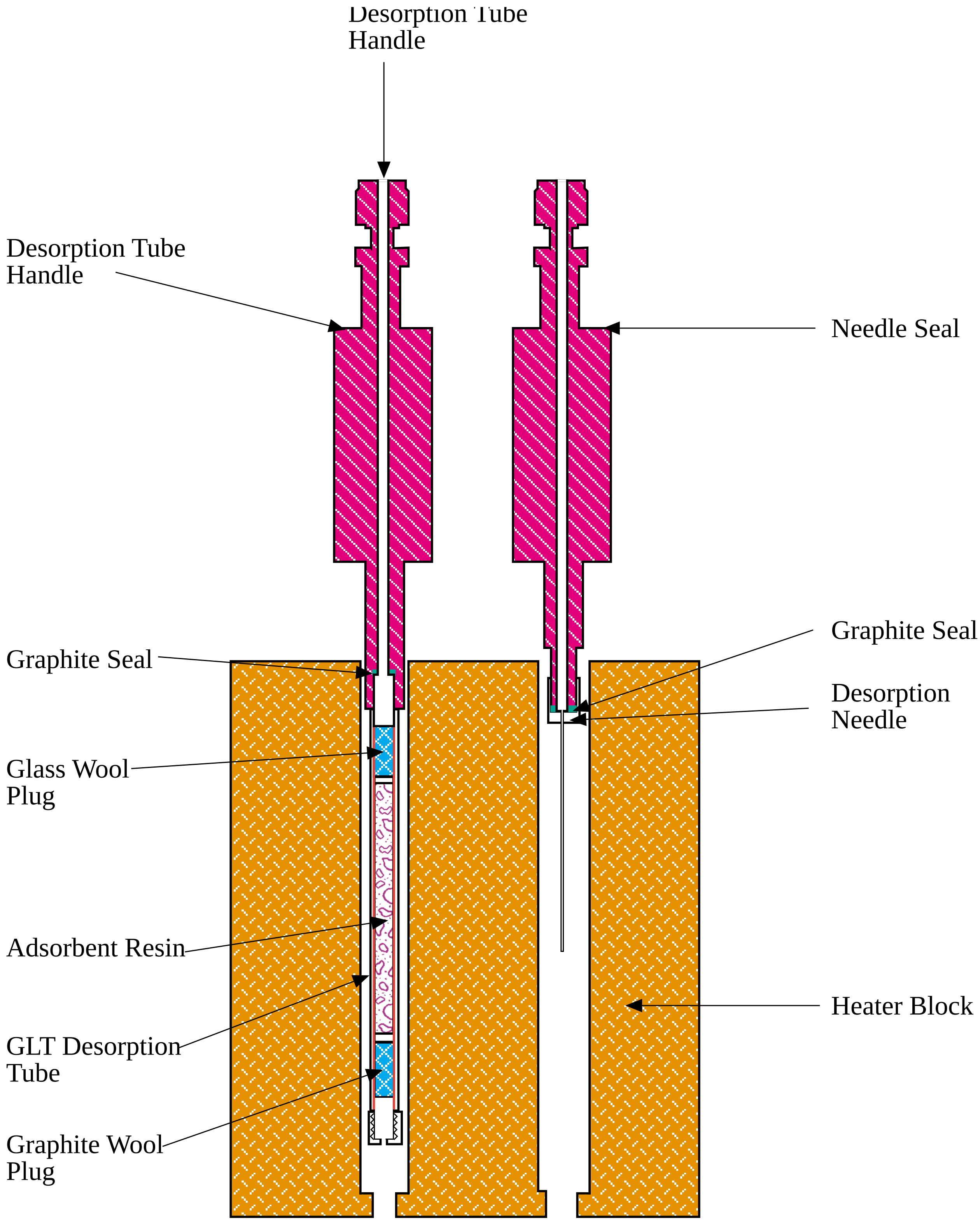


Figure 1 - Desorption Tube Conditioning Oven Theory of Operation

II. System Description

A. Front Panel Description (Fig. 2)

Three on/off switches are present on the front panel including a main power switch, a heater switch and a gas switch. The main power switch controls the power to the entire Conditioning Oven, to the temperature controller, and other switches. The heater switch turns on the power to the heater cartridges in the heater block and begins its heating cycle. The carrier gas switch turns on the carrier gas to permit its flow through the desorption tube. An L.E.D. digital readout displays the set temperature and the actual temperature of the system. A heater demand light indicates the actual heating demand of the heater blocks. If the demand light is bright red the heater is on; heater is off when there is no light. If the demand light is dim red, the heater is unplugged or an open circuit exists. Each of the flow rates for the desorption tubes and needles can be monitored by the six rotameters and flows adjusted with the high precision metering valves.

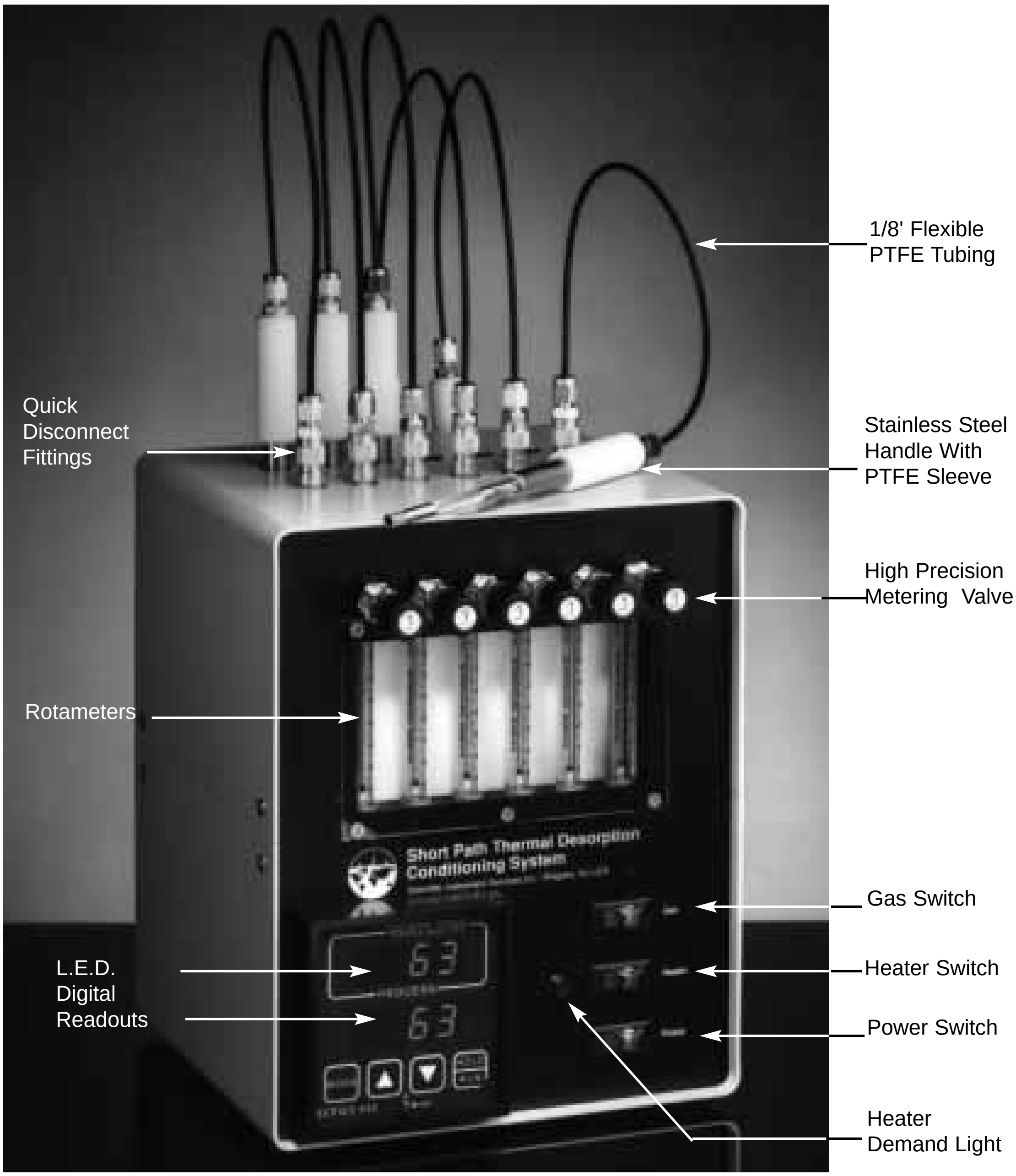


Figure 2 - Desorption Tube Conditioning Oven - Front View

The self contained system includes six adjustable bubble rotameters with flow ranges of 0 to 50 ml/min, one for each of the tubes or needles to be conditioned. The recommended flow rate is 20 ml/min. Flowmeter calibration data for air and helium are listed in **Table I**. Each of the flows through the tubes can be independently controlled or flow turned off to that port via a high precision metering valve. A single electrically operated solenoid valve turns the gas flow off or on to all the ports via the Gas Switch on the front panel. The quick disconnect fittings on the top of the Conditioning Oven turn the flow off to individual gas flow lines when disconnected **(Fig. 5)**.

TABLE 1

Flowmeter Calibration Data
Scale Readings at Center of Float

Scale Readings	Flow ml/min (Helium)	Flow ml/min (Air)
65	44	48.7
60	39	43.3
55	33	37.6
50	28	31.4
45	23	26.5
40	20	22.4
35	17	19.1
30	15	16.0
25	12	12.7
15	6	6.6
10	4	4.7
5	3	3.3

Two types of Conditioning Oven Handles are available, one for attaching the desorption tubes and the other for attaching stainless steel needles **(Fig. 3)**. All systems are shipped with six desorption tube handles and two needle handles. The handles are constructed from stainless steel with PTFE sleeves. The gas connection fitting is a standard 1/8" swagelok fitting. Graphite seals with metal inserts (part #781015) are recommended for sealing the desorption tubes to the handles during conditioning. Graphite seals are physically soft but have excellent sealing properties and temperature limits and are recommended for most applications with the Conditioning system. When soft materials such as graphite are used, a metal tube is inserted inside the center hole in the seal to prevent the graphite from closing and restricting gas flow. Other sealing washers could also be used if preferred for lower temperature cleaning.

