SIS AutoDesorb™ Operation Manual

AutoDesorb Model 2000

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NOTICE

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

Scientific Instrument Services™, Inc. (SIS™) 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.

SIS 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 or the AutoDesorb System as described in this manual.

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

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

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

Other Patents are pending.

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This Manual

This manual provides general information and covers the operation of the AutoDesorb™ system **(ADS)** from Scientific Instrument Services, Inc., including system care and maintenance. Information pertaining to installation and software can be found in the section of the manual dedicated to those subjects. Detailed information about system accessories can be found in the appendices. As new equipment is developed, additional appendices will be provided.

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

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

http://www.sisweb.com

Figure 1. The AutoDesorb System. Desorption unit (left) and Controller (right).

General Description

The SIS Short Path Thermal Desorption systems are intended for the thermal extraction and analysis of volatile and semi-volatile organic compounds from gas, liquid and solid matrix samples. A few of the applications for which they have been used include organics in air, flavors and fragrances in foods and cosmetics, chemical residues in pharmaceuticals, volatiles in packaging materials and building products, and residues in forensic samples.

These systems were developed specifically to provide a means of introducing volatile components into a GC with minimal carryover, sample degradation and solvent use. They were also designed to be easily installed and removed from the GC so that the instrument need not be dedicated to thermal desorption analysis. Once installed, the AutoDesorb system can be removed from the GC in seconds. The AutoDesorb system can analyze both volatile and semi-volatile analytes from a wide variety of sample matrices without any memory effects and with greater sensitivity than many solvent injection or direct headspace techniques. These improvements are accomplished in two ways:

(1) by concentrating analytes on a trap or extracting directly into the GC inlet
(2) by providing a very short path from the sample to the analytical column.

The AutoDesorb System has several unique features:

(1) It eliminates the use of solvents by using only heat to extract volatile and semi-volatile compounds. The AutoDesorb System can desorb samples ballistically at temperatures up to 450ºC or at ramp rates of up to 100º C per minute. Desorption times and other timers can be set from 1 second to 99 minutes. Multi-step desorption heater ramping with hold times can also be programmed.

(2) The desorbed sample component is easily and efficiently transferred into the injection port of the gas chromatograph from its GLT desorption tube and needle. The short transfer path in an inert environment minimizes degradation of labile sample components and condensation of semi-volatile material. Focusing of the analytes takes place on the analytical column or guard column rather than an intermediate sorbent bed.

(3) Each sample has its own unique adsorbent trap tube and needle to eliminate the possibility of cross-contamination and prevent "memory effects" due to overloading or residues from previous samples.

(4) The system does not dedicate the inlet or instrument to one analytical technique. Liquid, headspace, or automated injections can be resumed within minutes of demounting the thermal desorber.

(5) The automated system permits the unattended analysis of multiple samples, increasing laboratory productivity.

Two main thermal desorption sampling techniques are used: "Purge and Trap Thermal Desorption" (P&T) and "Direct Thermal Extraction " (DTE). With the P&T technique, analytes are first trapped on an adsorbent resin such as Tenax™ TA or activated carbon, then desorbed from the trap into the gas chromatograph. In DTE, volatile and semi-volatile materials are desorbed directly from the sample matrix into the GC inlet. For more information, see the 'Considerations for Method Development' section in the Operation Manual.

Hardware Description

The AutoDesorb System hardware consists of the desorption unit, controller (see Figure 1), and an optional Cryotrap (not pictured).

The desorption unit mounts over the GC inlet and contains the sample carousel, the pickup and injection mechanisms, desorption heater and flow control devices. Each GLT desorption tube is attached to a connecting tube and needle, and is placed in a numbered position on the carousel. The connecting tube contains an integrated molecular sieve filter and sealing element that may be removed and reconditioned as needed. The needle is used to puncture the septum in the GC inlet and provide the short transfer path.

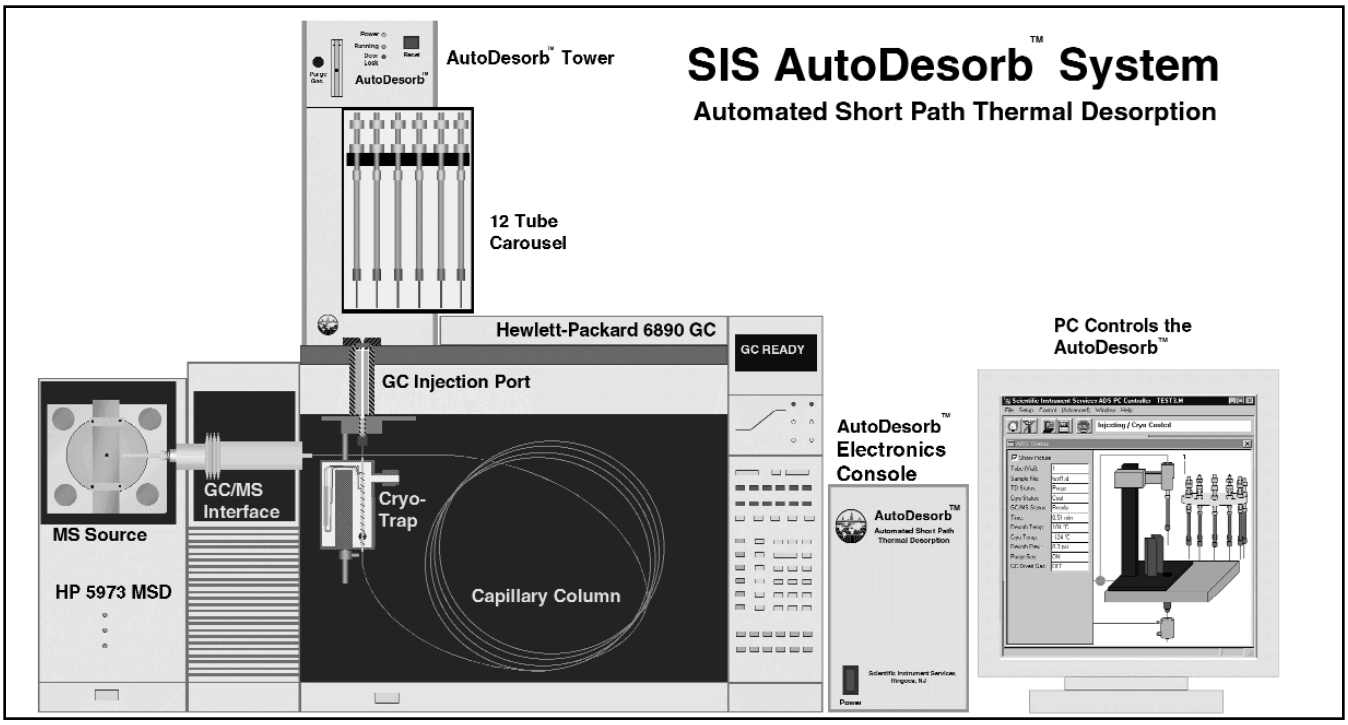


Figure 2 - AutoDesorb system components

The controller consists of the microprocessor and the electronics used to control and monitor the status of the desorption unit and cryotrap. The controller is connected to a serial communications port on the data system computer. It also sends 'START' and 'STOP' or receives 'READY' signals from the gas chromatograph.

The Cryotrap mounts inside the GC oven underneath the injection port. The analytical column or a short guard column runs through the trap and is connected to the inlet in the normal way. The Cryotrap provides a means to focus volatile materials at the head of the column. It releases the trapped analytes by heating rapidly, which results in excellent chromatographic resolution.

Software Description

The AutoDesorb hardware is controlled and interfaced to a chromatographic data system with software developed at SIS. The software includes a user-friendly graphical interface that allows the desorption system and cryotrap status to be monitored. The interface also allows run settings to be modified in real-time. Preset 'Run' and 'Data entry' views allow quick access to run status and the most commonly changed variables.

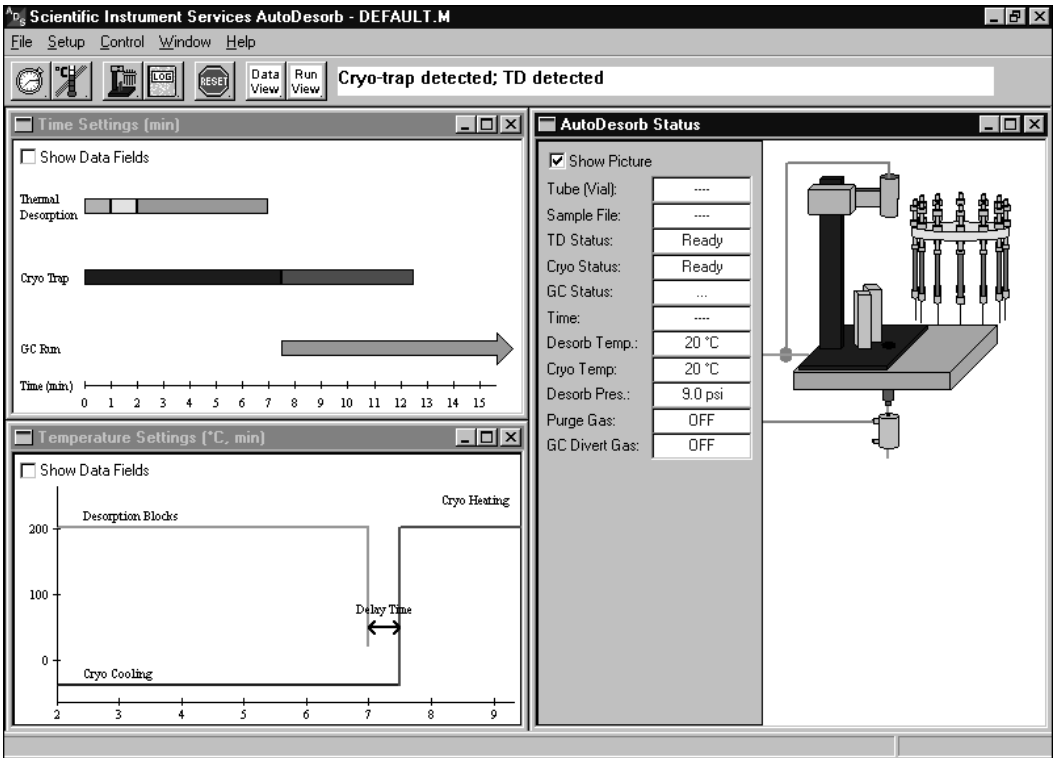


Figure 3 - AutoDesorb system software Run view

Single runs or entire sequences are initiated through the data system, and AutoDesorb method parameters are stored in data system method files. Data for each sample needs to be entered only once in the data system sequence table. The system documents each run in the Sample Log Book, in which any method changes or run errors are recorded. The log can be exported and used for Quality Assurance documentation or included in a lab notebook.

