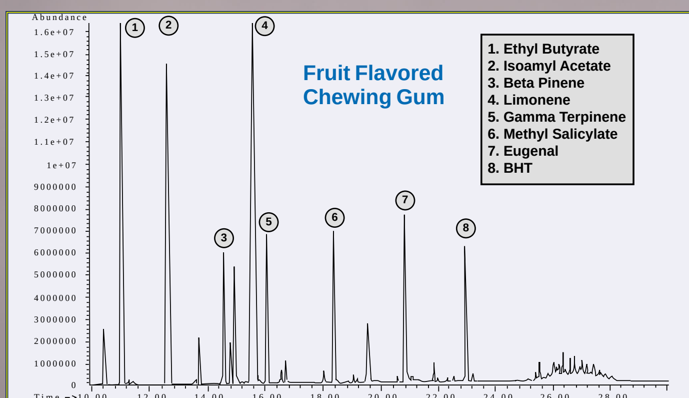
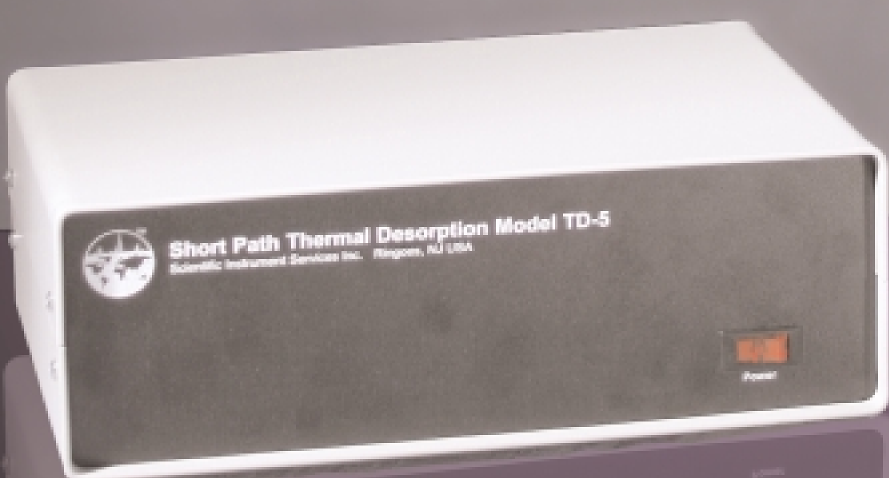
SHORT PATH THERMAL DESORPTION MODEL TD-5

A versatile technique for introducing volatile and semi-volatile organics in solid, liquid and gas matrices into a gas chromatograph (GC).



Technique: Direct Thermal Extraction of Solid Samples
Sample: 8.3 mg. of thinly sliced gum was placed inside the GLT desorption tube
Thermal Desorption: Block temperature: 100°C, Purge flow: 2.0 ml/min He,
Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D., 0.25 u film, -40° to 280°C at 10° /min.



Scientific Instrument Services Inc.

System Overview

S.I.S. TD-5

The new SIS TD-5 is a "short path" thermal desorption system. It provides unattended thermal extraction (thermal desorption) and injection of volatile and semi-volatile organics from solid, liquid and gas samples into a gas chromatograph (GC). The system delivers samples into the GC along an optimal "short path," thereby eliminating transfer lines and cross-sample contamination issues found in previous systems.

A sample for analysis is introduced into a desorption tube, which is then fitted with a needle and attached to the TD-5 Desorption Unit. The Desorption Unit sits over the GC injection port, where, upon signal from the PC software, it automatically injects and desorbs the sample into the GC injection port and column using the temperatures and times prescribed in the desorption method.

The TD-5 System consists of the Desorption Unit (which contains the sample analysis hardware), an Electronics Console (which contains a microprocessor and power supply), and a Windows-based Thermal Desorption software (which provides control and monitoring of the system). This software can be fully integrated with selected GC data systems (e.g. Agilent ChemStation), or it can operate fully independently from the GC data system. Various accessories are available such as the Cryo-Trap, which cryogenically cools and rapidly heats the GC column head for improved chromatographic resolution.

