

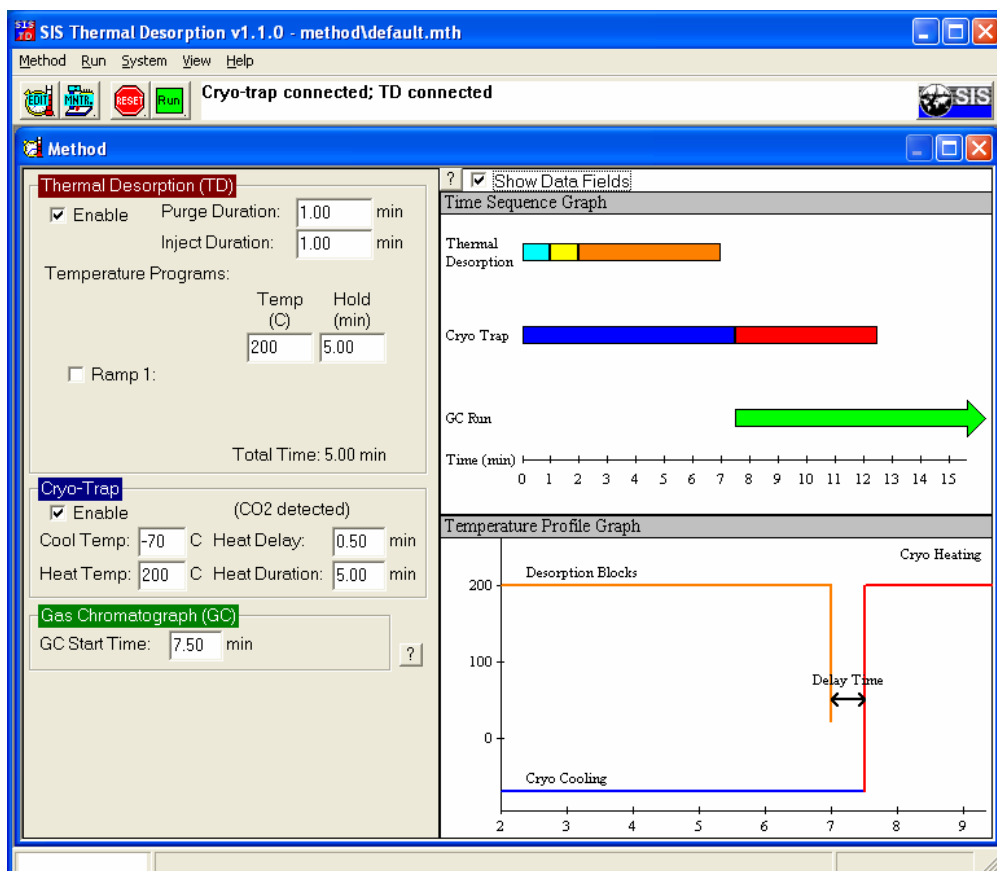
SIS Thermal Desorption Software User Manual

Software Version 1.1.0 – 2005-06-01

For the SIS TD-5 and AutoDesorb Short Path Thermal Desorption Systems

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

U.S. Patent #5,065,614

U.S. Patent #5,123,276

U.K. Patent #GB 2 253 161 B

U.S. Patent #5,596,876.

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About the Manual

This manual covers the installation and operation of the SIS Thermal Desorption Software for the SIS TD-5 and AutoDesorb thermal desorption systems.

The information in this manual is provided with the assumption that the user is familiar with general gas chromatography (GC) concepts and the operation of the instrument on which the thermal desorption system is installed. Refer to the manuals supplied by the manufacturer of your GC for specifics on your GC and GC data system software.

Additional information can be found on the thermal desorption section of the SIS web site:

SIS Short Path Thermal Desorption Home Page

<http://www.sisweb.com/sptd/>

This includes product information, application notes, and adsorbent resins data.

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1. Introduction

Thermal Desorption Software – The thermal desorption software provides a graphical user interface (GUI) from which the operator can configure the thermal desorption system settings/run parameters, control the thermal desorption system (e.g. start runs), and monitor the thermal desorption system (e.g. temperatures and pressures). The thermal desorption software resides on a computer that is connected to the Electronics Console via an RS-232 serial communication cable (with optional RS232 ↔ USB converter cable). Typically, this computer is the same computer on which resides the software that controls the GC (the **GC data system software**).