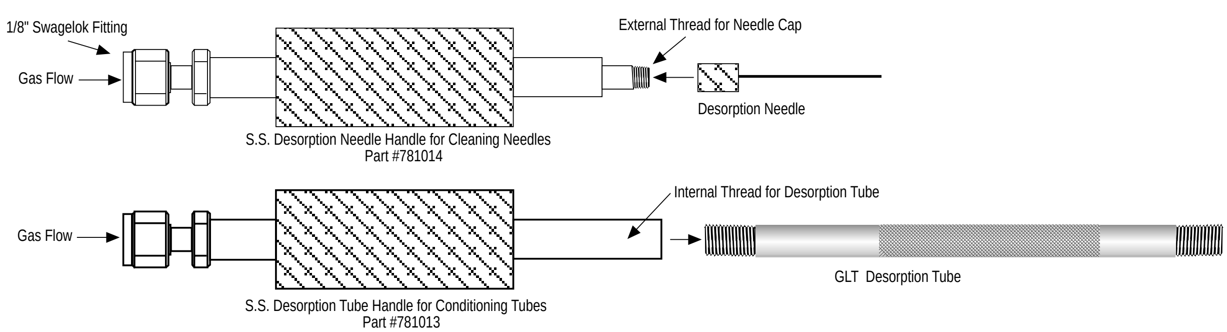


Figure 3 - Conditioning Oven Handles

B. Rear Panel Description (Fig 4)

Electrical power is provided from a standard 110 volt, 10 amp grounded outlet. A single 1/8" swagelok fitting on the back of the Conditioning System provides for the attachment of the carrier gas from its source. An external high temperature reset switch is located in the rear of the oven so that if temperatures exceed 350°C on the heater block, the external reset will open and heat to the block will stop. This switch can be reset by simply pushing this red button once the block has cooled. Two Slo Blo fuses are mounted in the rear of the Conditioning System to handle the initial surges of the Main Power and Heater switches when they are turned on.

In addition, a three inch fan enclosed on the back side of the Conditioning System provides a steady flow of air through the system to maintain the temperature of the temperature controller, rotameters, and other components inside the system at an acceptable level and to provide for cooling of the heater block once the heater circuitry is turned off.

After conditioning the desorption tubes and needles are removed and placed in a cooling rack attached to the back of the Conditioning System and cooled under constant flow. As soon as the tubes are touchable (5-10 min), they are immediately capped on both ends with stainless steel caps with PTFE seals that have also been conditioned.

III. Gas Flow CIrcuit

The Desorption Tube Conditioning Oven schematic **(Fig. 5)** pictorially represents the overall operation of the system including the gas flow and heater circuits. A 110 volt outlet is required and 10 amps provides the total electric power required for system operation.

The self contained system includes six adjustable bubble rotameters with flow ranges of 0 to 50 ml/min, one for each of the tubes or needle to be conditioned. Each of the flows through the tubes can be independently controlled via a microneedle valve and the flow turned off to that port if no flow is required. A single electrically operated solenoid valve (v) turns the gas flow off or on to all the ports via the Gas Switch on the front panel **(Fig. 5)**.

On top of the Conditioning Oven six quick disconnects connect to 1/8" flexible PTFE tubing to provide gas flow through the desorption tubes and needles during conditioning. When a quick disconnect is removed the gas flow is automatically closed to that port. By providing gas flow from a carrier gas such as high purity nitrogen or helium through the desorption tubes and needles while conditioning, it can be assured that no oxygen enters the GLT desorption tubes which could destroy the adsorbent material. Impurities from the inside of the desorption tubes and needles are flushed from the interior surface of these parts. A single 1/8" fitting on the back of the Conditioning System is provided for the attachment of the carrier gas from the source **(Fig. 5 - (Gas in))**.

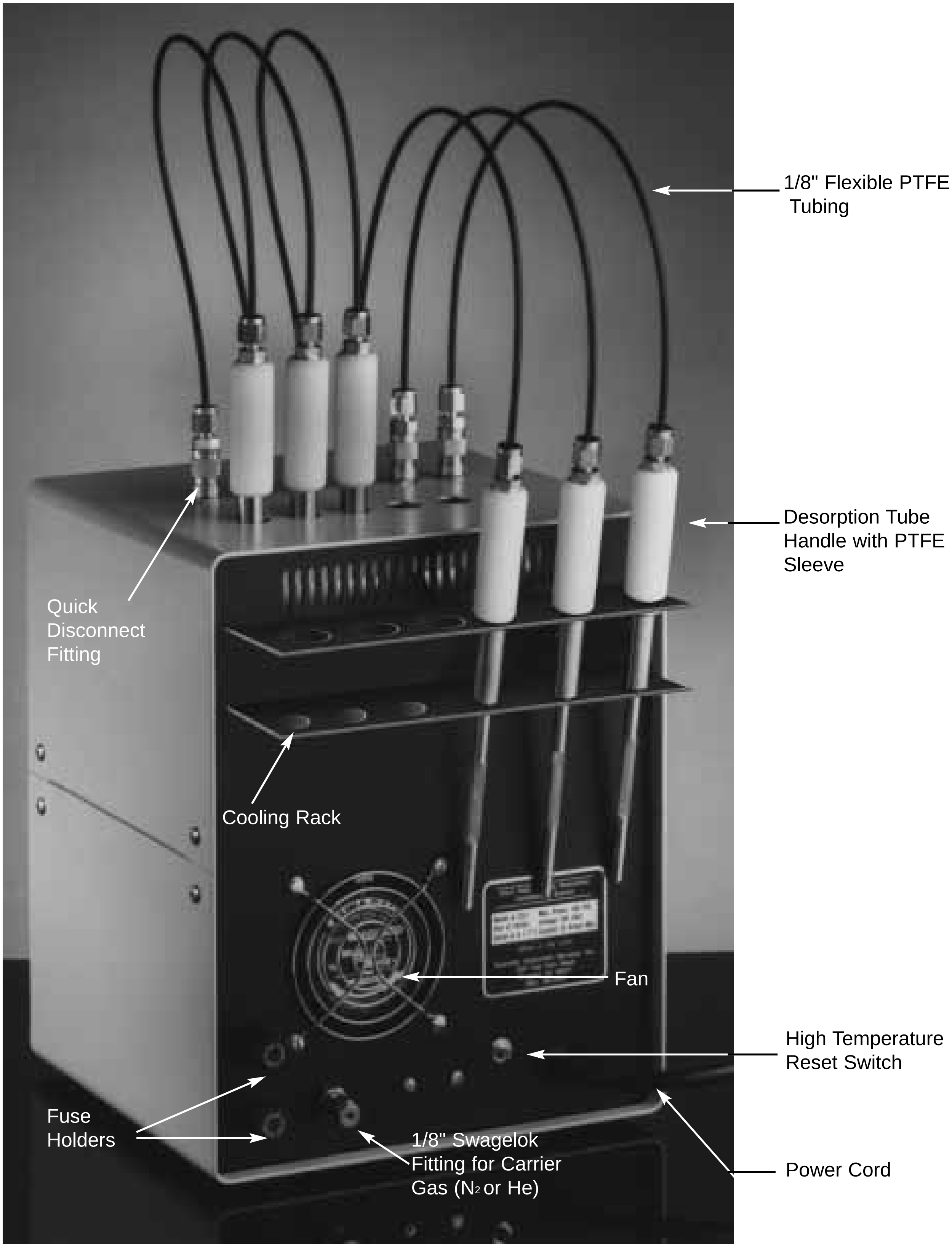
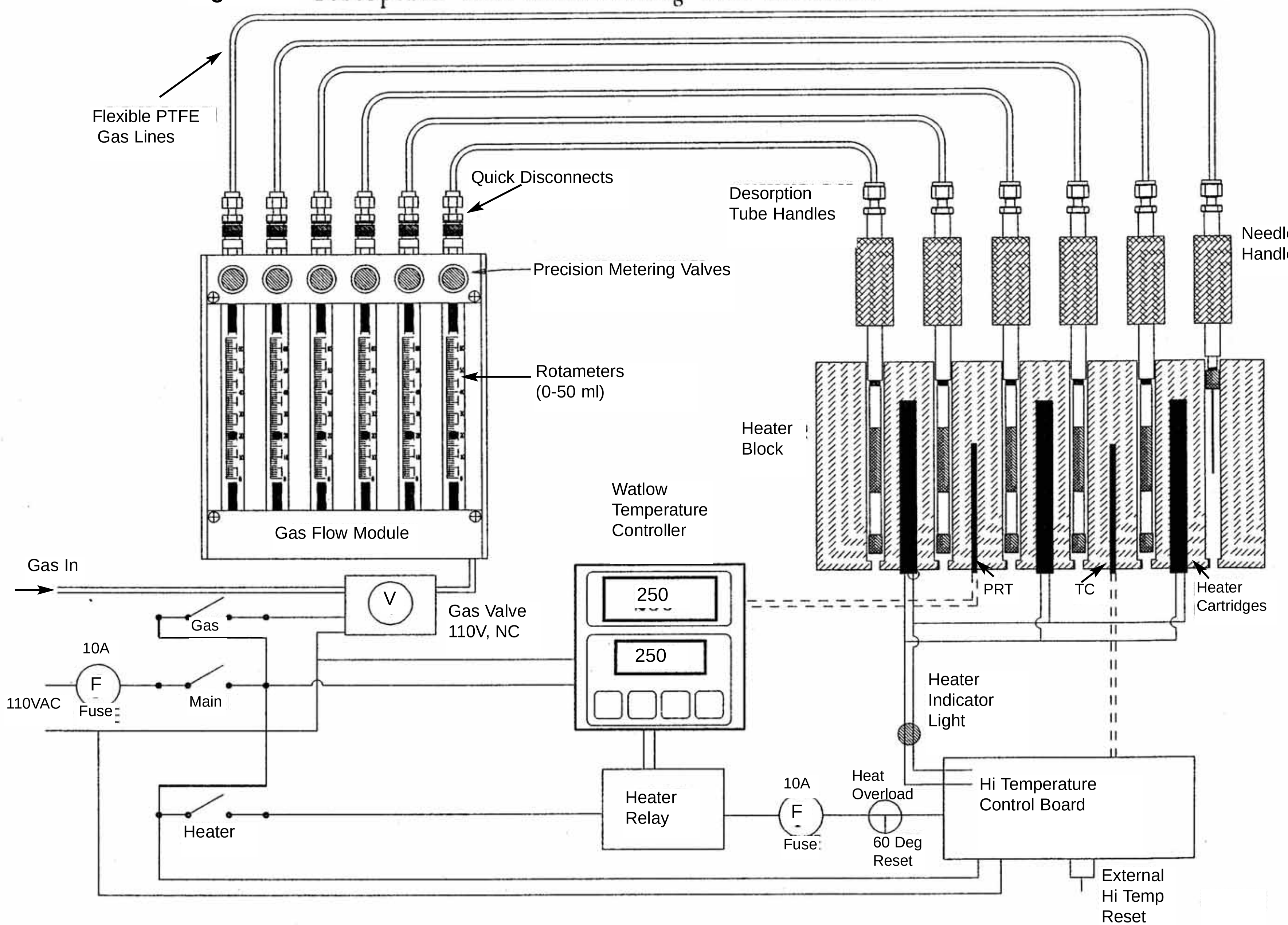


