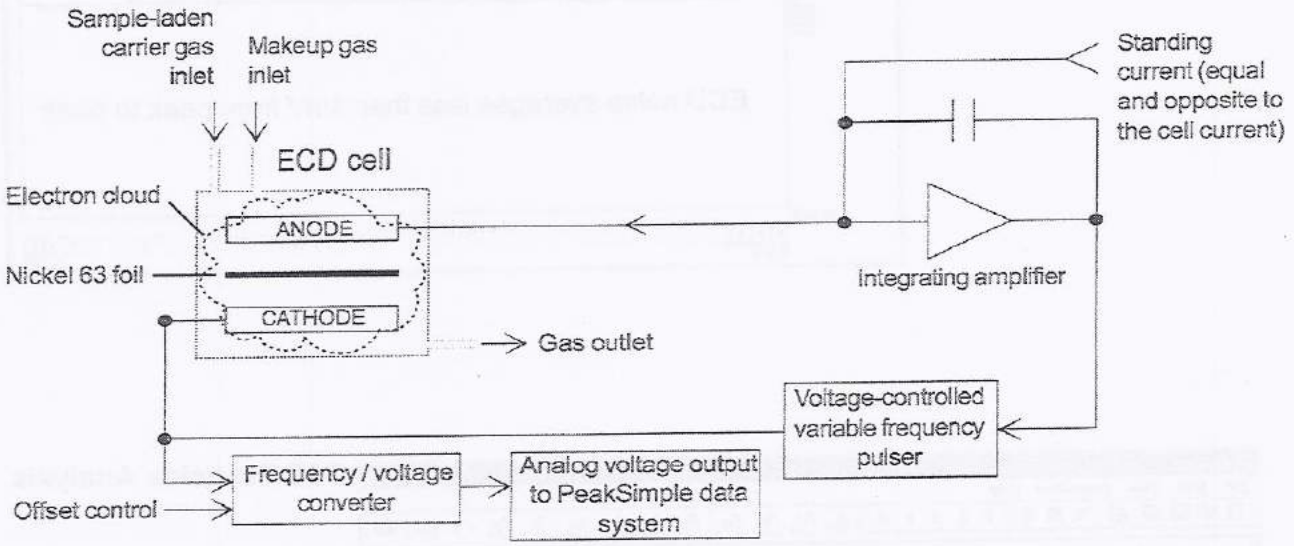
Theory of Operation

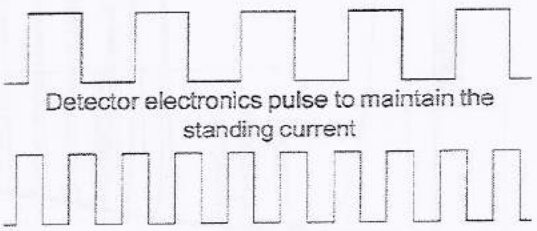
The radioactive Nickel 63 sealed inside the ECD detector emits electrons (beta particles) which collide with and ionize the make-up gas molecules (either nitrogen or P5). This reaction forms a stable cloud of free electrons in the ECD detector cell. The ECD electronics work to maintain a constant current equal to the standing current through the electron cloud by applying a periodic pulse to the anode and cathode. The standing current value is selected by the operator; the standing current value sets the pulse rate through the ECD cell. A standing current value of 300 means that the detector electronics will maintain a constant current of 0.3 nanoamperes through the ECD cell by periodically pulsing. If the current drops below the set standing current value, the number of pulses per second increases to maintain the standing current.

ECD Detector Operational Diagram



When electronegative compounds enter the ECD cell from the column, they immediately combine with some of the free electrons, temporarily reducing the number remaining in the electron cloud. When the electron population is decreased, the pulse rate is increased to maintain a constant current equal to the standing current. The pulse rate is converted to an analog output, which is acquired by the PeakSimple data system. Unlike other detectors which measure an increase in signal response, the ECD detector electronics measure the pulse rate needed to maintain the standing current.

Example Pulse Trains



Detector electronics pulse to maintain the standing current

The pulse rate is increased in the presence of electronegative compounds

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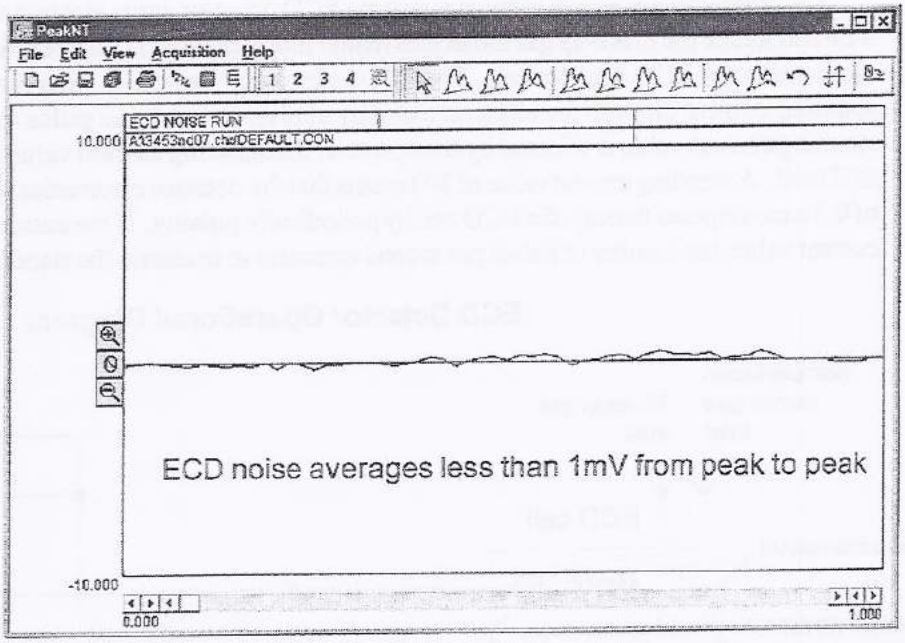
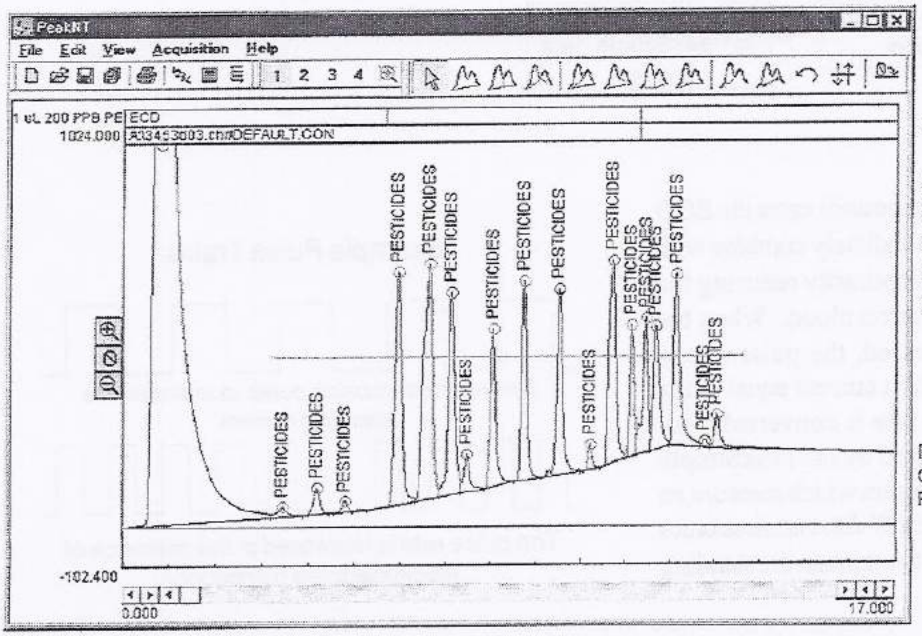
Expected Performance

ECD Noise Run

Column: 15m MXT-5 capillary
Carrier: Helium @ 10mL/min
Makeup: Nitrogen @ 60mL/min
ECD Temp: 250°C
ECD standing current: 300
Offset before zeroing the data system signal: 280mV





ECD Pesticide Analysis

Sample: 1µL 200ppb chlorinated pesticides
Column: 15m MXT-5 capillary
Carrier: Helium @ 10mL/min
Makeup: Nitrogen @ 60mL/min
ECD temp: 250°C
ECD standing current: 300

Results:

Component	Retention	Area
Pesticides	2.850	45792.4350

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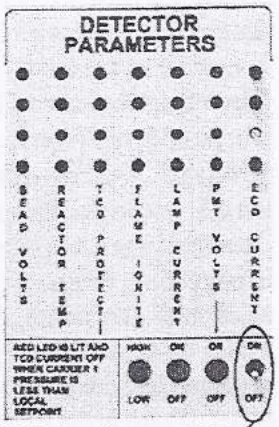
General Operating Procedure

The following suggestions are specific to your SRI ECD-equipped GC. Consult the Valco ECD detector manual for carrier gas purity requirements, carrier gas system configuration, and other general ECD detector information. Keep in mind that the electronics shematics in the Valco manual do not apply to your ECD-equipped SRI GC.



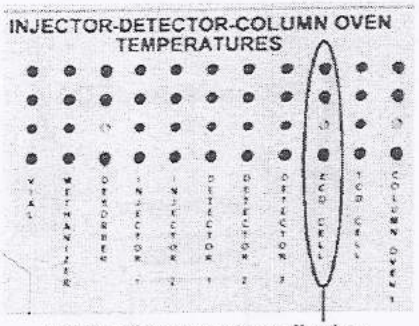
ECD standing current ON/OFF switch

1. Cap off the carrier inlet to the ECD cell (in the column oven).

2. Connect the makeup gas and let it flow through and purge the ECD cell. Makeup flow is 40-100mL; typically 60mL.

3. Heat the ECD detector to 150°C to verify that the baseline noise and offset are normal. 150°C is hot enough to evaporate off water but low enough to avoid oxidation of the nickel foil which can occur at high temperatures in the presence of oxygen. Once you have verified the ECD's operation at this temperature, you may heat it to higher temperatures.

4. Turn on the ECD standing current (the ECD current ON / OFF switch is located on the front control panel of the GC, under "DETECTOR PARAMETERS"). As a rule of thumb, an ECD detector requires enough nitrogen makeup flow (40-100mL/min) to significantly dilute the carrier in order to help keep detector noise down; the ECD can tolerate a 6:1 ratio of nitrogen to helium.



ECD cell temperature display

With the carrier and makeup gas connected and flowing, check the offset from zero. The millivolt reading should be between 100 and 500mV. If the signal offset is less than 100mV, the standing current needs to be increased. If the signal offset is higher than 500mV, the standing current needs to be decreased. Once the signal is relatively quiet and stable, set the temperature to whatever is appropriate for your analysis by adjusting the trimpot setpoint with the flat blade screwdriver provided.

5. When the ECD detector cell reaches temperature, let the system stand until you get a stable milliVolt reading. Once the system exhibits a stable baseline, reconnect the column. Observe the signal in the presence of the carrier flow. If it is significantly higher, it indicates contamination introduced on the carrier flow. If the milliVolt reading is still relatively stable in the presence of carrier flow, then sample may be injected. Avoid samples with high concentrations of electronegative compounds; they may effect ECD operation for some time thereafter, as they could take too long to dissipate.

6. You may need to adjust the ECD standing current using its trimpot setpoint. The trimpot setpoints are located on the top edge of the front control panel, directly above the display push-buttons for each controlled zone. Remember, increasing the standing current increases the ECD's sensitivity and raises the baseline offset.