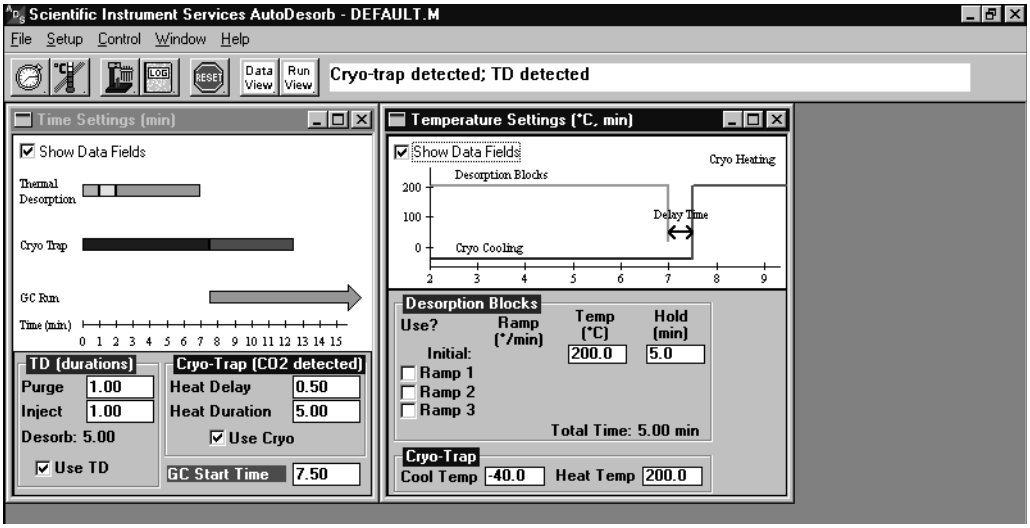


Figure 4 - AutoDesorb System software Data Entry view

The software also includes the ability to set system limits. Variables such as maximum desorption heating temperature and minimum system pressure may be set as global constraints to prevent accidental damage to the system or sample loss.

For more information, see the AutoDesorb Getting Started section of this manual.

Specifications

Power Requirements

115 Volt +/- 10% AC

10 amps

Weight & Dimensions

Desorption unit

Weight: 17 lbs

Dimensions: 6.5" W x 13" D x 17" H

Controller

Weight: 10 lbs

Dimensions: 7" W x 10" H x 14.5" D

Gas Requirements

Carrier Gas pressure: 40-60 psi (100 psi maximum)

Compressed Air pressure: 50-80 psi (100 psi maximum)

Desorption Heater Blocks

Cartridge Heaters (2 ea)

Heater Input: 115 volt, 200 Watts Each (Total 400 Watts)

Control Input: Platinum Resistance Thermometer (PRT) - 100 ohms

Three-Digit Readout for Set Temperature & Actual Temperature

Operational Temperature Range: 20° to 450°C

Temp. Ramp Range: 0° to 100°/min. (Programmable)

Temp. Ramp: >200°/min. (ballistic mode)

Cryo-Trap Accessory

Single Resistive Heating Element (32 ohms)

Heater Input: 18 VAC, 10 Watts

Control Input: Type K Thermocouple

Maximum Heating Temperature: 450°C

Operational Temperature Range: -180° to 450°C

Maximum Ramp Rate: 400°C per minute

Minimum Cooling Temperature:
CO2: -70°C
LN2: -180°C

Computer Requirements

(Computer Not Included)

Pentium Processor or greater Operating System: Windows NT/2000 or 95/98

3 MB available on Hard Disk

Spare RS232 Port Required

One of the following supported versions of ChemStation Software (Not Included)

HP 5973 - Version 1701 & higher

HP 5971/72 - Version 1034C & higher

HP GCD - Version 1074B & higer

HP 6890 - Version G2070AA

HP 5890 - Version G2070AA

Safety Information

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

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

Warning: Important Point

Warning: Electrical Hazard

Warning: Burn Hazard

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

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

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

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

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

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

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

Do not desorb samples above 450^{O} C. Exceeding this temperature may damage heater blocks and surrounding parts.

Do not operate the equipment when moving parts are exposed.

Compressed air is required for the operation of the desorption unit. The compressed air pressure should normally be set to 60 psi. In NO case should the air pressure exceed 100 psi.

The AutoDesorb is normally shipped and set up for 110 Volt AC operation. It is also available in a 220 Volt version. Check the AutoDesorb controller for voltage setting before use.

The AutoDesorb System must be installed exactly as described in the installation manual. If not installed correctly, system damage or personal injury may occur.

For proper system operation, use only desorption tubes and needles manufactured by SIS.

Registration

If you have not already done so, please fill out the enclosed registration card. This information will allow SIS to provide you with software and manual updates and keep you advised of new applications and accessories as they become available. Mail the completed registration form to the address below:

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

Warranty and Service

Warranty

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

Extended Warranty

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

Service and Repair

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

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

Phone: (908) 788-5550

Keep all Boxes and Packaging.

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

OPERATION

Getting Started
System Startup
Creating and Editing AutoDesorb Methods
Running a Single Sample
Running a Sequence
The Sample Log Book
Special Methods

Getting Started

Before you begin using the AutoDesorb system **(ADS)**, make sure that it has been properly installed. This pertains not only to the desorption unit, controller and Cryotrap, but also to the operating software. Installation by a technician trained by SIS is recommended, and certain warranty provisions may be void unless the installation is performed by certified personnel. Installation by an authorized agent of SIS is always accompanied by initial training in the use of the system using customer samples when possible. This is the best way to be sure that your system is correctly installed.

At a minimum, verify that carrier gas, compressed air, electrical power, and if applicable, cryogenic fluid have been correctly connected and supplies are adequate.

Verify that the desorption unit is in place over the GC inlet, and that the centering septum nut is present.

It is also recommended to READ THIS MANUAL THOROUGHLY before using the system.

System Startup

Turn on the power to the controller, start the data system for the instrument configured for thermal desorption analysis, and verify that the SIS AutoDesorb System (ADS) software starts, and that the system initializes. This is accompanied by a muted 'popping' sound from the desorption unit as the pneumatics are tested. Most difficulties, if they occur, will make themselves evident at system start-up. Low compressed air pressure, for instance, will cause the absence of the 'popping' noise, and an error message will appear on the screen. Likewise, if the controller is turned off, the ADS software will report a communication error. Usually the option will be given to correct the problem and re-initialize the controller.

Once the system has started and initialized, a method can be created, or samples may be run using a previously stored method.

Creating and Editing AutoDesorb Methods

Creating an ADS method is a matter of modifying an existing one to suit the current analysis. This is true even for a data system method that has been created on an instrument with which the ADS has never been used. When loading a method into the ADS instrument, the ADS software will look inside the method for a set of parameters to use, and if none are found, a default set will be supplied. When the method is saved or the instrument is shut down, ADS parameters will be saved along with the method.

Data Entry

It is recommended that most setting changes be made from the 'Data Entry' view. This view displays the Time Settings and Temperature Settings windows with the data fields expanded. Activate the Data Entry view by clicking the icon on the toolbar, or selecting **Window/Data Entry view** from the pull-down menus.

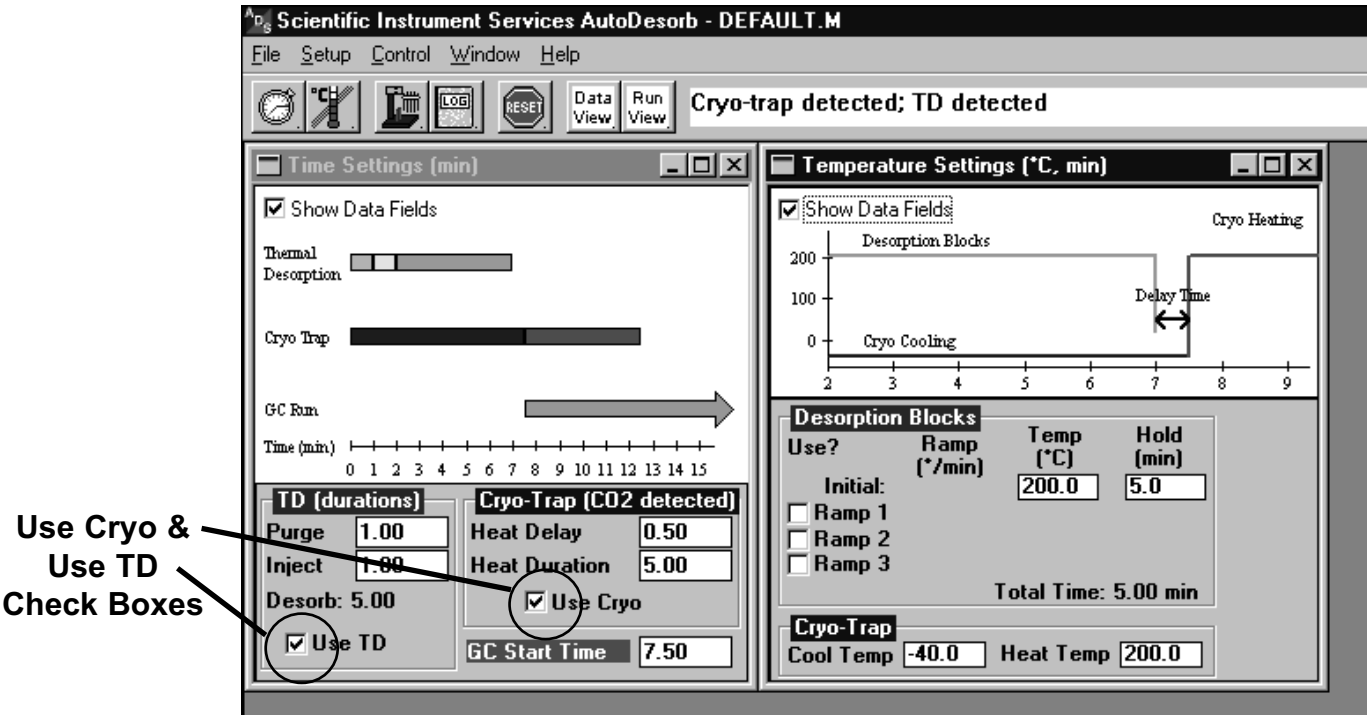


Figure 5 - AutoDesorb Data Entry View

The thermal desorber (TD) and Cryotrap can be independently enabled or disabled from the Data Entry view. An example of a method that would not use the cryotrap is the thermal extraction of high-boiling components from a soil sample (e.g. PCB's or Polynuclear Aromatic Hydrocarbons). In this case, materials that would be focused on the column cryogenically might only interfere with the analysis. Likewise, the Cryotrap may be used independently of the TD for focusing static headspace injections. The TD and Cryotrap may be enabled or disabled for each method by checking the **"Use TD"** or **"Use Cryo"** boxes in the Data Entry view.

Time Settings Window

The main parameters for the ADS are the Purge, Inject, and Desorb times and the temperature settings for the desorption heater and cryotrap. Other parameters include the GC start time and Cryotrap heat delay and duration.

