MICRO CRYO-TRAP Adds Cryo-cooling capabilities to your GC

New programmable Cryo-cooling and heating trap for the Cryo-focusing of volatiles and semivolatiles at the head of GC capillary columns.

A pplications

- Thermal Desorption Sample
 Trapping
- Purge and Trap Systems
- GC Headspace Sample Analysis
- Multi-dimensional GC applications
- Improve Chromatographic resolution of early eluting peaks





Scientific Instrument Services, Inc. 908-788-5550

F eatures of Micro Cryo-Trap

- Only 1.0" long uses minimum amount of cooling gas
- Reduction of CO2 or LN2 use by 97% as compared to cooling the entire oven.
- Improve chromatographic resolution of early eluting peaks
- Dual programmable cryo-cooling and heating cycles
- Trap compounds in the GC oven at the head of the GC column.
- Remote input connector for switching between cryo-cooling and heating cycle switching via GC, Desorption system or manually.
- Rapid heating up to 400°C at > 1000°C per minute.
- Remote start output signal for starting GC, MS or recorder.
- Clamp Mounts onto GC Injection Port

GC Septa GC Injection Port GC Injection Port Liner Vespel Insulator Injection Port Clamp Injection Port Nut Heater Coil Thermocouple Cooling Gas IN Cryo-Trap Cooling Chamber

Micro Cryo-Trap Theory of Operation

Mounts inside GC Oven on bottom of injection port



Micro Cryo-Trap Controller



Micro Cryo-Trap Mounted Inside GC Oven

he Cryo-Trap consists of a small heating/cooling chamber which is 3/4" in diameter and 1" long and mounts to the bottom of the injection port just inside the GC oven. In the center of the chamber is a small stainless steel capillary through which the capillary column freely passes. Capillary columns up to megabore (0.53mm I.D.) diameters can be used. Around the stainless steel capillary tube a heating coil is wound to provide for the rapid heating of the capillary tube. A thermocouple provides accurate measurement of both the cooling and heating temperatures. Either Liquid CO2 or Liquid Nitrogen for cooling is introduced into the Cryo-Trap (Cooling Gas In), and is exhausted through the outlet. The exhaust can either be vented into the GC Oven or a tube can be attached to vent the cooling gas external to the GC.

The control of the Cryo-Trap is provided via an independent Cryo-Trap Controller provided with the System. Both the Cryo-Cooling and heating temperatures are set via this digital temperature controller. The system can be used either manually to switch between cooling and heating or can be operated automatically via an input signal from a controlling device.

For the cooling operation the cooling gas is pulsed into the chamber. Liquid CO₂ (Model 971) will cool down to -70 degrees C. Liquid Nitrogen (Model 981) will cool down to -180 deg. C. The cooling temperature can be set to any temperature between room temperature and the lower limits of the cooling gas. The temperature controller will pulse the cooling gas into the chamber to accurately control the temperature to the value you set. The thermocouple provides the feedback to both regulate the temperature as well as display the GC Cryo-Trap temperature on the display of the temperature controller. The electronics of the Model 971 and Model 981 are different due to the different cooling properties of the liquid CO₂ and Liquid Nitrogen. Therefore parts are not interchangeable between the two models and the particular model can only be used with the cooling gas for which it was designed.

Each Micro Cryo-Trap System consists of the GC Cryo Trap which goes inside the GC oven, the Dual Temperature Controller and cooling gas tubing.

A pplications of the Micro Cryo-Trap

Headspace GC Analysis

Low boiling point volatiles from headspace samplers can be trapped in the GC Cryo-Trap for subsequent analysis. This will permit the analysis of large gas volume injections (0.5 to 100 ml or more) as well as multiple injections of headspace volumes into capillary GC columns. After trapping volatile organics at temperatures down to -180°C, the GC Cryo-Trap is rapidly heated to temperatures to 400° C to release the volatiles for separation on the capillary column. The resulting peaks are highly resolved, even for the very light volatiles such as butane and acetone.

In **Figure # 1**, 0.5 ul of gasoline was dissolved in 5.0 ml of water in a headspace vial. The sample was heated to 70°C in a CTC Headspace Sampler and then 1.0 ml of the headspace gas was injected over a 35 second time interval into the GC injection port and cryo-trapped in a narrow band on a 1.5u film thickness guard column in the GC Cryo-Trap at a temperature of -120°C. The sample was trapped for 5.0 minutes and then rapidly heated to 200°C to release the volatiles for GC analysis. More than 100 compounds were detected and identified including the low boiling volatiles butane and pentane. A lower trapping temperature of -180° C was able to trap ethane and propane. Applications of this technique could easily be expanded to the detection of low boiling volatiles in water, soil, food products, commercial products and other solid, liquid and gas matrix samples.