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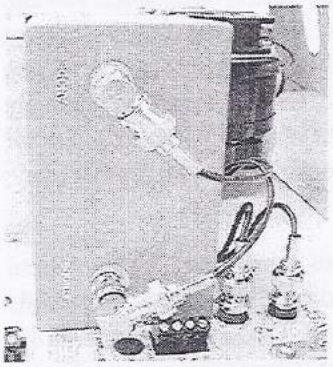
ECD Troubleshooting

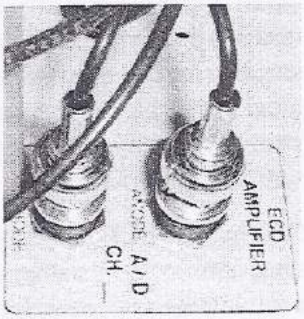
If you are experiencing baseline offset and noise problems withyour ECD detector, try the following two diagnostic tests:

1. Verify that the ECD amplifier electronics are working properly by removing the detector from the circuit and inserting a 1000MOhm test resistor in its place. The parts kit in the tackle box included with your GC under the red lid contains a 1000MOhm resistor for this test. Turn the ECD current off. The anode and cathode connections are BNC connectors located on the GC chassis near the base of the ECD detector housing. Disconnect these two BNC connectors from the detector electronics, and install the 1000MOhm test resistor as a jumper between the center conductor in the anode BNC jack and the center conductor in the cathode BNC jack. Zero the data system signal. Turn the ECD current back on, and check the signal offset (observe the mV reading in the upper right area of the PeakSimple chromatogram window. With the test resistor in the detector's place, the signal offset should be 120-150mV with the standing current at 300. If the signal offset is pegged up or down (5000mV or 1500mV, respectively), there is a problem with your ECD detector electronics. Try turning off the GC power for at least 30 seconds, with the test resistor still in place, then turning it back on to see if the signal offset still indicates a problem. If the signal offset is at zero with the test resistor in place, check to make sure that you are looking at the correct detector channel. If you are observing a signal offset of zero in the ECD detector channel, call technical support.

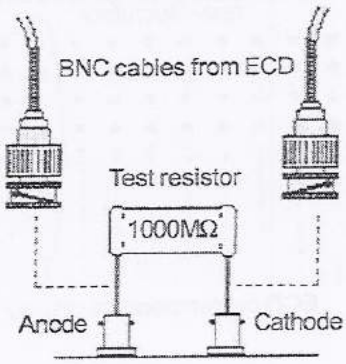
The ECD anode and cathode are connected to the ECD amplifier via BNC cables



Anode and cathode BNC connectors



ECD electronics test



2. Operate the ECD on make-up gas only by disconnecting the column from the ECD. With the standing current still set at 300, observe the signal offset and noise. If it drops, then the problem is being introduced into the GC and ECD by the carrier gas through the column.

Tip: In most situations, the ECD will be used to detect sample components that are reactive with metal. Use glass, fused silica, or fused silica lined metal capillary columns to help avoid reactive sites and ghost peaks.

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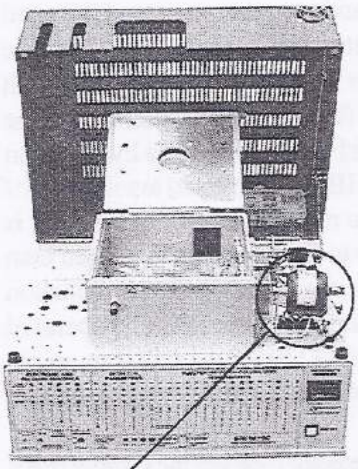


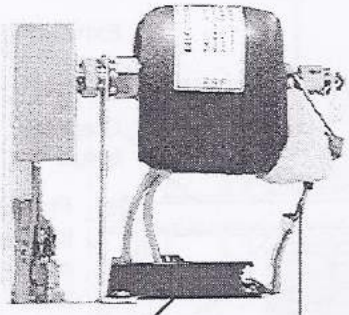
DELCD on an 8610C GC

Overview

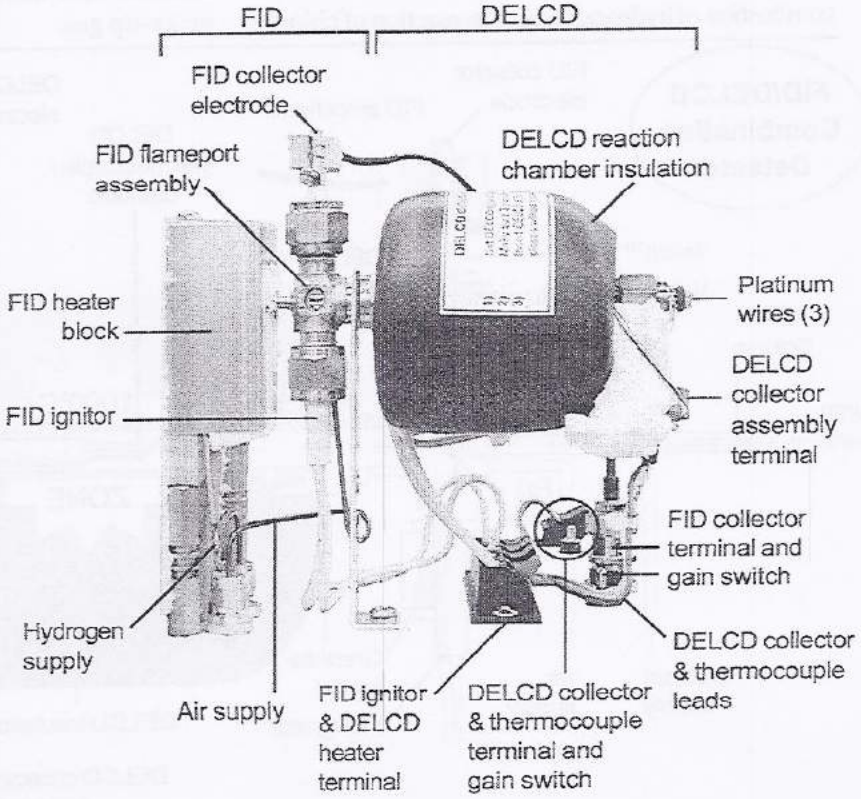
The Dry Electrolytic Conductivity detector, or DELCD, is selective to chlorinated and brominated molecules. It differs from the traditional wet ELCD in that it does not use a solvent electrolyte, and the reaction products are detected in the gaseous phase. The SRI DELCD is available alone or in combination with the FID detector. On its own, the detection limits of the DELCD are in the low ppb range. In combination with the FID, its detection limits are in the low ppm range. The FID/DELCD combination enables the operator to reliably identify hydrocarbon peaks detected by the FID as halogenated or not. Because the DELCD operates at 1000°C, it can tolerate the water-saturated FID effluent, measuring the chlorine and bromine content simultaneously with the FID measurement of the hydrocarbon content. All hydrocarbons are converted by the FID flame to CO_2 and H_2O prior to reaching the DELCD, thus preventing contamination of the DELCD by large hydrocarbon peaks.

DELCD - À la carte

Close-up of the same DELCD detector



FID / DELCD Combo Detector



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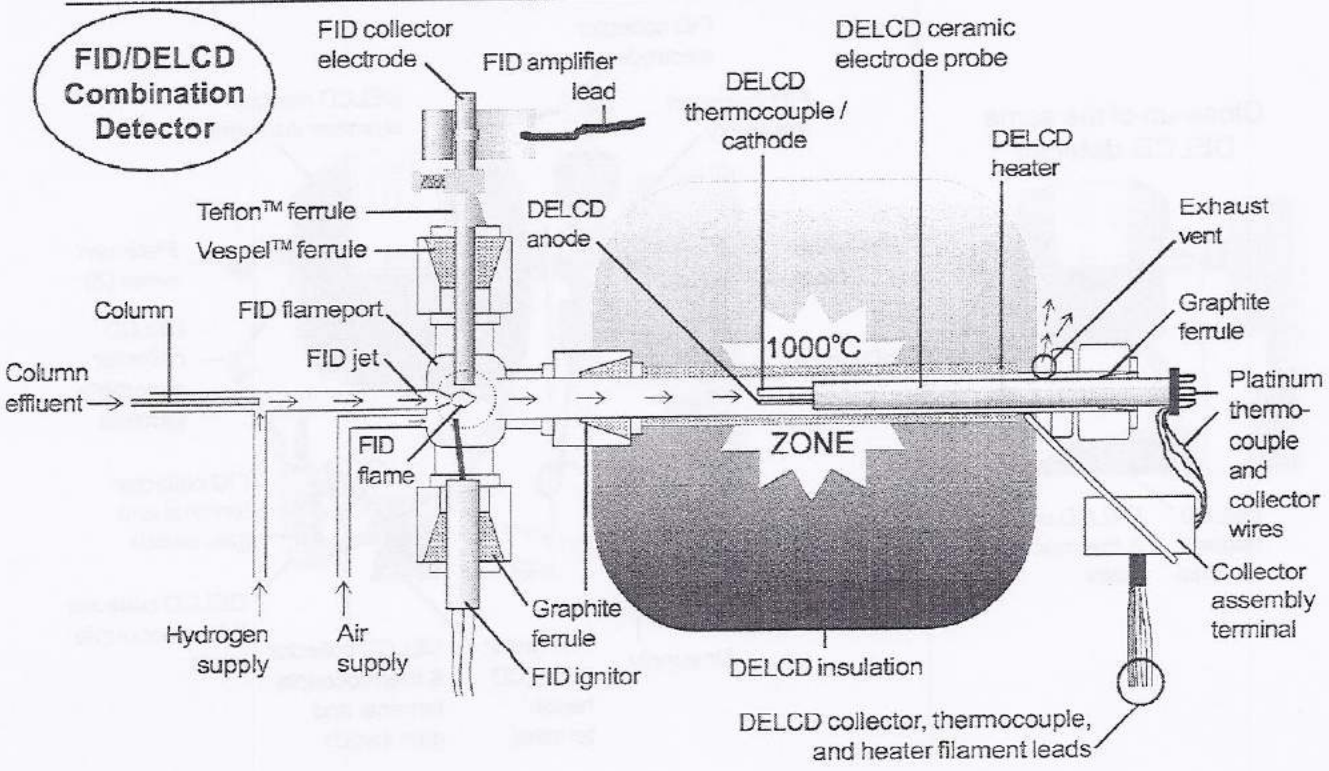
Dry Electrolytic Conductivity Detector - DELCD

Theory of Operation

The DELCD consists of a small ceramic tube—the DELCD reactor—heated to 1000°C. Inside the reactor, a platinum thermocouple measures the detector temperature, and a nichrome collector electrode measures the conductivity of the gases flowing through the DELCD. The detector response is dependent upon its temperature. Therefore, the control circuit must maintain the temperature, within a fraction of a degree, at 1000°C.

When combined with the FID detector, the DELCD is mounted on the FID exhaust. Column effluent enters the FID flame where hydrocarbons are ionized and combusted. Electrons freed in the ionization process are collected by the FID collector electrode, which has an internal diameter of 1mm (0.040"). Due to its small I.D., the collector electrode acts as a restrictor, splitting the FID exhaust gases so that it takes about half of the flow, and the remainder is directed to the DELCD. The FID exhaust gases consist of un-combusted hydrogen and oxygen, nitrogen, and water and carbon dioxide from the combustion of hydrocarbons. The reaction of chlorine or bromine and hydrogen forms HCl and HBr, and the reaction of chlorine or bromine and oxygen forms ClO_2 and BrO_2. The DELCD detects the oxidized species of chlorine and bromine, such as ClO_2 and BrO_2. It does not detect the acids HCl or HBr like the conventional wet ELCD. In the hydrogen rich effluent from the FID flame, the chlorine and bromine preferentially react with hydrogen (or the hydrogen in water) to make HCl-HBr. Given equal availability of hydrogen and oxygen molecules, a chlorine atom is 100 times more likely to react with the hydrogen than the oxygen. Therefore, the FID/DELCD combination is 100 times less sensitive than the DELCD operated with the FID off. The SRI FID/DELCD is operable as a combination detector, as an FID only, or as a DELCD only.

A DELCD only detector receives the sample laden carrier gas directly from the column or from a non-destructive detector outlet, like the PID. It is mounted on the heater block on the column oven wall so that the column effluent is maintained at a temperature consistent with the analysis. This type of high sensitivity DELCD uses helium or nitrogen carrier gas and air make-up gas.