The Time Settings and Temperature Settings windows provide data field display a graphical representation of the desorption process. Adjustable parameters in these windows are explained briefly below:

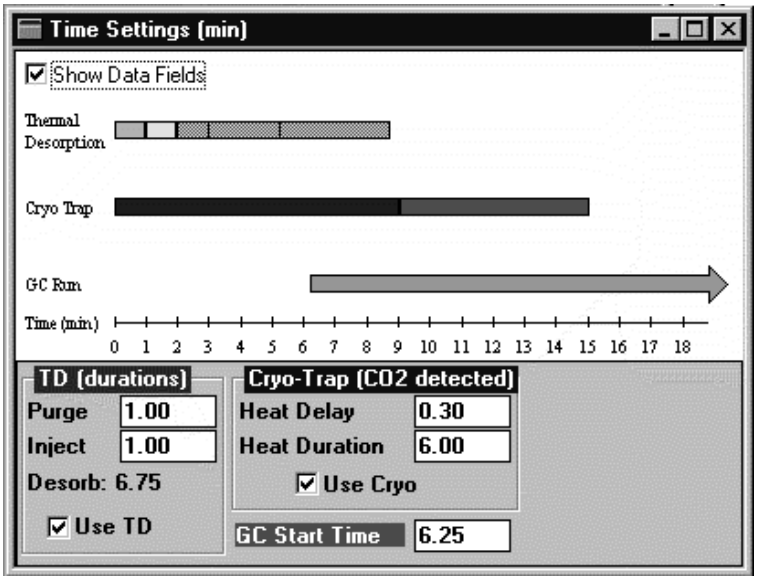


Figure 6 - Time Settings Window

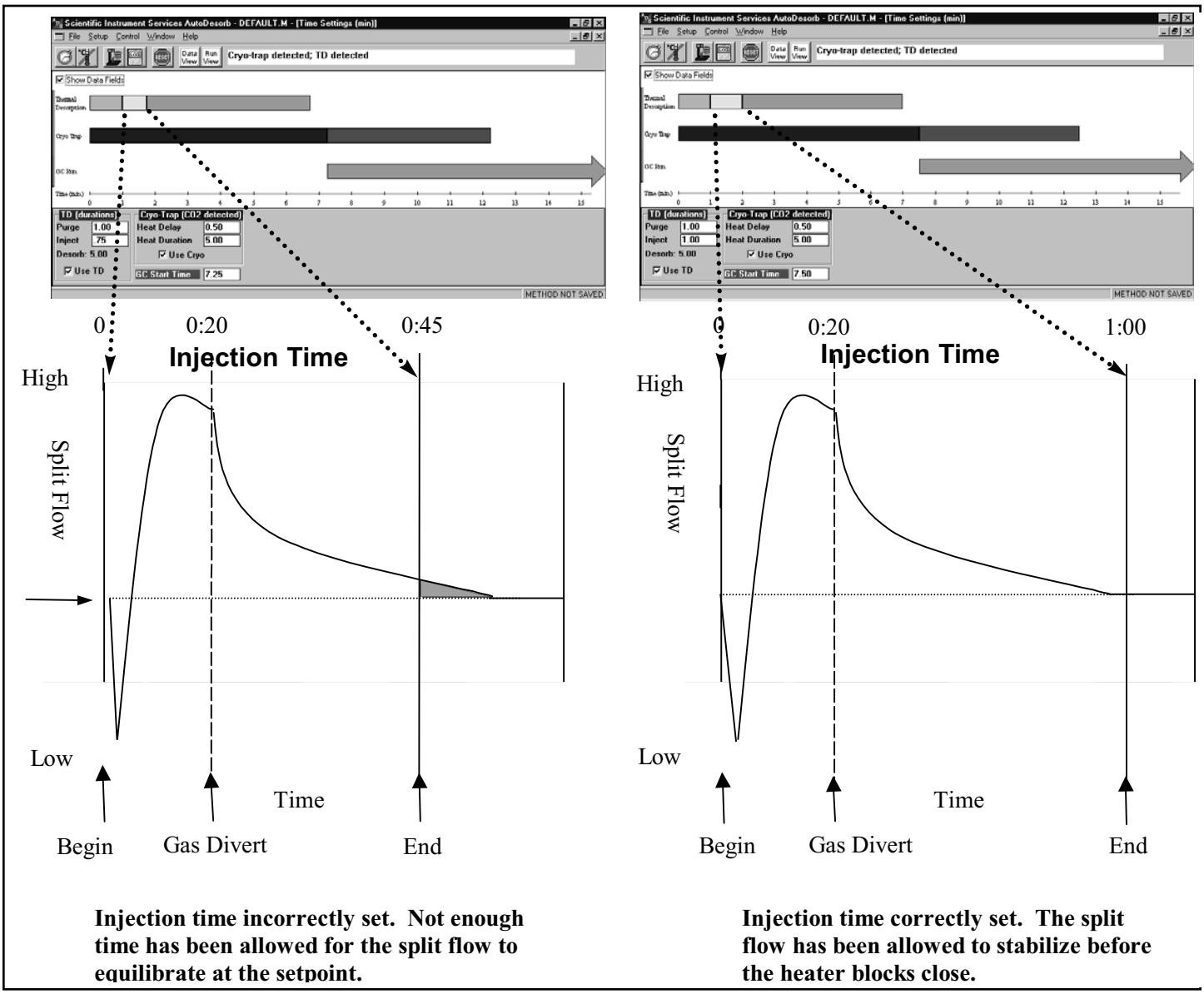
Purge:

This is the time allowed for gas to purge across the desorption tube before the needle is lowered into the GC inlet. This allows for a volume of carrier gas to remove oxygen, excess water, or other unwanted volatile materials that may be resident in the tube. The default setting for the purge gas time is one minute. Note that the retention of some analytes may be affected by this purge, and the use of a more appropriate trapping resin may be indicated if sample is lost due to this small volume. Purge gas flow is regulated by a mass flow controller mounted on the front of the desorption unit. The flow should be adjusted to provide adequate carrier to sustain Total Flow for the highest split to be used. The setting is best made by adjusting the flow controller during the Inject period, after the carrier has been diverted (see below).

Inject:

The inject period begins when the needle is lowered into the GC inlet. A drop in inlet pressure is normal as the septum is punctured; however, re-pressurization should follow rapidly as the desorption purge flow and normal GC carrier are both directed into the inlet. The dual flow lasts for a preset time after which the carrier gas regulated by the GC's pressure control feature is diverted to pass through the desorption unit. The default value of 20 seconds (0.3 minutes) for the dual flow period is changeable from the **Setup/System Limits** menu, using the **GC Gas Divert Time** setting. In this way, the pressure and flow control are handled by the GC for maximum consistency. Although an inject time setting of one minute is supplied in the default method, it should be set in conjunction with the Purge Gas flow to allow the injection port pressure to equilibrate and the proper split flow to be reached before the time expires. At least once for each method, it is advisable to check the split flow after the carrier gas has diverted, and adjust the time if necessary (See Figure 7). At the end of the Inject period, a pressure reading from the desorption unit is compared to the 'Minimum Pressure to Run' setpoint in the System Limits screen. If the minimum pressure is not met a leak is assumed, the tube is returned to the carousel, and the sequence advanced to the next sample.

SPLITLESS INJECTION IS NOT RECOMMENDED. The low flow rate associated with most splitless injections does not provide an efficient transfer of material from the desorption tube to the column. Most chromatographs will yield inconsistent results with splitless injection due to difficulty regulating the inlet flow through the desorption unit in this mode.



Injection time incorrectly set. Not enough time has been allowed for the split flow to equilibrate at the setpoint.

Injection time correctly set. The split flow has been allowed to stabilize before the heater blocks close.

Figure 7 - Setting the Inject Time Parameter

Desorb:

When the Inject time expires, the heater blocks close around the desorption tube and remain closed for the time specified here. The Desorb duration cannot be edited from the Time Settings window. It is affected by temperature ramp rates and hold times specified in the Temperature Settings window, and is calculated automatically. The value supplied in the default method is five minutes.

Cryo Heat Delay:

This provides a short equilibration period after the desorption needle is removed from the inlet. During this time, the Cryotrap is held in cooling mode to prevent pressure fluctuations from affecting chromatography. The default value for the cryo heat delay is thirty seconds. At the end of the delay, the Cryotrap is ballistically heated to release the focused analytes instantaneously, much like a liquid injection.

Cryo Heat Duration:

This setting controls the length of time that the Cryotrap is heated. Most volatile materials should be released in the first few seconds of heating. Keeping the Cryotrap heater on for long durations may shorten its life. The default setting of five minutes is recommended for most analyses.

GC Start Time:

The time specified here is relative to the beginning of the Purge time. The GC may be started at any time beginning with the activation of the desorption Purge. Normally the GC Start time is set to coincide with the start of the Cryotrap heating by using the sum of the Purge, Inject, Desorb, and Heat Delay times. This provides chromatographic results that most closely match those obtained by liquid injection. Starting the GC early may be useful in method development (particularly when choosing a Cryotrap cooling temperature) since compounds breaking through a Cryotrap that is too warm may go unnoticed unless the data system has been started. Early GC start times may also be used to take advantage of functions such as inlet pressure programming that are controlled from the GC time base.

Temperature Settings Window

Desorption Temperatures and Ramp Rates:

For isothermal desorption (recommended for most adsorbent trap methods) the only entries necessary are the initial temperature and desorption duration. For ramped desorption (recommended for Direct Thermal Extraction) one or more segments of the temperature program are selected for use by checking the box next to the segment label (Ramp 1, Ramp 2, etc.). When selected, heating rate (°C/minute), target temperature (°C) and hold time (minutes) for each segment may be entered. The maximum controlled heating rate is 100 °C/minute. A heating rate value of 0°C will result in the ballistic heating of the sample. Total desorption time is calculated automatically.

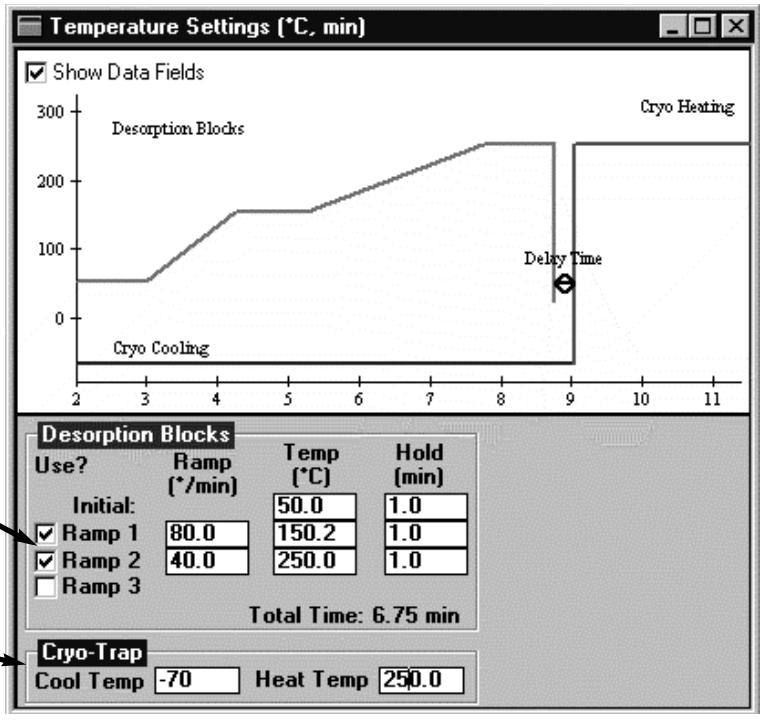


Figure 8 - Temperature Settings Window

Cryotrap Temperatures:

Cryotrap cooling temperature may be set between ambient temperature and -70°C for liquid CO_2 use or between ambient and -180 °C for liquid nitrogen. Cooling temperature should be set below the freezing point of the most volatile compound being analyzed. When using liquid CO_2, trapping efficiency may be enhanced by using thicker film guard columns. When using liquid nitrogen, trapping temperatures often go below the glass transition point of the polysiloxane bonded phases (~ -60 to -70 °C). Therefore the use of a bonded phase offers no advantage in terms of trapping efficiency, and may actually be detrimental to the column due to the rapid temperature changes. Deactivated fused silica guard columns are recommended for use with liquid nitrogen. For either liquid nitrogen or liquid CO_2 , the use of a wide bore capillary guard column will increase the trapping capacity and minimize the chance of an ice plug forming in the trap. The cryotrap heating temperature should be set slightly higher than the GC inlet. Keeping each successive stage of the chromatographic system hotter than the last helps to keep the system clean and prevent carryover.

