The Mass Spec Source

VOL. XXI, NO. 1 MARCH, 1999

For Users of Mass Spectrometers and Gas Chromatographs

FEATURES

CONTENTS

We're Back!!

We are proud to announce that the MASS SPEC SOURCE is back in publication after some time off. The staff of SIS is working to bring you this newsletter quarterly in the coming year. The SOURCE had taken some time off over the last year, and many of you communicated to us that you missed it and would like to see it again. Well, here we go.

The SOURCE is intended to provide useful and helpful information in the areas of mass spectrometry, gas chromatography, liquid chromatography, thermal desorption and related fields. It is our hope to be able to keep you up to date with S.I.S. and what we are doing, introduce new products to you which may be of interest, as well as provide you with tips and techniques and other information to make your work more productive. If there is information or techniques that you have developed or are aware of that would be interesting and helpful for our readers please let us know that as well.

Thanks for your interest in the MASS SPEC SOURCE and in S.I.S. Please take the opportunity to let us know how we are doing. We would love to hear from you. You can find our address, including our website and e-mail addresses on page two in the "Terms and Conditions" section. Thanks again and we hope you enjoy the newsletter.

S.I.S. News

A few things have happened since our last newsletter was published. Here are some of the highlights from a very busy year, plus some important items coming in the near future.

"We're Going to Disney"

Pittcon '99 is heading to Orlando in early March. The week of March 7th we hope to see all of you at Pittcon '99. S.I.S. will have our largest presence yet at this exhibit where we will have four booths packed with new instrumentation and will be providing demonstrations of new software. We will have our brand new Automated Short Path Thermal Desorption system at the show as well as our Direct Probe for the HP5973 MSD, Cryotraps for gas chromatography, and Electrospray tips and accessories for LC/MS. In addition we

SIS Poster Presentations at PittCon 1999

1. Design, Development and Testing of a Microprocessor Controlled Automated Short Path Thermal Desorption Apparatus. Sunday Poster Session. **Poster #1620P**
2. Volatile Compounds from Electron Beam Cured and Partially Electron Beam Cured Packaging Using Automated Short Path Thermal Desorption. Sunday Poster Session. **Poster #1630P**
3. Analysis of Perfumes and Their Effect on Indoor Air Quality. Sunday Poster Session. **Poster #1628P**
4. Rapid Bacterial Chemotaxonomy by Direct Probe/MS. Tuesday Poster Session. **Poster #2054P**

will be providing demos of new software that will be of interest to users of GC/MS and LC/MS. One of the featured software products will be the NIST98 software product for mass spectrometer users. This powerful, new product features a greatly expanded and improved database as well as a new deconvolution package which will provide you with even more tools to evaluate and understand the data you create. This new software is available now from S.I.S. and if you can't see it at PittCon you can download a copy of the demo from our website. http://www.sisweb.com

S.I.S. Expands!

Last year S.I.S. underwent a major expansion when we ***nearly tripled the size of our existing facility.*** The completion of this project was much anticipated by all of us here at S.I.S. and we are thrilled to be working in the new facility. All of our shop facilities are now housed in the new wing of our building with more room to expand the equipment we need to serve our customers. We will be adding new equipment and technology that will allow us to continue to provide you with new instruments and products. We are also evaluating new technology and techniques that will make ordering supplies and getting technical information even easier. Stay tuned to future issues of the SOURCE for information regarding these changes.

Giant Software Demo CD

S.I.S. will be distributing a new free Giant Software Demo CD. This demo disk is packed full with over 44 demos of new software which will enhance your lab. Some of the featured software packages on the CD include the new NIST98, SIMION ion optics modeling software, Chemical Inventory software and many training programs for chromatography, mass spectrometry and other techniques. In addition more than 100 other scientific software packages are described on the CD. Also included in the demo CD is a full copy of our website and a copy of the Hewlett-Packard ChemAssist.ChemAssist is a handy tool for finding the parts you need for your HP instruments. S.I.S. is now an authorized dealer for HP parts and supplies. Please see below for more information. If you are planning on going to Pittcon please stop by, and pick up your free Giant Software Demo Disk. If you can't visit us in Orlando, and would like a copy of the demo CD just give us a call and we will be pleased to send one to you.

