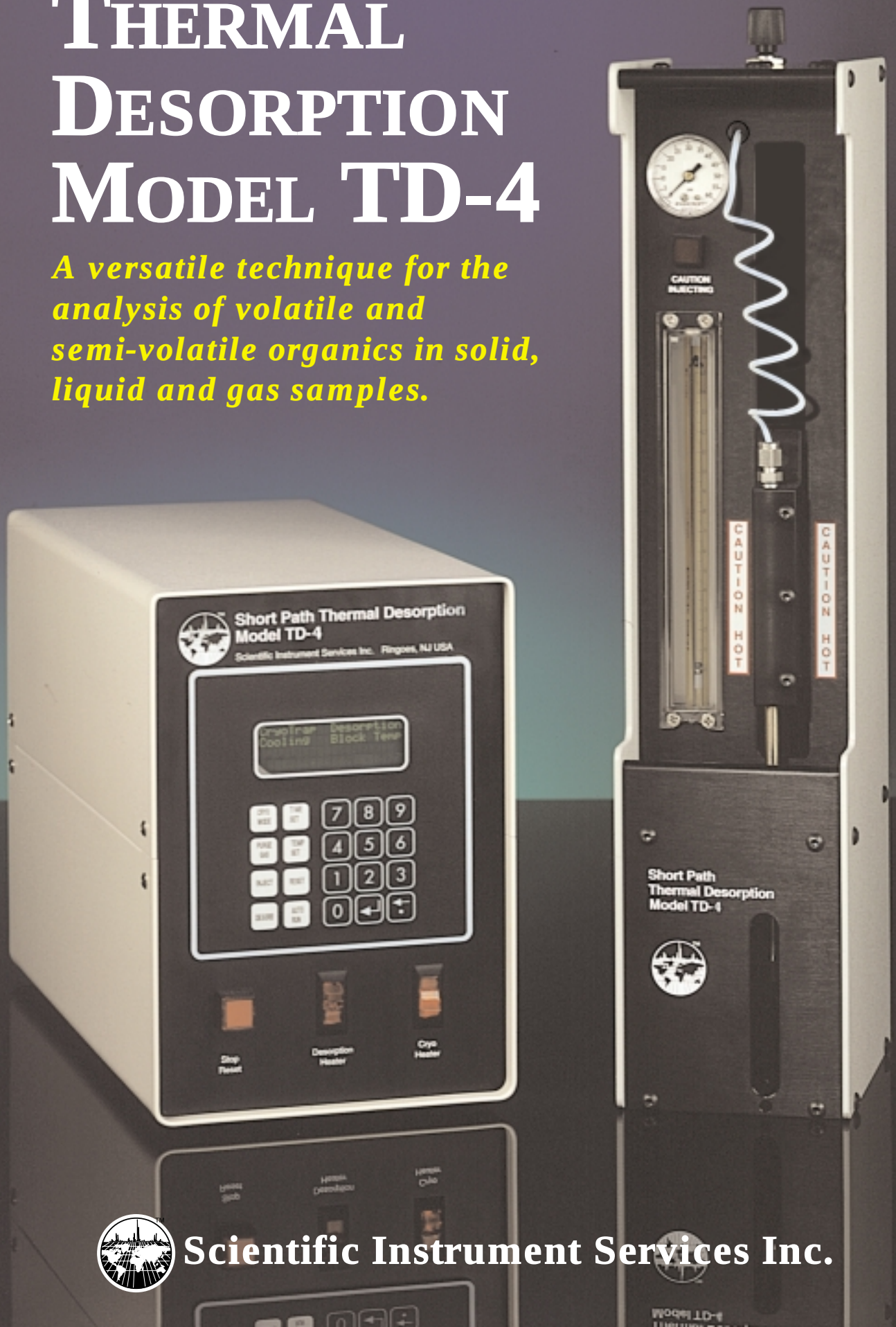
Short Path Thermal Desorption Model TD-4