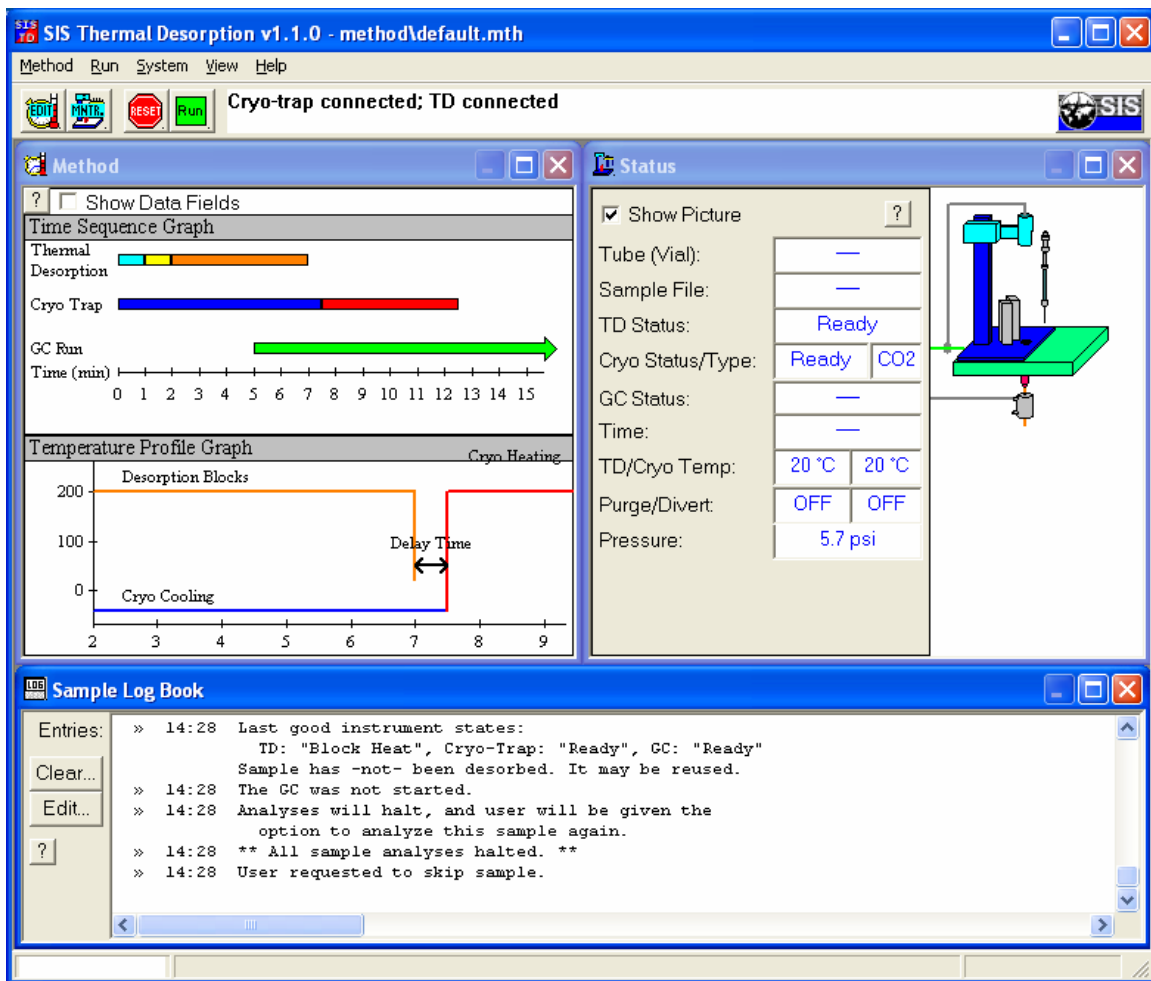


Figure 1-1 – Thermal Desorption Software

The thermal desorption system can be operated in either of two modes: *standalone mode* (independent of the GC data system software) and *integrated mode* (integrated with the GC data system software). Which you use will depend on whether the thermal desorption software is able to integrate with your particular type of GC data system software. Currently, the integrated mode only works on version of Agilent (HP)

1 - Introduction

ChemStation. (Check the online documentation for specific compatibility requirements or call SIS.) The standalone mode can work with any GC that the thermal desorption system is physically compatible with. The advantage of the integrated mode is that thermal desorption run settings are stored within (linked to) the GC method, and the run operation is a bit more coordinated (e.g. the thermal desorption system will start automatically when a new sample run is invoked from the GC data system). The integrated mode is highly recommended when running sequences on the AutoDesorb.

2. Software Installation

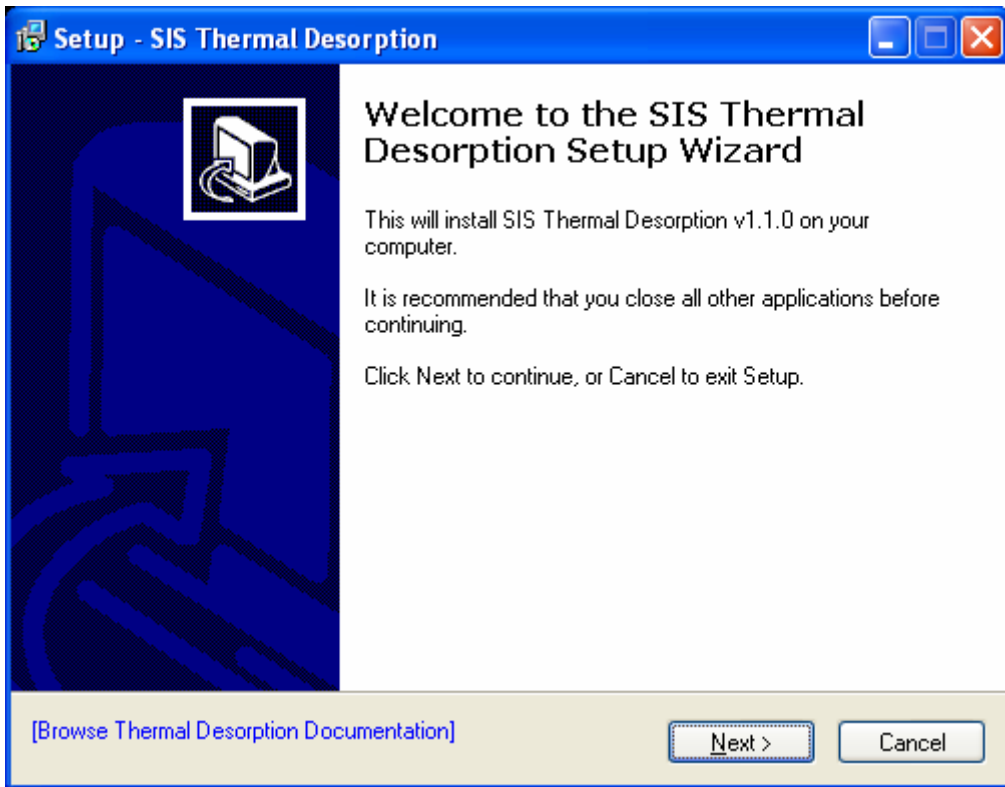


Figure 2-1 – Welcome Page for installation of the Thermal Desorption Software.

Overview

This section describes the installation process for the **Thermal Desorption Software** (Figure 1-1). See Chapter 1 for background information on this software. Installation of the software may be done before or after installation of the hardware (Chapter 2).

Note: the screenshots in this section might vary slightly from those you see.

Installation

Insert the SIS Thermal Desorption Software CD. Typically, the window in Figure 2-1 will display.

(If window does not display automatically, select from the **Windows Taskbar** the menu item “**Start | Run...**” and then enter “**D:\setup.exe**” where “D” is replaced by the drive letter of your CD-ROM drive on your system.)

Note: At any time, you can optionally click the blue “**Browse Thermal Desorption Documentation**” link to view the online documentation.