New Scientific Supplies Catalog

The new S.I.S. Scientific Supplies Catalog for 1999-2000 will be available soon. This new catalog is even bigger with many new products and services for users of mass spectrometers, gas chromatographs and liquid chromatographs. The catalog should be in your mailbox sometime late in March. Some of the new products featured in the catalog are highlighted below.

New Products from S.I.S.

As mentioned previously, S.I.S. will be presenting its new ***Automated Short Path Thermal Desorption*** system at Pittcon. This long awaited new product is based on our patented Short Path Thermal Desorption system. This new system allows the unattended automatic analysis of 12 thermal desorption samples. See details on page 6.

S.I.S. is pleased to announce that we are now authorized dealers for ***Hewlett-Packard Consumables and Supplies***. We can now provide any of the parts that are listed in the HP Consumables and Supplies catalog as well as many other replacement parts. From replacement GC inlet parts to new electron multipliers and LC detectors we have the parts you need for your HP instruments.

S.I.S.has also become an authorized dealer for products from ***New Objective Technologies.*** New Objective Technologies is a leader in spray tips for electrospray and nanospray LC/MS. Replacement tips are available for most common LC/MS systems. Visit our website for more information on these exciting new products.

In addition to these new product lines S.I.S. is now offering ***Whatman nitrogen generators for LC/MS systems.*** We have two systems available. The small system is designed to meet the requirements of an individual LC/MS instrument, while our larger unit will handle up to three LC/MS systems.

S.I.S. now offers the widest range of ***electron multipliers*** for mass spectrometers. In addition to the Galileo multipliers that we have sold for many years, we have added the original HP electron multipliers, and multipliers from ETP and Detech. Give us a call or visit our website for additional information.

Terms and Conditions

Scientific Instrument Services (S.I.S.) continues to supply "The Mass Spec Source" newsletter as a service to our customers. Printed four times a year, it includes articles and notes on new products and procedures of interest to mass spec and GC users. Papers from all fields of scientific inquiry in which mass spectrometry and gas chromatography can play a role will be considered and subject to review. However, S.I.S. reserves the right to reject any article that is in direct competition with S.I.S. products.

Articles and Application Notes

Editorials and reviews on new instrumentation and techniques for GC/MS will be considered for publication. These articles may be any length and our Graphics Department will aid you in any way you may need.

Mass Spec Tips

Any new ideas or tips that could benefit other mass spectroscopists can be submitted for inclusion in this section.

For More Information

Anyone interested in writing in any of the areas above should contact the editor of the Mass Spec Source, at (908) 788-5550. We are always trying to improve this newsletter, if you have any suggestions please give us a call. Thanks for your continued support.

Warranty

S.I.S. does not warranty that the items described herein are usable or fit for a particular purpose. Our company makes no representation as to condition or character of the merchandise. S.I.S. will not be responsible for consequential or special damages.

"The Mass Spec Source"

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NIST 98/S.I.S. Giant Demo CD

NIST 98

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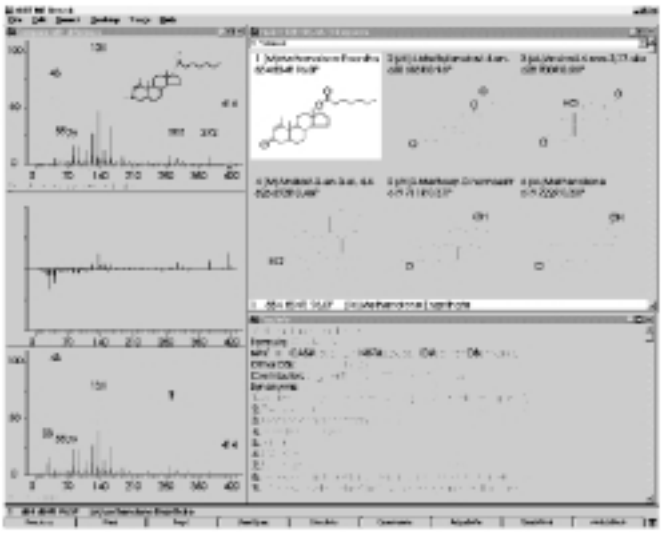