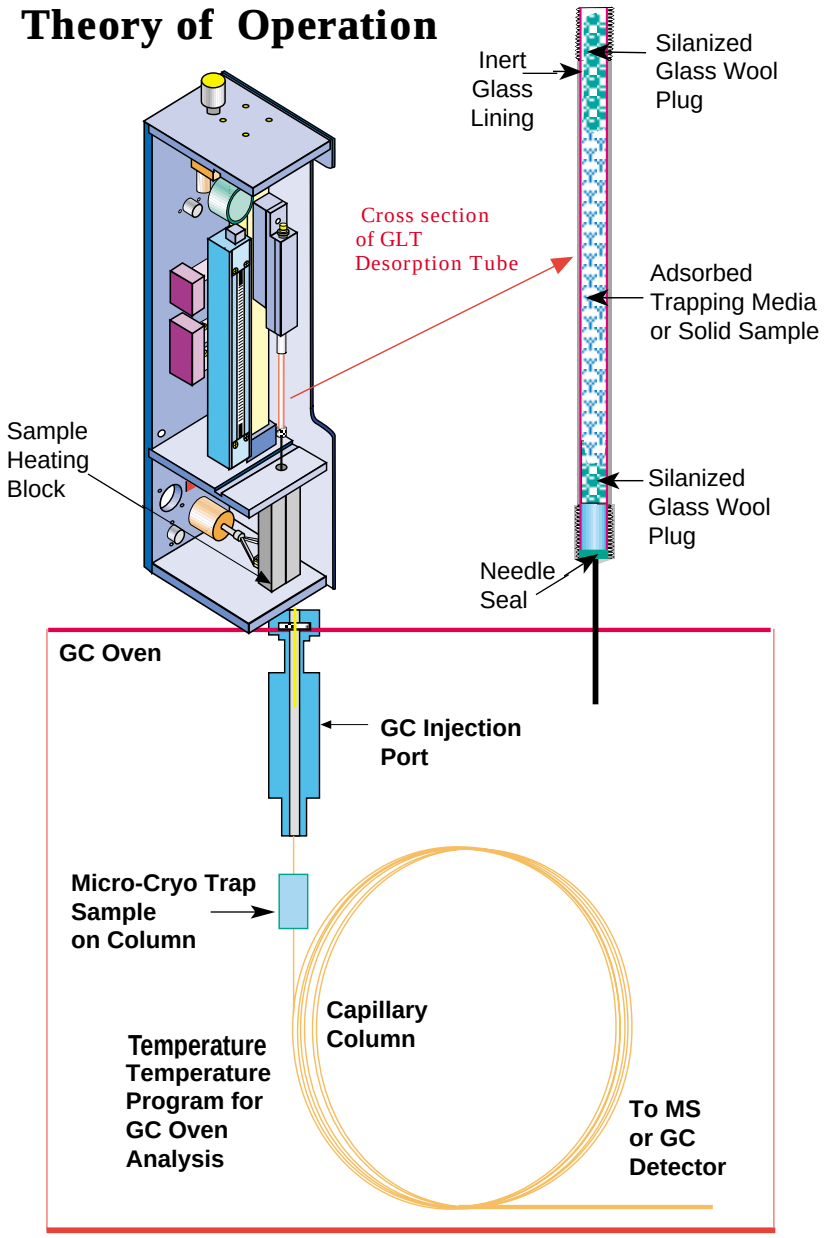
A versatile technique for the analysis of volatile and semi-volatile organics in solid, liquid and gas samples.



Scientific Instrument Services Inc.

This new technique permits the direct thermal desorption of volatiles into the GC injection port.

Theory of Operation



The Thermal Desorption Unit sits directly on top of the injection port of most GC's, where it is utilized for the direct desorption of both volatile and semi-volatile samples into the GC injection port and column. Due to its "short path" of sample flow, this new system overcomes the short comings of previous desorption systems by eliminating transfer lines (which are easily contaminated by samples) and by providing for the optimum delivery (and therefore maximum sensitivity) of samples to the GC.