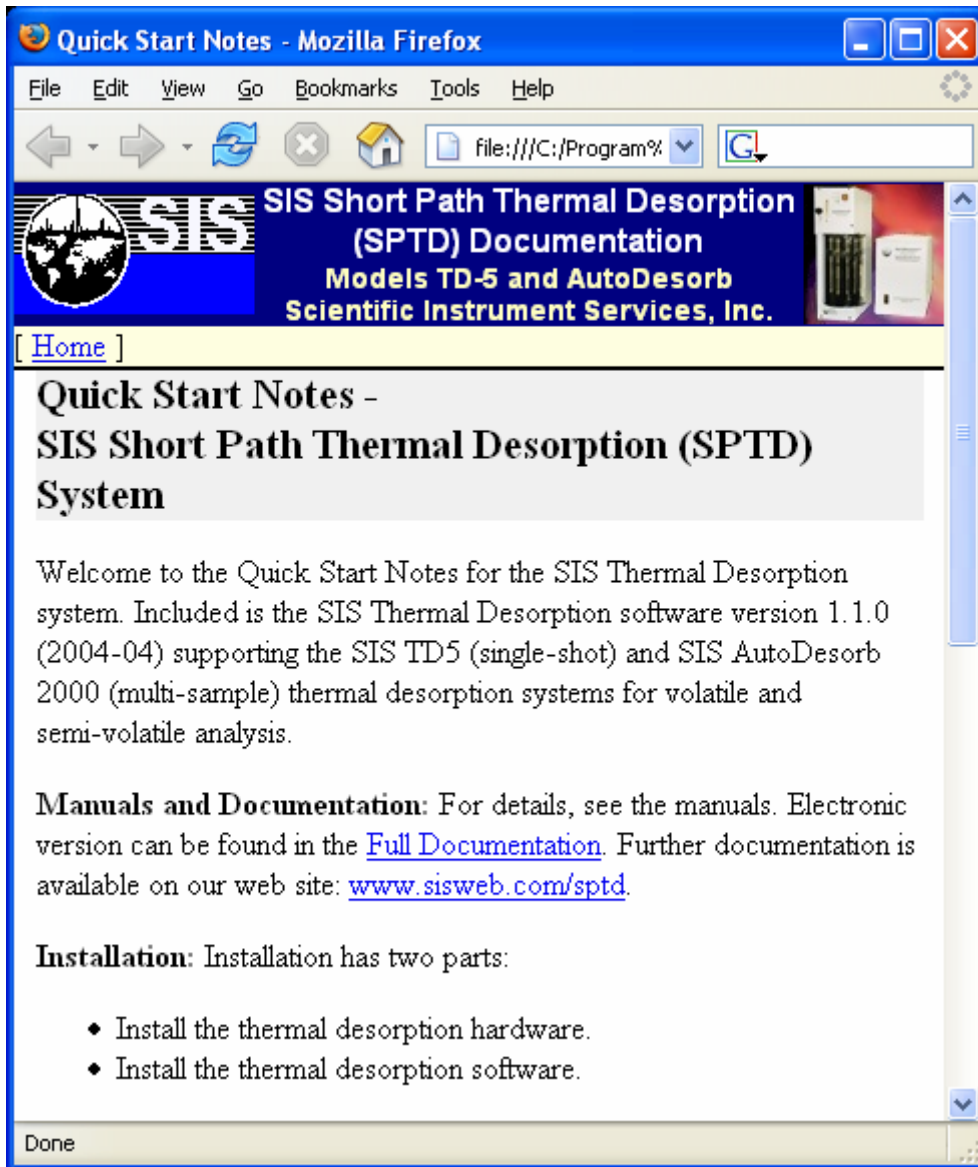


Figure 2-2 – Online documentation

The first part of the installation process is fairly typical. Press **Next** on the installation screen shown in Figure 2-1.

2 - Software Installation

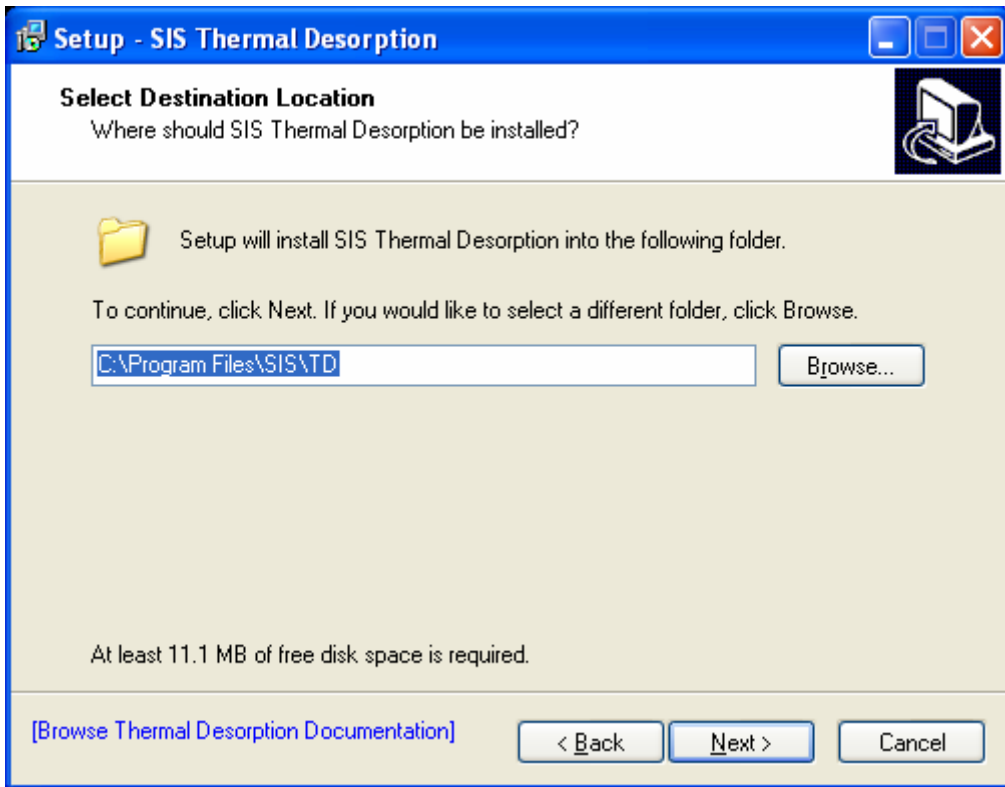


Figure 2-3 – Installation program, selection of installation location.

The default installation location for this software is at “C:\Program Files\SIS\TD”, but you may change this to any location as shown in Figure 2-3. Press **Next**. When the window in Figure 2-4 displays, press **Install**.