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PITTCON '99

March 7-12, 1999
Orlando, Florida

Booth #1933
Scientific Instrument Services
Also see a running demo of NIST '98

FEATURE

Precision and Reproducibility in Automated Short Path Thermal Desorption Analyses

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[1]Center for Advanced Food Technology, Rutgers University, New Brunswick, NJ

[2]Scientific Instrument Services, Inc., Ringoes, NJ

Introduction

In March 1999, Scientific Instrument Services, Inc. will introduce the new AutoDesorb™System at Pittcon in Orlando, FL.This is the automated sampling version of the SIS Short Path Thermal Desorption System. The AutoDesorb System is designed for the unattended pickup, injection, and thermal extraction of volatile and semi-volatile organics from solid, liquid, and gas samples for analysis by GC and GC-MS. The unit has the capacity to run up to 12 samples and is controlled with a Windows™based software package, developed by SIS. The entire system is designed to operate within Hewlett-Packard ChemStation™software on the HP 6890 GC. All desorption parameters such as temperature, cryo-trap cooling, and temperature ramping are fully programmable via the software interface. Furthermore, any method can be stored and changed between samples allowing great flexibility during analyses and even automated methods development! As with the earlier versions of Short Path Thermal Desorbers (TD-4), the unit operates directly on top of the Hewlett-Packard 6890 GC or HP 5973 GC/MS and is NOT permanently attached. The AutoDesorb was designed to run up to 12 samples unattended to make your GC analyses more productive and less costly.

The purpose of this article is to demonstrate the accuracy and reproducibility of the new auto loading, injecting and desorbing Short Path Thermal Desorption System. Good reproducibility will assure that the system is consistently performing its functions in a predictable manner. By automating the thermal desorption system, temperature and time operations including sample loading, injecting, desorbing, cryotrapping, and GC starting, the final GC and GC/MS analysis results will be more reproducible and accurate. Therefore in this paper the reproducibility of multiple injections of samples without an internal standard is first studied. A second study is the preparation of a calibration curve for an analyte over 3 decades of sensitivity using an internal standard. These two studies will verify the accuracy and reproducibility of the new AutoDesorb System.

Determining Reproducibility

With automated chromatography analyses it is important to have a way to measure reproducibility. This is normally calculated using a statistical parameter called relative standard deviation (RSD):

$$RSD = \frac{\sigma}{\bar{x}} \times 100\%$$

where σ= standard deviation

$\bar{x}$= sample mean(average)

This equation can be described as giving a "window" to repeatability. The standard deviation relays to the analyst how widely values are dispersed from the average value. When this number is divided by the mean (average), the ratio of the deviation to the mean is calculated. The key is to minimize the standard deviation while maximizing the value of the mean. This has the desirable effect of lowering the RSD value.

Reproducibility Using the AutoDesorb System - No Internal Standard

A standard solution of n-dodecane and n-tetradecane in methanol was prepared at a concentration of 100 ng/µl for each alkane. The GLT desorption tubes were packed with 100 mg of Tenax TA™ between glass wool plugs and flow conditioned at 300°C for 120 minutes. Then, 1 µl aliquots of the standard solution was injected directly onto the eight packed desorption tubes using the SIS desorption tube injection head. After sample loading the solvent (methanol) was removed by purging the thermal desorption tube with 120 ml of helium. The eight samples were then thermally desorbed unattended using the AutoDesorb™System in conjunction with an HP 6890 GC with a 5973 Mass Selective Detector. The analysis conditions are given in **Table 1**.