"Due to its "short path" of sample flow, this new system overcomes the short comings of previous desorption systems by the elimination of transfer lines"

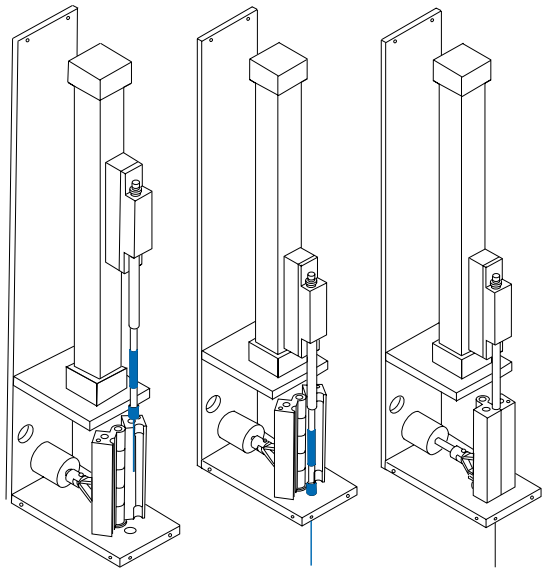
Samples to be analyzed are collected on Glass Lined Stainless Steel (GLT) Desorption Tubes containing an adsorbent resin such as Tenax™ TA or activated carbon. Alternatively, analytes of interest can be packed into the tube and subjected to direct thermal desorption.

"Volatiles can be thermally desorbed from adsorbent resins, or solid samples can be thermally sparged directly into the GC injection port."

When ready to be analyzed, a syringe needle is attached to the desorption tube which is then attached to the connector tube on the Desorption Unit.

The Electronics Console processor is activated to inject the desorption tube with needle attached (INJECTING). The desorption tube will pass through the opening in the middle plate of the Desorption Unit base to position the desorption tube in proper alignment with the GC injection port and the normally open desorption block assembly.

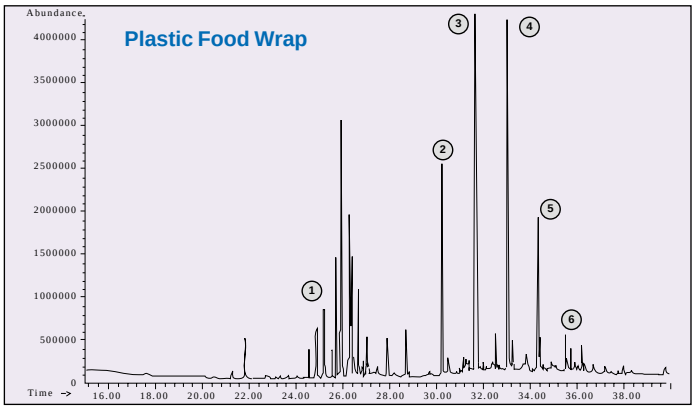
When injection is complete (INJECTION COMPLETE), the flows are readjusted as required by the method of analysis, i.e. split/splitless, etc.The hinged heating blocks close around the desorption tube (HEAT & DESORB). The desorption tube will ballistically heat up to the set temperature or the temperature program ramp for the heater blocked will begin. 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. The TD-4 permits the control (both heating and cooling) of the Micro Cryo-Trap™ for the trapping of volatiles at the front of the GC column inside the GC oven. This enables the desired components to be collected in a narrow band on the front of the GC column over a long period of time (5 to 15 minutes). When the sample has been fully desorbed into the GC column, temperature programming is commenced to volatilize the organics and to elute and separate them into the desired components.