2 - Software Installation

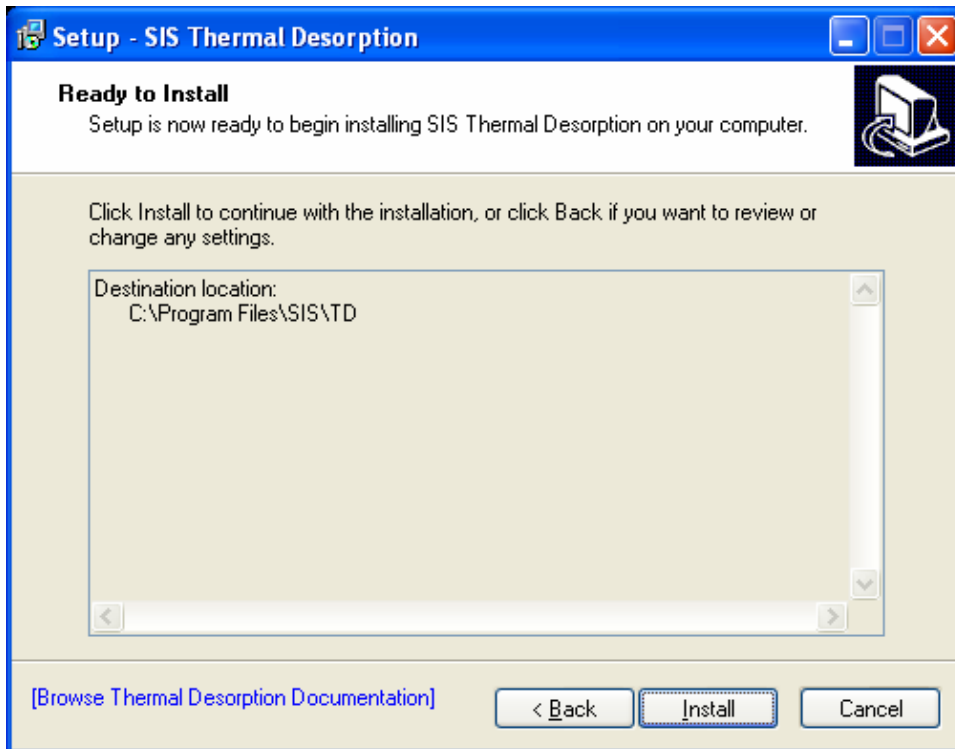


Figure 2-4 – Installation program, confirmation page

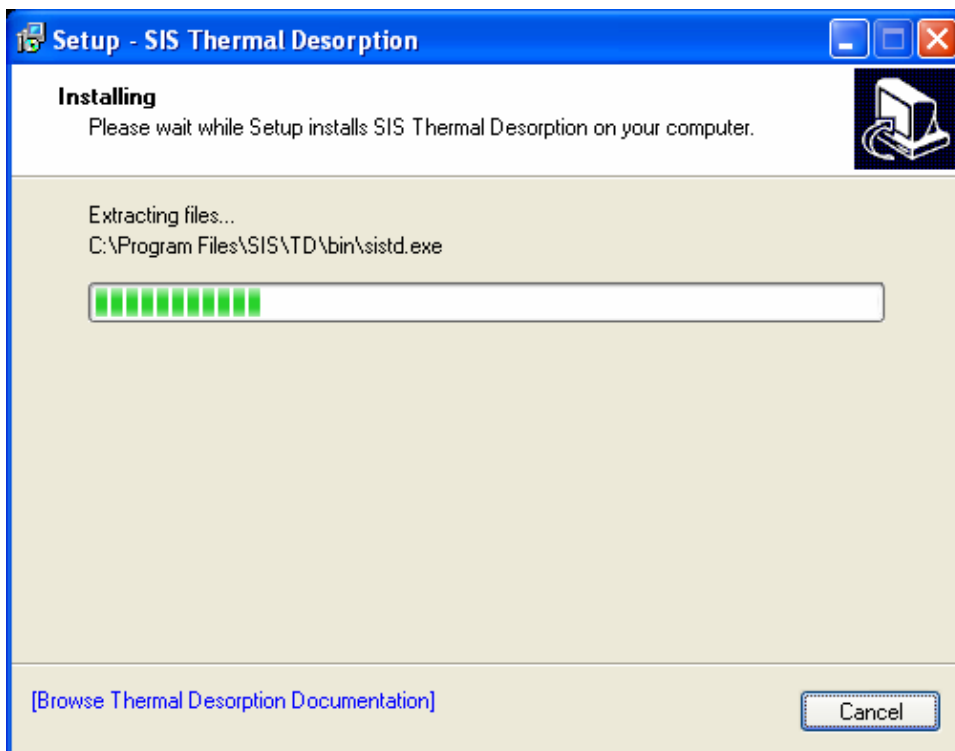


Figure 2-5 – Installation program, installing files

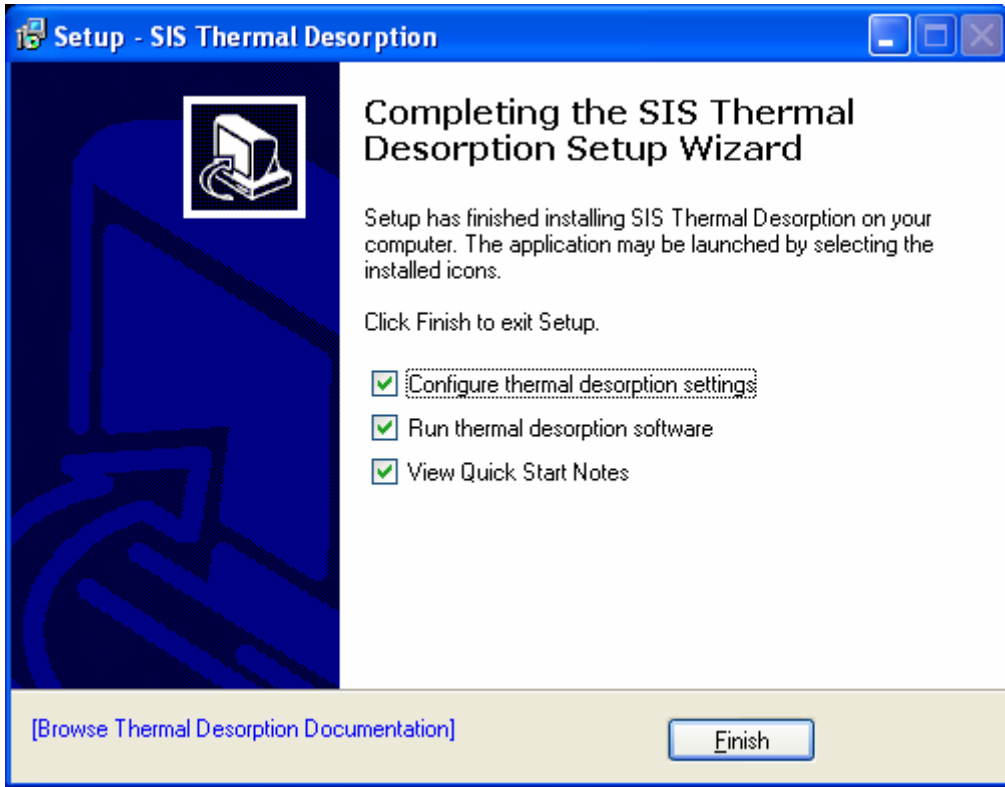


Figure 2-6 – Installation program, installation complete.

The software is installed, and the window in Figure 2-6 displays. The software still must be configured.

Configuration

It is now time to configure the software. The Configure System window (Figure 2-7) will display following the installation.