Features

Analytical quality

- High sensitivity thermal desorption and direct thermal extraction of solid, liquid, and gas samples
- No memory effects – each sample has its own independent and "short" flow path from sample to GC, eliminating transfer lines as a source of contamination
- Stainless steel sample tubes (glass-lined GLT or silco treated) are inert to samples and strong for sample handling and transporting.
- Rapid cryogenic (CO_2 or LN_2) cooling and heating of the GC column head for improved chromatographic resolution (optional Cryo-Trap accessory).

Functions/Convenience

- Mounts overtop most GC injection ports—easily removable and transferable.
- Automatic injection and desorption of sample into GC (pneumatic driven injector).
- **NEW -** Windows-based software for control and monitoring of the system
- **NEW -** Seamless integration into Agilent ChemStation (optional)

Specifications

- **NEW -** Desorption: temp range: 20° to 400°C, either isothermal or temperature programmed. Max heat rate: 100°C/min.
- Cryo-trap: min temp: -70°C (CO2) or -180°C (LN2). Max temp: 400°C. Max heat rate: 10°C/sec.

Multi-sample versions of our Thermal Desorption System are also available.

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Theory of Operation

The Thermal Desorption Unit sits directly on top of the injection port of most GC's (Fig.2), where it provides direct desorption of both volatile and semi-volatile samples into the GC injection port and column. The system delivers samples into the GC along an optimal "short path," thereby eliminating transfer lines and cross-sample contamination issues found in previous systems.

Samples to be analyzed are collected into stainless steel desorption tubes (either glass lined GLT or silco-treated)**(Fig.1)**. A solid sample of interest may be packed directly into the tube and subjected to direct thermal desorption. Alternately, the tube may be packed with adsorbent resin such as Tenax™ TA or activated carbon for indirectly trapping analytes from liquid or air samples.

