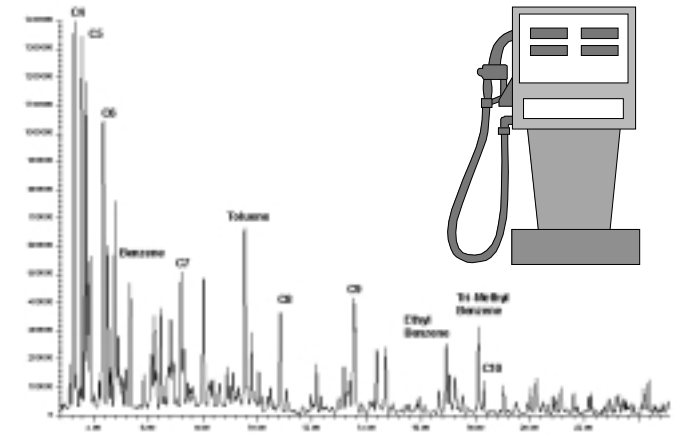
Applications of the GC Cryo-Trap and Micro Cryo-Trap™

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 µl 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.

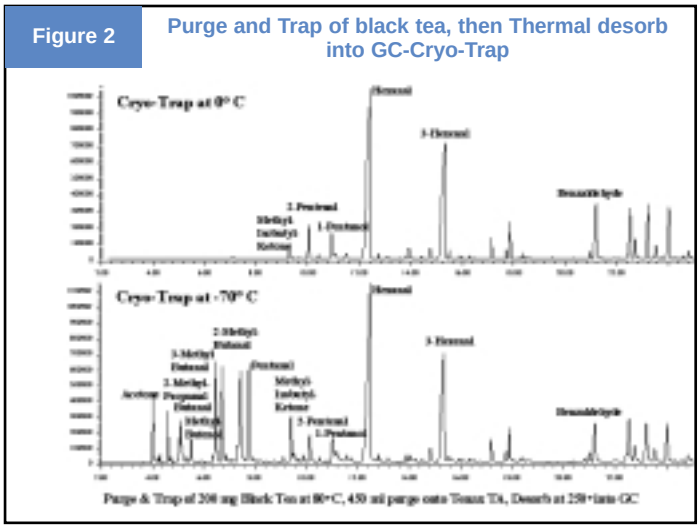
Figure 1 Headspace analysis of gasoline in water using GC Cryo-Trap



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.

Figure 2 Purge and Trap of black tea, then Thermal desorb into GC-Cryo-Trap



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 CO_2 (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.

Figure 3 Trapping Efficiency of Hydrocarbons on .53mm DB-5 (1.5u) trapped at various GC Cryo-Trap temperatures