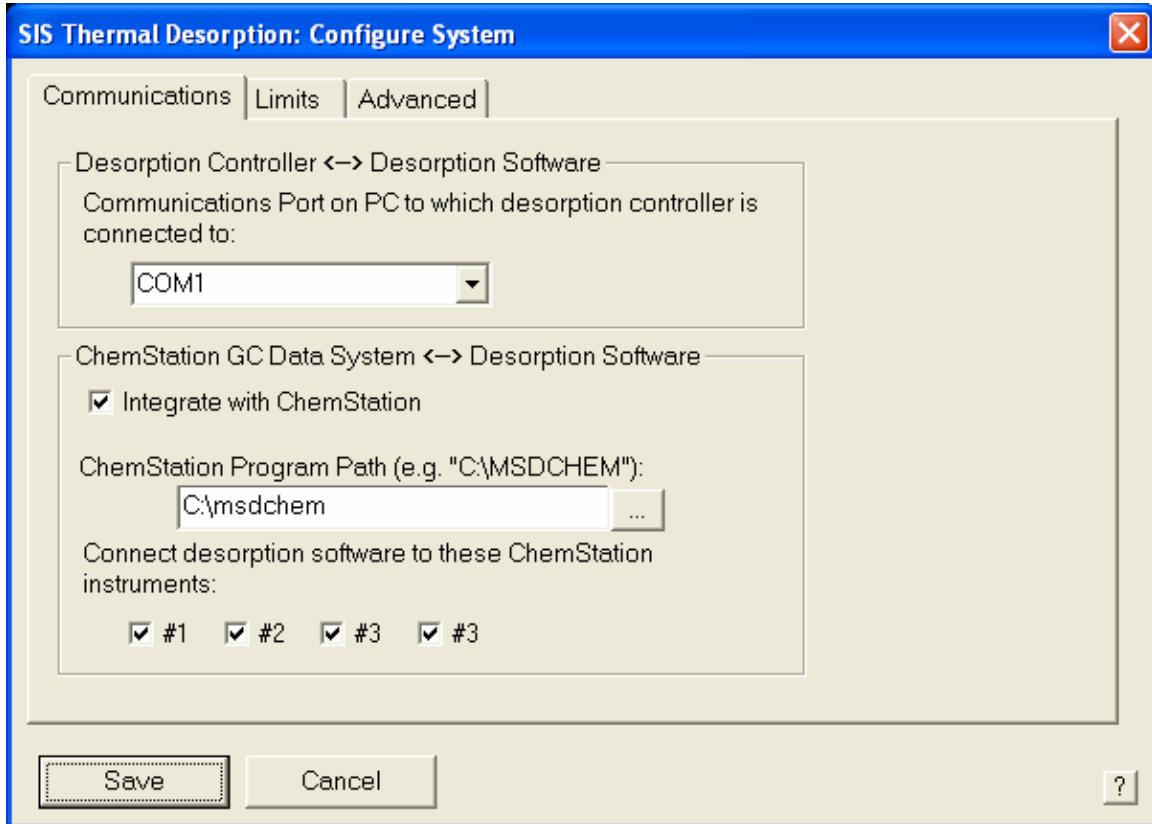


Figure 2-7 – Configure System window

Only two items need be configured at this time:

- The **communications port**
- The **ChemStation program path** (only if you wish to use the software in integrated mode with the Agilent ChemStation GC data system).

Both of these items display on the “**Communications**” Tab.

Note: These items and others can be changed at any later time. In particular, the configuration window is accessible from the **Windows Start Menu (“Programs | SIS Thermal Desorption | SIS Thermal Desorption | Configure”)** as shown in Figure 2-9.

ChemStation Integration

The thermal desorption software interfaces to Agilent (HP) ChemStation via ChemStation macros. The thermal desorption software installs its own macros (sistd.mac) and makes a few minor modifications to the standard ChemStation macros.

As shown in Figure 2-7, ChemStation integration is enabled by checking the “**Integrate with ChemStation**” checkbox. You must specify the **ChemStation Program Path** (typically, “C:\MSDCHEM”) in order that the thermal desorption software knows which ChemStation files to modify. In addition, you can enable/disable the thermal desorption system only on specific ChemStation instrument numbers. By default, all instrument numbers are enabled (note: there is no harm if certain instrument numbers do not exist).

When pressing **Save**, the thermal desorption software will attempt to update the ChemStation macros. If successful, you will see a series of dialog boxes as shown in Figure 2-8.

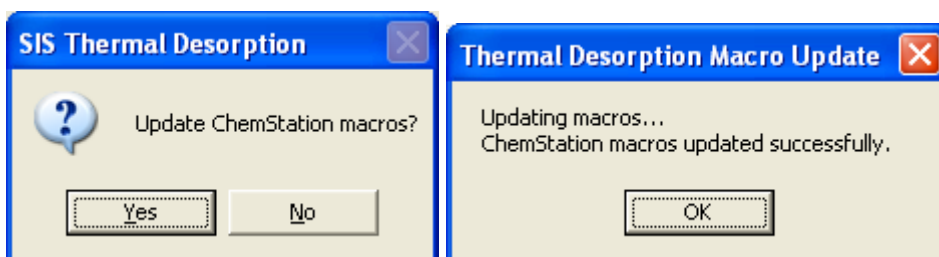


Figure 2-8 – updating ChemStation macros

Reinstalling or Upgrading ChemStation

Warning: If you ever reinstall, upgrade, or move ChemStation, you will need to re-update the ChemStation macros since they may have been overwritten. This can be done by removing and re-enabling ChemStation integration (i.e. disable **Integrate with ChemStation**, do a **Save**, **Enable Integrate with ChemStation**, and do a **Save** again).

Starting the Thermal Desorption Software

The thermal desorption software is by default set to start automatically following the installation and configuration. The software can also be started from the Windows Start Menu | Programs | SIS Thermal Desorption | SIS Thermal Desorption Program as shown in Figure 2-9.

2 - Software Installation

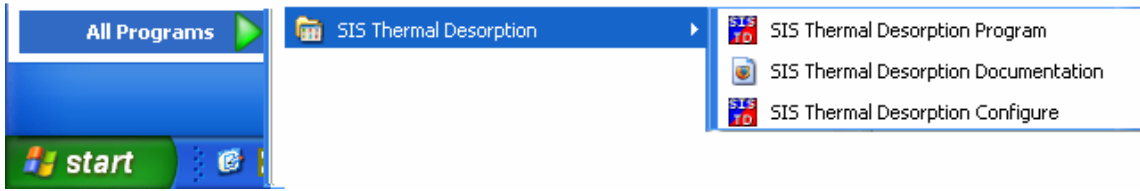


Figure 2-9 – Accessing the thermal desorption software from the Windows Start Menu.