Figure 2 - Desorption Unit mounted on an Agilent 6890 GC

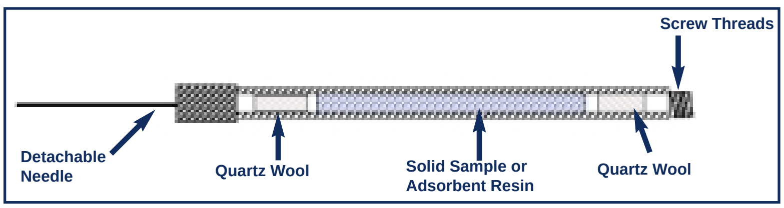


Figure 1 - Desorption (sample) tube

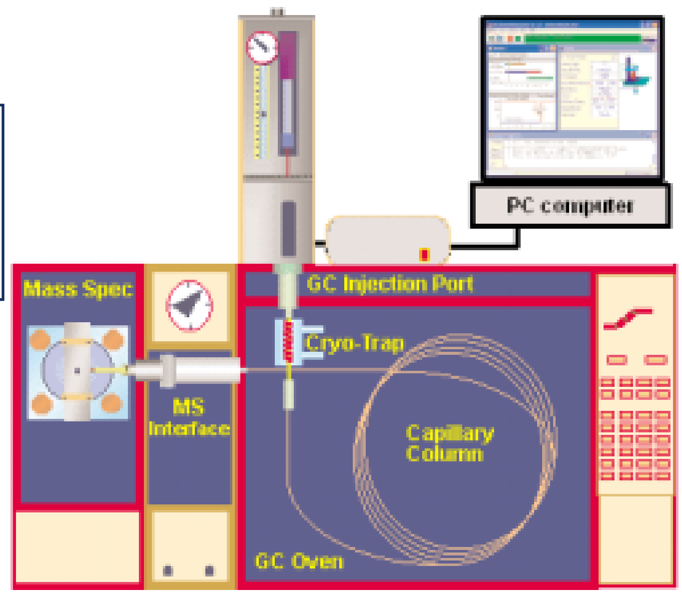
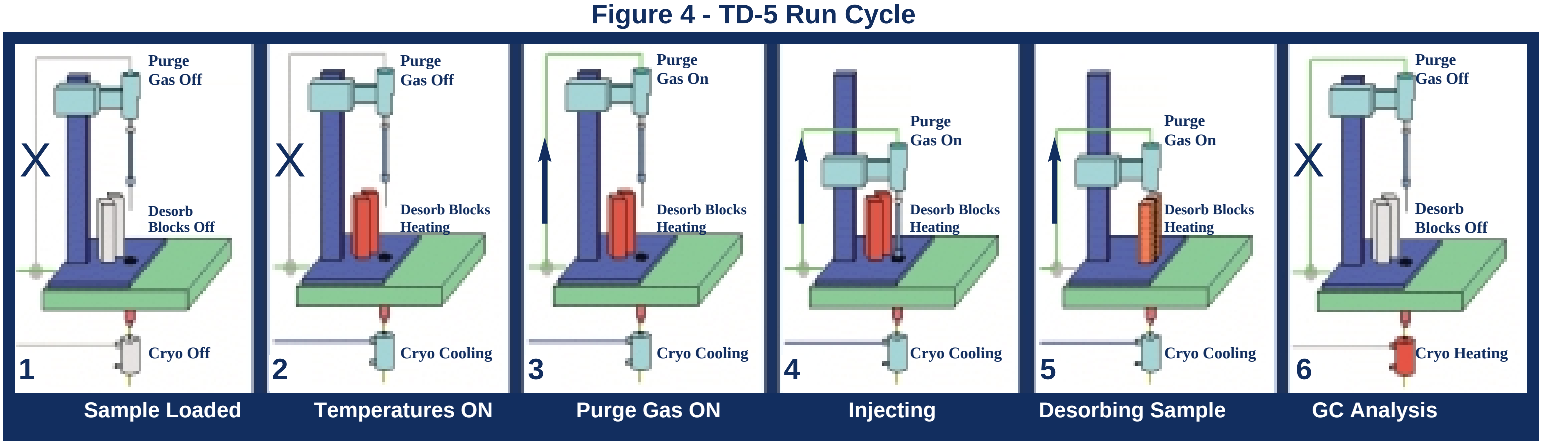


Figure 3 - Schematic of TD-5 System Connected to GC

To analyze a prepared desorption tube (Fig.4), a syringe needle is first attached to the desorption tube, which is then attached to the connector tube on the Desorption Unit **(#1)**. The desorption blocks heat to initial temperature **(#2)**. Carrier gas is sent through the desorption tube for an initial purge time **(#3)**. The system then injects the desorption tube into the GC injection port **(#4)** where flows are readjusted as required by the method of analysis, i.e. split/splitless, etc. The hinged heating blocks close around the desorption tube **(#5)** to ballistically heat the sample tube, optionally with a temperature ramp. The combination of the heat applied and the carrier gas flow through the desorption tube will purge the desired components into the GC injection port and onto the front of the GC column. To obtain sharp chromatographic peaks when desorption typically lasts 5 to 15 minutes, it can be desirable to focus desorbed components at the head of the GC column. This focusing can be improved by installing a Cryo-Trap, which cryogenically cools the head of the GC column (with liquid CO_2 or LN_2) during desorption to trap desorbed analytes. After desorption, the Cryo-Trap rapidly heats to volatilize the trapped analytes and quickly release them through the GC column for separation **(#6)**.