Thermal Desorption - Purge and Trap Applications

In the thermal desorption technique, large volume gas samples are typically purged from the sample or adsorbent resin, into the GC injection port for analysis. Utilizing the GC Cryo-Trap, the volatile organics from this large gas volume can be cryo-trapped or cryo-focused in a narrow plug in the guard column in the GC Cryo-Trap. In Figure # 2, 200 milligram of black tea in water at 80°C was purged with 450 ml of gas and the volatiles trapped on a Tenax TA desorption trap. The volatiles on the adsorbent resin were then thermally desorbed off the resin at a temperature of 250°C utilizing the S.I.S. Short Path Thermal Desorption System, purged into the GC injection port and cryo-trapped on a 5.0 u film thickness guard column in the GC Cryo-Trap at two different temperatures (0° and -70°C). The GC Cryo-Trap was then heated to 220°C to release the trapped volatiles for subsequent GC/MS analysis. At a cryo-trapping temperature of 0°C, volatiles down to methyl isobutyl ketone were trapped. At a trapping temperature of -70°C, eight additional volatiles including acetone were trapped and identified.

Direct Injection Applications

In **Figure # 3**, a series of neat hydrocarbons from ethane through nonane were direct injected into the GC injection port utilizing the split mode and trapped on a 1.5 u film thickness guard column in the GC Cryo-Trap at a variety of temperatures. This chart demonstrates the range of volatiles that can be trapped as a function of the GC Cryo-Trap temperature. Utilizing the Model 971 with liquid CO₂



For additional applications request the FREE Application Notes available from S.I.S.

(minimum temperature -70°C), volatiles down to pentane can be cryo-trapped. Utilizing the new Model 981 GC Cryo-Trap which uses liquid nitrogen for cooling to a minimum temperature of -180°C permits the trapping of ethane on this guard column. The utilization of PLOT guard columns will permit the trapping of even lower volatiles such as methane, formaldehyde and ethylene oxide.

Micro Cryo-Trap

New - programmable Micro Cryo-Trap for the Cryo-focusing of Volatiles and Semivolatiles at the Head of GC Columns



Micro Cryo-Trap

Two models of the Micro Cryo-Trap are now available. The Model 971 is designed for liquid CO₂ for cryo-trapping temperatures down to -70° C. The Model 981 is designed for Liquid Nitrogen for cryo-trapping temperatures down to -180° C. Each model can maintain any cooling temperature within + / - 3° C from its minimum temperature up to room temperature. In order to release the trapped volatiles from the GC Cryo-Traps, both models can heat the GC Cryo-Trap at temperatures up to 400° C at a heating ramp rate in excess of 1000° /minute. Most users prefer to use the CO₂ version of the GC Cryo-Trap (Model 971) due to the ease of handling liquid CO₂ and for applications where -70° C is an acceptable lower temperature limit.

If lower temperatures are required, then the liquid nitrogen version of the GC Cryo-Trap (Model 981) must be used. The liquid nitrogen delivery lines are larger than the CO_2 lines and the liquid nitrogen lines must be insulated. This makes installation somewhat more cumbersome. In addition the Liquid Nitrogen version uses more cooling gas and is slightly more audible.

Depending on the make and model of your GC, a Micro Cryo Trap can be ordered as described below. First you must decide which model of Cryo-Trap is required for your application; the Model 971 for use with CO_2 , or the Model 981 for use with liquid Nitrogen. The parts of the two models are not interchangeable. Neither the GC Cryo-Trap or its related electronics are interchangeable between the two cooling gases.

Visit our Web Home Page http://www.sisweb.com for further information and application notes.



Micro Cryo-Trap - Ordering

Model 971 Micro Cryo-Trap for use with Liquid C02

The Model 971 Micro Cryo-Trap is designed for use with liquid CO_2 tanks with a DIP tube. The minimum cooling temperature is -70° C.

Part # 971001 - Model 971 Micro Cryo-Trap for use with liquid CO₂ on HP Gas Chromatographs, Electronics Controller, connecting cables, relay and stainless steel connecting lines -

Part # 971002 - Model 971 Micro Cryo-Trap for use with liquid CO₂ on Varian Gas Chromatographs, Electronics Controller, connecting cables, and stainless steel connecting lines -

Part # 971003 - Model 971 Micro Cryo-Trap for use with liquid C0₂ on Shimadzu Gas Chromatographs, Electronics Controller, connecting cables, and stainless steel connecting lines -

Model 981 GC Cryo-Trap for use with Liquid Nitrogen

The Model 981 GC Cryo-Trap is designed for use with liquid nitrogen tanks (low pressure). The minimum cooling temperature is -180° C.

Part # 981001 - Model 981 Micro Cryo-Trap for use with liquid LN₂ on HP Gas Chromatographs, Electronics Controller, connecting cables, HP relay switch and copper connecting lines -

Part # 981002 - Model 981 Micro Cryo-Trap for use with liquid LN_2 on Varian Gas Chromatographs, Electronics Controller, connecting cables, and copper connecting lines -

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