Figure 4 - Desorption Tube Conditioning Oven - Rear View

Figure 5 - Desorption Tube Conditioning Oven Schematic



IV. Heater Circuit

The Conditioning Oven includes a heater block with six ports (I.D. 0.40” x 4.0” deep) for the cleaning of six desorption tubes or needles simultaneously at temperatures up to 350° C. A Watlow precision programmable temperature controller permits the programming of the temperature at which the tubes are to be conditioned. The heater block contains a platinum resistance thermometer (PRT) for accurate (+/-.1%) temperature readout and provides the feedback to the temperature controller to maintain the heater block temperature. A J type thermocouple (TC) is also present in the heater block and serves as the temperature sensor for the high temperature limit control circuit. Four 3/8” diameter, 150 Watt heater cartridges heat the aluminum heater block. The heater cartridges are wired in parallel and are fused by their own 10 amp slow-blow fuse. A separate heater switch on the front panel permits the manual turning OFF and ON of the power to the heater cartridges. A 60° C heat overload sensor is attached to the inside of the Conditioning Oven case to protect against the excessive heating of the circuitry inside the Oven case. Two overheat protection circuits are built into the Conditioning Oven as described below. The heater indicator light (on the front panel) displays the power demand for the heater cartridges. When brightly lit, power is being supplied to the heater cartridges; when off, no power is supplied to the heaters. When dimly lit, the circuitry to the heater cartridges is open within the Conditioning Oven.

A. Heater Protection Circuitry

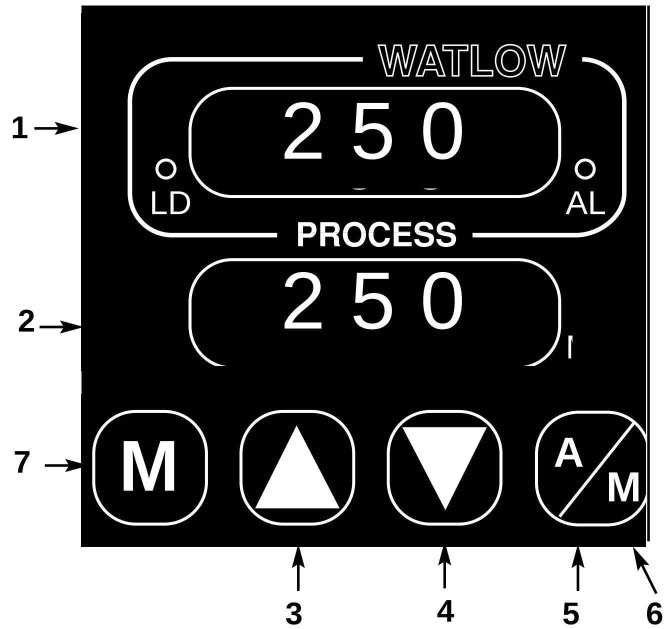
A heat overload thermostat (internal 60° C reset switch) is located inside the Conditioning Oven case to prevent the interior case and electrical components from being subjected to excessive heat. If the Oven case should exceed 60° C due to a circuit failure (such as failure of the cooling fan to operate), this overload sensor will open the circuit providing power to the heater cartridges. This overheat sensor can only be reset by allowing the system to cool, opening the Conditioning Oven case, and manually resetting the internal overload reset switch. If the heat overload thermostat should open when operating the system, the entire Condition Oven should be returned to the factory for service. Service should only be performed by qualified electrical technicians knowledgeable of the system electrical circuits.

A high temperature limit control board with an external high temperature reset switch **(Figs 4 & 5** - Hi temp reset) prevents the heater block temperature from exceeding 350° C. If the block temperature exceeds 350° C, the external reset switch will automatically open the power circuitry to the heater cartridges. Power cannot be restored to the heater cartridges until the heater blocks are allowed to cool and the external reset switch is manually reset **(Fig 5)**. Note : Do not exceed 350° C for the Conditioning Oven heater blocks. Higher temperatures will damage internal circuitry and create a potential meltdown and electrical hazard.

B. Watlow Temperature Controller

The Watlow microprocessor based temperature controller provides the heater output to the Conditioning Oven heater blocks. The system can either be run isothermally (Manual Operation) at a set and constant temperature or alternatively can be temperature programmed or temperature ramped (Automatic Operation) via a user selected temperature program. Procedures of up to six steps with various ramp cycles and hold times can be stored in the temperature controllers memory for the automatic ramping of temperatures as the desorption tubes are conditioned. Accuracy of the programming temperatures is +/- .1% of the full scale reading. Two 0.56" red LED’s display the set and actual temperatures for the heater block.

Description of the Keys and Display



1. **Upper Display (Actual heater block temperature)**
   The red 0.56" (14 mm) high, seven segment, four digit LED display indicates the actual heater block temperature, in addition to parameter values, or an open sensor. When powering up, the displays will be blank for 8 seconds.

2. **Lower Display (SET temperature)**
   The red 0.56 (14 mm) high, seven segment, four digit LED display, indicates the temperature SET point for the system. This value can either be set manually via the up/down arrows or is defined and displayed in the automatic mode by the user selected temperature program.

   UP/DOWN keys
   The UP/DOWN arrow keys are used to manually set the desired value for the SET temperature as displayed in the lower LED display. When pressed down simultaneously for 3 seconds, the SETUP Menu appears.

3. **UP Key**
   Increases the value of the SET temperature LED display. A single light touch increases the value by one. Hold the key down to increase the value at a rapid rate.

4. **DOWN Key**
   Decreases the value of the SET temperature LED display. A single light touch decreases the value by one. Hold the key down to decrease the value at a rapid rate.

5. **HOLD/RUN Key**
   Used to run or hold a temperature program. Press once to load the temperature program. Press a second time to start the temperature program. Pressing a third time will stop the temperature program and restore the controller to the manual (isothermal mode of operation).

6. **HOLD/RUN LED**
   Lit when the control in RUNning. When blinking, press the HOLD/RUN key again to begin RUNning the temperature program.

7. **MODE Key**
   Steps through the various menus such as the temperature program menu. Also automatically enters data before proceeding to the next parameter.

For additional features, control and system setup refer to the Watlow Controller Manual.

Temperature Controller System SETUP

The purpose of the SETUP menu is to define the various operating parameters and condition of the temperature controller. These user selectable parameters include the conditions of controller operation, the calibration parameters, rate of heating, sensor input type, temperature scale (°C or °F), display decimal point location, and temperature range. The Watlow temperature controller has already been preset to the correct operating parameters at the factory and the user should not normally have need to alter these factory settings. The SETUP Menu is entered by pressing both the UP and DOWN arrow keys simultaneously for 3 seconds. The values presently in the controller on the Conditioning Oven have been factory selected for the correct operation of the Conditioning Oven. <u>It is NOT recommended that they be changed</u>. For a detailed description of these parameters and the selections available refer to the Watlow Users Manual.

The factory preset values are listed below.

Setup Menu		**Operation Menu**	
LOC	O	Prog	no
In	rtd	Pb1	6
C_F	C	rE1	0.36
rL	0	rA1	0.27
rH	400	Ct1	5
Ot1	ht	CAL	0
HYS1	2	AUt	0
rtd	din		
PtYP	rAte		
gSd	0		
POUt	Cont		

Isothermal or Manual Mode Operation of the Watlow Controller

To operate the Watlow controller in the isothermal or manual mode of operation the following steps are used.

1. Turn on the Main Power Switch on the Conditioning Oven.
2. Turn on the Gas Switch.
3. Use the UP/DOWN arrow keys to select the desired temperature of the heater blocks (SET temperature is displayed on the lower LED display of the Watlow controller).
4. Turn on the heater switch. Within 5 minutes the heater blocks temperature (Upper display) should reach the same value as the SET temperature (Lower display).
5. Attach the GLT desorption tubes and needles to the appropriate handles.
6. Insert the GLT tubes and needles into the heater block ports.

Programmed Temperature Ramp or Automatic Mode of Operation

In order to operate the Watlow temperature controller in an automatic or preprogrammed temperature ramp the following steps should be followed. Before running the system with a temperature program, the user must have previously entered the ramp temperature program stored in the systems memory as described in a subsequent section of this manual.