Table 1 AutoDesorb Conditions

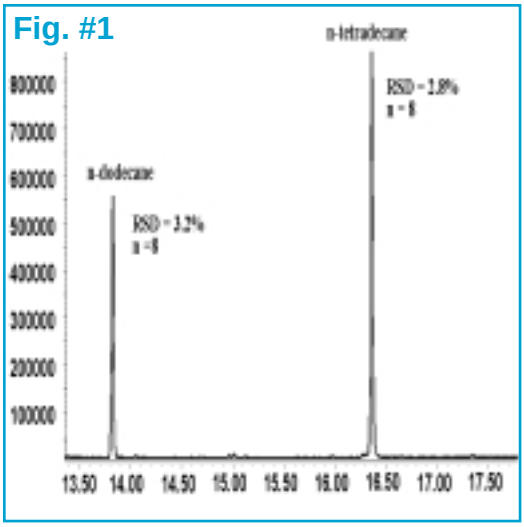
Initial Desorp.Temp.	150°C
Final Desorp. Temp.	280°C
Desorp. Ramp Rate	50°C/min
Desorp. Time	5 min.
Sample Prepurge	2min.
Initial Cryotrap Temp.	-65°C
Final Cryotrap Temp.	250°C
GC Conditions	
Column	H.P. 35ms, 60m x 250µm x .25µm
GC Column Init. Temp.	50°C
GC Column Final Temp.	250°C
GC Column Ramp Rate	10°C/min
Injection Split Ratio	40:1
Mass Spectrometer Conditions	
MS Mode	EI
Mass Range	35-350
Scans/second	2.36

The resultant chromatogram for the two hydrocarbons is shown in **Figure 1**.

The area under each of the peaks was integrated for each of the eight GC runs and the RSD was calculated as described previously. The results of the analysis are in **Table 2**.

The errors observed are the result of an accumulation of error sources including sample loading, thermal desorption

Fig. #1



efficiency, GC injection port stability, split accuracy, as well as chromatography, GC stability and MS reproducibility. The results observed are quite good especially considering no internal standard was used. In general a relative standard deviation of 0 -10% is considered acceptable. An internal standard would produce better results by minimizing any errors generated by sample preparation, thermal desorption, and sample splitting.

Table 2	Dodecane
Sample mean (TIC area counts)	8669130.2
Standard Deviation	276901.7
RSD	3.2%
Tetradecane	
Sample mean (TIC area counts)	12852783
Standard Deviation	358452.7
RSD	2.8%

Generating a Calibration Curve With Internal Standards

In this experiment eight solutions of butylated hydroxytoluene (BHT) were prepared in methanol using d-20 BHT as the internal standard. The concentration of BHT increased from 5 ng/µl to 100 ng/µl while the concentration of d-20 BHT, the internal standard, was constant at 138 ng/l. Desorption tubes packed with Tenax™TA were prepared as described previously. The next step was to inject 1µl aliquots of each solution onto each of eight Tenax packed GLT tubes. The tubes were then purged with 120 ml of helium to remove the methanol solvent. The samples were then analyzed unattended using the AutoDesorb System in conjunction with an HP 6890 GC with a 5973 MSD. The details of the analysis conditions are given in Table 3. A representative chromatogram is shown in **Figure 2**.

After the chromatograms were generated the integrated area counts from the BHT relative to the internal standard were calculated and a calibration curve was generated. The result is a plot of BHT levels on the x-axis while the ratio of BHT detector counts to d-20 BHT detector counts is on the y-axis. This yields a linear response factor curve. The linearity of sample transfer is quantified using linear regression on the data points. If the y-intercept is sufficiently close to zero, we assume that an efficient transfer occurred. A graph of the calibration curve obtained from the above experiment is shown in **Figure 3**. From the equation for the line, we see that the R^2 (correlation coefficient) value of .999 is acceptably close to 1 indicating a near perfect straight line.

Fig. #2

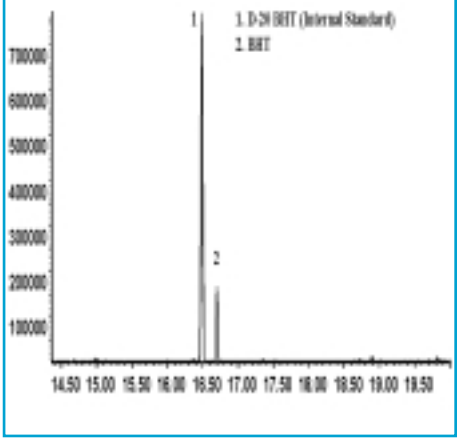
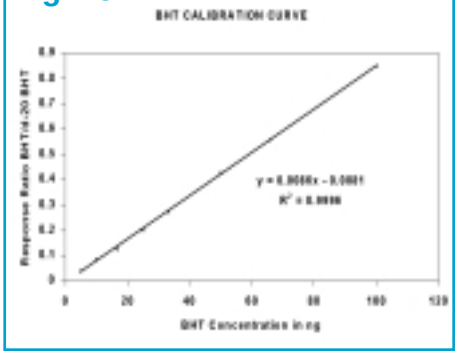