3. Software Operation

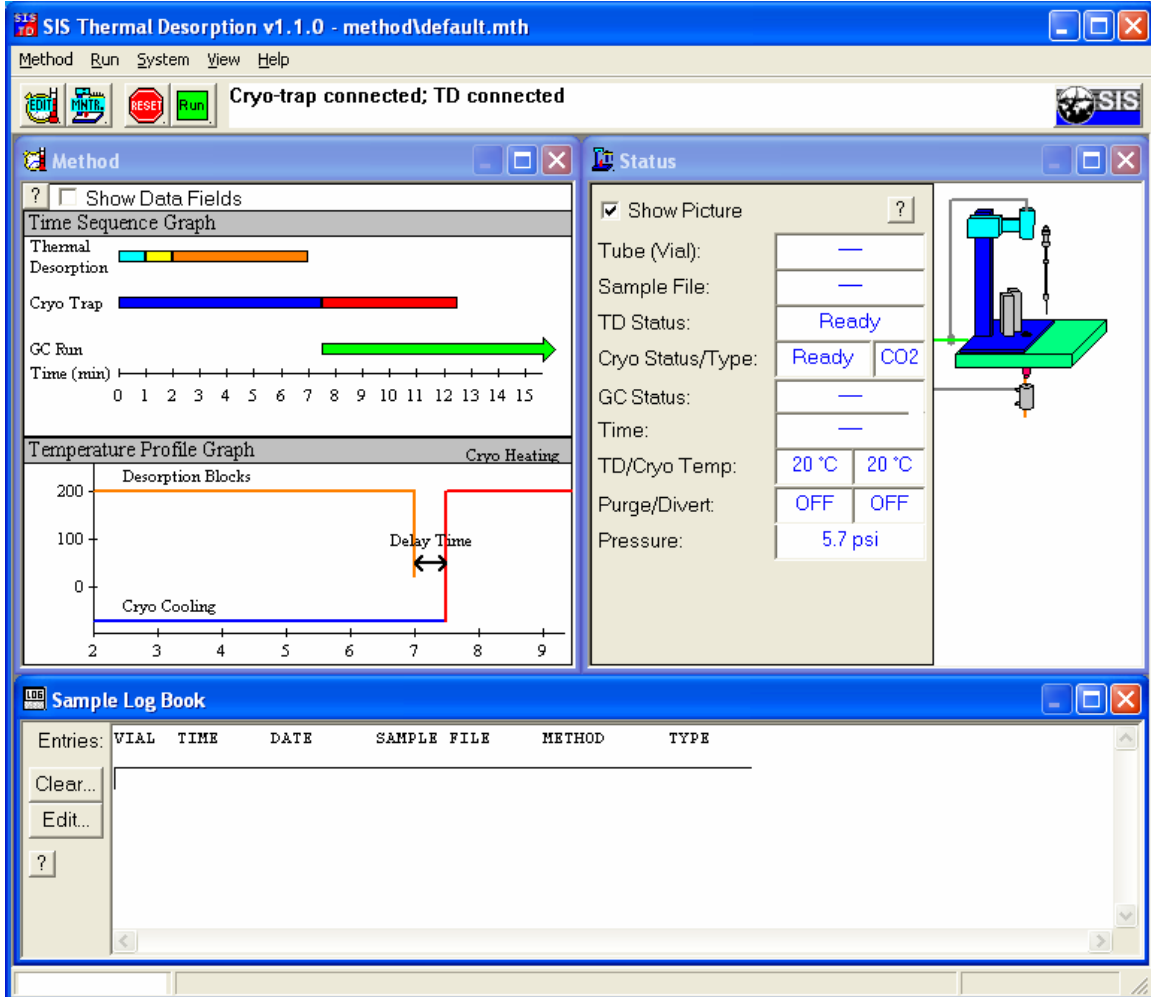


Figure 3-1 – Thermal Desorption Software window

Overview

This section details the Thermal Desorption Software and its use in configuring, running, and monitoring the Thermal Desorption Unit. See Chapter 1 for a background on the Thermal Desorption Software.

Getting Started

Before you begin using the thermal desorption system, make sure that it has been properly installed. This pertains not only to the Desorption Unit, Electronics Console and Cryo-Trap, but also to the Thermal Desorption 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

First, turn on the power to the Electronics Console.

If the Thermal Desorption Software is running in integrated mode (integrated with your GC data system), the Thermal Desorption Software will open automatically when the GC data system is opened. If not, start the Thermal Desorption Software manually as shown in Figure 2-9.

When the Thermal Desorption software is started, it will attempt to communicate with the Electronics Console. Any error messages will be displayed at this time.

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

Thermal Desorption Methods

Parameter settings for one or more thermal desorption runs are contained in a Thermal Desorption Method, which can be saved as file of extension “.mth”. You can have


3 - Software Operation

multiple methods, which can be loaded, saved, and edited as needed using the Thermal Desorption Software.

Furthermore, if the Thermal Desorption Software is used in integrated mode, the Thermal Desorption Method will be saved (and linked to) the GC method in your GC data system so that appropriate thermal desorption parameters will be loaded automatically when a GC method is loaded in your GC data system.

Creating a thermal desorption method is a matter of modifying an existing one to suit the current analysis. A default method is loaded when the Thermal Desorption Software is started in standalone mode or if a GC method without thermal desorption settings is loaded in integrated mode.

Method Edit View

The thermal desorption method settings can be edited from the **Method Edit View** of the **Method Window**, which is accessible from the “**View | Method Edit View**” menu item or the  button on the toolbar.