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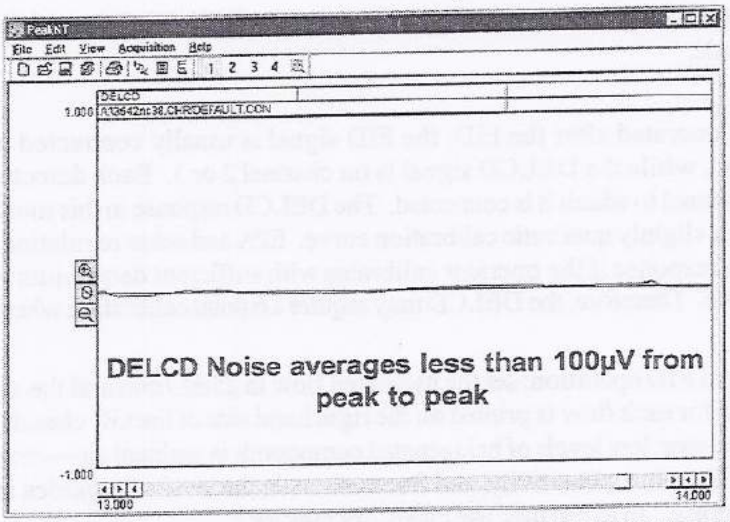
Expected Performance



DELCD Noise Run

Column: 15m MXT-VOL
Carrier: helium @ 10mL/min
DELCD gain: LOW
DELCD heater block temp: 150°C
DELCD reactor setpoint: 260

Temperature program:

Initial	Hold	Ramp	Final
80°C	20.00	0.00	80°C

FID / DELCD Combo Test Run

Sample: 1µL 100ppm BTEX Plus
Column: 15m MXT-VOL
Carrier: helium @ 10mL/min

Temperature program:

Initial	Hold	Ramp	Final
40°C	2.00	15.00	240°C

DELCD gain: LOW
DELCD heater block temp: 150°C
DELCD reactor setpoint: 260

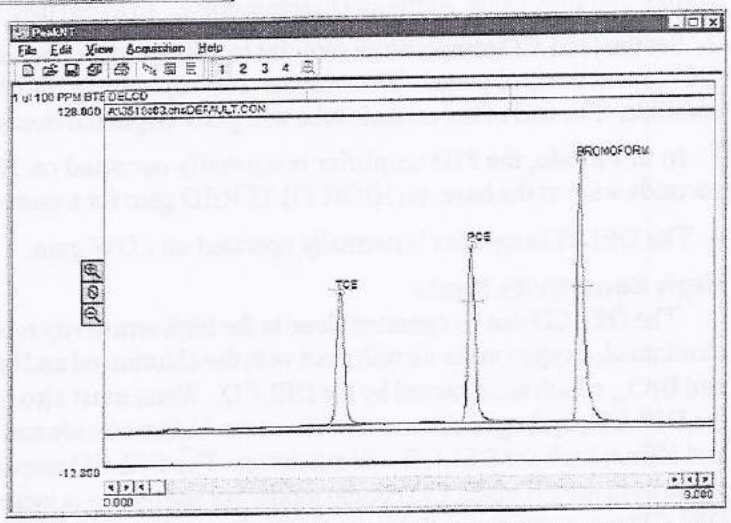
DELCD Results:

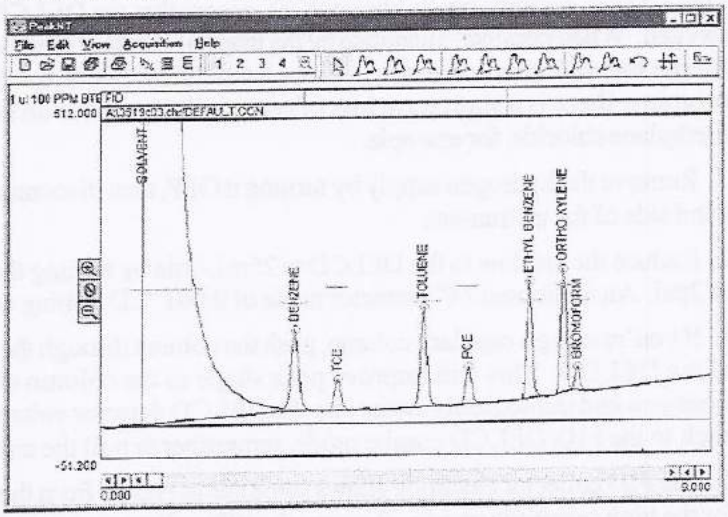
Component	Retention	Area
TCE	3.483	463.5080
PCE	5.416	532.2900
Bromoform	7.016	759.6650
	Total	1755.4630

FID gain: HIGH
FID temp: 150°C
FID ignitor: -400

FID Results:

Component	Retention	Area
Solvent	0.600	144406.8420
Benzene	2.850	1074.0740
TCE	3.500	378.3505
Toluene	4.766	1109.8590
PCE	5.416	364.5700
Ethyl Benzene	6.316	1103.6370
Ortho Xylene	6.800	1135.6855
Bromoform	7.016	220.3325
	Total	149793.3505





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DETECTORS
Dry Electrolytic Conductivity Detector - DELCD

General Operating Procedure

The FID/DELCD combination detector can be operated in the Combo Mode, the High Sensitivity Mode (DELCD only), or the FID only mode.

Combo Mode

In the Combo Mode, the DELCD is operated after the FID; the FID signal is usually connected to Channel 1 on the PeakSimple data system, while the DELCD signal is on channel 2 or 3. Each detector amplifier is factory labeled with the data channel to which it is connected. The DELCD response in this mode is useable from 1 to 1000 nanograms with a slightly quadratic calibration curve. EPA and other regulations allow the use of detectors with non-linear response if the operator calibrates with sufficient data points to accurately model the detector response curve. Therefore, the DELCD may require a 6 point calibration where 5 point calibration is normally required.

1. Set the hydrogen and air flows for normal FID operation: set the hydrogen flow to 25mL/min and the air flow to 250mL/min. The pressure required for each flow is printed on the right hand side of the GC chassis. (**NOTE:** If you're using a built-in air compressor, low levels of halogenated compounds in ambient air—even levels below 1ppm—can cause the DELCD to lose sensitivity, and fluctuations in the level of organics in ambient air may cause additional baseline noise. To avoid this, use clean, dry tank air.)

2. Set the DELCD temperature setpoint to 260 by adjusting the appropriate trimpot on the top edge of the GC's front control panel. The number 260 represents 1000°C; the DELCD will heat to about 254 and stabilize. The end of the ceramic tube will glow bright red due to the high temperature.

3. In this mode, the FID amplifier is normally operated on HIGH gain or, if the peaks are more than 20 seconds wide at the base, on HIGH FILTERED gain for a more quiet baseline.

4. The DELCD amplifier is normally operated on LOW gain.

High Sensitivity Mode

The DELCD can be operated alone in the high sensitivity mode by eliminating hydrogen. With hydrogen eliminated, oxygen in the air will react with the chlorinated and brominated molecules at 1000°C to form ClO_2 and BrO_2, which are detected by the DELCD. Water must also be eliminated; at the high temperatures inside the DELCD, hydrogen disassociates from the H_2O molecule and becomes available as a reactant to form HCl and HBr, which the DELCD will not detect. The DELCD response curve is quadratic in the high sensitivity mode as in the FID/DELCD combo mode, but sensitivity is increased by 100 to 1000 times. In this mode, the DELCD can perform much like an ECD, except that the DELCD is more selective for halogens and blind to oxygen. When possible, quantitate by the internal standard method, using a chlorinated/brominated compound for the internal standard peak. Although the DELCD will not be damaged by large quantities of chlorine/bromine, there is a short term loss of sensitivity for about an hour following the injection of 1µL of pure methylene chloride, for example.

1. Remove the hydrogen supply by turning it OFF, then disconnecting it at the GC's inlet bulkhead on the left hand side of the instrument.

2. Reduce the air flow to the DELCD to 25mL/min by turning the the air pressure trimpot setpoint down to 1 or 2psi. An additional 24" restrictor made of 0.001" I.D. tubing would be useful for fine pressure adjustment.

3. If you're using a capillary column, push the column through the FID jet until it just enters the ceramic tubing of the DELCD. This will improve peak shape as the column effluent will be discharged into the flowing airstream and immediately swept into the DELCD detector volume by the air make-up gas. (When switching back to the FID/DELCD combo mode, remember to pull the column back into the FID jet.)

4. The FID collector electrode allows some gas to escape from the FID combustion area, which is undesirable for the high sensitivity mode. Remove the FID collector electrode and replace it with a 1/4" cap fitting.

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General Operating Procedure continued

FID/DELCD - FID Only

1. Remove the DELCD heater wires from the push terminals. Remove the three DELCD collector and thermocouple wires (yellow, white and red) from the scew terminals.

2. Disconnect the DELCD detector assembly from the FID exhaust by using a wrench to loosen the 1/4" Swagelok fitting securing the two detector parts together.

3. Use a cap nut to seal the DELCD connection on the FID flameport.

4. Set the FID amplifier gain switch to HIGH for most hydrocarbon applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM. When peaks of interest are 20 seconds wide or more at the base and extra noise immunity is desired, set the gain switch to HIGH (filtered). This setting broadens the peaks slightly.

5. Set the FID hydrogen flow to 25mL/min, and the FID air supply flow to 250mL/min. The approximate pressures required are printed in the gas flow chart on the right-hand side of the GC.

6. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. The ignitor switch is located on the front panel of your SRI GC under the "DETECTOR PARAMETERS" heading (it is labelled vertically: "FLAME IGNITE").

7. Verify that the FID flame is lit by holding the shiny side of a chromed wrench directly in front of the collector outlet. If condensation becomes visible on the wrench surface, the flame is lit.

DELCD Only

1. Set the helium carrier gas flow to 10mL/min and the air make-up flow to 25mL/min. Clean, dry tank air helps to obtain the best achievable DELCD sensitivity and signal stability.

2. Set the DELCD reactor temperature setpoint to 260 (= 1000°C) by adjusting the trimpot on the top edge of the GC's front control panel. The DELCD will heat to about 254 and stabilize. The ceramic tube will glow bright red from the heat.

3. By adjusting the appropriate trimpot, set the thermostatted DELCD heater block temperature to 25°C higher than the "Final" temperature you have entered in the temperature program.

4. The DELCD amplifier is normally operated on LOW or MEDIUM gain.

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Troubleshooting and Maintenance

Installing the Spare DELCD Cell

Each SRI DELCD detector is shipped with a spare DELCD cell. Because the DELCD heater operates close to 1000°C, it will burn out and fail eventually. Follow the instructions below to remove the old cell and install the new one.

1. With the GC power OFF, remove the DELCD heater wires (2) from the push terminals and the DELCD thermocouple and collector wires (3) from the screw terminals.

2. Remove the DELCD cell by using a wrench to loosen the 1/4" fitting that secures it on the FID exhaust port or on the heater block. You may have to hold the insulation aside to freely access the fitting; it is soft and may be compressed by hand.

3. Position the new cell on the fitting with the label facing up, as the DELCDs are shown on the ***Overview*** page. Be sure to push the DELCD cell all the way into the FID.

4. Secure the new DELCD cell into place by tightening with a wrench the fitting that holds it onto the FID exhaust or the heater block.

5. Carefully lower the red lid to make sure that it does not touch the DELCD cell; the cell will crack if the lid hits it. There should be at least 0.5" of clearance between the red lid and the edge of the DELCD cell.