Saving Settings

When modifying an ADS method, it is important to remember to save the changed settings by selecting File/Save Settings to This Method from the menu bar or by saving the entire method from the data system. If the setting changes are not saved, the prior settings will be restored the next time the method is loaded. As a reminder, the text "METHOD NOT SAVED" appears in the lower right-hand corner of the ADS software screen whenever a setting change is made. Setting changes can be made at any time, even in the middle of a run. Although not recommended for normal operation, this flexibility can assist in method development. Any changes that are made during a run are recorded in the log book; however, changes must be saved if successive runs in a sequence are to use the new settings.

Exporting Settings

ADS method changes can be exported to a different data system method by selecting File/Export Settings from the AutoDesorb menu bar. The data system method file must exist to take advantage of this feature. The ability to export changes can save time when evaluating the effect of one or two desorption system parameters on an analysis. For example, you might want to evaluate the effect of different desorption temperatures on the recovery of a peak. In that case you would create the basic method in your data system (e.g. a ChemStation method named BASIC.M) and then save it with different names (BASIC1.M,BASIC2.M, etc.). Next, make a single change to the desorption temperature in the ADS software, and export the settings to BASIC1.M. Change the same parameter again, and then export to BASIC2.M, and so on. This feature allows you to change ADS parameters easily without having to load each ChemStation method. By running a sequence with a number of desorption tubes containing the same sample, but with different ADS settings, desorption parameters can be optimized while the instrument runs unattended.

Running a Single Sample

Once the data system and ADS parameters have been set, a single sample may be run from the data system in the same way a single sample would be run using a liquid autosampler.

In ChemStation, for instance, the following steps are used:

1. From the Instrument Control or Run Control window, select Method/Run to activate the Start Run window.
2. Edit sample information, using the ADS carousel position where 'Vial' is indicated.
3. Click the Run Method button in the Start Run window.

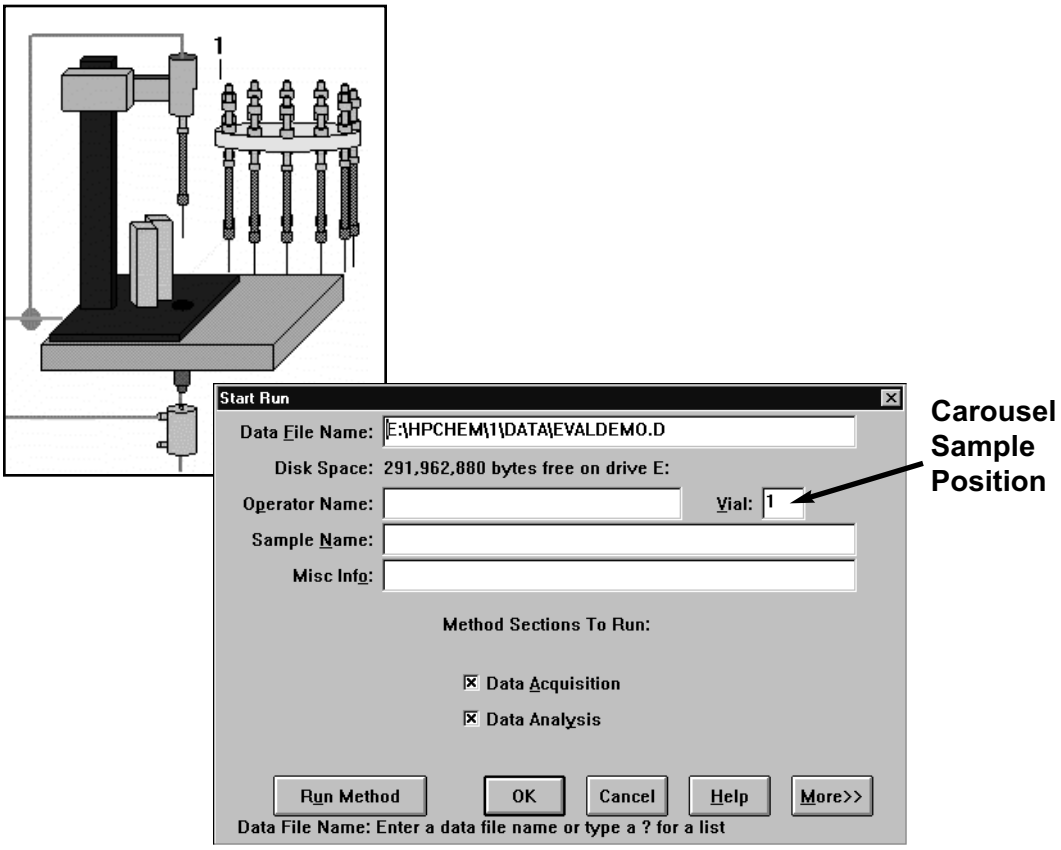


Figure 9 - Agilent (HP) Sample Run Screen

The entire data system method, including thermal desorption settings, will be executed when the GC becomes ready.

If for any reason the sample must be aborted or stopped during the run, do so by clicking the Reset icon in the ADS software. The ADS software contains error handling protocols that will assure that the GC oven temperature program is run if the desorption blocks have closed. Stopping the run from ChemStation may result in desorbed material remaining on the column into the next run.

Running a Sequence

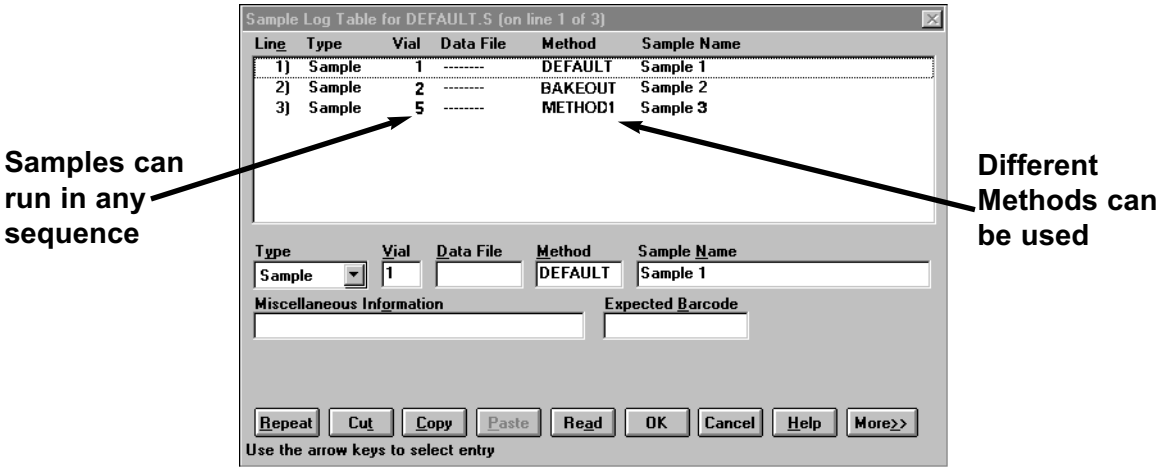


Figure 10 - Agilent (HP) Sequence analysis Screen

The ADS software was designed to interoperate with your data system's sequence files. As is the case with single samples, the same procedure used when running a liquid autosampler will work with the AutoDesorb.

Again using ChemStation as an example, perform the following steps:

1. From the Top window, select Sequence/Edit Sample Log Table from the menu bar.
2. Enter all the sample information as you would for liquid samples, including vial numbers (carousel positions), data file name, method and comments.
3. Click OK and return to the Top window.
4. Select Sequence/Save from the menu bar, and name the sequence file.
5. Select Sequence/Run from the menu bar.

The sequence will then proceed as normal.

NOTE: Samples need not be run in numerical order and may even be run multiple times or with different methods.

Tubes may be loaded and unloaded during the sequence whenever the door on the desorption unit is unlocked. Sequences may also have as many lines as necessary and are not restricted to the number of tube positions on the carousel. Each Vial Number entry, however, must correspond to a position on the carousel, and values must therefore be between 1 and 12.

If the sample or sequence must be stopped prematurely, do so by clicking the Reset icon on the ADS software. Depending on the desorption system state at the time the controller is reset, the choice may be given to retry or skip the sample. Check the sample log book to see whether the tube may be re-analyzed or if the sample has been compromised by going through an incomplete desorption cycle. If the entire sequence is to be aborted, that should be done through the data system before responding to the Skip/Retry request from the ADS software.

The Sample Log Book

The ADS software has extensive error and status logging functions. The Sample Log Book keeps track of all errors and method changes that occur during a run or sequence. It is an excellent tool to use for diagnosing problems, verifying that sequences have run completely, and tracking method changes. The Log Book can be accessed by clicking on the Log Book icon on the toolbar or by selecting Window/Log Book from the pull-down menus. An example of the Log Book screen is shown below in Figure 10.

An entry in the Log Book is created every time the data system begins a run. The initial entry consists of one line of text and contains the Vial (carousel position) number, Time, Date, Datafile name, Method name and the names of active instruments in the method being used (e.g., Cryotrap or TD). Most often, only one more entry is made for each sample when the data acquisition is complete, and it contains the time and a message confirming the successful completion of the sample. Entries for different samples are separated by a solid line. When an error or a method change occurs, the event is logged with the time, the error or change, and sometimes extra information about the sample. The first entry in the example in Figure 11 illustrates a successful run, and the second shows that the controller was reset from the ADS software during a run. Note that the last instrument states are recorded, and that the program has indicated that the controller was reset before desorption had occurred, and that the sample may be re-analyzed.

Sample Log Book data is stored in a file named **Default.log** in the SIS\AutoDesorb directory of the computer that runs the data system. The Log Book may be opened in Notepad and saved as an ASCII file, or the contents may be selected and copied to the Windows clipboard for insertion into other applications. Periodically, the Sample Log Book should be archived (opened in Notepad and saved with a different name) and cleared. Information about other .LOG files and their use can be found in the Software manual. When running samples, Default.log should be selected in the Log Book window.