1. Turn on the Main Power Switch on the Conditioning Oven.
2. If the temperature program has not been installed, refer to the next section on how to install the temperature ramp program before proceeding.

3. Turn on the heater switch. Within 5 minutes the heater blocks temperature (Upper display) should reach the same value as the SET temperature (Lower display).
4. Attach the GLT desorption tubes and needles to the appropriate handles.
5. Insert the GLT tubes and needles into the heater block ports.
6. Turn on the Gas Switch.
7. Press the HOLD/RUN switch to load the temperature program. The first step # of the program will be displayed. You can either begin the temperature program at this first step or you can use the UP/DOWN arrows to increment to a subsequent step in the program.
8. Press the HOLD/RUN switch again to begin the temperature program
9. The lower display will indicate the program set temperature and the upper display will indicate the actual temperature of the heater blocks. Both displays should be within 1° of each other during the temperature program cycle.
10. The system will reset itself when the temperature program is done. To manually stop or interrupt the temperature program during a run, push the HOLD/RUN switch. This will return the controller to the manual (isothermal) mode of operation.

For further information refer to the Watlow Controller User's Manual.

Setting up the Temperature Program for the Watlow Controller

It is often preferred to ramp the temperature of the Conditioning Oven heaters up to a predetermined rate of temperature rise to a set temperature, hold at that temperature for a predetermined time, and then allow the blocks to automatically cool down to a predetermined temperature. For example, GLT desorption tubes packed with Tenax are routinely temperature programmed from ambient temperature up to 300° C at a rate of 4° C per minute while constantly purging with helium or other high purity carrier gas at flows not less than 20 ml per minute. The traps are held at the high temperature for four hours. After this time the power to the heater blocks are turned off, the desorption tubes are removed, allowed to cool, and are finally capped for storage. Traps prepared in this manner exhibit excellent adsorptive capacity and contain no organic background (bleed or artifact peaks) when analyzed by GC/MS. The temperature controller can be quickly and easily set up to conduct this automatic temperature ramp. Programs set up are stored in the controllers memory for future use. Programs are retained in memory, even when power is turned off to the Conditioning Oven. The controller can be programmed for multiple ramp profiles. You can continue entering program parameters until you run out of steps, there are a total of 24 steps available. The following program steps are used to create the program described above for the conditioning of the Tenax desorption tubes. Refer to the Watlow manual for detailed directions on how to enter these values into the Watlow temperature controller and for a detailed description of the Menu selection descriptions.

1. When the lower display reads set point, press MODE once until you see Prog (program parameter). Use the UP arrow key to select YES in the upper display. Press the MODE key once again.

2. The controller asks you for the StEP (Step Number). The upper display reads 1 (Step #1).

3. Press the MODE key to enter Step # 1 and you are then asked for StYP (step type). The default is END. Use the UP/DOWN arrow keys to select SoAh (soak) and then press the MODE key again to enter this value.

4. Use the below table to continue entering the parameters from left to right through the table. Remember that the MODE key is used to progress through the menu, and the UP/DOWN keys are used to select parameters and their values.

5. The program must end with the END statement

6. At the last step (Step # 5), when the program asks for rtn (Return), select YES. This will save the program and corrections in the controller memory, exit the program menu, and return to normal system operation.

Step	StYP Step TYpe	SP Set Point	HOUr	Min	Sec	Ent1 *	Ent2 *	rATE	End	rtn
1	SoAh		0	10	0	OFF	OFF	—	—	nO
2	StPt	300				On	OFF	4.0	—	nO
3	SoAh			4	0	0	On	OFF	—	— nO
4	StPt	20				On	OFF	0.0	—	nO
5	END							—	OFF	YES

*Ent1 and Ent2 values are not used on all models of the controllers.

Step 1 initializes the beginning set point to the current set point which has been manually entered and will hold it at this temperature for 10 minutes. This is designed to permit sufficient gas to flow through the desorption tube to remove all traces of oxygen from the Tenax contained therein before the heating cycle is begun.

Step 2 sets a final temperature of 300° C which will be attained at a ramp rate of 4° per minute.

Step 3 holds the heater blocks at the high temperature (300° C) for a total of 4 hours.

Step 4 reinitializes the system to 35° C and begins the cooling cycle.

Step 5 ends the program.

V. Standard Procedures For Preparing And Conditioning Desorption Tubes

Upon installation, a carrier gas line from a high purity gas source such as nitrogen or helium is attached to a single 1/8" swagelok fitting on the back of the Conditioning System. On top of the Conditioning System six quick disconnects are connected to 1/8" flexible PTFE lines to provide gas flow through the desorption tubes and needles during conditioning. The electrical connection is made with a 110 VAC plug which is grounded. Desorption tubes which have graphite seals and needles which have either graphite or graphitized vespel seals screw into the Conditioning Oven handles.

Desorption Tubes are prepared in the following manner for use in the "Short Path Thermal Desorption System".

A. Washing - All desorption tubes should be thoroughly cleaned by a detergent and water rinsing followed by an acetone rinse in an ultrasonic bath and air dried. Appropriate ventilation, safety glasses and gloves should be used. Tubes are baked out in an oven at 100°C. Cleaning by additional solvents such as methanol may be required depending on the applications of the user. It is important to remove all solvent residues by thoroughly baking out the tubes.

B. Silylating - Silylation with a suitable silation reagent such as Dimethyldichlorosilane (DMDCS) under the trademark Silon™ T (Pierce Co.) is recommended depending on the nature of the components being analyzed and the requirements of the user. This is done under a hood with protective glasses and gloves. Tubes are capped on one end without a seal, placed in a beaker and filled with the reagent via a pipette for approximately one minute and then the reagent is discarded into a labelled hazardous waste

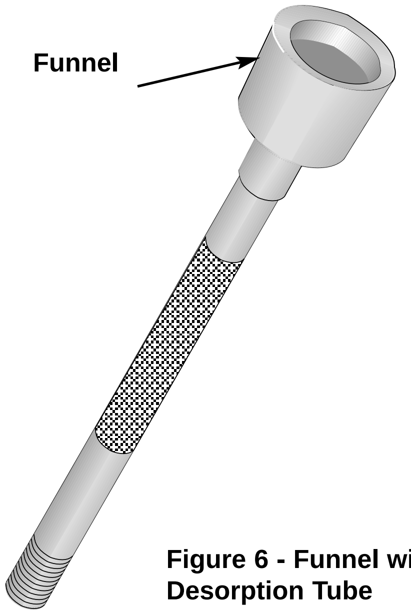


Figure 6 - Funnel with Desorption Tube

container. This is preferred to dipping the entire desorption tube in the silation reagent due to the reaction of the reagent with the outer metal covering resulting in the discoloration of the surface.

C. Rinsing - This is followed by removing the caps and rinsing (2X) with methanol: methylene chloride (1:1) in a beaker in an ultrasonic bath for approximately 30 minutes each.

D. Drying - Tubes should be baked out in an oven at 100°C.

E. Packing - The glass-lined stainless steel (GLT) sample tubes (3 & 4 mm i.d.) are next packed with a predetermined porous polymer resin such as 2, 6-diphenyl-p-phenyleneoxide sold under the trademark Tenax™ TA or the activated graphitized carbon sold under the trademark Carbotrap™ or combination thereof. Both of the above enumerated trapping agents have a high affinity for non-polar organic compounds and a very low affinity for water vapor and other low molecular weight polar compounds such as alcohols with less than three carbon atoms. Different trapping agents have affinities for different types of organic compounds and it is important to choose one which is known to have a high affinity for the analyte of interest. Tenax is ideal for aromatics, heterocyclics, aldehydes, ketones, alcohols etc. as long as the alkyl chain lengths are C-3 or longer. Carbotrap is good for the same type of compounds but also effectively traps aliphatic, olefinic and other types of paraffinic hydrocarbons which lack any other type of functionality. For these reasons we routinely make combination traps containing two or more trapping agents combined which then provides us with a broad spectrum general purpose trap. Two other trapping agents include Carbosieve S-III which is excellent for trapping small airborne molecules such as the C-2 hydrocarbons and Carbotrap C which is an ideal adsorbent for trapping a wide range of airborne organic compounds from C-4 to C-5 to polychlorinated biphenyls, polynuclear aromatics, and other large molecules. There are many other adsorbent materials which can function equally as well which are available from many manufacturers (Supelco, Alltech).

Three and 4mm i.d. desorption sample tubes are packed with approximately 40-250 mgs of adsorbent material using an S.I.S. funnel (Part# 781122) **(FIg 6)**. The funnel fits snugly over the o.d. of the desorption tube providing easy packing of the adsorbent material. The amount of adsorbent used depends on the users requirements. The ends of the tubes are plugged with silanized glass wool approximately 1 cm on each end to hold the adsorbent in place **(Fig 7)**.

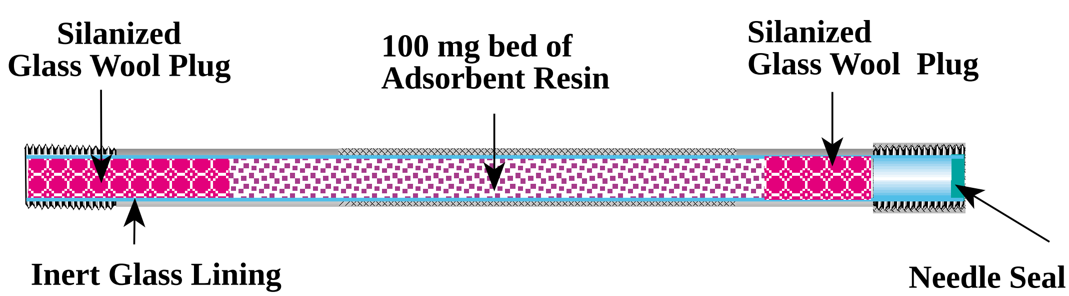


Figure 7 - Cross Section of Packed GLT Tube

F. Conditioning - In order to prepare the packed desorption tubes for the collection of samples, the adsorbent resins contained therein must be conditioned to remove all foreign materials including water vapor. This is done utilizing the Desorption Tube Conditioning Oven. The following procedure or a suitable version of this method should be used to condition the desorption tubes. The maximum temperature utilized will be determined by the properties of the adsorbent material used in the desorption tubes. Empty GLT desorption tubes are conditioned in the same manner.