6. Sensitivity may improve for the first 24 hours of operating time with the new cell installed.

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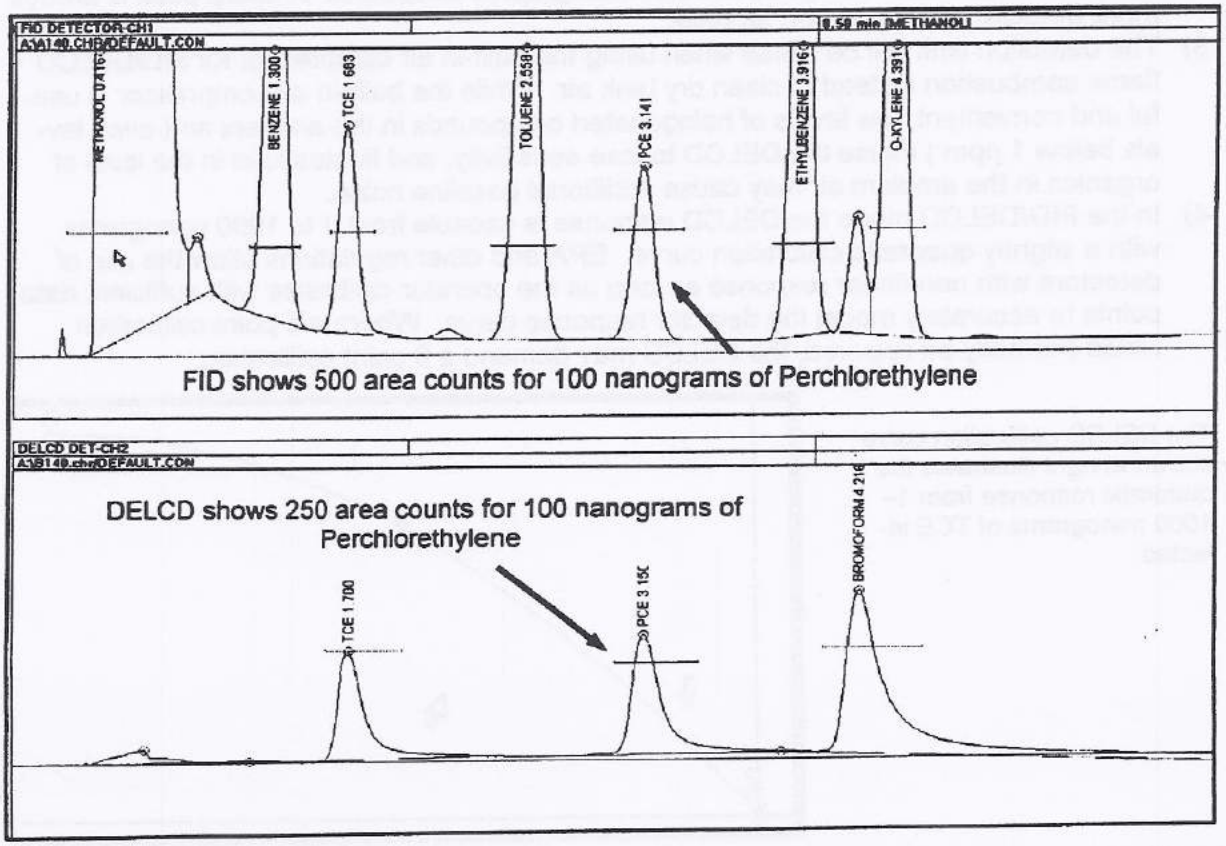
Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

In the combo mode, the DELCD is operated after the FID. The FID signal is usually connected to Channel 1 on the PeakSimple data system. The DELCD signal may be on Channel 2 or 3. Each detector amplifier is labeled at the factory with the data channel to which it has been connected. Detector signals may of course be connected to any available data channel by simply attaching the white and black signal wires to the screw terminals on the A/D board inside the GC.

1) Set the FID hydrogen and air flows for normal FID operation. This is typically 25 ml/min hydrogen (corresponds to 25 psi) and 250 ml/min air (typically 6 psi). The exact pressure required for each flow is labeled on the GC's right hand side.
2) Set the DELCD temperature setpoint to 260 using the front panel adjustments. This number actually represents 1000°C. The DELCD will heat up to about 254 and stabilize. The quartz collector electrode will appear a bright red color due to the 1000C temperature.
3) In the FID/DELCD combo mode, the FID is normally operated on high gain or on hi-filtered gain if the peaks are more than 10 second wide at the base. The hi-filtered gain position is identical to the high gain except that extra noise filtering results in a quieter baseline. The DELCD amplifier is normally operated on low gain. In this configuration the FID and DELCD produce approximately the same response to chlorinated peaks such as TCE (same peak area counts). The FID will generate approximately 4 area counts per nanogram injected on column while the DELCD will generate 2-4 area counts per nanogram of chlorinated hydrocarbon. (see example chromatogram below).



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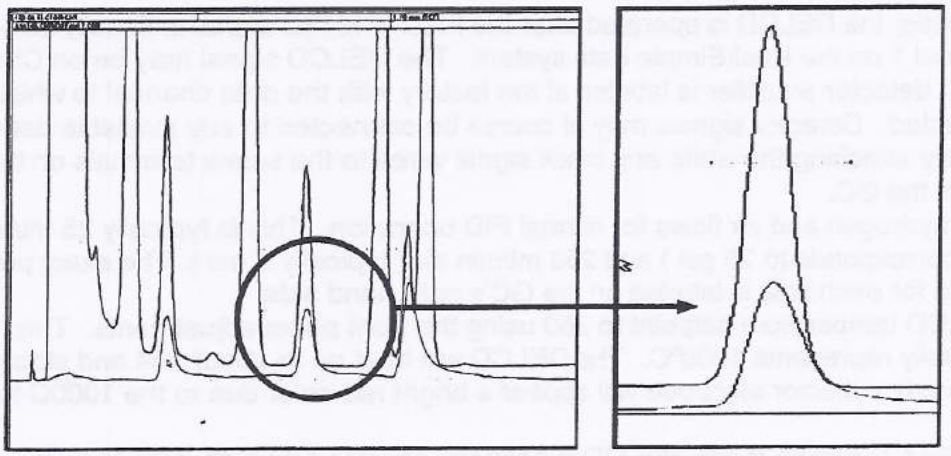
Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

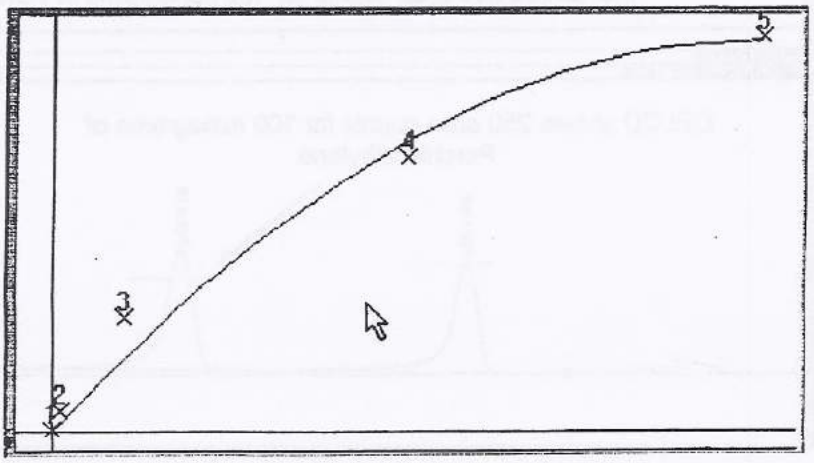


DELCD peak overlaid on FID peak for PCE, then expanded for clarity.

The smaller peak is the DELCD response.

1) As shown in the chromatogram above, the DELCD peak for PCE occurs at the same time as the FID peak for PCE. Notice that the DELCD peak exhibits a little bit of tailing compared to the FID response.
2) In the FID/DELCD combo mode, the minimum detectable amount is approximately 1 nanogram. Assuming a 1 microliter injection, this translates into approximately 1 ppm. The exact detection limit will depend on the analyte molecule (how much chlorine/ bromine in the compound) and the chromatographic conditions. A sharp peak is always more detectable than a short fat peak.
3) The detection limit will be worse when using the built-in air compressor for FID/DELCD flame combustion instead of clean dry tank air. While the built-in air compressor is useful and convenient, low levels of halogenated compounds in the ambient air (even levels below 1 ppm) cause the DELCD to lose sensitivity, and fluctuations in the level of organics in the ambient air may cause additional baseline noise.
4) In the FID/DELCD mode the DELCD response is useable from 1 to 1000 nanograms with a slightly quadratic calibration curve. EPA and other regulations allow the use of detectors with non-linear response as long as the operator calibrates with sufficient data points to accurately model the detector response curve. Where a 5 point calibration would normally be required, the DELCD may demand a 6 point calibration.

The DELCD calibration curve shown at right illustrates the quadratic response from 1–1000 nanograms of TCE injected



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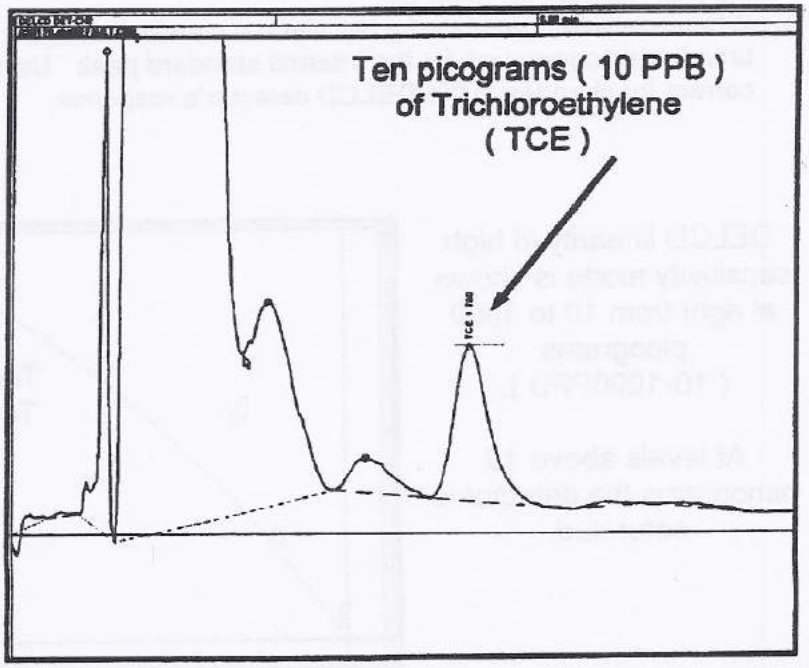
Chapter: Detectors

Topic: Operating the FID/DELCD in the high sensitivity DELCD only mode

1) The DELCD can be operated in a high sensitivity mode by eliminating the hydrogen from the reactions which lead up to the detection of the ClO2-BrO2. Because the chlorine/bromine atoms prefer to react with hydrogen to form non-detectable HCl-Hbr, than with oxygen to form detectable ClO2-BrO2 by a factor of 100-1000 to 1, eliminating the hydrogen improves the DELCD sensitivity by at least 100 times. Water must also be eliminated as at the high temperatures inside the DELCD, hydrogen becomes dissassociated from the H2O molecule and available as a reactant. In practice, this means turning off the hydrogen and using clean dry tank air (not the built-in air compressor).
2) Remove the hydrogen supply from the GC by disconnecting the hydrogen supply at the GC's inlet bulkhead on the left hand side of the instrument. Reduce the air flow to the DELCD to 50 ml/min by turning the air pressure setpoint down to 1-2 psi. An additional air flow restrictor consisting of 12" of .067 tubing (1/16', 1.58mm) with an internal diameter of .010 (.25mm) can easily be added to the air supply immediately below the detector to enable the flow to be controlled more precisely at higher pressures. With the extra restrictor installed a pressure setpoint of 10 psi will deliver an air flow of approximately 50 ml/min.
3) If using a capillary column, push the column through the FID jet until it just enters the ceramic tubing of the DELCD. This will improve the peak shape somewhat because the column effluent will be discharged into the flowing airstream and will be immediately swept into the DELCD detector volume. When switching back to FID/DELCD combo mode remember to pull the column back into the FID jet.
4) Remove the FID collector electrode and replace it with a 1/4' cap fitting. The FID collector electrode allows some gas to escape from the FID combustion area, and this is not desirable when operating in the high sensitivity mode.

The DELCD chromatogram shown at right illustrates the response to 10 picograms (1ul of 10 PPB) of TCE in the high senstivity mode.

Note that in high sensitivity mode, there is some response to the methanol solvent.