NOTE: The Log Book is not designed to be a text reader. Do not use the log book to open ASCII files or files with a .LOG extension that have not been generated by the ADS software.

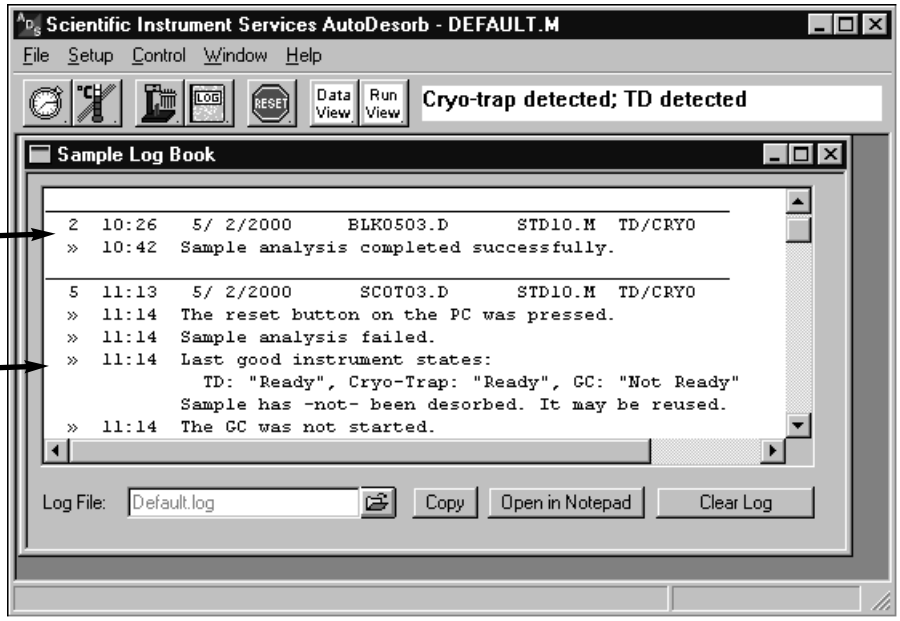


Figure 11 - Sample Log Book

Special Methods

Gas chromatographs that are used often for liquid injections may harbor contamination in the injection port that may be seen in a thermal desorption blank but may not be evident with a liquid injection. This can be due to the turbulent flow of preheated helium entering the GC inlet during thermal desorption. The usual assumption is that the desorption unit has been contaminated; however, this is hardly ever the case, especially with a new unit. If the first blank (and sometimes subsequent ones) display a high baseline with many peaks, then it is probably time to run a bakeout method.

Bakeout Method - Recommend that the Bakeout Method be run before any samples are analyzed or when a sample is overloaded

Create a bakeout method with parameters similar to these:

Oven temperature:	At or near the column maximum, isothermal
Oven Hold Time:	10 minutes
Inlet Temperature:	Column maximum up to 350°C
Inlet Mode:	Split (=100:1)
Detector or Transfer line:	At or near the column maximum

Thermal Desorber Settings:

Cryotrap:	Disabled
Heater Blocks:	300 to 350 °C, isothermal
Purge Time:	1 minute, purge flow controller wide open
*Inject Time:	Less than GC Gas Divert Time specified in System Limits
Desorb Time:	20 minutes
GC Start Time:	15 minutes

Note - By setting the Inject time shorter than the GC Gas Divert Time, the carrier diverter valves are not activated, allowing flow to enter the injector from both the desorption unit and the regular GC carrier inlet for the duration of the desorption process. The excess flow helps remove contaminants from the injector through the split vent. The default setting for GC Gas Divert Time is 20 seconds (0.33 minutes). A typical Inject time setting for a bakeout method is 0.2 minutes.

Save the data system method with a convenient name such as BAKEOUT.M. Run the method either by itself or as part of a sequence whenever inlet contamination is suspected, or as part of a regular inlet maintenance program. Remember to change the bakeout method parameters to include appropriate oven temperatures for your GC column.

Figure 12 illustrates the carrier flow path during the Purge and Inject times. In Figure 12A carrier gas (indicated by the dotted line) is delivered to the column from the Electronic Pressure Control (EPC) system of the GC, and purge gas is directed through the thermal desorption unit by a separate route. Figure 12B illustrates the flow immediately after the needle has penetrated the septum (two separate flows are entering the inlet). This is the state that is maintained during the system bakeout. Figure 12C shows the flow path of the carrier after the diverter valves have been activated. Note that the EPC now supplies the inlet through the thermal desorption system. Locations where flow has been valved off are indicated by a symbol. ⊗

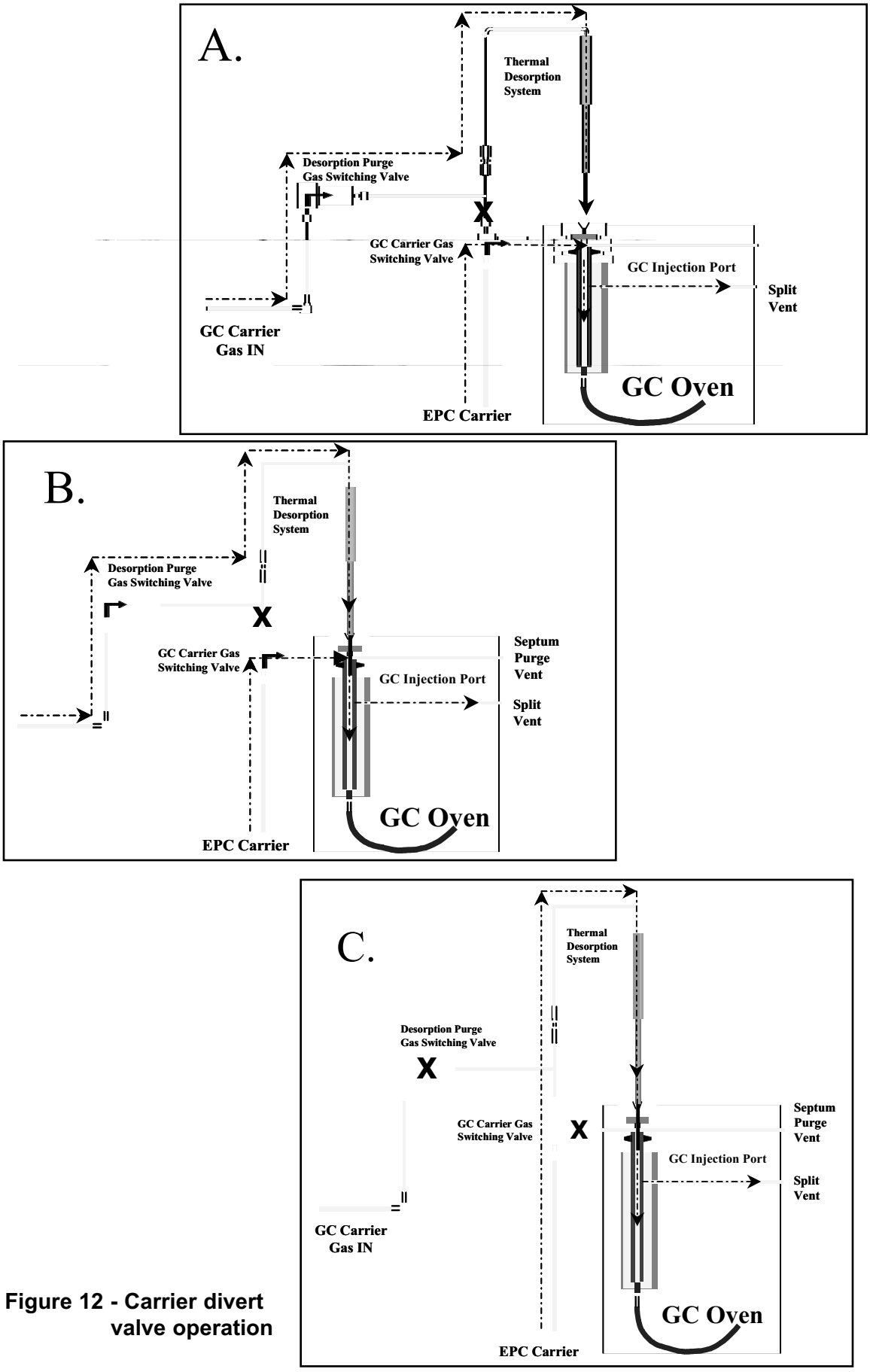


Figure 12 - Carrier divert valve operation

System Care and Maintenance

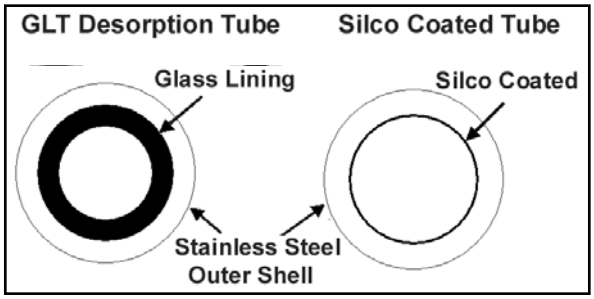
Desorption Tubes, Caps, Needles and Seals
Connecting Tubes
Cleaning, Packing and Conditioning
Desorption Tubes
AutoDesorb System Supplies and Spare Parts

Desorption Tubes, Caps, Needles, and Seals

Figure 13 GLT desorption tube

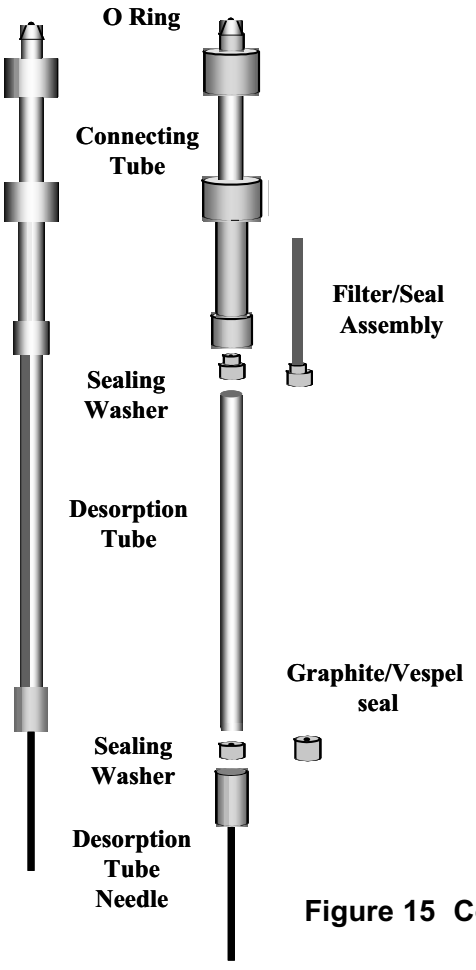
Three styles of desorption tubes are available for the AutoDesorb System.

Glass Lined Stainless Steel	I.D. 3.0 mm
Stainless Steel	I.D. 4.0 mm
SilcoSteel® deactivated Stainless Steel	I.D. 4.0 mm



The glass lined stainless steel desorption tubes are available with an inside diameter of 3.0 mm. These are the most durable and inert of the desorption tubes and are recommended for most applications. These tubes have a strong stainless steel outer shell with an inert glass lining that can be further deactivated by silylating if so desired.