Fig. #3



Conclusion

The accuracy and reproducibility of the AutoDesorb System has been verified. Multiple injections of identical samples produced a relative standard deviation (RSD) of better than 3.5% for the samples studied without the use of an internal standard. In addition the generation of a calibration curve with an internal standard had a correlation coefficient of 0.999 over 3 decades of sample size. The new Automated Short Path Thermal Desorption System (AutoDesorb) is an invaluable tool for increased productivity using the thermal desorption technique while also producing accurate and reproducible results.

For additional information, stop by the SIS booth #1933 at Pittcon or visit our website at http://www.sisweb.com.

AutoDesorb™ is a trademark of Scientific Instrument Services, Inc.

ChemStation™ is a trademark of Hewlett-Packard Corporation

Tenax™TA is trademark of Buchem Inc.

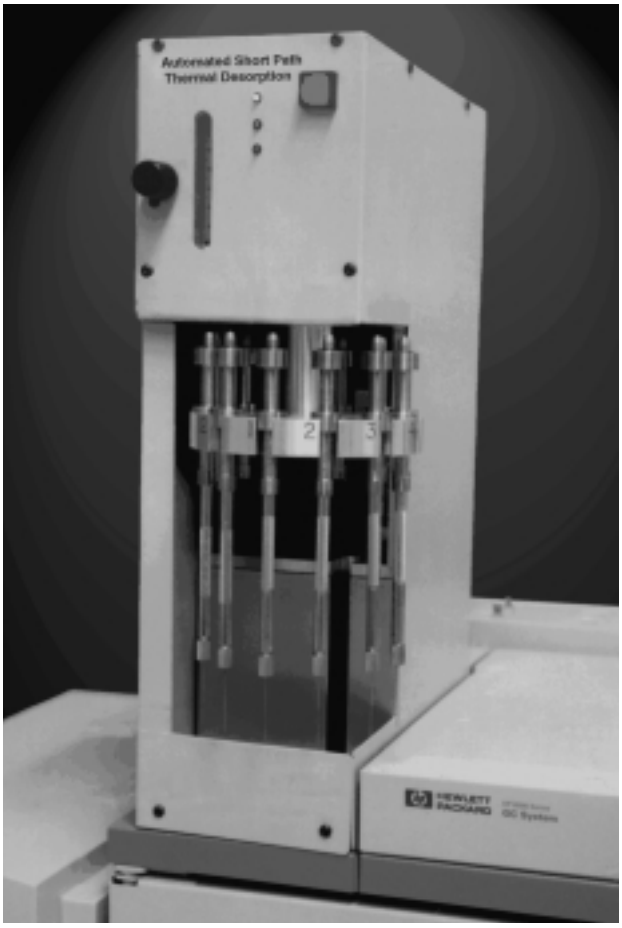
Windows™ is trademark of Microsoft Corporation

Table 3	AutoDesorb Conditions
Initial Desorption Temperature	150°C
Final Desorption Temperature	280°C
Desorption Ramp Rate	50°C/min
Desorption Time	5 min.
Sample Prepurge	2min.
Initial Cryotrap Temperature	-65°C
Final Cryotrap Temperature	250°C
GC Conditions	
Column	Hewlett-Packard 35ms, 60m x 250µm x .25µm
GC Column Initial Temperature	50°C
GC Column Final Temperature	250°C
GC Column Ramp Rate	10°C/min
Injection Split Ratio	40:1

AutoDesorb

NEW - Automated Short Path Thermal Desorption- Introduced at Pittcon '99

The PC Controlled Automated Short Path Thermal Desorption System is designed for the continuous and unattended thermal extraction of volatile and semi-volatile organics from solid, liquid and gas samples for analysis by GC and GC/MS.