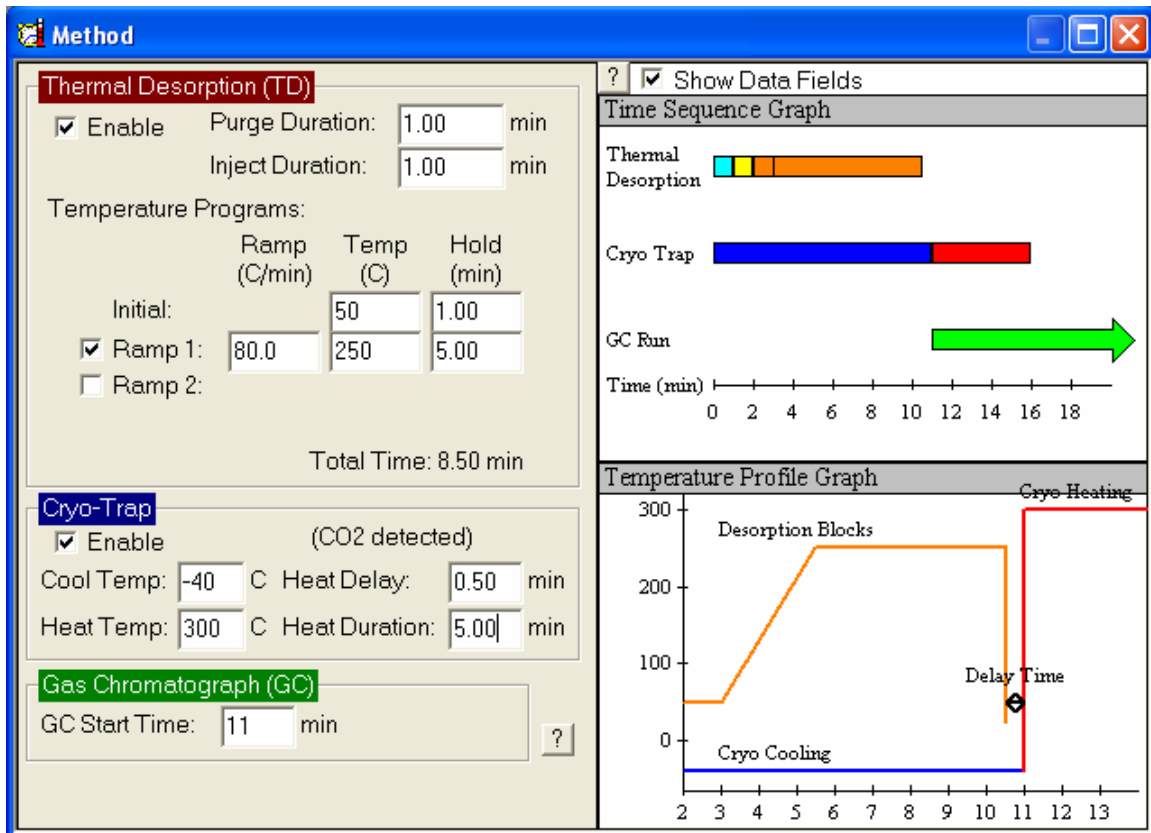


Figure 3-2 – Thermal Desorption Method Window

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The Desorption Unit (TD) and Cryo-Trap can be independently enabled or disabled from in the method by checking the “*Use TD*” or “*Use Cryo*” boxes. An example of a method that would not use the Cryo-Trap 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 Cryo-Trap may be used with the Desorption Unit disabled and removed from the GC injection port in order to do focusing static headspace injections.

The main parameters for the thermal desorption are the Purge, Inject, and Desorb times and the temperature settings for the desorption heater and cryo-trap. Other parameters include the GC start time and Cryo-Trap heat delay and duration. Adjustable parameters are explained briefly below:

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).

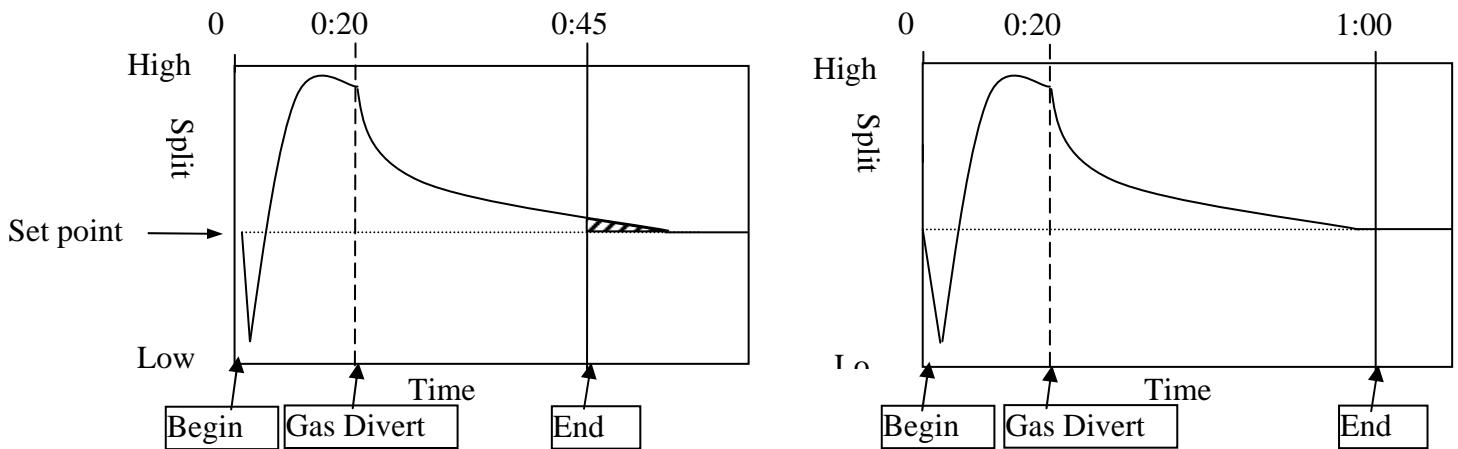
Inject: The inject period begins when the needle is lowered into the GC inlet. A drop in inlet pressure is normal as the septum is punctured; however, re-pressurization should follow rapidly as the desorption purge flow and normal GC carrier are both directed into the inlet. The dual flow lasts for a 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 for the dual flow period** is changeable from the “**System | Configure... | Limits**” and changing GC Gas Divert Time.. 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 4). At the end of the Inject period, a pressure reading from the Desorption Unit is compared to the **Minimum Pressure to Run** set-point in “**System | Configure... | Limits**.” If the minimum pressure is not met a leak is assumed, the sample is terminated.

WARNING: 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.

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 cryo-trap 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 cryo-trap is ballistically heated to release the focused analytes instantaneously, much like a liquid injection.



Injection time incorrectly set. Insufficient time allowed for split flow to equilibrate at the set point.

Injection time correctly set. The split flow has been allowed to stabilize

Figure 3-3 - Setting the Inject Time Parameter

Cryo Heat Duration:

This setting controls the length of time that the cryo-trap is heated. Most volatile materials should be released in the first few seconds of heating. Keeping the cryo-trap 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 cryo-trap 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 cryo-trap cooling temperature) since compounds breaking through a cryo-trap 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.

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 ($^{\circ}\text{C}/\text{minute}$), target temperature ($^{\circ}\text{C}$) and hold time (minutes) for each segment may be entered. The maximum controlled heating rate is $100\text{ }^{\circ}\text{C}/\text{minute}$. A heating rate value of 0°C will result in the ballistic heating of the sample. Total desorption time is calculated automatically.

Cryo-trap Temperatures:

Cryo-trap cooling temperature may be set between ambient temperature and -70°C for liquid CO_2 use or between ambient and $-180\text{ }^{\circ}\text{C}$ 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 $-70\text{ }^{\circ}\text{C}$). 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 cryo-trap 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 thermal desorption method, it is important to remember to save the changed settings by selecting “**Method | Save Method**” or “**Method | Save Method As**” from the menu bar or (in integrated mode) by saving the entire method from the GC 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 Thermal Desorption Software 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.

Importing settings

In integrated mode, it can be useful to import thermal desorption settings from one GC method into another. This is made possible by the “**Method | Import Method...**” menu item.

Running a Single Sample

Once the data system and thermal desorption 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 standalone mode, select the “**Run | Run Sample...**” menu item or click the



button. Then click “**Start Run**”.

In integrated mode using ChemStation the following procedure is used. From the Instrument Control or Run Control window, select **Method | Run** to activate the Start Run window. Edit sample information and click the **Run Method** button in the Start Run window. The entire data system method, including thermal desorption settings, will be executed when the GC becomes ready.