Figure 14 - Cross section of desorption tubes



Where larger samples are required, desorption tubes with an inside diameter of 4.0 mm are available in stainless steel. The larger I.D. permits almost twice the amount of sample or adsorbent resin to be contained in the tube. Where a more inert environment is required the stainless steel tubes are available with SilcoSteel® deactivation.

All of the desorption tubes have an outside diameter of ¼ inch (0.250") and are 4.0" long. The threads at the ends of the desorption tubes provide compression against a Vespel® seal with minimum torque. It is important to remember that the flat end of the tube, not the threads, forms the seal. The threads are specially pitched to allow pieces to be joined and heated without seizing. To avoid damage to the threads and possible binding, carefully inspect them on both the desorption tube and the cap, needle or connecting tube prior to assembly. Remove any foreign material before joining the pieces, and NEVER use Teflon tape or other thread sealing compound on the desorption tubes. For added protection against binding, especially in high-temperature applications (e.g. routine desorption temperatures of over 300°C), Neolube lubricant may be applied to the threads. Neolube is a suspension of highly pure graphite in isopropanol, and is the only lubricant recommended for use with the desorption tubes. Hydrocarbon oils and greases are to be avoided especially.

Figure 15 Complete desorption tube, needle and connecting tube

Desorption Tube Caps and Seals

Conditioned desorption tubes should remain capped until samples are taken, and the caps should be replaced after sampling. Remove the caps only when ready to place the tubes on the carousel for analysis. Desorption tube caps should contain a Teflon seal to prevent contamination or diffusive loss. The Teflon seal is used whenever an unheated leak-tight connection to a desorption tube is needed (e.g., spiking or sampling apparatus, tube caps, etc.). Tighten the connection with your fingers until you feel the seals compress to achieve an effective barrier. When a leak-tight connection is to be heated (e.g., when connecting to a desorption needle or when 'blanking off' a tube in the conditioning oven), a Vespel® seal must be used. Vespel® does not compress like Teflon does, however the sealing system has been designed to give finger-tight connections that are leak-free. DO NOT USE TOOLS TO TIGHTEN CONNECTIONS.

Desorption Tube Needles

Needles for the AutoDesorb system are composed of two pieces: a threaded cap, and a needle/hub assembly. The two parts are held together with a circlip. The cap turns freely around the hub, compressing a single graphite/Vespel® seal between the hub and desorption tube to form a leak-tight connection. Tools are not required and should never be used to tighten needles onto desorption tubes. Needles should not be disassembled. They may be routinely cleaned in the SIS conditioning oven using a needle handle or manifold supplied with the oven. Gross contamination may be removed by rinsing or sonicating in an appropriate solvent, followed by conditioning. The needles themselves are of a side-port design to prevent coring. Two styles of needles are available, a stainless steel needle and a and a SilcoSteel® deactivated style needle. Discard needles if they become clogged (indicated by high backpressure within the desorption unit, but insufficient pressure in the GC inlet), severely bent, or if there is any evidence of damage to the threads.

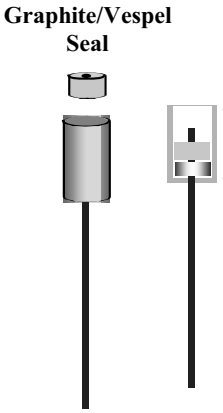


Figure 16 AutoDesorb needle and seal

Connecting Tubes

The AutoDesorb system uses stainless steel connecting tubes to hold and manipulate the desorption tubes. At the top, the connecting tubes have a ball-and-spring seal to retard backward flows of gas through the desorption unit, and prevent diffusion of volatiles into and out of the desorption tube. A Viton o-ring forms a positive seal with the carrier flow path. To prevent contamination of the carrier gas, the o-ring is separated from the carrier flow path by an unswept volume and is sufficiently removed from the heated portion of the desorption tube.

Connecting Tube Filter and Seal Assembly

A unique feature of the connecting tubes is the integrated seal/filter assembly. The Vespel piece that forms the seal between the desorption tube and connecting tube is attached to a filter containing a carbon molecular sieve. This assembly serves as a final trap to remove any organic impurities from the carrier gas before it enters the desorption tube. It also prevents volatile material or particulates from being carried backward into the desorption unit and contaminating the instrument. The filter and seal are pressed into place, and are removable with a special tool supplied with your AutoDesorb system. To use the tool, grip it by the knurled knob and insert the threaded end into the connecting tube as shown in Figure 17. Turn the knob clockwise until it seats firmly in the connecting tube, then give an additional ¼ to ½ turn to remove the seal from its seat.

DO NOT PULL BACK ON THE REMOVAL TOOL until the seal is free. Doing so may damage the seal and make it difficult to remove. Turn the removal tool clockwise to thread the stem into the seal and keep turning until the seal is removed.

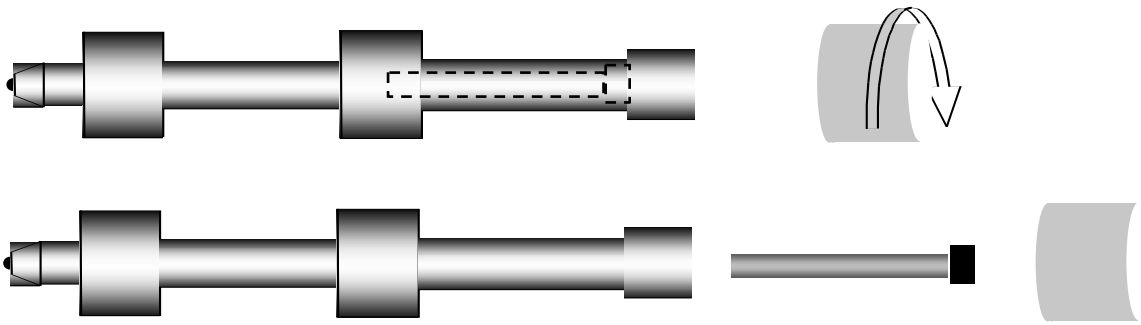


Figure 17 Removing the filter/seal assembly

Remove the seal from the tool by unscrewing the threaded end. Once removed, the filters can be replaced or reconditioned. The SIS conditioning oven is used to recondition the filters. The filters are placed (seal-end-in) into a conditioning oven handle or manifold and are held in place with 4.0 mm I.D. desorption tubes (Figures 18 & 19). Flow is supplied through the handle or manifold, and the whole assembly is lowered into the conditioning oven. Filters should be conditioned at 350 °C for a minimum of two hours with at least 10 ml/minute clean gas flowing through each filter. Allow the filters to cool and, handling them as little as possible, replace them into the connecting tubes or store them in a airtight glass vial.

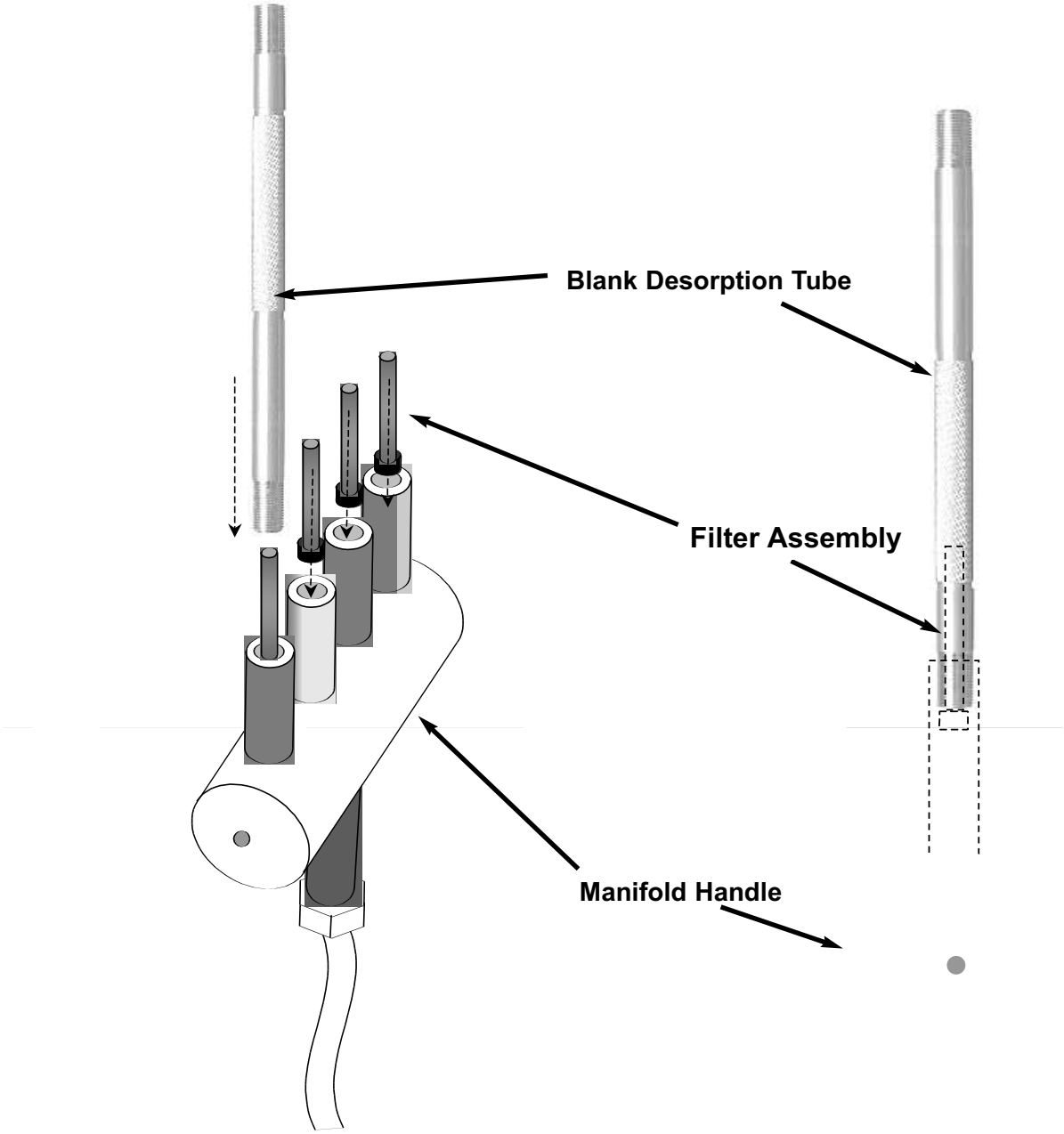


Figure 18 Filters inserted in conditioning oven manifold

Figure 19 Filters held in place by 4.0 mm ID desorption tubes

Filters must be pressed into connecting tubes by attaching a desorption tube and firmly threading it into the connecting tube as far as it will go. Filters are fully seated when no thread from the desorption tube is visible past the end of the connecting tube (Figure 20).