Features

- Automated Thermal Desorption Sample Introduction System
- Carousel holds 12 samples for unattended operation
- Uses the SIS 'Short Path Thermal Desorption' technology
- Designed to Operate with the HP-6890 GC or HP-5973 GC/MS
- PC Controlled Operation - User-friendly Graphical Interface
- Fully integrated with the HP ChemStation Software
- Permits the analysis of volatile and semi-volatile organics
- P&T Thermal Desorption and Direct Thermal Extraction
- No "memory effect" - individual flow path for each sample
- Mounts overtop the GC injection port
- Not permanently installed to the GC - easily removable
- Desorb samples at temperatures from room temperature up to 450°C, either isothermal or at ramp rates up to 100°C per minute
- Glass Lined stainless steel desorption sample tubes are inert and strong for sample handling
- GC Cryo-Trap Accessory for cryo trapping volatiles during desorption
- Lowest cost Automated TD System on the Market

AutoDesorb™ - Automated Short Path Thermal Desorption

The new AutoDesorb System is the automated sampling version of the patented SIS Short Path Thermal Desorption System. It is designed for the automatic and unattended pickup, injection and thermal extraction of volatile and semi-volatile organics from solid, liquid and gas samples for analysis by GC and GC/MS.

The system is operated and controlled by a PC Windows based software package developed by SIS. The AutoDesorb System has been developed to operate within the Hewlett-Packard ChemStation software package on the HP 6890 GC or HP 5973 GC/MS to provide for a seamless integration of the HP GC or MSD with the SIS AutoDesorb System.

The carousel enables up to 12 samples to be loaded for the unattended analysis by thermal desorption. The carousel is powered by a stepper motor to position each sample for pickup and injection into the GC.

The desorption tube with samples for analysis is fitted with a needle and is attached to a connecting tube. Each connecting tube is sealed at the top to prevent sample contamination. The connecting tube enables the pickup of the desorption tubes and air tight sealing of the tubes during the thermal desorption process. Each sample therefore, has an individual flow path to eliminate the possibility of sample cross-contamination.

A door is closed around the carousel and locked during the desorption process. When signaled by the HP ChemStation software, the system automatically locates, loads and analyzes the correct sample using a predefined method. Samples can be run in any sequence - this is controlled by the vial number selection in the HP ChemStation software. Additional samples can be loaded to the carousel between runs and during the GC run for continuous sample analysis.

The AutoDesorb System and Hewlett-Packard HP ChemStation Software

The AutoDesorb software is fully integrated with the HP ChemStation software. Therefore sample information must only be entered once. All data such as sample name and vial number are entered into the HP ChemStation window for the analysis of samples. All thermal desorption methods are saved as part of HP Method file.

PC Thermal Desorption Windows and Screens

The AutoDesorb software system provides for the visualization and control of all the AutoDesorb operations including sample purging and injecting times, desorption temperatures and times, GC start times and Cryo-Trap heating and cooling parameters. A user-friendly Graphical Interface displays the pictorial as well as digital status of the thermal desorption system operation and GC Cryo-Trap operation.

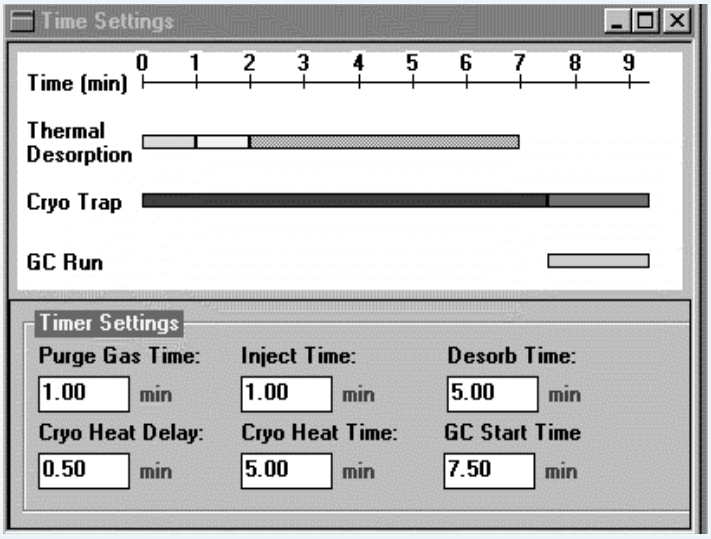
The first of these windows is the Thermal Desorption Status Window. This screen displays graphically and digitally the current status of the AutoDesorb system. This display is for information and status only. The graphics are updated immediately as the system status changes.