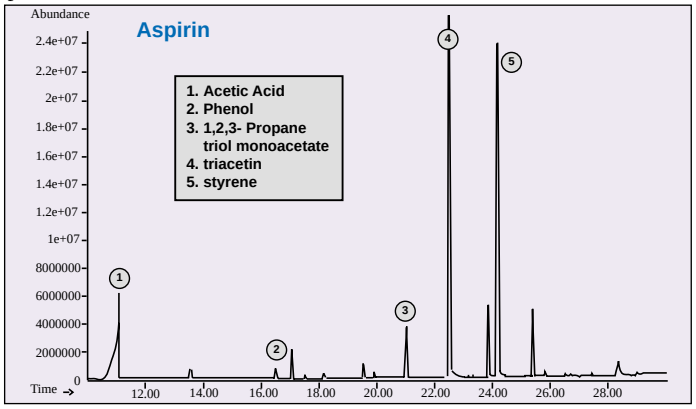
Injecting **Injection Complete** **Heat & Desorb**

Direct Thermal Extraction Applications

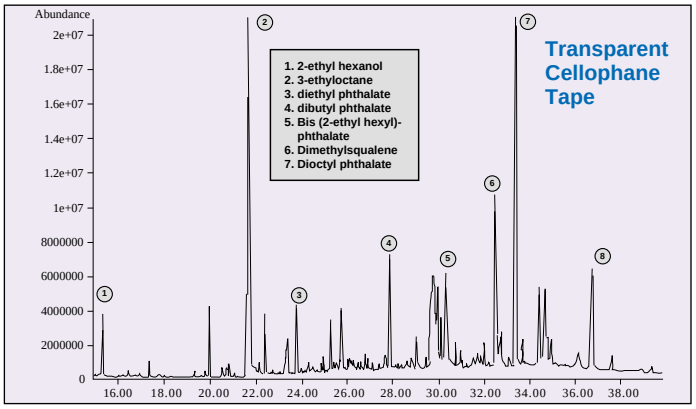
The technique of Direct Thermal Extraction permits the analysis of solid samples without any prior solvent extraction or other sample preparation. Solid samples between 1 and 500 milligrams are placed directly inside the GLT desorption tube between two glass wool plugs. The desorption tube needle is attached to one end of the sample tube. The desorption tube with sample enclosed is then attached to the Short Path Thermal Desorption System, purged with carrier gas to remove all traces of oxygen and injected into the GC injection port. The preheated heater blocks are closed around the desorption tube thereby permitting the thermal extraction of the volatiles and semi-volatiles present into the GC injection port. After the desorption is complete, these components, which have been trapped on the front of the GC column, are eluted and separated via a temperature program of the GC oven. 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. By the proper selection of the desorption block temperature, the number and molecular weight distributions of components in the samples can be selected.



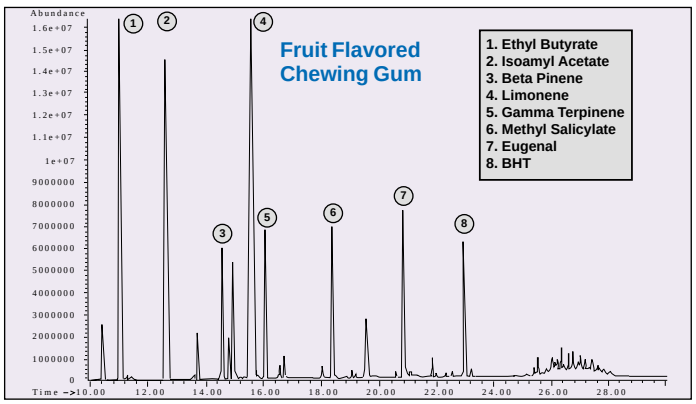
Technique: Direct Thermal Extraction of Solid Samples
Sample: 2.0 sq. in. piece of a clear plastic food wrap was placed inside the GLT desorption tube
Thermal Desorption: Block temperature: 100°C, Purge flow: 2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5,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