Figure 4 - TD-5 Run Cycle

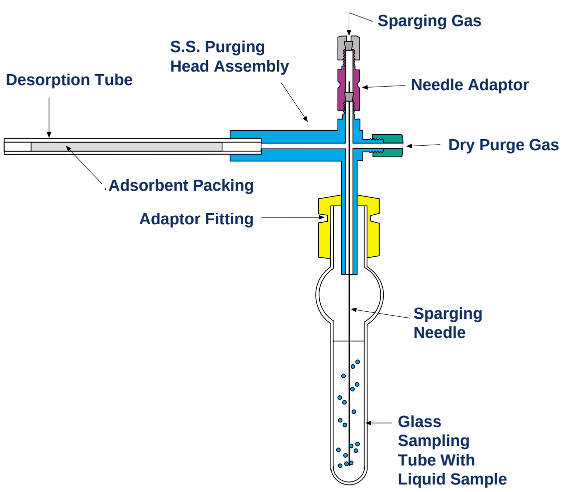


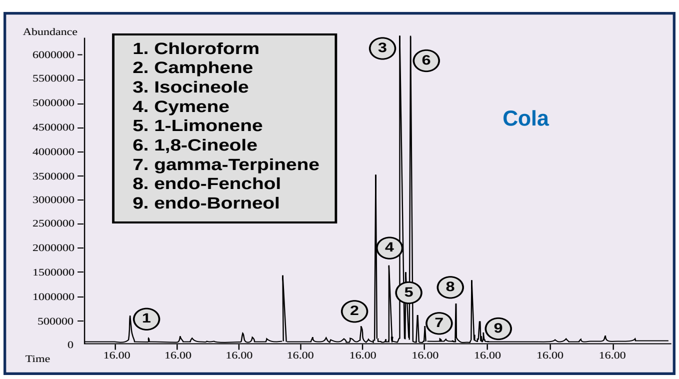
Analysis Methods

A number of analysis methods are available including Purge and Trap (for liquid samples), Direct Thermal Extraction (for solid samples), and Air Sampling Methods (for gas samples).

Purge and Trap System

The S.I.S. Purge and Trap System extracts volatiles from a wide diversity of liquid matrix samples such as flavors, fragrances, off-odors, and manufacturing by-products. A liquid sample is placed in a glass sampling tube (optionally heated) and then purged by bubbling high purity Helium or Nitrogen through the sample. The purged volatiles get trapped onto a Tenax or other suitable resin in an attached desorption tube, which may later be placed in the TD-5 Desorption Unit to complete the analysis.

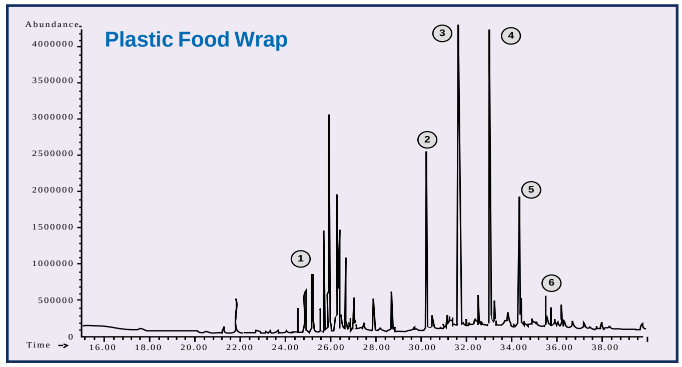




Technique: Purge and Trap of Volatiles in Liquid Samples
Sample: 25 ml of a Carbonated Cola was purged with 150 ml of high purity He at 15 ml/min and a dry purge of 15 ml/min. The volatiles were trapped on Tenax traps.
Thermal Desorption: Block temperature: 150°C, Purge flow:2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D., 0.25 u film, -40° to 280°C at 10°/min.

Direct Thermal Extraction

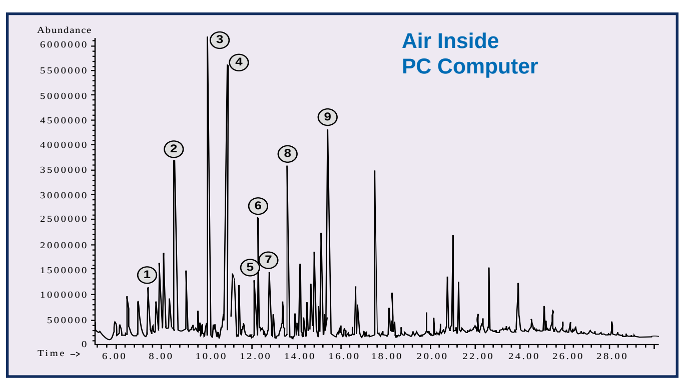
The Direct Thermal Extraction technique permits the direct analysis of solid samples without any prior solvent extraction or other sample preparation. This technique is useful for the analysis of a wide variety of low moisture content solid samples including vegetation, food products, pharmaceuticals, building materials, forensic samples and packaging products. A solid sample between 1 and 500 milligrams is placed directly inside the desorption tube between two glass wool plugs. The desorption tube is then attached to the TD-5 Desorption Unit for analysis. In the analysis, the sample is purged with carrier gas to remove all traces of oxygen and injected into the GC injection port. By the proper selection of the desorption block temperature, the number and molecular weight distributions of components in the samples can be selected.