The second screen is the Temperature Settings Screen. On this screen are entered changes in the thermal desorption and cryo-trap parameters. Method setup is done here as well as monitoring the current status of the thermal desorption block heaters and cryo-trap temperatures.

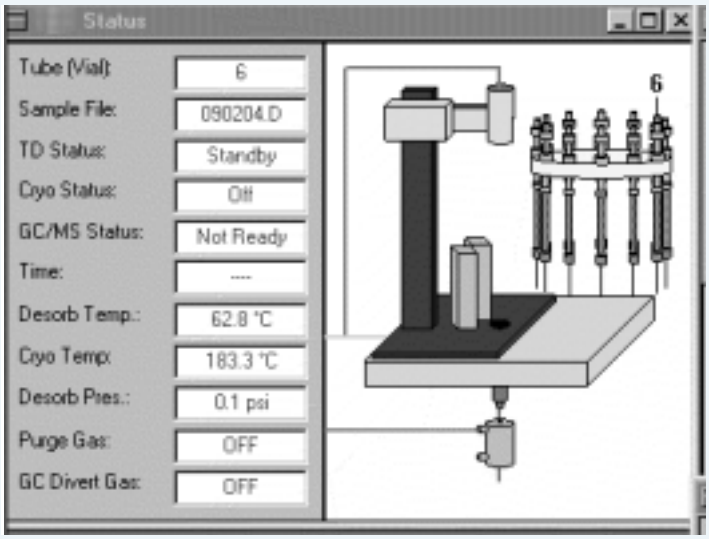
The third screen is the Time Settings Screen. Method setup is done here as well monitoring the current status of Purge Gas Time, Injection Time, Desorption Time, Cryo-Trap cool and heat times and the GC Start Time. The GC start time can be at set at anytime from the time of injection until after the desorption process is complete.

The fourth screen is the Sample Log Screen which maintains a log of all samples run as well as a sample error report. This report can be printed out to report the samples that were analyzed.

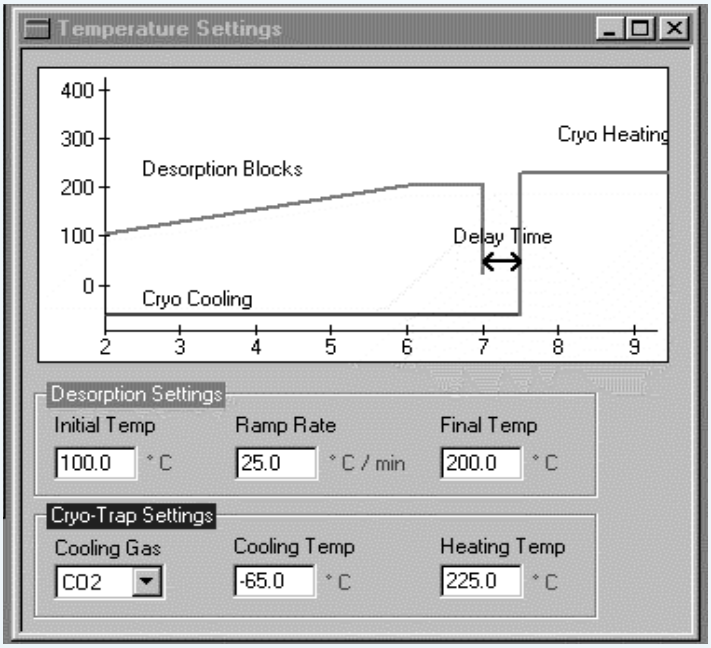
For additional details visit the S.I.S.web site at http://www.sisweb.com.



Time Settings Window In AutoDesorb System



Thermal Desorption System Status Window



Temperature Settings Window in AutoDesorb

Explore
The World of Mass Spec

S.I.S. invites you to explore our web site.

http://www.sisweb.com

The S.I.S.Web Site is continually expanding. Below are a few of the latest additions:

- *Application Notes (over 75 online)*
- *NIST 98*
- *AutoDesorb*

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