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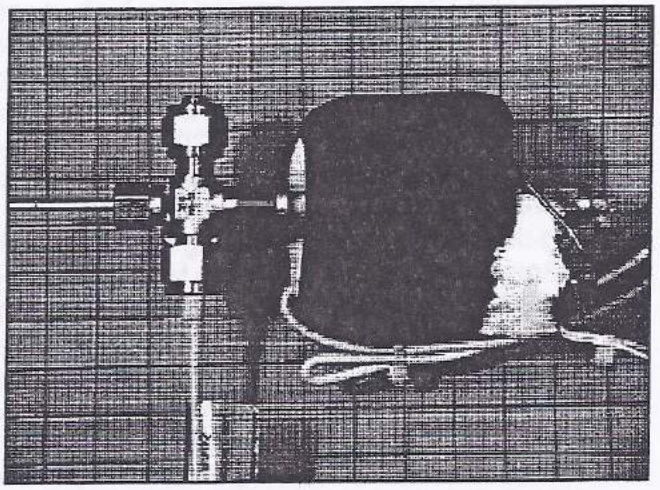
Chapter: Detectors

Topic: Operating the FID/DELCD in the high sensitivity DELCD only mode

The FID/DELCD detector is shown at right configured for the high sensitivity mode.

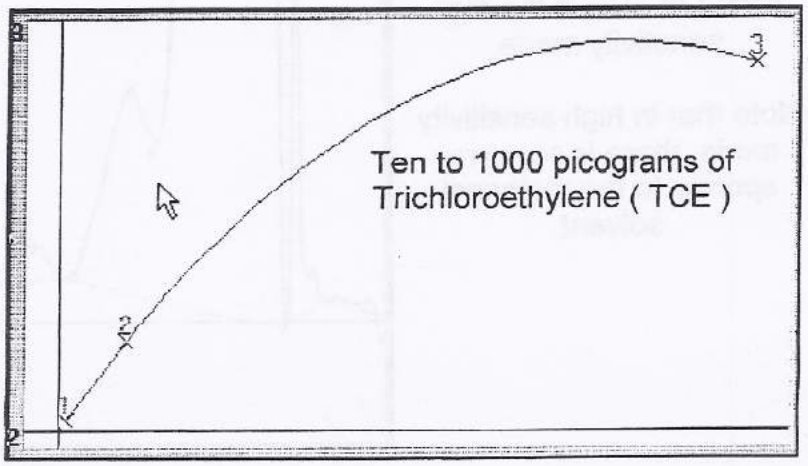
The collector electrode is removed and a 1/4" cap installed instead.



1) Just as the DELCD response curve is quadratic in the FID/DELCD combo mode, the response is also quadratic in the high sensitivity mode, but sensitivity is increased by 100-1000 times. In the high sensitivity mode the DELCD is most useful in the range of 1-1000 picograms which assuming a 1 microliter injection translates into 1-1000 PPB.
2) In the high sensitivity mode, the DELCD can perform much like an Electron Capture Detector (ECD) except that the DELCD is more selective for halogens and blind to oxygen.
3) Although the DELCD will not be damaged by large quantities of chlorine/bromine, there is a short term loss of sensitivity for an hour or so following the injection of 1 ul of Methylene Chloride for example.
4) When possible quantitate by the internal standard method, using a chlorinated/ brominated compound for the internal standard peak. Using an internal standard will correct for changes in the DELCD detector's response.

DELCD linearity in high sensitivity mode is shown at right from 10 to 1000 picograms (10-1000PPB).

At levels above 10 nanograms the detector is saturated.



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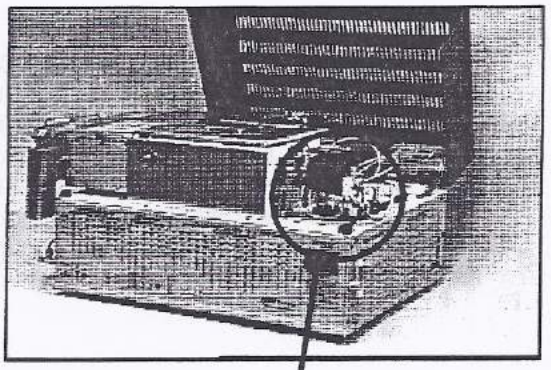
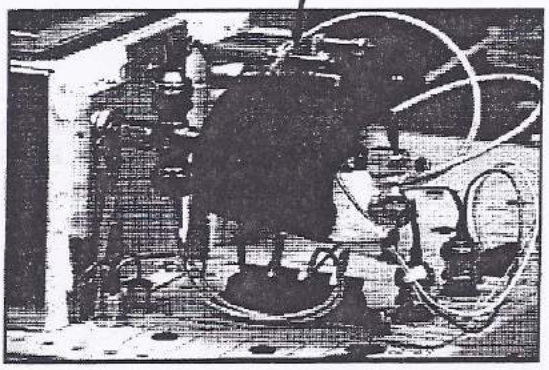
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

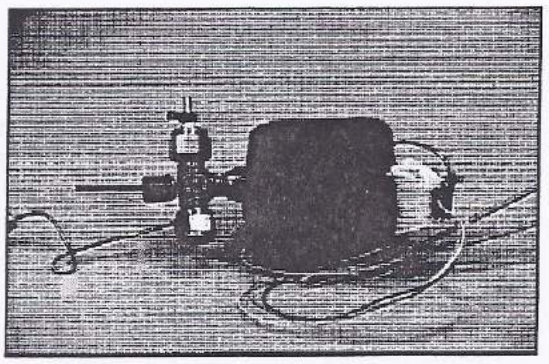
The DELCD detector is only available in combination with the FID detector.



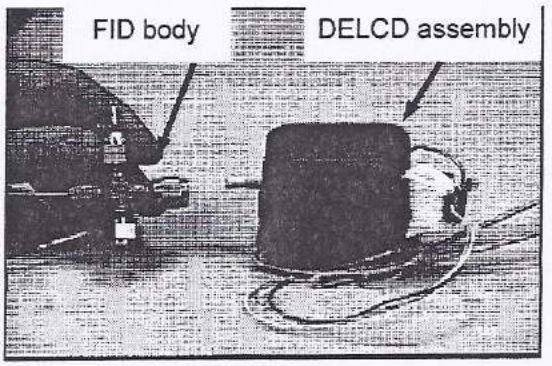
The FID/DELCD combo detector is mounted to a thermostatted aluminum heater block on the right hand side of the column oven.



The FID/DELCD combo detector is shown at right removed from the GC.



The DELCD part of the detector is the large black cylinder which mounts into the right hand port of the FID detector body. It can be separated from the FID body by loosening the 1/4" Swagelok nut and graphite ferrule which secures it in place.



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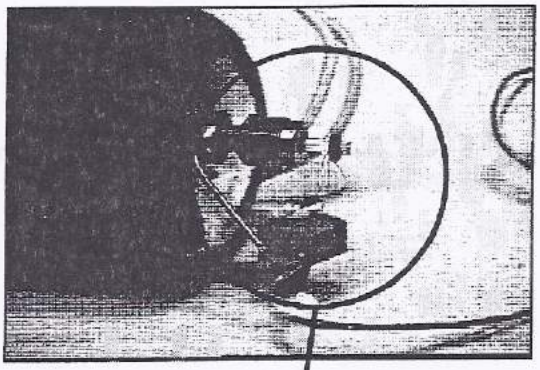
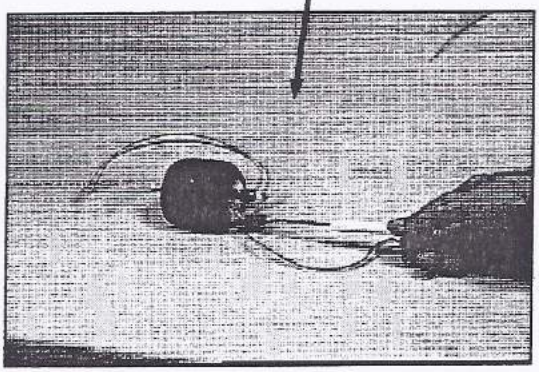
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

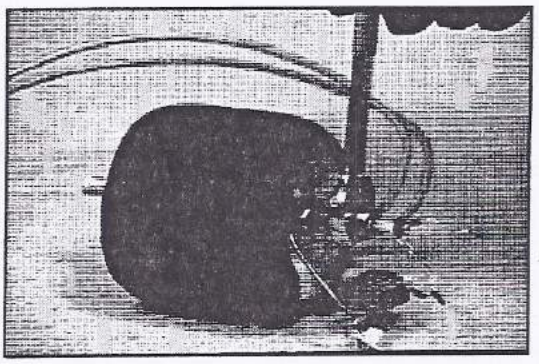
The DELCD collector electrode (part# 8670-1028) can be removed from the heater. Because the heater operates at close to 1000°C, it will fail eventually. A new heater (part # 8670-1027) is less expensive than the complete heater/collector assembly (part# 8670-1029), so it may make sense to remove the collector from the bad heater and re-install it into a new heater.



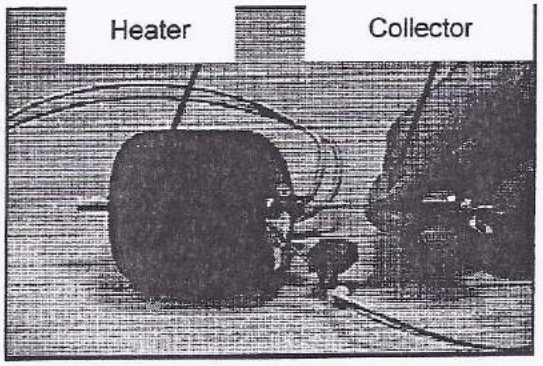
Dis-connect the three small platinum wires from the screw terminals and gently move them aside.



Using two wrenches to avoid rotating the fitting, loosen the 1/8" Swagelok nut and graphite ferrule which secures the collector electrode into the heater.



The collector can then be withdrawn from the heater.



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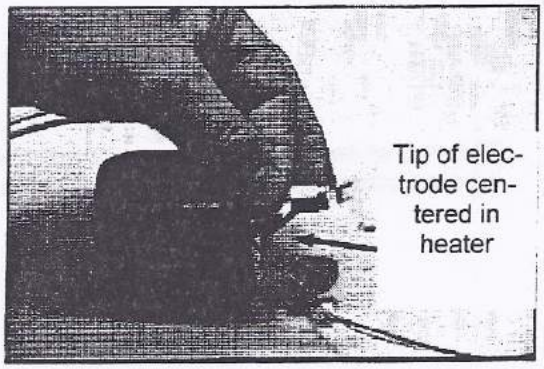
Chapter: Detectors

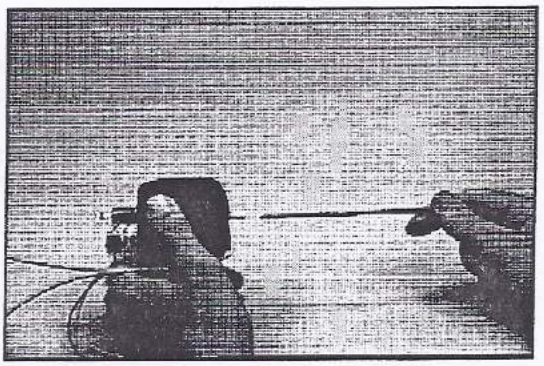
Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

When the collector electrode is re-installed in the new heater, it is important that the tip of the electrode is positioned in the center of the heater.