(1) Attach desorption tubes with graphite seals with metal inserts to the Conditioning Oven handles.
(2) Loosely attach caps with holes approximately 1.0 mm in diameter (Part#781007) on the bottom of the desorption tubes to prevent glass wool and adsorbent from blowing out the end of the desorption tube during conditioning under flow. Do Not tighten, otherwise caps may seize after heating. Do not attach needles to desorption tubes for conditioning.
(3) Turn on gas flow and adjust with microneedle valve to achieve flow of 20 ml/min through each desorption tube.
(4) Insert desorption tube into heater block.
(5) Set up temperature program for heater block. Refer to Watlow manual for details.
(6) Flow gas through tubes for 3-10 minutes to remove all air/oxygen from inside tubes before heating.
(7) Begin temperature program.
(8) Flow condition for a minimum of 4 hours at maximum temperature.
(9) When complete place tubes in the cooling rack on back of the cabinet. WARNING-HOT Do Not Touch. Use PTFE handles when removing tubes and needles from the heater block.
(10) Allow to flow cool for 5-10 minutes. Do Not flow longer than necessary when cooling or you will trap trace materials from the carrier gas.
(11) Turn off heater. Allow to cool to less than 60°C and then turn off main power.
(12) Condition solid caps with PTFE seals in GC oven at 200°C for 1-2 hours while desorption tube are conditioning. Remove and allow to cool.
(13) Cap conditioned desorption tubes with stainless steel caps and seals.

G. Storage - The tubes are then immediately capped on both ends with stainless steel solid caps with PTFE seals that have also been conditioned. Tubes prepared in this manner exhibit excellent adsorptive capacity and contain no organic background when analyzed by GC/MS. Preconditioned tubes can be stored at room temperature for two to ten weeks.

H. Needle Conditioning - In the Short Path Thermal Desorption System, the needle serves as the transfer line for sample introduction into the gas chromatograph from the glass-lined thermal desorption tube containing the samples being analyzed. This "short path" for sample transmission is a key advantage of the Short Path Thermal Desorption System minimizing sample decomposition, eliminating sample contamination (memory effects) of the transfer lines, and providing for maximum delivery of samples into the gas chromatograph (maximum sensitivity). It is therefore essential that the needles be baked out at 300°C for 15 minutes while purging with helium or another suitable gas at 20 ml/min in the Conditioning System prior to sample introduction into the GC. Repeat this after every sample. Attach the needles to the Conditioning Needle handles with the appropriate seals (graphite or graphitized vespel). Cool before use.

Note: Empty GLT desorption tubes for direct thermal desorption are conditioned in the same manner.

Conditioning Oven and Replacement Parts

Part No.	Description
781051	Desorption Tube Conditioning Oven, Conditions 6 sample tubes simultaneously, 6 rotameters, programmable temperature controller, 6 desorption tube handles, 2 needle handles and seals
781013	Connection Handle for Desorption Tube
781014	Connection Handle for needle
781015	Graphite top seal with metal insert
781007	S.S. Cap with 0.040" hole for conditioning GLT tube
781122	Funnel
781006	Cap, Solid
781004	PTFE Seals
782007	Fuse, 10A slo-blow

Sample Oven


Short Path Thermal Collection System
Scientific Instrument Services, Inc., Ringoes, NJ USA
WATLOW
Heat Control
Main Power
Heater
Gas

Sample Oven

I. Introduction

A. Theory of Operation

The Sample Collection Oven **(Fig. 1)** permits the collection of volatiles and semi-volatile compounds present in solid materials into desorption tubes packed with an adsorbent resin for subsequent analysis by desorption utilizing the Short Path Thermal Desorption System. Volatile organics can be collected from sample sizes ranging from less than 0.5 grams up to 20.0 grams. The Sample Collection Oven consists of a sample tube oven with ports for four 0.5" or 0.25" diameter sample tubes. Up to four samples can be collected simultaneously with the system. A Watlow precision temperature controller provides accurate control of the temperature of the Sample Collection Oven up to 250° C. A digital readout indicates the actual oven temperature. The actual Sample Collection Oven consists of a large aluminum plate with holes drilled for the sample tubes and is heated by cartridge heaters. Heat transfer to the sample tubes occurs via direct thermal transfer from the aluminum block to the sample tubes. Temperatures can be maintained within 1% of the full scale reading. Four rotameters regulate the gas flow through each of the samples independently of one another.

B. Specifications

1. Safety - Warning Messages

WARNING Do not heat samples above 250° C otherwise damage may occur to oven or internal circuitry.

WARNING Maximum gas pressure is 60 P.S.I.

WARNING Do Not use Hydrogen Gas in the Sample Collection Oven. Use only He or N_2.

WARNING Make sure that only fuses with the required rating and of the specified type are used for replacement. The use of incorrect or makeshift fuses or the short-circuiting of fuse holders creates a shock hazard for the operator and can damage the instrument.

WARNING Do Not increase temperature range of heat overload circuit.

WARNING Do Not leave Sample Collection Oven heaters in the heated ON position unattended overnight. The Sample Collection Oven heaters rapidly heat and cool to their final operating temperatures and therefore, in order to prolong their life and avoid premature failure of the system they should be turned off when not actively being utilized.

WARNING Use grounded outlet only. Connecting the Sample Collection Oven to a power source which is not equipped with a protective earth ground contact creates a shock hazard for the operator and can damage the instrument.

WARNING The Sample Oven should be used in a laboratory hood.

WARNING Do not expose persons to the direct line exposure of the ends of the sample tubes. Fittings can slip off under pressure and cause injury if the user is not protected.

WARNING Safety glasses must be worn when using the Sample Oven. High gas pressures are present and glass sample tubes are used which may present a hazard.

2. Electrical Specifications

Power Requirements
Voltage - 110 VAC
Current - 10 amps maximum
Electrical Cord - 110 v/grounded outlet

Temperature Controller

Heater Circuit - Accuracy - +/- .1% of full scale- Range - up to 250° C
Heater Output - 115 volt, 1200 watts
Sensor Input - Platinum Resistance Thermometer
Heater Cartridges - 4 each - 115 v, 300 watts
Digital Readout for set & actual temperatures 3 or 4 digit
Temperature Range - Room Temperature to 250° C

3. Gas / Pressure Specification

Type - Nitrogen or Helium (high purity)
Maximum pressure - 60 P.S.I.
Flow - maximum 50 ml / min per tube
    - Maximum total 200 ml / min
Quick disconnects - automatically turn off gas

4. Weight and Dimensions

Weight - 18 pounds
Size - 9" wide x 12" deep x 11" high

Warranty

The Sample Collection Oven is warranted against defects in material or workmanship for a period of 90 days commencing from the date of shipment from the warehouse of Scientific Instrument Services in Ringoes, N.J. hereafter referred to as the company. The company's liability on the Sample Collection Oven and accessories is limited to the cost of correcting the defect in the product. In no case shall the company be liable for consequential or special damages. The system should not be run unattended overnight. The company will not correct defects caused by buyers negligence. The company does not guarantee or warrantee the product for any particular purpose. The companies warranty shall end 90 days after shipment.

Extended Warranty

An extended one year warranty for parts and labor is available if purchased within 30 days of shipment of the unit. The one year extended warranty will cover parts and labor to repair the Sample Collection Oven within the facilities of Scientific Instrument Services. Service on customers facilities is not available.

Service and Repair

The Collection Oven should be serviced only by qualified S.I.S. staff. Any shipment to be serviced under warranty or otherwise should be sent to the repair facilities of Scientific Instrument Services in Ringoes, N.J. 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 returned.

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

RA # _______________
Phone: (908) 788-5550

II. System Description

A. Front Panel Description (Fig. 1)

Three on/off switches are present on the front panel including a main power switch, a heater switch and a gas switch. The main power switch controls the power to the entire Collection Oven, to the temperature controller, and other switches. The heater switch turns on the power to the heater cartridges in the heater block and begins its heating cycle. This switch should be turned off at night or when the system is not in use. The carrier gas switch turns on the carrier gas to permit its flow through the samples. An L.E.D. digital readout displays the set temperature and the actual temperature of the system. A heater demand light indicates the actual heating demand of the heater blocks. If the demand light is bright red the heater is on; heater is off when there is no light. If the demand light is dim red, the heater is unplugged or an open circuit exists.

The self contained system includes four adjustable bubble rotameters with flow ranges of 0 to 50 ml/min, one for each of the sample tubes. The recommended flow rate is 20 ml/min. Flowmeter calibration data for air and helium are listed in **Table 1**. Each of the flows through the tubes can be independently controlled or flow turned off to that port via a high precision metering valve. A single electrically operated solenoid valve turns the gas flow off or on to all ports via the gas switch on the front panel. Four quick disconnect fittings connect to a 1/8" flexible transfer line to provide gas flow through each of the sample tubes during sampling. The quick disconnects turn the flow off to individual gas flow lines when disconnected.