Technique: Direct Thermal Extraction of Solid Samples
Sample: 2.0 sq. in. piece of a clear plastic food wrap was placed inside the GLT desorption tube
Thermal Desorption: Block temperature: 100°C, Purge flow: 2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D., 0.25 u film, -40° to 280°C at 10° / min.

Environmental Air Testing

Volatiles present in the environmental air can be readily analyzed using the TD-5 System. Air to be analyzed is pumped through a desorption tube packed with an adsorbent resin such as Tenax. A small air sampling pump is used to pass air through the tube by pulling so as not to add any contaminants from the pump itself or the connecting tubing. A variety of fittings are available to adapt the desorption tube and permit air sampling at 10 to 100 milliliters per minute. The total amount of air sampled is dependent on the concentrations of the volatiles in the air, the breakthrough volume for the volatiles in the adsorbent resins and the detection limits of the GC or GC/MS.



Technique: Air Sampling of Volatiles from Adsorbent Traps
Sample: A 6.0 liter sample of air from inside a two year old PC Computer was pumped through a Tenax GLT desorption trap.
Thermal Desorption: Block temperature: 200°C, Purge flow:2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D., 0.25 u film, -40° to 280°C at 10°/min.

Software

The SIS Thermal Desorption Software included provides visualization and control of all thermal desorption operations through a user-friendly graphical user interface. The user may configure system and thermal desorption methods settings (e.g. times and temperatures) as well as monitor the system status (e.g. temperatures and pressures) during a run.

The Thermal Desorption Software can be used fully independent of the GC data system, or it can be fully integrated with certain GC data systems (e.g. Agilent ChemStation™). When the software is integrated with ChemStation, thermal desorption parameters get stored directly into the current ChemStation™ method so that GC and thermal desorption method settings are linked. The run is initiated from ChemStation™, and methods may be changed between samples to provide different desorption parameters for different samples. For example, a separate high temperature bakeout or blank method can be setup and run between samples to assure the system is clean for the next sample.

Features

- Configure system parameters and limits
- Configure thermal desorption methods including desorption and cryo-trap times and temperatures.
- Monitor system status including actual times, temperatures, pressures and status during a run.
- Log sample status and errors.
- View online help system.
- RS-232 (optional USB bridge) communication with TD-5 instrument.
- Optional Agilent ChemStation integration for method load/save and sample run.
- The Cryo-Trap may be operated separately for syringe injections.