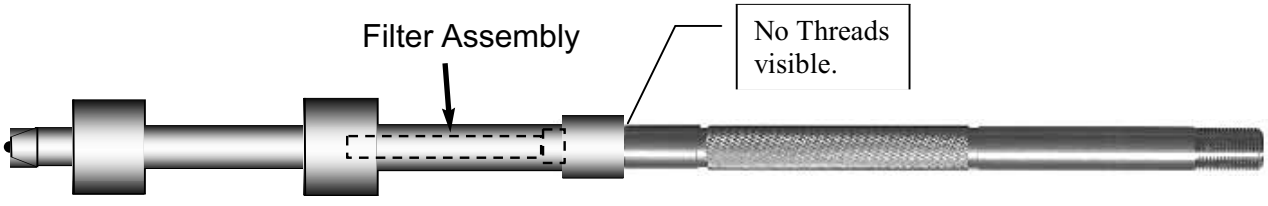


Figure 20 - Filter and Seal properly seated

Cleaning, Packing and Conditioning Desorption Tubes

The preparation of Desorption Tubes for use with the AutoDesorb is usually a multi-step process including:

1. Washing (using water and a detergent such as Alconox)
2. Silylating (optional)
3. Rinsing
4. Drying
5. Packing (optional)
6. Conditioning (at elevated temperatures with flow)
7. Sealing and Storage

Cleaning Desorption Tubes

Desorption tubes may be cleaned using water and a laboratory-grade detergent. An ultrasonic cleaner or a small stainless steel brush can aid in cleaning. Do not use a stainless brush on Silcosteel treated desorption tubes, or the integrity of the deactivation layer may be compromised. Rinse the tubes thoroughly with clean water, and dry in a laboratory or GC oven. If solvents are required for cleaning, be sure to remove all residues before using the tubes for analysis.

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 of the prepared tubes.

Packing Desorption Tubes

Normally samples are collected on desorption tubes packed with a 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 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 http://www.sisweb.com/index/referenc/resins.htm .

Thermal desorption sampling tubes are usually packed with approximately 20-200 mgs of adsorbent. The amount of adsorbent used will depend on the specific analysis requirements. The ends of the tubes are plugged with approximately 1 cm of silanized glass wool on each end to hold the adsorbent in place.

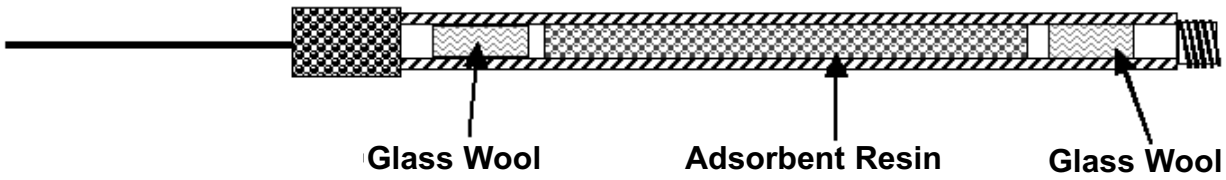


Figure 21 Thermal Desorption Tube for Purge &Trap Analysis

Alternatively, tubes can be packed with the actual materials to be analyzed for controlled direct thermal analysis of residual components in samples such as packaging and construction materials, fibers, paint chips, etc. This technique is called Direct Thermal Extraction, and is a preferred method of analysis of many solid-matrix materials, because it involves very little sample preparation and because all of the analytes extracted from the sample are delivered to the GC injection port - nothing is lost.

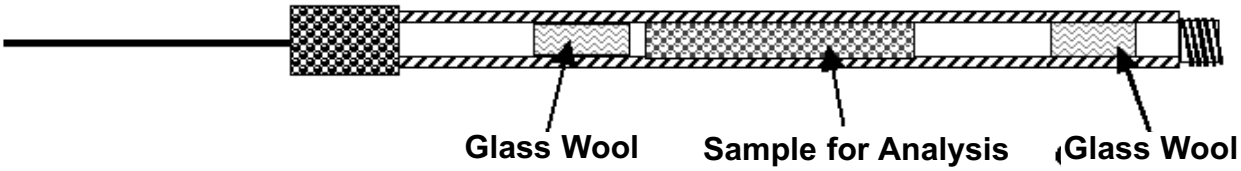


Figure 22 Desorption Tube for Direct Thermal Extraction

Conditioning Thermal Desorption Tubes

In order to prepare the adsorbent-packed desorption tubes (or empty desorption tubes) for thermal desorption, they must first be flow-conditioned at elevated temperatures to remove all foreign materials. The following procedure or a suitable version of this method should be used to condition the tubes. The maximum temperature will be determined by the properties of the adsorbent material used in the tubes.

The desorption tubes containing the adsorbent are attached to the thermal desorption conditioning system. The tubes are flow-conditioned at room temperature for several minutes to remove any oxygen from the interior of the tubes. This step is especially important when using resins such as Tenax (which decomposes easily at elevated temperatures in the presence of oxygen). The tubes are normally purged with nitrogen or helium at a flow rate of 10 to 100 ml/min during all stages of the conditioning process. After the initial purge at room temperature, the tubes are heated from ambient temperature to 300°C at a rate of 4°/min while purging with carrier gas. The tubes are held at the upper temperature limit for not less than four hours under continuous flow. Packed tubes may also be heated ballistically by placing them in a pre-heated conditioning oven provided that oxygen has been purged away at room temperature. Afterward the tubes are removed from the conditioning oven and placed in the cooling rack on the rear of the oven and allowed to cool for 5 minutes. When cool, the tubes are immediately capped on both ends. The stainless steel caps with liners should also be conditioned by baking in a laboratory or GC oven. Tubes prepared in this manner exhibit excellent adsorptive capacity and contain no organic background when analyzed by GC/MS.

Desorption Tube Conditioning Oven

A tube conditioning system is available from S.I.S. Two models of the oven are available, one that will condition 6 tubes or needles simultaneously and a second model that has a 24-tube capacity. The self contained system includes 6 rotameters, handles for desorption tubes, needle handles and a programmable digital temperature controller. The controller permits the temperature program described above to proceed unattended. A procedure of up to 6 steps with various ramp cycles and hold times can be programmed.

The desorption tube conditioning oven is also used to clean the desorption tube needles. The needle handles are included with each unit. The needles are normally conditioned at 300°C with gas flow. For a complete detailed description of the conditioning oven and cleaning of desorption tubes see the Conditioning Oven Manual.

Figure 23 Thermal desorption tube conditioning oven

AutoDesorb System Supplies and Spare Parts

Part #	Description
786005	3.0 mm ID GLT desorption tube
786002	4.0 mm ID SilcoSteel deactivated desorption tube
786003	4.0 mm ID Stainless desorption tube
781006	Stainless cap for desorption tube
781007	Stainless cap for desorption tube with 0.040" hole
781004	Teflon seals for desorption tube caps
786035	AutoDesorb needle
786135	AutoDesorb needle, SilcoSteel deactivated
786018	Graphitized Vespel seal
786009	AutoDesorb connecting tube
786017	Connecting tube filter/seal
786910	Filter/seal extractor tool
V107	O-ring for connecting tube
789003	Neolube, 2oz.
979302	Tenax TA 10g
979402	Tenax GR 10g
781122	Funnel for loading desorption tubes
781099	Spatula for loading DTE samples
783092	Direct injection adapter for loading liquids, standards, etc.
786030	Accessory Supply Kit

We recommend that at least 1 kit (# 786030) be purchased with each desorption system. Each kit contains the following items:

Accessory Supply Kit Components (#786030)		
Part #	**Description**	**Quantity**
786005	GLT Desorption Tubes, 3 mm, empty	12
786035	AutoDesorb Needle	12
786018	Graphitized Vespel® seal	12

Troubleshooting

General
Flow and Mechanical
ADS Controller
Software
Data

General

This chapter is concerned with the diagnostic procedures to follow when problems occur with the AutoDesorb system. The following areas are addressed:

Flow & Mechanical System
ADS controller
Software
Data

Most mechanical and software problems can be easily diagnosed by resetting the controller through the software. The ADS software runs a set of diagnostic tests when the controller is reset, and will post messages to alert the user of any fault conditions. When confronted with a problem, reset the controller first. The Log Book can also be used to help trace the cause of errors and when they originate.

This manual section is intended to help users diagnose and correct routine errors and faults that may occur. It is not intended as a service manual or guide. Unauthorized service of the AutoDesorb system will void the warranty. PROTECT YOUR INVESTMENT-CALL SIS TECHNICAL SUPPORT AT (908) 788-5550 OR YOUR AUTHORIZED SIS REPRESENTATIVE IF YOU ENCOUNTER A PROBLEM THAT MAY NEED SERVICE.

Flow and Mechanical

High Pressure

Problem - Pressure reading in the Status window is higher than normal when desorbing a sample.

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 desorption tube needle. Compare the AutoDesorb pressure displayed in the Status window with the injection port pressure displayed on the GC panel. Normally these two readings should be within 1 or 2 pounds of each other. If the desorption pressure is much higher, then there is an obstruction in the desorption system. If the desorption pressure is much lower, then there is a leak in the desorption system. Abort the sample by clicking the reset button in the AutoDesorb software. The pressure reading should drop quickly. If it does not, then the needle is probably clogged. Remove the needle to confirm the clog. With use, the needle can become clogged with septum particles or residue from a sample. The needles can be cleaned by rinsing and/or sonicating with solvent. Needles should be conditioned in the conditioning oven before use.

Solution (b)

See Ice Plugs (below).

Low Pressure

Problem - Zero reading or low pressure reading in the AutoDesorb Status window when desorbing. The inlet shuts down due to a low flow condition.

Possible Causes

(a) Leaking seal at the connection between the needle and desorption tube
(b) Leaking seal at the connection between top of desorption tube and connecting tube
(c) Bad GC septum
(d) No carrier gas is being supplied to the desorption system
(e) Broken GC column

Solutions

The most likely cause of low or zero pressure readings is a leak in the desorption tube. The most common 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 removing the desorption tube and needle from the injection port and sliding an old GC septum over the needle. When the purge gas turns on, the pressure should rise fairly quickly and equilibrate at the carrier gas pressure being used. If the pressure does not rise or rises too slowly there is a leak in the system.

If the problem is not located in the desorption system, then it may be in the GC injection port. Check the GC 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.

No Gas Flow

Problem - Obstruction in the rotameter

Cause

Dirt or contamination has entered the rotameter glass tube.

Solution

Remove the rotameter glass tube and clean it. Call SIS Tech Support for more information.

Bent Needles

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 samples in the carousel, visually inspect them to be sure the needle is hanging straight from the desorption tube and is not bent. If the needle is misaligned, gently straighten to the approximate center position. Also check that the septum nut adapter is in place and that the desorption unit is sitting squarely over the septum nut.

No Motion in Desorption Unit

Problem - Desorption unit will not home, pick up tubes, etc.

Cause

Air pressure too low

Solution

Reset the controller. If the compressed air pressure is not sufficient, initialization will fail and a message will appear in the main ADS window. Correct the problem and reinitialize the controller.