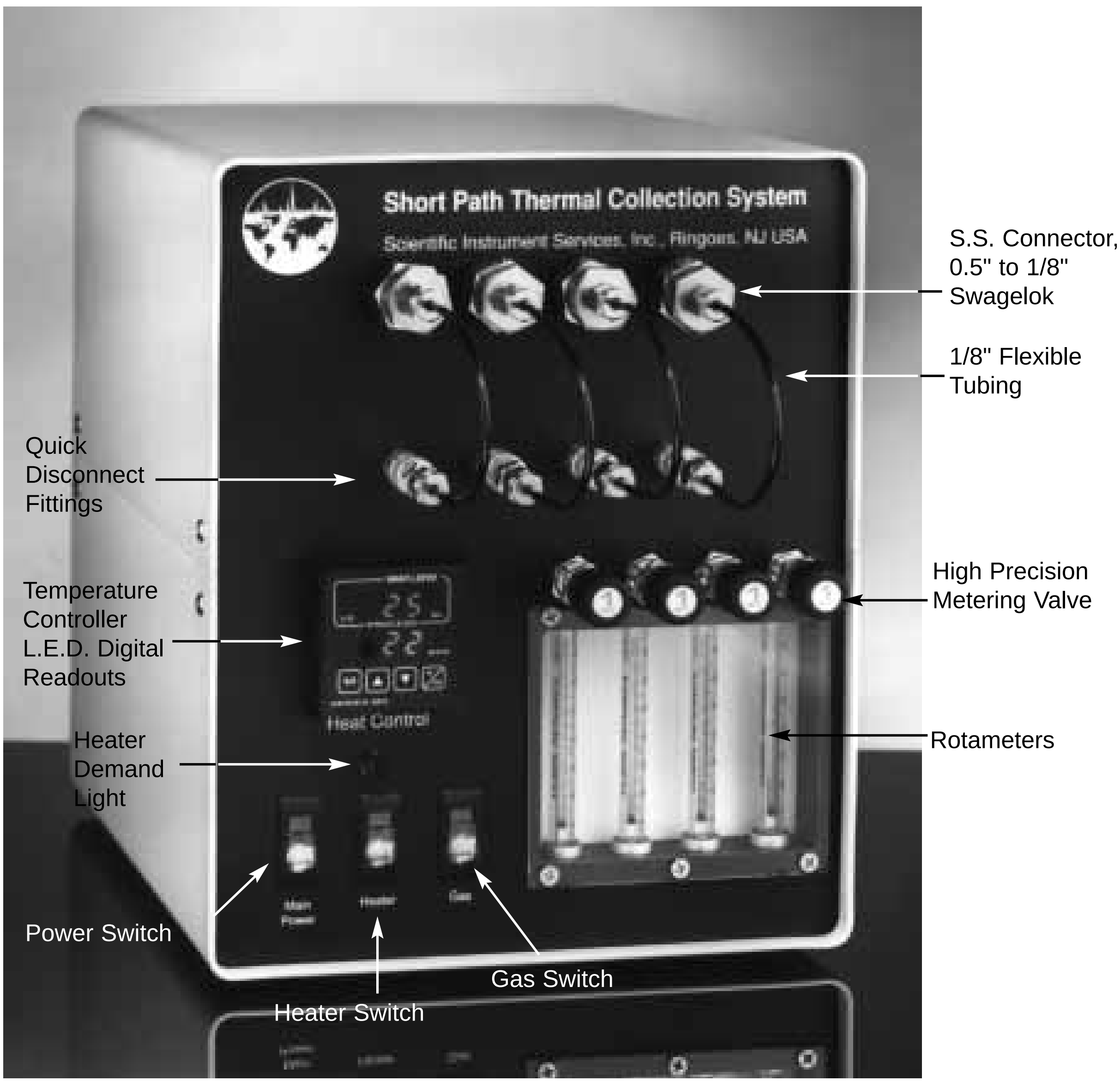


Figure 1 - Sample Collection Oven - Front View

TABLE 1

Flowmeter Calibration Data
Scale Readings at Center of Float

Scale Readings	Flow ml/min (Helium)	Flow ml/min (Air)
65	44	48.7
60	39	43.3
55	33	37.6
50	28	31.4
45	23	26.5
40	20	22.4
35	17	19.1
30	15	16.0
25	12	12.7
15	6	6.6
10	4	4.7
5	3	3.3

B. Rear Panel Description (Fig. 2)

Electrical power is provided from a standard 110 volt, 10 amp grounded outlet. A single 1/8" swagelok fitting on the back of the Sample Collection Oven provides for the attachment of the carrier gas from its source. An external high temperature reset switch is located in the rear of the oven so that if temperatures exceed 250° C on the heater block, the external reset will open and heat to the block will stop. This switch can be reset by simply pushing in this red button once the block has cooled. Two Slo Blo fuses are mounted in the rear of the Collection Oven to handle the initial surges of the Main Power and Heater switches when they are turned on.

In addition, a three inch fan enclosed on the back side of the Collection Oven provides a steady flow of air through the system to maintain the temperature of the temperature controller, rotameters, and other components inside the system at an acceptable level and to provide for cooling of the heater block once the heater circuitry is turned off.

III. Electrical and Gas Flow Circuit

The Sample Collection Oven schematic (**Fig. 3**) pictorially represents the overall operation of the system including the gas flow and heater circuits. A 110 volt outlet is required and 10 amps provides the total electric power required for system operation.

The self contained system includes four adjustable bubble rotameters with flow ranges of 0 to 50 ml/min, one for each of the sample tubes. Each of the flows through the tubes can be independently controlled via a microneedle valve and the flow turned off to that port if no flow is required. A single electrically operated solenoid valve (v) turns the gas flow off or on to all ports via the gas switch on the front panel (**Fig. 3**).

On the front of the Sample Collection Oven four quick disconnect fittings connect to a 1/8" flexible transfer line to provide gas flow through each of the sample tubes during sampling. When a quick disconnect is removed the gas flow is automatically closed to that port. By providing gas flow from a carrier gas such as high purity nitrogen or helium through the sample tubes while sampling, it can be assured that no oxygen enters the GLT desorption tube which could destroy the adsorbent material. A single 1/8" swagelok fitting on the back of the Collection Oven is provided for the attachment of the carrier gas from the source (**Fig. 2)**.