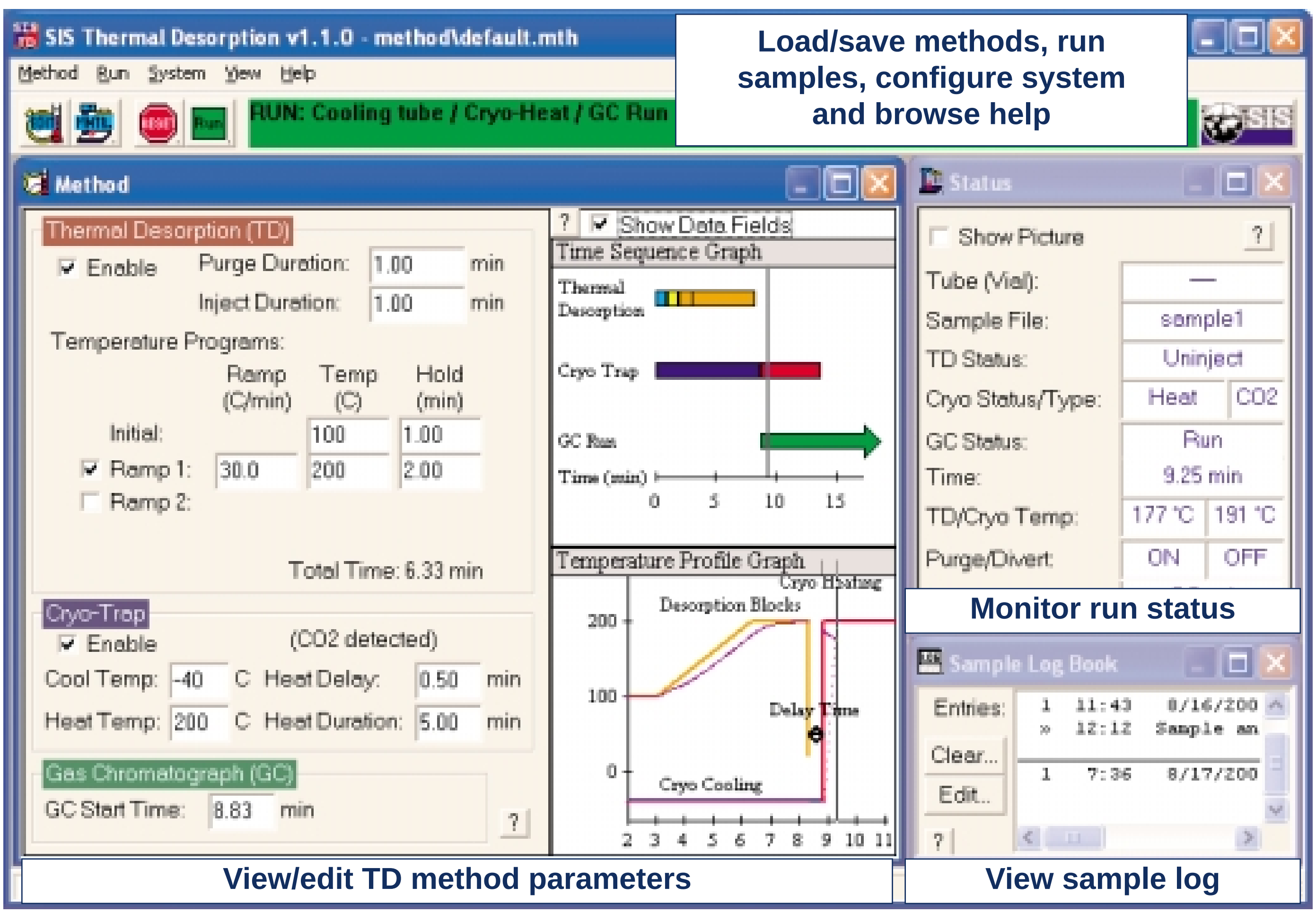


Figure 5 - SIS Thermal Desorption Software

Applications of the S.I.S. Short Path Thermal Desorption System

For additional information call Scientific Instrument Services, Inc. at 908-788-5550 or visit our Short Path Thermal Desorption web page: http://www.sisweb.com/sptd/

Thermal Desorption Applications

Environmental Air Analysis
Indoor Air Pollution
Flavor and Fragrance Analysis
Off-odor/Off-flavor Analysis
Forensic Arson Analysis

Residual Gas, Solvents and Chemicals in:

Pharmaceuticals
Packaging Materials
Building Products
Food Products
Natural Products

Direct Thermal Extraction of:

Plastics
Synthetic Fibers and Other Materials
Spices
Natural Products
Pharmaceuticals
Finished Products for Solvent Residues

APPLICATION NOTES:

1. Determination of Off-Odors and Other Volatile Organics in Food Packaging Films by Direct Thermal Analysis-GC-MS. (781301)
2. Detection of Arson Accelerants Using Dynamic Headspace with Tenax Cartridges Thermal Desorption and Cryofocusing (781302)
3. Indoor Air Pollution (781303)
4. Direct Analysis of Spices and Coffee (781304)
5. Direct Analysis Using the Short Path Thermal Desorption System: A new technique to permit the analysis of volatiles and semi-volatiles in solid samples without solvent extraction. (781305)
6. Direct Thermal Analysis of Plastic Food Wraps Using the Short Path Thermal Desorption System. (781306)
7. Chemical Residue Analysis of Pharmaceuticals Using The Short Path Thermal Desorption System. (781307)
8. Detection of Volatile Organic Compounds in Liquids Using the Short Path Thermal Desorption System. (781308)
9. Methodologies for the Quantification of Purge and Trap Thermal Desorption and Direct Thermal Analyses. (781309)
10. Quantification of Naphthalene in a Contaminated Pharmaceutical Product by Short Path Thermal Desorption (781310)
11. Flavor/Fragrance Profiles of Instant and Ground Coffees by Short Path Thermal Desorption. (781311)
12. Identification of the Volatile and Semi-Volatile Organics in Chewing Gums by Direct Thermal Desorption. (781312)
13. Detection and Quantification of PNA's and PCB's in Soil by Direct Thermal Desorption (781313)
14. Analysis of Carbonated Beverages by P&T Thermal Desorption Quantification of Limonene and Cymene (781314)
16. Analysis of Indoor Air and Sources of Indoor Air Contamination by Thermal Desorption. (781316)
17. Identification of Volatile Organics in Wine over Time (781317)
18. Determination of Volatile Organic Compounds in Mushrooms (781318)
19. Design and Application of the SIS GC Cryo-Trap (781319)
20. Using Direct Thermal Desorption to Assess the Potential Pool of Styrene and 4-Phenylcyclohexane in Latex-Backed Carpets. (781320)
21. Detection and Identification of Volatile and Semi-Volatile Organics in Synthetic Polymers Used in Food and Pharmaceutical Packaging. (781321)
22. Comparison of Volatile Compounds in Latex Paints (781322)
23. Fragrance Qualities in Colognes (781323)
24. Selection of GC Guard Columns for Use with the GC Cryo-Trap (781324)
25. Flavor and Aroma in Natural Bee Honey (781325)
26. Volatile Organics Present in Recycled Air Aboard a Commercial Airliner (781326)
27. Analysis of Volatile Organics in Soils by Automated Headspace GC (781327)
28. Analysis of Volatile Organics in Latex Paints by Automated Headspace Sampling and GC-Cryo-Focusing (781328)
29. Analysis of Volatile Organics in Oil Base Paints by Automated Headspace Sampling and GC Cryo-Focusing (781329)
30. Comparison of Cooking Oils by "Direct Thermal Extraction" and Purge and Trap GC/MS (781330)
31. Volatile Organic Composition in Several Cultivars of Peach (781331)
32. Selection and Use of Adsorbent Resins for Purge and Trap Thermal Desorption Applications (781332)
33. Changes in Volatiles in Milk Over Time (781333)
34. Selection of Thermal Desorption and Cryo-Trap Parameters in the Analysis of Teas
35. Volatile Organic Composition of Cranberries
36. Identification of Volatile Organic Compounds in a New Automobile
37. Volatile Organic Emissions from Automobile Tires
38. A New Micro Cryo-Trap for Trapping of Volatiles at the front of a GC Capillary Column
39. Comparison of Sensitivity of Headspace GC, Purge and Trap Thermal Desorption and Direct Thermal Extraction Techniques for Volatile Organics
40. Composition of Septa by Direct Thermal Extraction
41. Hydrocarbon Production in Pine by Direct Thermal Extraction
42. The Influence of Pump Oil Purity on Roughing Pumps
43. Volatile Organic Composition in Blueberry
54. Identification of Volatile Organic Compounds in Office Products
55. Seasonal Variation in Flower Volatiles
57. Aroma Profiles of Various Lavandula Species
58. Direct Probe Analysis and Identification of Multi-component Pharmaceutical Samples via Electron Impact MS
60. Programmable Temperature Ramping of Samples Analyzed via Direct Thermal Extraction GC/MS
63. Organics in Printer Toners using Thermal Desorption
64. Comparison of Various GC/MS Techniques for the Analysis of Black Pepper
65. Determination of Ethylene by Adsorbent Trapping and Thermal Desorption GC
71. Flavor Profile Determination of Rice Samples Using Thermal Desorption
73. The Analysis of Perfumes and Their Effect on Indoor Air Pollution
75. An Apparatus for Sampling Volatile Organics from Live Plant Material Using Short Path Thermal Desorption
77. The Determination of Volatile Organics in Vacuum System Components
79. Volatile Organic Compounds From Electron Beam Cured and Partially Electron Beam Cured Packaging Using Automated Short Path Thermal Desorption
80. Design, Development and Testing of a Microprocessor Controlled Automated Short Path Thermal Desorption Apparatus

Also: huge collection of breakthrough volumes for 200 organic analytes for 6 adsorbent resins including Tenax TA, Tenax GR, Carbotrap, Carboxen 569, and Glass beads as a function of temperature. This data is useful for designing methods for collecting and desorbing samples using thermal desorption systems.

Visit **www.sisweb.com/sptd** for online articles and documentation

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