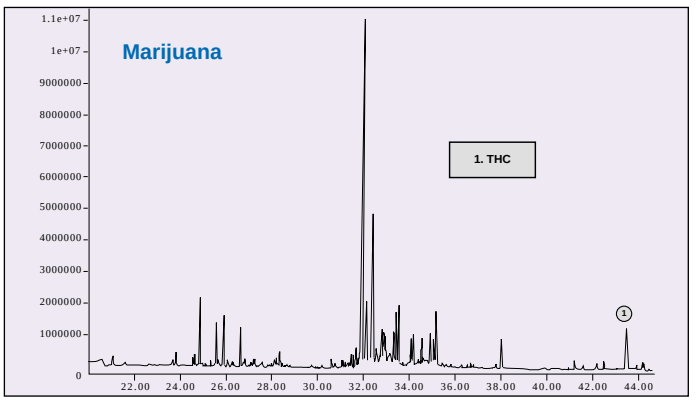
Technique: Direct Thermal Extraction of Solid Samples
Sample: 2.0 mg. crushed aspirin was placed inside the GLT desorption tube
Thermal Desorption: Block temperature: 100°C, Purge flow: 2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D.,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



Technique: Direct Thermal Extraction of Solid Samples
Sample: 2.0 sq. mm. of a transparent tape was placed inside the GLT desorption tube
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.



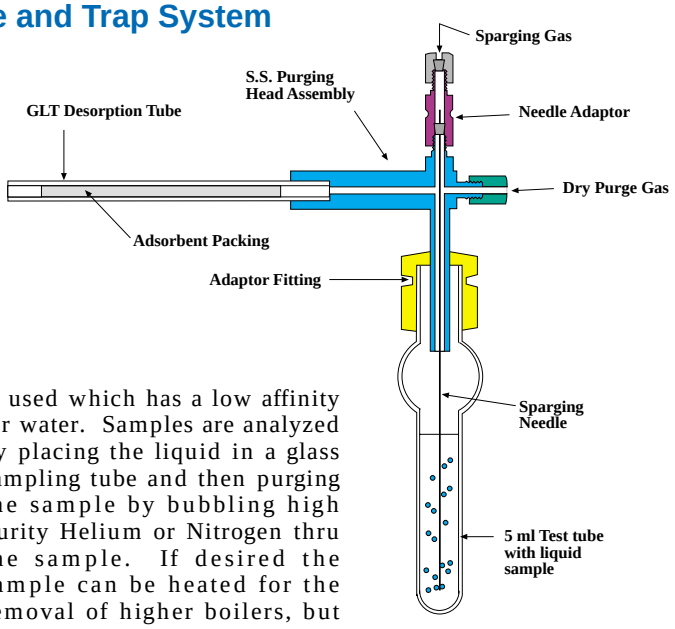
Technique: Direct Thermal Extraction of Solid Samples
Sample: 8.3 mg. of thinly sliced gum was placed inside the GLT desorption tube
Thermal Desorption: Block temperature: 100°C, Purge flow: 2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25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



Technique: Direct Thermal Extraction of Solid Samples
Sample: 3.0 mg. of marijuana was placed inside the GLT desorption tube. Data courtesy NJSP
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.