Improper Injection Speed

Problem - Injection or Uninjection too slow or too fast

Cause

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 60-80 psi of compressed air for all the air solenoids and columns in the system. Check the air pressure to the desorption unit 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 50 psi) will cause the system to operate more slowly.

Ice Plugs

Problem - Ice plugs in GC column when cryofocusing.

Cause

Water content of sample being analyzed is too high.

Solution

1. Use smaller samples
2. Install a megabore deactivated fused silica guard column in front of the regular GC column to minimize the chance of ice plugs.
3. Use a larger diameter capillary column or a packed GC column.
4. Trap volatiles purged from the samples onto Tenax or other adsorbent resin that has a low affinity for water, and then desorb the traps into the GC.

Heater Blocks Do Not Close

Problem - Desorption system heater blocks will not close around the sample. Error message indicates that the sample was not desorbed due to low pressure in the desorption unit.

Cause

(a) Desorption system head pressure is less than the value set in System Limits window 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
(6) the settings in the System Limits window is not appropriate for the analysis

(b) Mechanical problem with the heater blocks prevents them from closing.

Solutions

Check the items listed above for (a) and correct if necessary. When the cause of this problem is a suspected leak, an error message from the ADS software indicates that the sample was not desorbed due to low pressure in the desorption unit. For item (6), make sure the setting in the System Limits window is at or below the initial head pressure for your GC method. Many short or large-bore columns require head pressures less than 2.5 psi at low initial column temperatures. If you have recently changed columns or initial oven temperature, the setpoint in the ADS software may be too low.

If the desorption unit pressure is adequate and the blocks still do not close, a mechanical problem with the desorption blocks may exist. Call SIS Technical Support.

ADS Controller

Heater Blocks Do Not Heat

Problem - Desorption system heater blocks will not heat

Causes

(a) Heater fuse in the controller is blown
(b) Overheat sensor switch in the desorption unit has been activated.
(c) Heater cartridge on the heater block is defective
(d) Platinum resistance thermometer is defective
(e) Heater power cable between controller and desorption unit is disconnected or defective

Solutions

Check the main power fuses on the desorption system controller and make sure the heater power cable is connected. If the problem persists, remove the side from the desorption unit and reset the over-temperature sensor circuit breaker on the back plate(red button). If this does not solve the problem, the heater cartridge or PRT may be bad. Call SIS Technical Support.

Cryotrap Does Not Cool

Problem - Cryotrap does not cool to temperature setting within the required time. Sample or sequence is halted, and the ADS software reports that the Cryotrap was taking too long to cool.

Cause

(a) Coolant level low
(b) Obstructed coolant line
(c) Coolant valve not connected to controller
(d) Faulty or obstructed coolant valve

Solutions

Low coolant level is the main cause of this symptom. After verifying adequate coolant level, make sure the cryotrap valve is connected to the controller.

LIQUID NITROGEN OR LIQUID CARBON DIOXIDE MAY BE PRESENT AT HIGH PRESSURE. SEVERE BURNS MAY RESULT FROM CONTACT WITH LIQUID CRYOGEN. SHUT OFF THE CRYOGEN SUPPLY AT THE TANK AND CAREFULLY VENT THE SUPPLY LINES BEFORE PROCEEDING TO TROUBLESHOOT THE CRYOTRAP VALVE. Connect a low pressure (<30 psi) air line to the valve to test it and the cryotrap supply line for faults or obstructions.

LINE VOLTAGE IS USED TO ACTIVATE THE CRYOTRAP VALVE. SHUT THE POWER TO THE CONTROLLER OFF BEFORE CONNECTING OR DISCONNECTING THE CRYOTRAP VALVE. USE CAUTION WHEN TROUBLESHOOTING THE VALVE. UNTRAINED PERSONNEL SHOULD NOT ATTEMPT TO DIAGNOSE OR REPAIR ELECTRICAL PROBLEMS.

Disconnect the coolant supply line at the Cryotrap and verify an unobstructed line between the valve and the trap by activating the valve through the ADS software. If no flow is observed, reset the controller to turn the valve off, and carefully disconnect the line at the outlet of the valve. Repeat the test to verify that the valve is working. After the defective part has been isolated in this manner, it can be replaced.

Cryotrap Does Not Heat

Problem - The Cryotrap does not heat or heats very slowly

Cause

(a) Heater cable not connected to controller
(b) Cryotrap heater is faulty
(c) Cryotrap heater fuse in the controller is blown

The following procedures are for diagnostic purposes only and should only be attempted by a trained individual with the proper equipment. LINE VOLTAGE IS USED TO ACTIVATE THE CRYOTRAP VALVE. UNTRAINED PERSONNEL SHOULD NOT ATTEMPT TO DIAGNOSE OR REPAIR ELECTRICAL PROBLEMS.

Solutions

Verify that the heater cable is connected to the controller. The cryotrap heater is supplied with 18 volts DC from the controller. Verify that this voltage is present from the connector at the back of the controller when the cryotrap should be heating. If the voltage is present, measure the resistance across the heater element. The resistance should be approximately 32 ohms. Infinite resistance or negligible resistance indicates a fault. If the fault is in the cable, it may be repairable. Contact SIS Technical Support. DO NOT ATTEMPT TO DISMANTLE THE CRYOTRAP. THERE ARE NO REPAIRABLE OR REPLACEABLE PARTS INSIDE. EVIDENCE OF TAMPERING WILL VOID ANY WARRANTY.

If the heater voltage is not present, contact SIS Technical Support for assistance in locating and checking the fuse for the heater circuit.

No Power

Problem - No power to the system. Controller or desorption unit does not light up.

Causes

(a) System not plugged in or power line is dead
(b) Fuses blown on back of desorption unit or controller

Solution

Check that the power cord is plugged into the electrical socket. Check and/or replace the main fuses at the back of the controller. If fuses are blown and continue to blow, the unit should be serviced by a qualified technician at SIS.

UNPLUG THE POWER CORD FROM THE ELECTRICAL SUPPLY BEFORE ATTEMPTING TO CHECK OR CHANGE FUSES.

Temperature Readings Out of Range

Problem - Temperature readings are unsteady, false, or consistently do not reach setpoints.

Causes

(a) Faulty temperature sensor(s)
(b) Temperature sensor(s) out of calibration
(c) Temperature control constants in software corrupted

Solution

Problems with temperature sensors including faulty sensors as well as those that are out of calibration must be corrected by servicing the instrument. Contact SIS Technical Support to verify the problem and arrange service.

In the unlikely event that temperature control constants in the software become corrupted or are altered, a reinstallation of the software will restore the proper values. Contact SIS Technical Support for assistance.

Software

ADS Software Does Not Start

Problem - The ADS software does not automatically start when the data system is started.

Causes

(a) Unsupported data system or unrecognized version.
(b) Data system instrument not configured for use with the AutoDesorb

Solution

Supported data systems and versions are listed in the README.TXT file on the AutoDesorb Software Library CD-ROM, and in the Specifications section of the operating manual. A standalone version is expected to be released sometime in 2001. If your data system is listed and you still experience problems setting up and running the software, contact SIS Technical Support for assistance.

It is recommended to establish an instrument configuration specifically for use with the AutoDesorb system, otherwise the AutoDesorb may be enabled or disabled each time the data system is started. Enabling the AutoDesorb can be accomplished by shutting down the data system and entering the AutoDesorb Configuration Editor from the SIS program group in the Windows Start menu. Select the instrument to be used with the AutoDesorb and the appropriate COM port, and save the settings. Exit the Configuration Editor and re-start the data system.

Software Cannot Communicate With Controller

Problem - The software displays a message that it cannot communicate with the controller.

Causes

(a) The controller is not plugged in or not turned on.
(b) The RS-232 communications cable is not connected to the controller or to the data system computer.
(c) The wrong COM port is assigned to the AutoDesorb system.

Solutions

Verify that there is power to the controller and that the unit is on.

Verify that the RS-232 communications cable is correctly connected to both the controller and a working serial port on the data system computer. Swap the cable with one that is known to work to eliminate the cable itself as the problem.

Through the ADS Configuration Editor (from the program group SIS on the Windows Start menu), verify that the correct COM port is selected, and save any changes to the configuration.

Data

No Peaks

Problem - No peaks are present in the chromatogram.

Causes

(a) Detector not turned on
(b) Leaks in the desorption system
(c) Bad GC Septum
(d) Broken GC column
(e) Split ratio too high
(f) Sample size too small
(g) Ice plug in column
(h) Clogged desorption tube needle

Solution

Verify that the GC method uses the detector, that the detector is operational and that data is being acquired.

Leaks in the flow path of the desorption system can cause the absence of peaks in the chromatogram. Leaks usually occur at the desorption needle during injection and desorption. Check the pressure readings during the desorption process. Refer to the Low Pressure symptom in section A above.

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 as required. Measure the flow at the split vent using a flow meter. Adjust the Inject time parameter to assure the correct split flow when the Desorption time begins (see Data Entry in the Creating and Editing AutoDesorb Methods section of this manual).

For solutions to the other causes of blank chromatograms, refer to the Flow and Mechanical section above.

Broad Peaks in Chromatogram

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 the best results are achieved using a megabore deactivated fused silica precolumn or guard column with all capillary columns.

Extraneous Peaks in Chromatogram

Problem - Extraneous Peaks in Chromatogram

Cause

(a) Contaminated desorption tube
(b) Contaminated needle
(c) Contaminated connecting tube
(d) Contaminated Septum
(e) Contaminated injection port liner or injection port
(f) Bleeding GC Septum
(g) Injection port too hot
(h) Bad guard column
(i) Desorption temperature too hot
(j) Contaminated GC carrier gas
(k) Breakdown of adsorbent resins in packed thermal desorption tubes
(l) Contaminated carrier gas line traps
(m) Injection port and carrier gas lines are contaminated

Solutions

Often three peaks are present in the chromatogram resulting from either septum bleed or GC column coatings. If the analysis is being done on a mass spectrometer, these three peaks will have the most abundant ions at 207, 281 and 355 (or 267) consecutively. These correspond to siloxanes resulting from breakdown of the bonded phases used in capillary GC columns or bleed from silicone septa. The three most abundant 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 55 & 267

In a normal GC run these compounds result in a slight elevation of the 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 and will result in distinct chromatographic peaks. These peaks 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, regularly replacing 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 coating. Also note that higher desorption block temperatures and injection port temperatures result in more pronounced siloxane peaks. Best results are always obtained at the lowest possible desorption and injection port temperatures required to perform the analysis.

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 performing the injection port bakeout procedure outlined in the Special Methods section of this manual. If the source of the contamination is the injection port, repeating the analysis should result in the elimination of, or at least a dramatic reduction in, the contamination peaks. If the peaks still appear, remove the connecting tube filter and condition it as described in the System Care and Maintenance section of this manual, and repeat the analysis.

If the problem still persists, the GC column and the GC carrier gas purity should be checked. Replace the GC column. Install a new carrier gas tank and replace the hydrocarbon and oxygen traps on the carrier gas lines.

If your instrument is routinely used for liquid injections or dirty samples, the injection port may become severely contaminated. For assistance in cleaning it up, contact SIS or visit our website at www.sisweb.com .