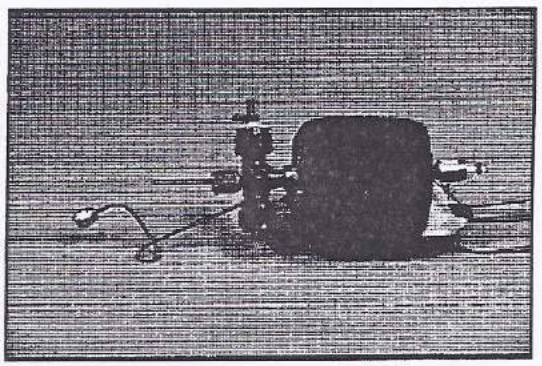


Tip of electrode centered in heater

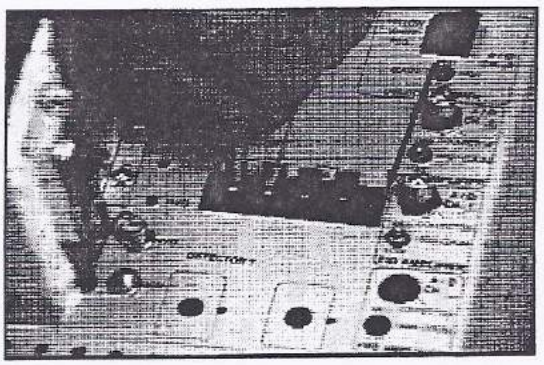
Use a file, rod, screwdriver or other long thin object as a gauge to verify that the electrode tip is centered in the new heater body. Gently re-position the electrode by sliding it through the graphite ferrule to get the proper adjustment. Finally, look down the bore of the heater and check to make sure that the tip of the electrode is centered in the bore of the heater, and is not bent to one side, touching the heater wall.



Connect the heater/collector assembly back onto the FID body. The heater/collector assembly should be inserted as far as possible into the FID body.



The two DELCD heater wires are connected to the push terminals on the deck of the GC which are labeled DELCD heater.



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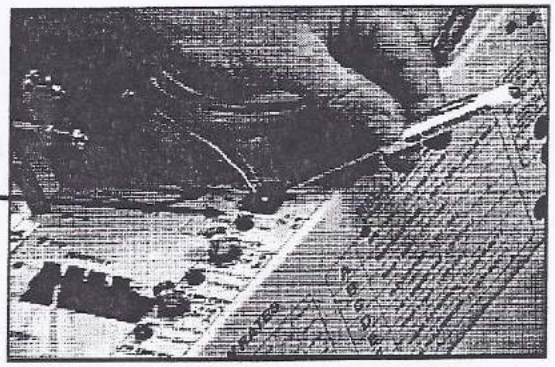
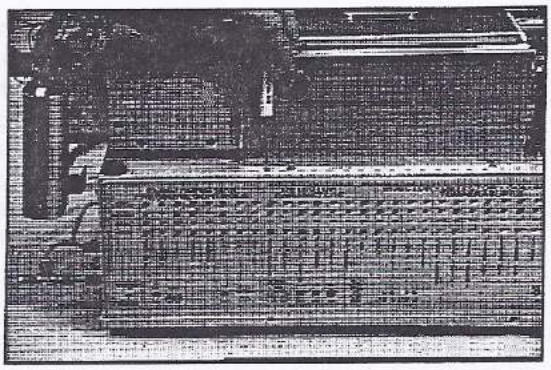
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

The red, white and yellow wires are inserted into the labelled screw terminals on the deck of the GC.

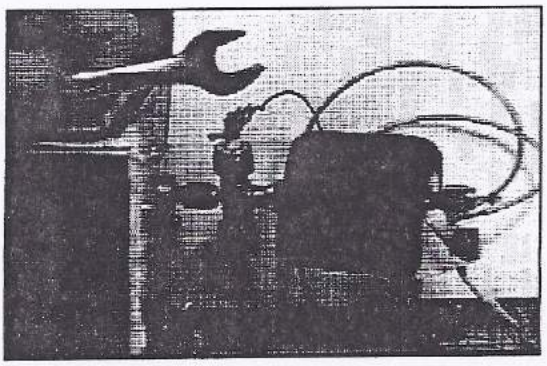


Set the DELCD heater setpoint temperature to 250. This is an arbitrary number which actually corresponds to a temperature of 1000°C. Better sensitivity can be obtained by raising the setpoint to 260 or 270, but at the cost of reduced heater lifetimes.



The actual temperature of the DELCD heater will equilibrate to about 7 degrees less than the setpoint within 10 minutes

Verify that the FID flame is lit by holding a shiny wrench or mirror above the FID collector electrode and looking for droplets of water condensation.



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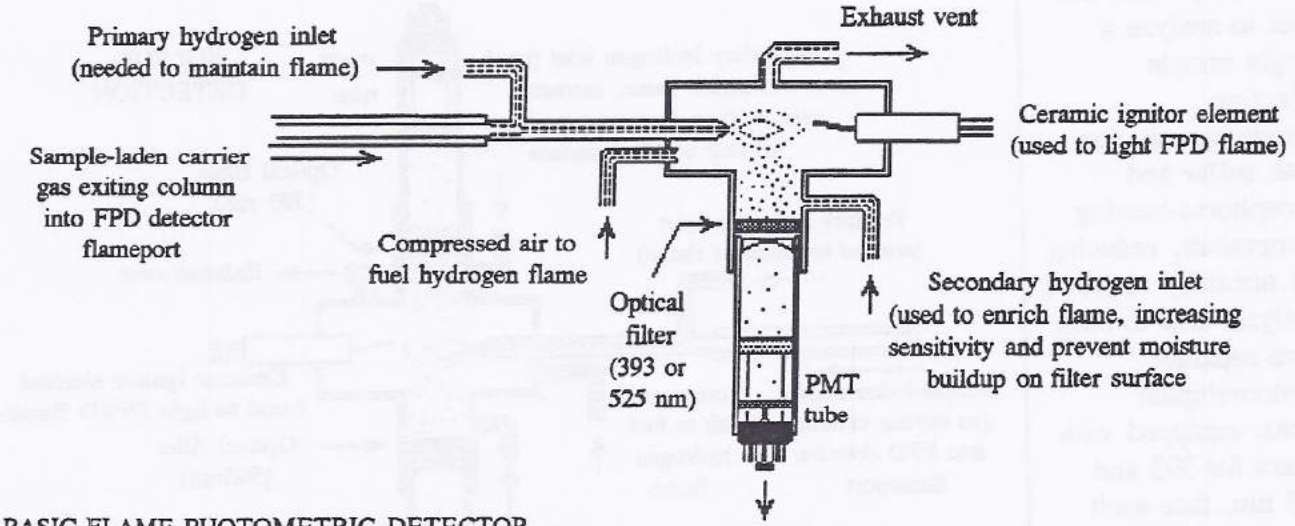
The Flame Photometric Detector, or FPD, as it is referred to, is typically used to analyze for sulfur or phosphorus-containing compounds, as the photomultiplier tube is extremely sensitive to a broad portion of the visible light spectrum, including those specific wavelengths emitted by the combustion of sulfur and/or phosphorus molecules in a hydrogen flame. Specific filtration inserted in the light path allows only the specifically desired wavelengths to pass unimpeded into the photomultiplier tube for quantitation.

Specificity for sulfur and phosphorus is obtained by the used of precision optical filters designed for use at 393 and 525 nanometers, the wavelengths of light that are emitted as the sulfur or phosphorus compounds elute from the analytical column and enter the flame of the detector. The hydrogen flame in the detector is invisible to the unaided eye because it does not give off any visible light, yet permits the photomultiplier tube electronics to establish a reference as a baseline or background value. When a sulfur molecule, for instance, enters the flame, a measurable quantity of blue light is emitted by the flame and this specific frequency of light is allowed to pass unimpeded through the filter and onto the measuring surface of the photomultiplier tube (PMT). The electrically-operated photomultiplier tube converts the quantitated emission of light into an analog signal that can be delivered to, and used by a data system for display and integration. The in-line optical filtration eliminates any interference from other compounds present in the sample-bearing carrier gas stream.



BASIC FLAME PHOTOMETRIC DETECTOR DIAGRAM

In addition to the standard single FPD detector configuration that may be used to quantitate either sulfur or phosphorus compounds with the appropriate in-line optical filter, SRI offers a Dual Flame Photometric Detector that permits the user to analyze a single sample simultaneously for both sulfur and phosphorus compounds. Two photomultiplier tubes, equipped with filters for 393 and 525 nm, face each other across the flame and produce simultaneous analyses from the same sample injection. SRI offers the only Dual Flame Photometric detector (DFPD) available today.

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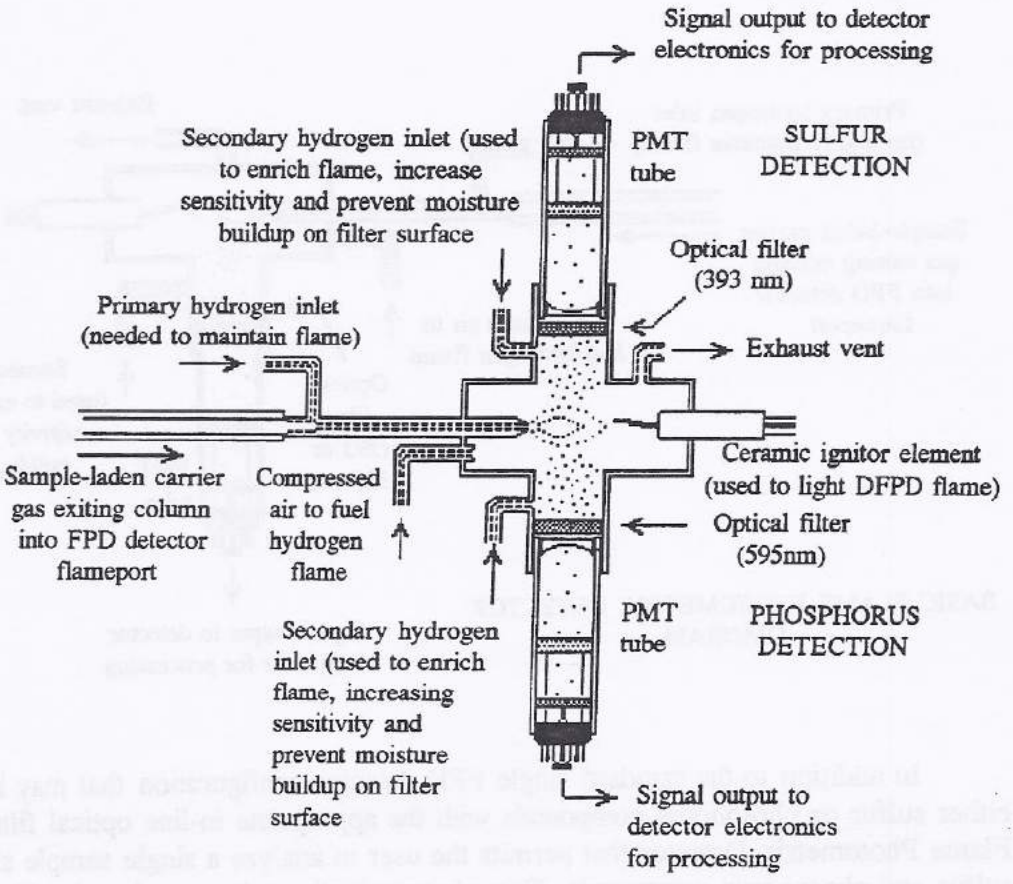
Chapter: DETECTORS

Topic: DFPD - Theory of Operation and General Information

The Dual Flame Photometric Detector, or DFPD, as it is referred to, is typically used to simultaneously analyze for sulfur and phosphorus-containing compounds. The photomultiplier tubes employed are extremely sensitive to a broad portion of the visible light spectrum. Specific filtration inserted into the light paths permits only the desired wavelengths to pass into the photomultiplier tubes and be quantitated. Specificity for sulfur and phosphorus is obtained by the used of precision optical filters designed for use at 393 and 525 nanometers, respectively, the wavelengths of light that are emitted as the sulfur and phosphorus compounds elute from the analytical column and enter the flame of the detector. The hydrogen flame in the detector is invisible to the unaided eye because it does not give off any visible light, but establishes a background value for the photomultiplier tube to reference as the baseline measurement.

When a sulfur molecule, for instance, enters the flame, a measurable quantity of blue light is emitted by the flame and this specific frequency of light is allowed to pass through the filter and onto the measuring surface of the photomultiplier tube (PMT). The electrically-operated photomultiplier tube converts the quantitated emission into an analog signal that can be delivered to the data system for display and integration. The process is similar for phosphorus-containing compounds. Each photomultiplier tube is equipped with full amplifier and data acquisition electronics to permit the simultaneous acquisition of both signals by a data system or other device.