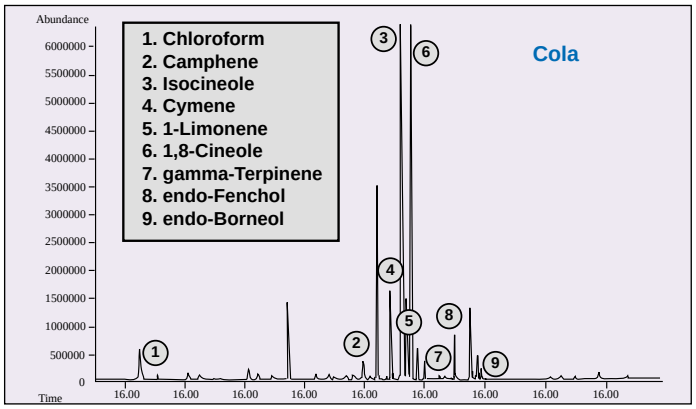
Purge and Trap Applications

Purge and Trap System

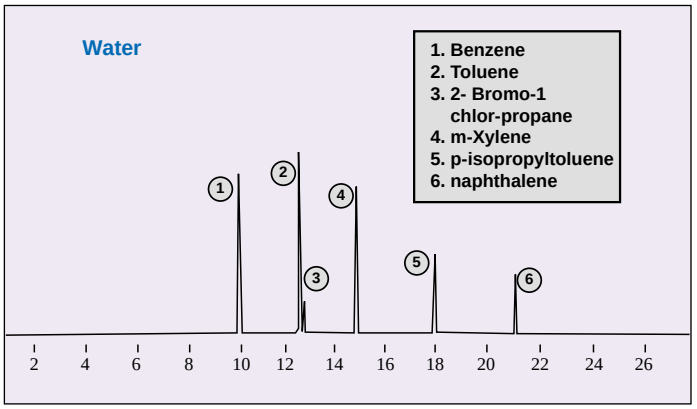


Purge and Trap of Liquids

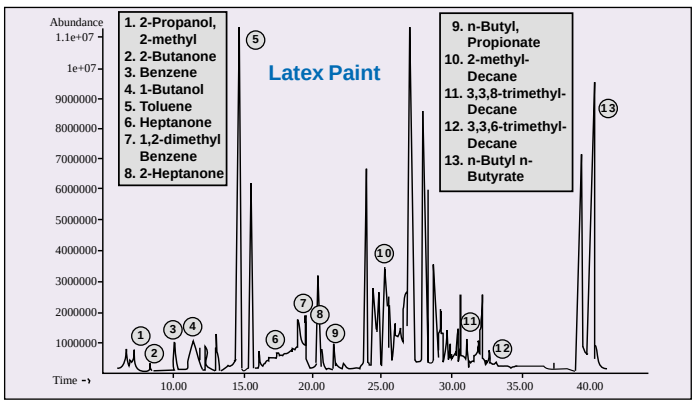
The Short Path Thermal Desorption System can be used for the analysis of volatile organics (VOC's) in commercial water based products including liquid formulations, colloidal suspensions, and liquid pastes. Using the S.I.S. Purge and Trap System for the purging of the volatiles from the liquid based samples followed by trapping on GLT desorption traps packed with Tenax or other suitable resins, the samples thus collected can be analyzed using the Short Path Thermal Desorption System. Using this technique it is possible to detect and identify various flavors, fragrances, off-odors, and manufacturing by-products in a wide diversity of liquid matrix samples. Water does not present any problem, since a dry gas purge is added just before the desorption tube to aid in the removal of water from the adsorbent resin, and an adsorbent resin such as Tenax is used which has a low affinity for water. Samples are analyzed by placing the liquid in a glass sampling tube and then purging the sample by bubbling high purity Helium or Nitrogen thru the sample. If desired the sample can be heated for the removal of higher boilers, but the temperature should be kept well below the boiling point of the liquid. The volatiles thus purged from the sample are trapped on the adsorbent trap. They are subsequently placed on the Short Path Thermal Desorption System to complete the analysis.



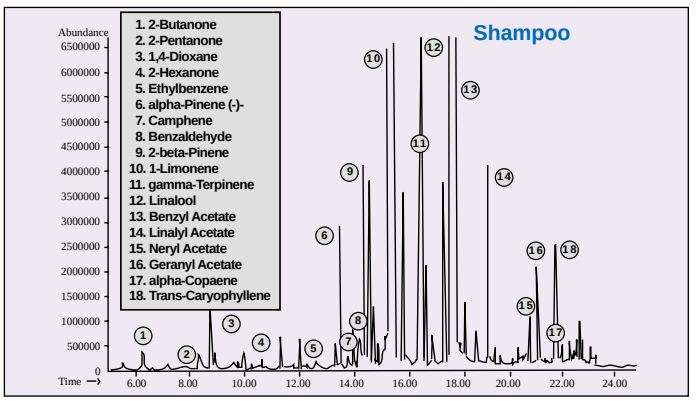
Technique: Purge and Trap of Volatiles in Liquid Samples
Sample: 25 ml of a Carbonated Cola was purged with 150 ml of high purity He at 15 ml/min and a dry purge of 15 ml/min. The volatiles were trapped on Tenax traps.
Thermal Desorption: Block temperature: 150°C, Purge flow:2.0 ml/min He, Desorption 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.