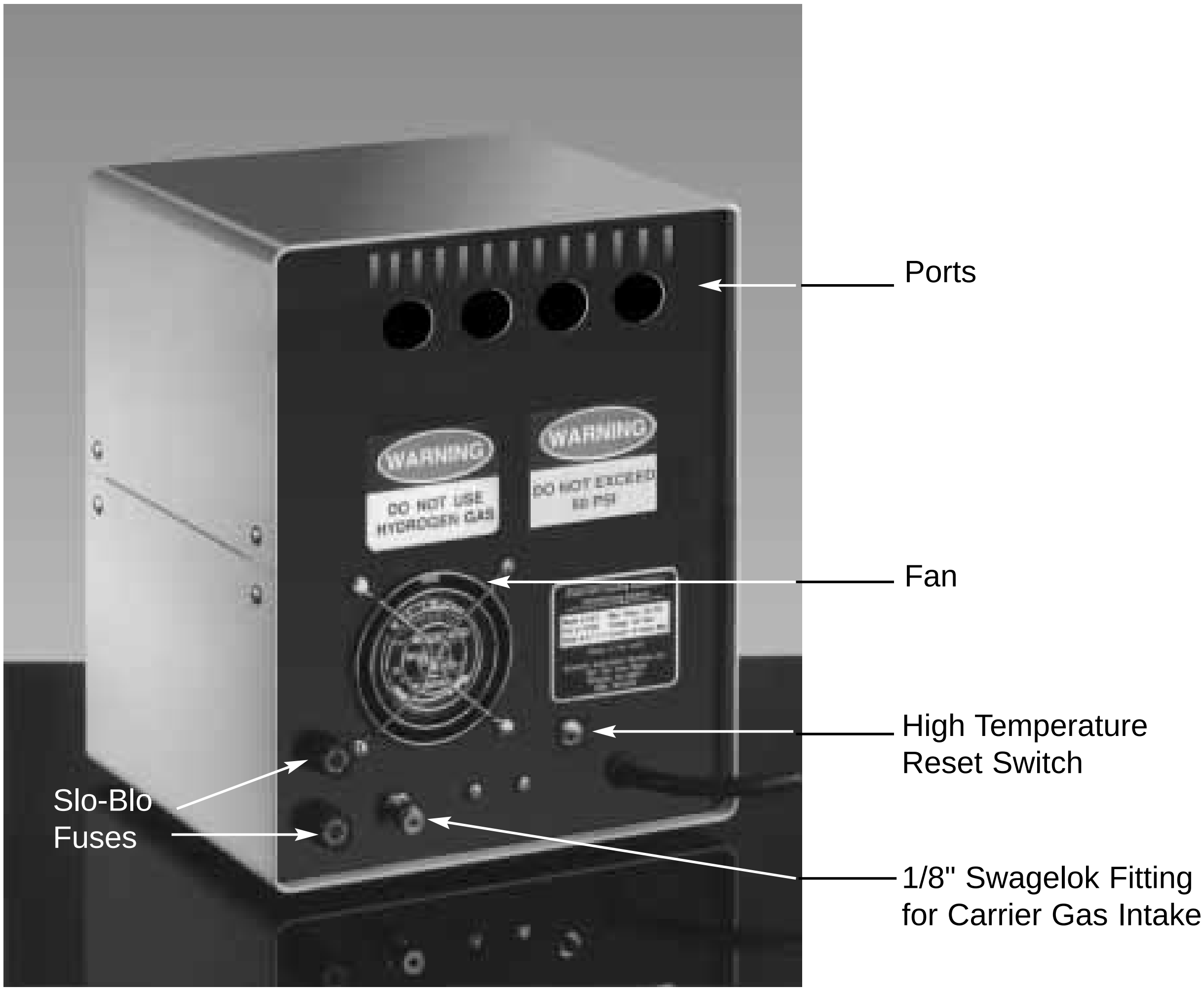


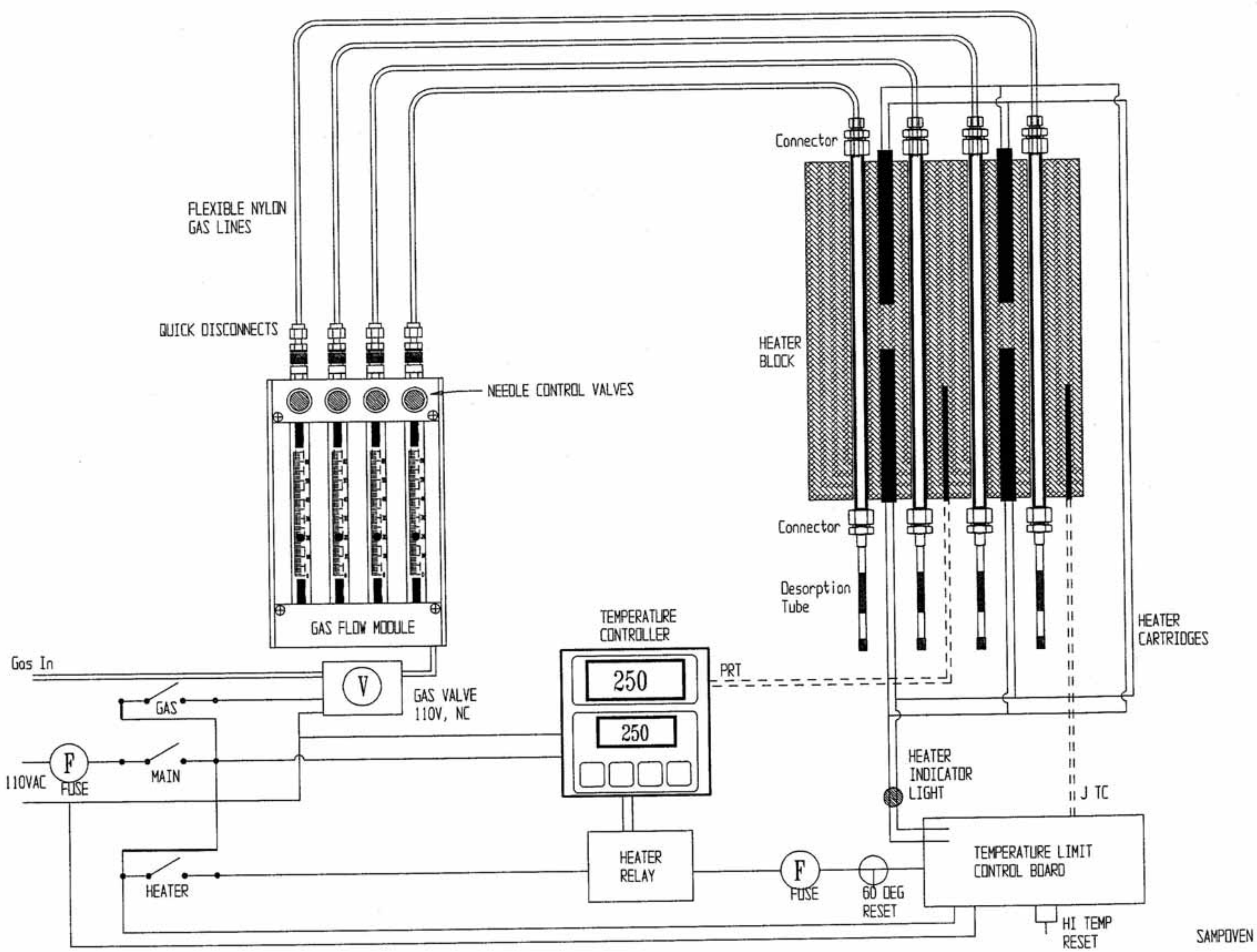
Figure 2 - Sample Oven - Back View

IV. Heater Circuit

The Sample Collection Oven includes a heater block with ports for four 0.5" diameter sample tubes. Up to four samples can be collected simultaneously at temperatures up to 250° C with the system. A Watlow Microprocessor Based Auto-tuning temperature controller permits the setting of the temperature at which the samples are to be collected. A platinum resistance thermometer (PRT) in the heater block provides for accurate temperature readout and also provides the feedback to the temperature controller to maintain the heater block temperatures. Accuracy of the programming temperatures is +/- .1% of the full scale reading. An L.E.D. digital readout displays the set temperature and the actual temperature of the system.

Four 3/8" diameter 300 watt heater cartridges heat the aluminum heater block. The heater cartridges are wired in parallel and are protected by their own 10 amp fuse. The heater indicator light displays the current demand for the heater cartridges. When off, the heaters are not receiving any power and therefore are not heating. When brightly lit the heater cartridges are in the heating cycle. If dimly lit, the heater is unplugged or an open circuit exists.

Figure 3 - Sampling Oven Schematic

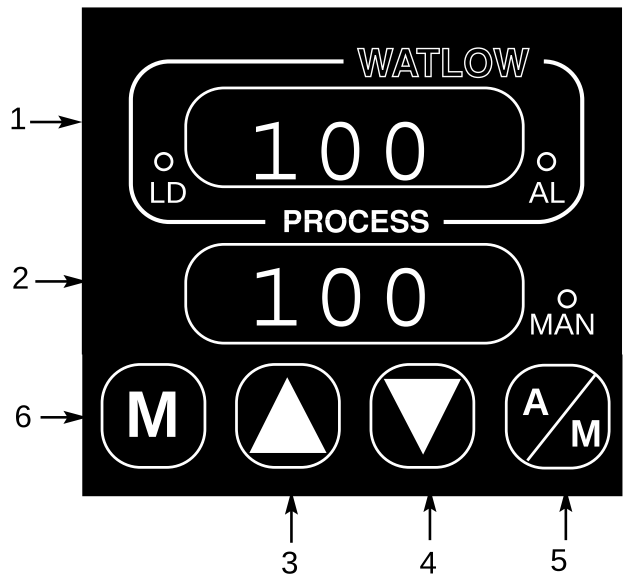


A. Heater Protection Circuitry

A heat overload thermostat (internal reset switch) is located inside the case of the Sample Collection Oven to prevent temperatures from exceeding 60° C inside the case, thereby providing a protective circuitry for the system. If the Oven case should exceed 60 °C due to a circuit failure (such as failure of the cooling fan to operate), this overload sensor will open the circuit providing power to the heater cartridges. This overheat sensor can only be reset by allowing the system to cool, opening the Oven case, and manually resetting the internal overload reset switch. If the heat overload thermostat should open when operating the system, the entire Sampling Oven should be returned to the factory for service. Service should only be performed by qualified electrical technicians knowledgeable of the system electrical circuits. In addition, a high temperature limit control board with an external high temperature reset switch (**Fig. 2 - Hi temp reset**) prevents heater block temperatures from exceeding 250° C. If block temperatures exceed 250° C, the external reset will open and heat to the block will be terminated until the block cools and the external reset switch is reset (**Fig. 2)**. Note: Do not exceed 250° C for the heater block.

B. Watlow Temperature Controller

Description of the Keys and Display



1. **Upper Display (Actual heater block temperature)**
   The red, 0.3" (8 mm) high, seven segment, three digit LED display, indicates the heater block temperature, the operating parameter values, or an open sensor. When powering up, the Process display will be blank for 5 seconds.

2. **Lower Display (SET temperature)**
   The red 0.3" (8 mm) high, seven segment, three digit LED display, indicates the set point, output value, prompts for data in the upper display, or error and alarm codes.

3. **UP Key**
   Increases the value of the temperature set point. A light touch increases the value by one. Holding the key down increases the value at a rapid rate. New data is self entering in 5 seconds.

4. **DOWN Key**
   Decreases the value of the temperature set point (lower display). A light touch decreases the value by one. Holding the key down decreases the displayed value at a rapid rate. New data is self entering in 5 seconds.

5. **AUTO/MAN Key**
   This key is inoperable as set by the factory. The System always runs in the automatic mode. Refer to the Watlow 965 controller manual for more information.

6. **Mode key**
   Allows the user to step through the operation menu. This key is inoperable as set by the factory. For more information see the Watlow 965 controller manual.

Note: The following is a description of the factory preset values. This is given only for reference in case of a problem. The values should never be changed. Refer to the Watlow 965 manual for more information.

Temperature Controller System SETUP

The purpose of the SETUP menu is to define the various operating parameters and condition of the temperature controller. These user selectable parameters include the conditions of controller operation, the calibration parameters, rate of heating, sensor input type, temperature scale (°C or °F), display decimal point location, and temperature range. The Watlow temperature controller has already been preset to the correct operating parameters at the factory and the user should not normally have need to alter these factory settings. The SETUP Menu is entered by pressing both the UP and DOWN arrow keys simultaneously for 3 seconds. The values presently in the controller on the Sampling Oven have been factory selected for the correct operation of the Sampling Oven. It is NOT recommended that they be changed. For a detailed description of these parameters and the selections available refer to the Watlow Users Manual.

The factory preset values are listed below.

Setup Menu		Operation Menu	
LOC	3	Pb1	13
In	rtd	rE1	0.06
C_F	C	rA1	1.25
rL	0	Ct1	5
rH	250	CAL	0
Ot1	ht	AUt	0
HYS	2		
Al1	no		
rtd	din		

V. Operation of the Sample Oven System

The Sample Collection Oven permits the analysis of trace components in larger sample sizes than could be analyzed by direct thermal desorption in the Short Path Thermal Desorption System. Samples to be analyzed are placed inside glass tubes and held in place by glass wool plugs. The system is designed to sample from 0.5" diameter tubes, and also from 0.25" diameter tubes with optional adaptors. Sample tubes are each 14" long. Samples up to 20 grams can be sampled in the 0.5" diameter tubes. When ready for sample collecting, the long tube containing the sample is placed in the Sample Collection Oven, fitted at one end with a supply of carrier gas to flush out the residues, and fitted at the opposite end with an adaptor fitting and a preconditioned desorption tube with adsorbent resin (**Fig. 4**). The Oven is set for the predetermined temperature and the desired components are collected onto the desorption tubes. Oven temperatures can range from room temperature up to 250°C. The temperature used, as well as the time of sampling, depends on the nature of the samples being analyzed and the requirements of the analyst. By providing gas flow from a carrier gas such as Nitrogen or Helium through the heated sample tubes while sampling, the volatile and semi-volatile organics will be purged from the sample and will be trapped by the adsorbent in the desorption tube.