The SRI Dual Flame Photometric Detector (DFPD) permits the user to analyze a single sample injection simultaneously for both sulfur and phosphorus-bearing compounds, reducing the normally required analysis time to half. Two separate photomultiplier tubes, equipped with filters for 393 and 525 nm, face each other across the flame and produce concurrent signals that may be analyzed by the data system. SRI offers the only Dual Flame Photometric detector (DFPD) available today.



BASIC DUAL FLAME PHOTOMETRIC DETECTOR DIAGRAM

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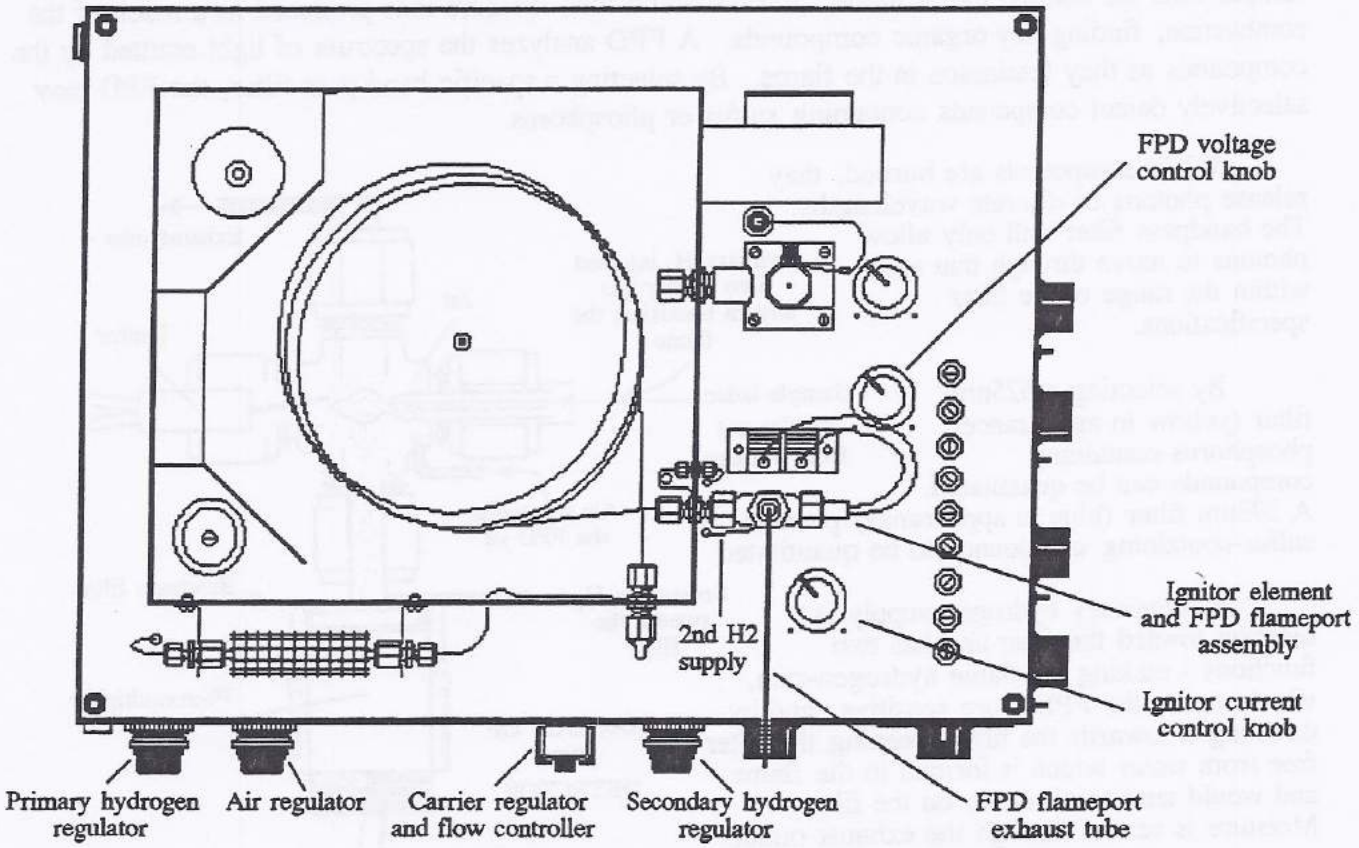
Chapter: DETECTORS

Topic: Operating The Flame Photometric Detector



The flame photometric detector (FPD) is primarily used for the analysis of compounds containing sulphur or phosphorus. The FPD consists of a flameport similar to a flame ionization detector (FID), but it lacks the collector electrode used to quantitate ionization. In the FPD detector, a photomultiplier tube (PMT) is positioned beneath the flameport for the purpose of monitoring the spectra emitted from the flame. A narrow wavelength optical bandpass filter is located between the flame and the photomultiplier tube window in order to selectively permit the passage of specific wavelengths of light into the photomultiplier tube. When testing for phosphorus-based components, a 525 nanometer filter is utilized. This filter appears as a yellow disk. When testing for sulphur-based components, a 393 namometer filter is utilized. This filter appears as a blue disk. When a sample component containing one of the specific chemicals elutes from the column and into the flame, the specific spectrum sought is emitted and is permitted to pass through the appropriate filter and strike the photomultiplier tube. This produces a quantitatable signal into and response from the detector electronics, which is relayed to the data system to be interpreted as a peak.

The SRI FPD incorporates several innovations: a compact PMT which can be mounted in a very small space, digital display of the PMT voltage being used, a variable output high voltage power supply and a secondary hydrogen inlet for purging the light path and enriching the flame boundary region with extra hydrogen.

It is important to note that the photomultiplier tube may be damaged by exposure to stray light when energized. De-energize the chromatograph prior to performing any maintenance that requires exposing the photomultiplier tube to ambient light.

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Chapter: DETECTORS

Topic: Operating The FPD Detector

The flame photometric detector (FPD) is similar to a flame ionization detector in that the sample exits the column into a flame. A FID would then measure ions produced as a result of the combustion, finding any organic compounds. A FPD analyzes the spectrum of light emitted by the compounds as they luminesce in the flame. By selecting a specific band pass filter, the FPD may selectively detect compounds containing sulfur or phosphorus.

When compounds are burned, they release photons of discrete wavelengths. The bandpass filter will only allow photons to move through that are within the range of the filter specifications.

By selecting a 525nm filter (yellow in appearance), phosphorus-containing compounds can be quantitated. A 393nm filter (blue in appearance) permits sulfur-containing compounds to be quantitated.

A secondary hydrogen supply is directed toward the filter and has two functions - making the flame hydrogen-rich, which makes the FPD more sensitive, and by directing it towards the filter, keeping the filter free from water which is formed in the flame and would tend to condense on the filter. Moisture is vented through the exhaust outlet.

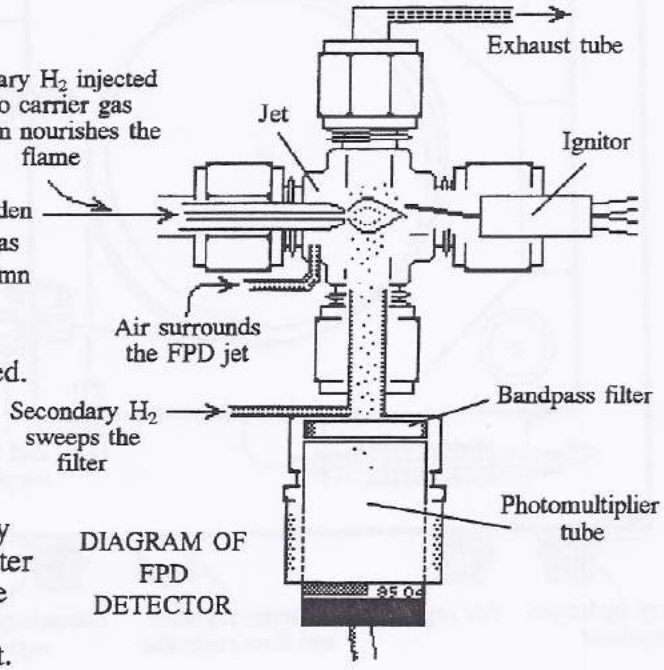

DIAGRAM OF FPD DETECTOR

Light must be kept out of the detector chamber so that only light from the flame will be analyzed.

FPD PRELIMINARY TESTING:

Verify that all gases are set to the proper flows. The primary hydrogen that feeds the flame is set to 30 ml/min (30 psi). The secondary hydrogen that sweeps the filter surface, and enriches the atmosphere over it, is set to 30 ml/min (psi). The flame's air supply is set to 100 ml/min. (5psi), and the carrier gas is set to between 20 and 50 ml/min.

Before the FPD is operated, perform a few simple tests. With the voltage to the PMT off and the flame unlit, remove the FPD exhaust tube. Look directly down into the FPD detector body. You should see the reflection of your eye deep in the center of the detector cavity. This verifies that the bandpass filter is properly aligned. If the reflection of your eye is not visible, realign the bandpass filter.

The next test is to check for possible light leaks. Replace the FPD exhaust tube. Set the FPD gain switch to LOW. With the flame unlit, set the FPD voltage to 500 volts. The voltage will read negative on the GC's LCD display. Lower the red protective oven cover. Take note of the millivolt reading produced by the FPD. The millivolt signal should be close to zero. Now raise the red protective oven cover and observe any change in the millivolt signal. When the detector is light-tight, the millivolt signal should rise no more than 10 millivolts. If the millivolt signal rises above this amount, inspect the FPD detector assembly for light leak sources.

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Chapter: DETECTORS

Topic: Servicing The FPD Detector

As has been stated before, the flame photometric detector (FPD) is similar to a flame ionization detector (FID) in that the sample exits the column into a flame. It differs in that the FID measures ions produced by organic compounds during combustion, while the FPD analyzes the spectrum of light emitted by the compounds as they luminesce in the flame. One of two filters available determines whether the detector "sees" sulphurous or phosphorous compounds.

Regardless of which filter is in use, it is important that the filter be properly installed, clean, and free from dust, debris, and particulate matter. The bandpass filter will only allow photons within the bandpass of the filter specifications to pass, but sensitivity may be reduced if the filter is improperly installed or dirty.

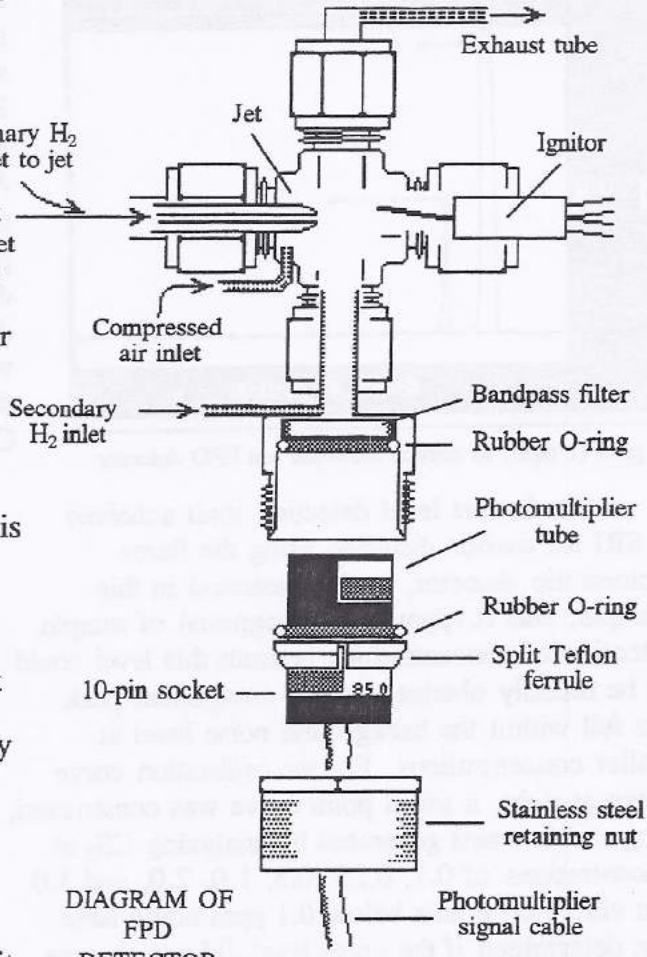

DIAGRAM OF FPD DETECTOR

As illustrated in the diagram at right, either of the filters (393nm or 525nm) installs into the lower extension of the FPD assembly with the mirrored surface of the lens facing the flame (up). The blue (sulphur) or yellow (phosphorus) side of the filter should face the lens of the photomultiplier tube (down). A rubber O-ring is inserted just below the lens in the lower assembly extension to secure the lens in the stainless steel body. The lens should seat fully in the stainless steel body, so that if the operator temporarily removes the FPD exhaust tube assembly and looks down into the FPD body, the reflection of the eye should be clearly seen in the visible mirrored surface. A misaligned lens will not permit viewing the eye's reflection.