Technique: Purge and Trap of Volatiles in Liquid Samples
Sample: 50 ml of water spiked with several aromatics at 20 ppm was purged with 300 ml of high purity He at 20 ml/min and a dry purge of 20 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.



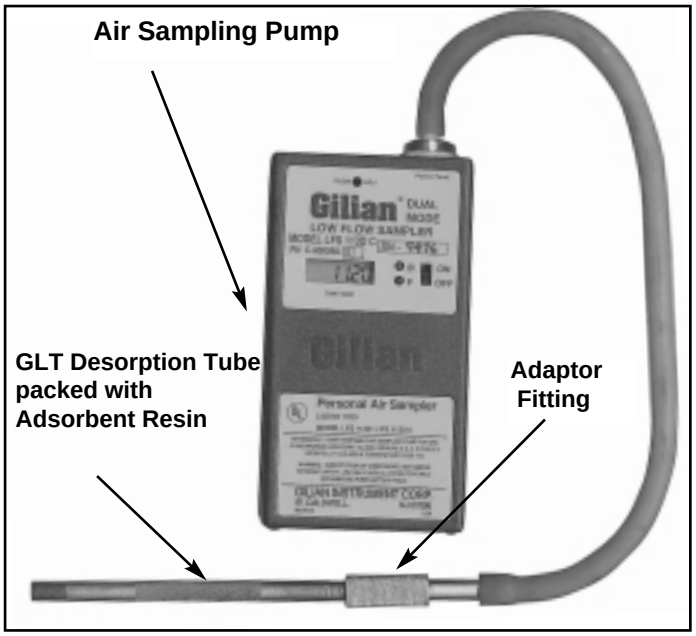
Technique: Purge and Trap of Volatiles in Liquid Samples
Sample: 1.0 ml latex Enamel paint was purged with 150 ml of high purity He at 15 ml/min and dry purge of 15 ml/min.
Thermal Desorption: Block temperature: 150°C, Purge flow:2.0 ml/min He, Desorption time: 10.0 min. Initial column trap temperature: -40°C
Column: DB-5, 25 meter x 0.25 mm I.D., 0.25 u film, -40° to 280°C at 10°/min.

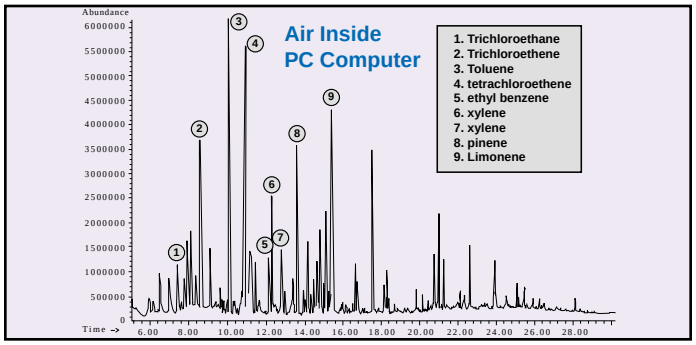


Technique: Purge and Trap of Volatiles in Liquid Samples
Sample: 5.0 ml of Liquid Shampoo was purged by passing 200ml of high purity He over the surface of the liquid shampoo at the rate of 20 ml/min. and a dry purge of 20 ml/min (Dynamic Headspace Purge). The volatiles were trapped on Tenax traps.
Thermal 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.