This technique also permits the analysis of volatiles from organic samples with high moisture content. A desorption tube adaptor fitting with a dry purge inlet can be used to reduce the water vapor condensation on the adsorbent trap. This problem can be especially troublesome when isolating volatiles from high moisture content samples at high temperatures. Although the adsorbent traps packed with Tenax have a low affinity for water it is inevitable that some condensation will occur when the heated sparge gas contacts the cool desorption tube adaptor fitting as the gas exits the apparatus. When moisture condenses on the adsorbent it can block the pores of the resin matrix and thereby drastically reduce the diffusion of volatile organics into the trapping agents. This will result in reduced trapping efficiency. With an additional dry purge, the gas is kept in the vapor phase and passes through the adsorbent resin without condensing onto the adsorbent.

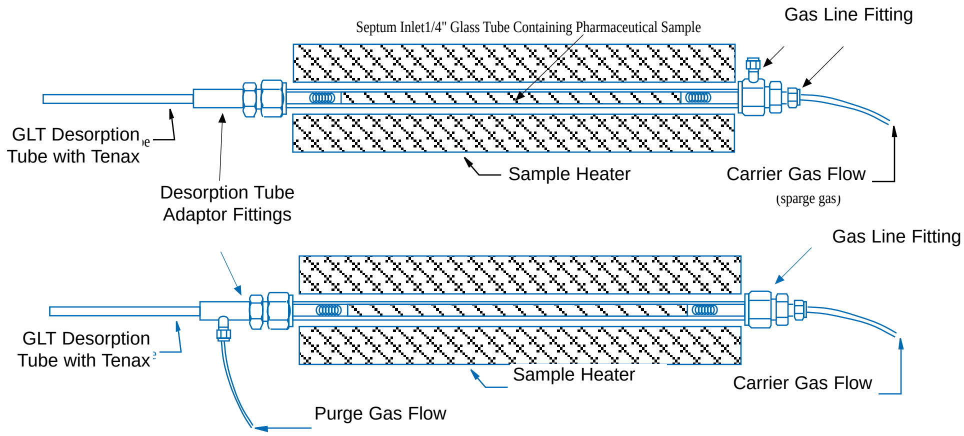


Figure 4 - Cross Section of Sample Collection Oven for the collection of thermally sparged volatile samples

The following procedure or a suitable version of this method should be used to sample solid samples.

I. Turn on Procedure

1. Turn on main power. **Note:** The Sample Oven should be used in a laboratory hood. See page 11-2 for caution notes. The Sample Oven operates under pressure with glass tubes. Extreme caution must be exercised when using. Eye protection must be worn when using the Sample Oven. Do not expose face to direct line exposure of sample tubes.
2. Use the UP/DOWN arrow keys to select the desired temperature of the heater block (SET temperature is displayed on the lower LED display of the Watlow controller).
3. Turn on the heater switch. Within 5 minutes the heater block temperature (Upper display) should reach the same value as the SET temperature (Lower display).
4. Safety glasses must be worn when using the Sample Oven. High gas pressures are present and glass sample tubes are used which may present a hazard.

II. Cleaning & Conditioning Sample Tubes

1. Wash sample tubes in detergent and water in appropriate container for 15 minutes, then rinse in a solvent such as acetone for 15 minutes and let air dry.
2. Condition sample glass tubes with glass wool plugs 50°C above sample temperature to be used (maximum temperature - 250°C) for 15 minutes in the Sample Oven under low gas flow (10 ml/min) prior to collecting sample, then remove the tubes from the Sample Oven and allow to cool to room temperature.
3. Glass wool can be conditioned separately and stored until ready for use.

III. Sampling

1. Insert preconditioned glass wool plug in one end of sample tube, then place sample to be analyzed inside sample tube and hold in place with additional glass wool.
2. Place preconditioned sample tube containing sample into Sample Collection Oven.
3. On front of Collection Oven connect quick disconnect fitting to 1/8" flexible transfer line via S.S. Connector, 0.5" to 1/8" Swagelok to provide gas flow through sample tube **(Figs. 4 & 5)**.
4. At the opposite end of the sample tube connect a preconditioned desorption tube with adsorbent resin to the sample tube via a S.S. Connector, 0.5" Swagelok to desorption tube **(Figs. 4 & 5**). Refer to Conditioning Oven Manual for conditioning desorption tubes.
5. Turn on carrier gas and begin collecting VOC's.

6. Do not expose persons to the direct line exposure of the ends of the sample tubes. Fittings can slip off under pressure and cause injury if the user is not protected.

7. When VOC's have been collected removed desorption tube and cap until ready for analysis.

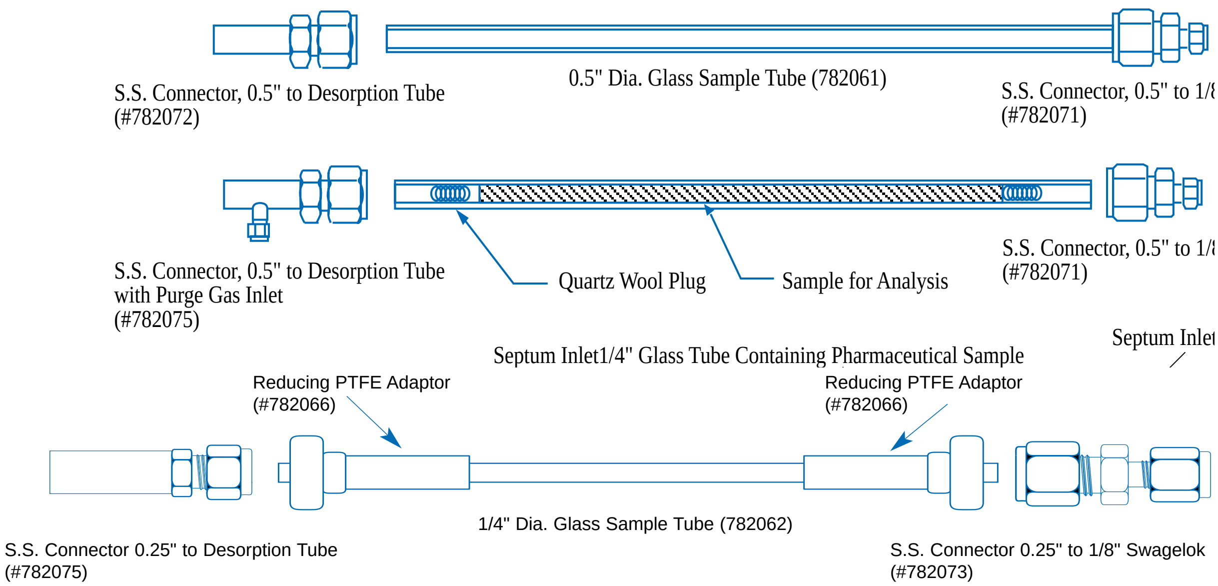
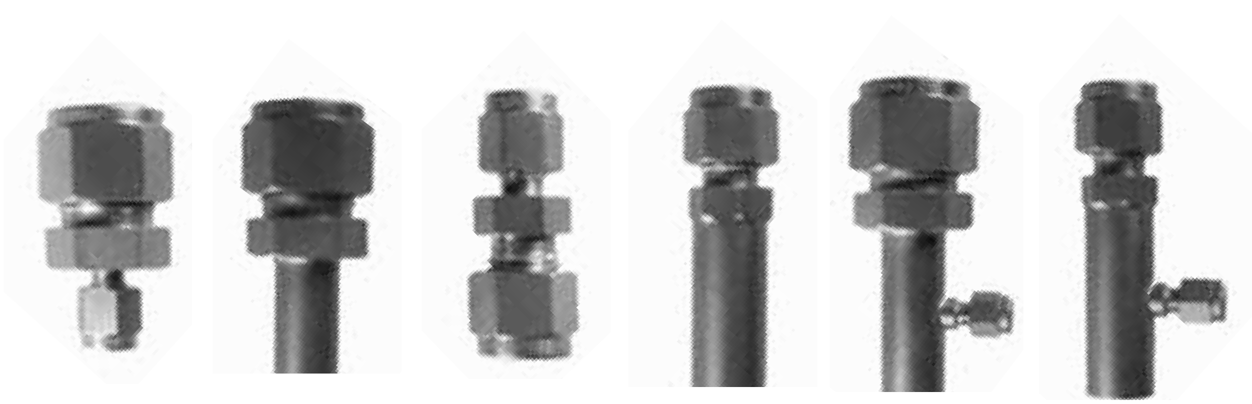


Figure 5 - Sample tube with connectors

Fittings & Adaptors for Sample Collection Oven



782071 782072 782073 782074 782075 782076

Sample Collection Oven System and Accessories

Part No.	Description
782050	Sample Collection Oven for 4 samples with 4 rotameters 4-0.5" ports, 4-0.5" glass sample tubes with fittings
782061	Sample Tube, Glass, 0.5" O.D. x .36" I.D. x 14" long
782062	Sample Tube, Glass, 0.25" O.D. x .15" I.D. x 14" long
782063	Sample Tube, Stainless Steel, 0.5" O.D. x .36" I.D. x 14" long
782064	Sample Tube, Stainless Steel, 0.25" O.D. x .15" I.D. x 14" long
782065	Reducing Adaptor Vespel, 0.5" to 0.25" sample tube
782066	Reducing Adaptor PTFE, 0.5" to 0.25" sample tube
782071	S.S. Connector, 0.5" to 1/8" Swagelok
782072	S.S. Connector, 0.5" Swagelok to Desorption Tube
782073	S.S. Connector, 0.25" to 1/8" Swagelok
782074	S.S. Connector, 0.25" Swagelok to Desorption Tube
782081	0.5" PTFE seal
782082	0.25" PTFE seal
782075	S.S. Connector, 0.5" to Desorption Tube with 1/16"purge gas inlet
782076	S.S. Connector, 0.25" to Desorption Tube with 1/16" purge gas inlet