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Start Run

Data File Name: E:\HPCHEM\1\DATA\EVALDEMO.D

Disk Space: 291,962,880 bytes free on drive E:

Operator Name: Vial: 1

Sample Name:

Misc Info:

Method Sections To Run:


Data Acquisition

Data Analysis

Run Method OK Cancel Help More>>

Data File Name: Enter a data file name or type a ? for a list

Figure 3-4 – Running a single sample from ChemStation.

Terminating a run: If for any reason the sample must be aborted or stopped during the run, do so by selecting the “**Run | Reset Controller...**” menu item or clicking the  button on the toolbar.

The thermal desorption software contains error handling protocols that will assure that the GC oven temperature program is run if there is an error following the desorption blocks having been closed. Stopping the GC run from ChemStation may result in desorbed analytes remaining on the column into the next run.

Running a Sequence of Sample (AutoDesorb)

The AutoDesorb supports running up to 12 samples unattended.

When integrated with ChemStation, the **ChemStation sequence table** is used to control the entire sequence.

Using ChemStation, 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.

3 - Software Operation

The sequence will then proceed as normal.

Line	Type	Vial	Data File	Method	Sample Name
1)	Sample	1	-----	DEFAULT	Sample 1
2)	Sample	1	-----	DEFAULT	Sample 2
3)	Sample	1	-----	DEFAULT	Sample 2

Type: Sample, Vial: 1, Data File: , Method: DEFAULT, Sample Name: Sample 1

Miscellaneous Information: , Expected Barcode:

Repeat, Cut, Copy, Paste, Read, OK, Cancel, Help, More>>

Use the arrow keys to select entry

Figure 3-5 – Running a sequence from ChemStation.


Caution: You may notice that the Thermal Desorption Software provides a sequence table of its own (**Run | Sequence**). **This is not used** when operated in integrated mode. This sequence table is provided for situations where integrated mode cannot be used (e.g. non-ChemStation GC data systems).

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

NOTE: 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 thermal desorption 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 Thermal Desorption Software.

Status Window

During the run, the actual temperatures, pressures, and states will be displayed in the **Status Window** (Figure 3-6). This window also includes an animated graphical representation of the Desorption Unit. This window is displayed by selecting the Run Monitoring View (“View | Run Monitoring View” from the menu or the  button).

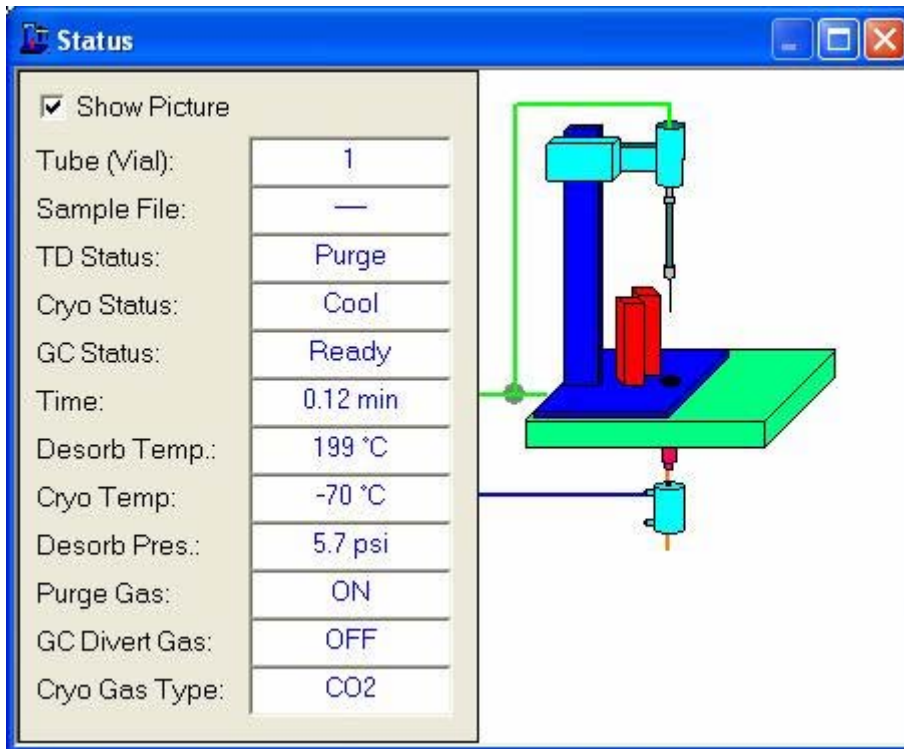


Figure 3-6 - Status Window

The Sample Log Book

The Thermal Desorption Software has extensive error and status logging functions. The **Sample Log Book** (Figure 3-7) 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 Sample Log Book can be accessed by clicking selecting the “View | Run Monitoring View” menu

item or clicking the  button on the toolbar.

3 - Software Operation

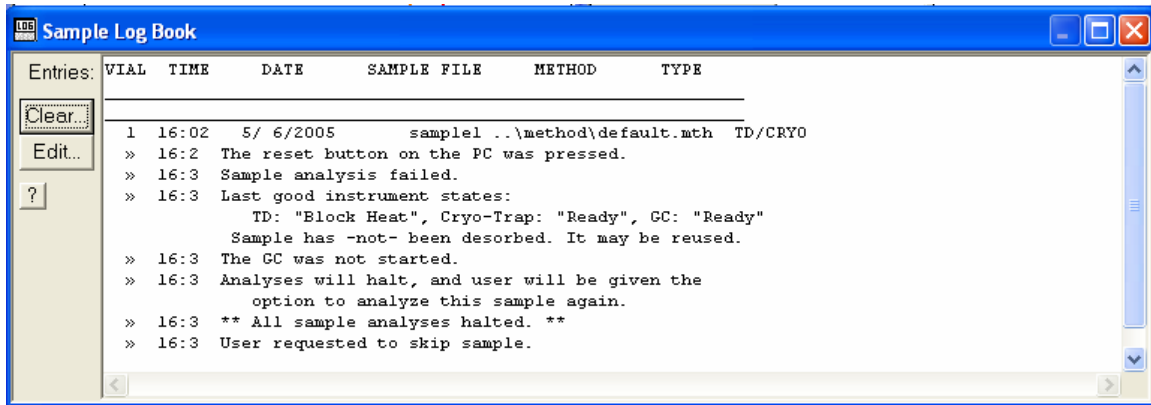


Figure 3-7 - Sample Log Book

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 number, Time, Date, Data file name, Method name and the names of active instruments in the method being used (e.g., cryo-trap 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.

Sample Log Book data is stored in a file named **default.log** in the C:\Program Files\SIS\TD\logs directory. The Sample Log Book may be opened in Notepad by clicking the "Edit..." button on the Sample Log Book. Periodically, the Sample Log Book should be archived (opened in Notepad and saved with a different name) and cleared via the "Clear..." button.

Online Help

Additional details can be found from the online help (**Help** menu) or by clicking the "?" buttons.