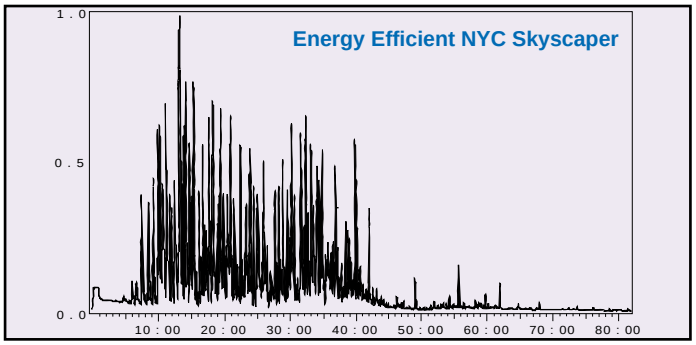
Environmental Air Testing

The Volatiles present in the environmental air can be readily analyzed using the Short Path Thermal Desorption System. The air to be analyzed is pumped through a GLT desorption tube which has been packed with an adsorbent resin such as Tenax. The packed desorption tube has been previously cleaned and conditioned to remove any contaminants from the resin. Any of a wide variety of small air pumps (vacuum) that are commercially available can be used to pump the air through the desorption tube. Some of the pumps available are battery powered and portable which makes them ideal for field use. A vacuum must be used to pull the air through the tube 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, 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.



Technique: Air Sampling of Volatiles from Adsorbent Traps
Sample: A 100 liter sample of air from a New York City Energy Efficient Skyscaper was pumped through a Tenax GLT desorption trap. Data Courtesy CAFT Rutgers Univ.
Thermal Desorption: Block temperature: 250°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 40° at 10°/min, 40° to 280° at 4°/min

Short Path Thermal Desorption Features

- High sensitivity Thermal Desorption and Direct Thermal Extraction System
- No memory effects - individual flow path for each sample preventing contamination of transfer lines.
- Short Path from sample to GC
- Eliminate tedious sample cleanup by other techniques such as solvent extraction
- Mounts overtop GC injection port, easily removable and transferable
- Microprocessor controlled electronics system
- Automatic injection of sample into GC via pneumatic driven injector
- Desorb samples at temperatures from 20° to 350°C either isothermal or temperature program at up to 40°/min.
- Program desorption time from 1 second to 100 minutes
- Automatic programming and control of GC Cryo-Trap Accessory.
- Automatic desorption cycling and GC remote start
- Glass lined stainless steel (GLT) sample tubes are both inert to samples and strong for samplehandling and transporting

The Short Path Thermal Desorption System provides several unique advantages over other desorption systems:

First, it enables the sample, which is trapped on an adsorbent media contained in a glass lined stainless steel tube (GLT desorption tube), to be subjected to rapid heating.

Second, the desorbed component can be easily and efficiently transferred into the injection port of the gas chromatograph from a glass lined stainless steel sample tube and its associated injection needle. This provides for a short transfer path for the sample in an inert environment to minimize the degradation of labile sample components which often decompose upon contact with the hot catalytic metal wall surfaces of the transfer path of other systems.

Third, each sample has its own individual adsorbent trap tube and needle to eliminate the possibility of cross-contamination from sample to sample, thus preventing any "memory effect" due to overloading of the sample in the GLT desorption tube or due to residues from previous samples.

Samples to be analyzed are collected on Glass Lined Stainless Steel (GLT) Desorption Tubes containing an adsorbent resin such as Tenax™ TA or activated carbon. Alternatively, samples of small size (mg) can be packed into the tube and subjected to direct thermal extraction.

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

For additional information call Scientific Instrument Services, Inc. at 908-788-5550

Visit Our WEB site for information o

Applications-Thermal Desorption

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

- **System Components**
- **System Description and Theory of Operation**
- **Ordering Information**
- **Accessory Equipment and Supplies**
- **Installation Directions**
- **Application Notes**

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Scientific Instrument Services, Inc.
1027 Old York Rd., Ringoes, NJ 08551
908-788-5550

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