The miniature photomultiplier tube (PMT) is connected to its 10-pin socket, and a rubber O-ring is slid over the PMT body until it is just above the point where the photomultiplier tube body meets the socket. Just beneath this O-ring, a split Teflon ferrule is mounted so that it sits on the socket, just below the point where the tube body meets the socket. This is also just below the rubber O-ring. Inspect the lens of the photomultiplier tube for dirt or debris. Use care in cleaning this lens, if cleaning is necessary.

The tube / socket assembly is then inserted into the lower FPD assembly body and held in place by the knurled stainless steel retaining nut. When the FPD detector assembly is fully reassembled, pack any excess signal cable from the socket back into the FPD chassis orifice.

This discussion of the servicing of the FPD detector will be especially useful for users who must change filters on a regular basis to switch from sulphur mode over to phosphorus mode and back again. The FPD detector is provided with one user-specified filter. Replacement and secondary filters are also available from SRI Instruments.

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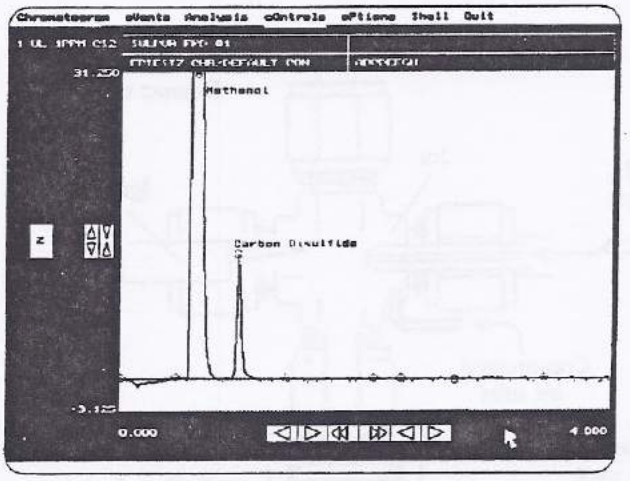
Chapter: DETECTORS

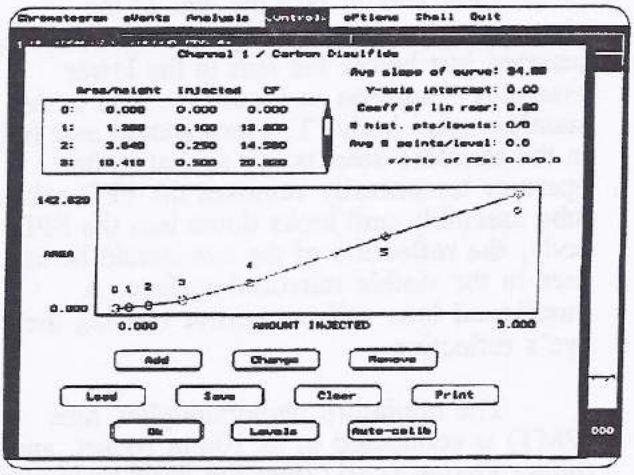
Topic: Demonstration of The Detection Limit of The FPD Detector

The flame photometric detector, or FPD, as it is commonly referred to, is capable of extreme sensitivity and selectivity. The selectivity is obtained by the use of optical filters placed in the light path to the photomultiplier tube. These filters permit the specific passage of 393 and 595 nm wavelength light emitted when either sulphur or phosphorus-containing compounds are burned in a hydrogen flame. The sensitivity of the detector is the product of the combination of the design of the photomultiplier tube and the SRI detector electronics design coupled with the optimization of gas flow through the detector. The SRI design is simple and readily permits being coupled with an FID detector or a second photomultiplier tube for simultaneous sulphur/phosphorus analysis. Although the one ppm (1 nanogram) carbon disulfide (CS_2) peak shown at left is a clean, crisp peak, it is by no means close to the limit of detectability of the SRI FPD detector. Carbon disulfide produces the following calibration curve when concentrations, as in this example (shown in the screen illustrated below) of 0.1 to 3.0 ppm CS_2, are plotted on the data system screen.

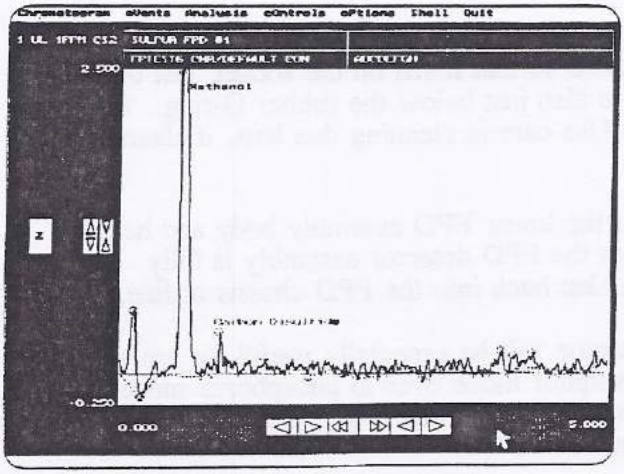


1 ppm (1 ngm) of carbon disulfide via FPD detector

The lowest level detection limit acheived by SRI for carbon disulfide using the flame photometric detector, as demonstrated in this example, was 0.1ppm (0.1 nanograms) of sample. Detection of concentrations beneath this level could not be reliably obtained, as the component peak area fell within the background noise level at smaller concentrations. For the calibration curve shown at right, a seven point curve was constructed, using six data sets generated by analyzing CS_2 at concentrations of 0.1, 0.25, 0.5, 1.0, 2.0, and 3.0 ppm via FPD. Points below 0.1 ppm could have been determined if the noise level did not obscure the peak areas, preventing integration. The 0.1 ppm peak is at a level approximately twice the background noise level. It is not recommended to attempt to establish detection limits below this level. As can be seen in the screen at left, the CS_2 peak clearly stands out above the background noise even at the one-tenth of one nanogram level (0.1 ppm or 100 ppb), facilitating quantitation of the carbon disulfide. Comparable detection limits can be expected for other similar compounds when operating the flame photometric detector in the sulphur or phosphorus mode.



PeakSimple calibration curve for CS_2 via SRI FPD



0.1 ppm of CS_2 detectable well above background

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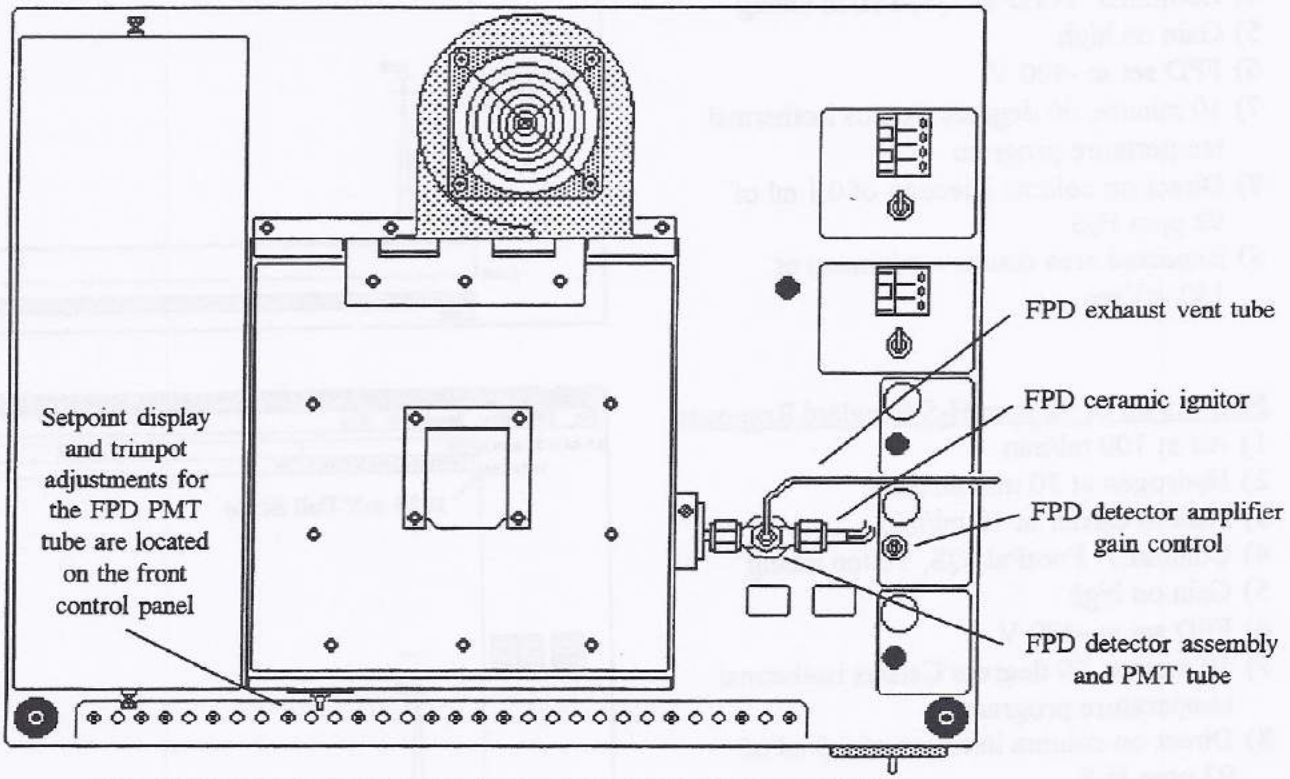
Chapter: DETECTORS

Topic: Proper Photomultiplier Tube Operating Voltage For FPD

The photomultiplier tube employed in the SRI flame photometric detector (FPD) is a new compact design that offers optimum performance and a long service period when operated at the recommended operating voltage. Photomultiplier tubes used by different manufacturers require distinct voltage levels to drive the tube for proper response. The SRI version requires a drive voltage that is lower than many, and in fact, is lower than the operating voltage used in earlier FPD detectors manufactured by SRI prior to use of this compact photomultiplier tube.



TOP VIEW OF FPD DETECTOR LOCATION ON 8610C GC CHASSIS

The Hamamatsu compact photomultiplier tube (PMT) used in current SRI FPD detectors has a recommended operating voltage of 300 volts. At no time is it necessary or advisable to operate this PMT tube at a voltage higher than this. Operation of the PMT tube at voltages higher than 300 volts will result in reduced PMT tube life, and a consequential loss of analysis time. The FPD tube voltage may be displayed and adjusted from the GC front control panel and the associated setpoint trimpot. The PMT tube is a consumable part, like septa and photoionization lamps, and is not covered by an SRI warranty. Any photomultiplier tube warranty issues or concerns must be communicated directly to Hamamatsu. A conservative PMT drive voltage is inherently more beneficial than any perceived gain to be obtained at higher drive voltages. Replacement photomultiplier tubes are available for purchase from SRI Instruments and directly from Hamamatsu.

DO NOT EXCEED 300 VOLTS

Compact photomultiplier tube used in current SRI FPD detector design (side view, shown with O-ring and Teflon seat on tube body)

Top view of PMT tube, with lens visible

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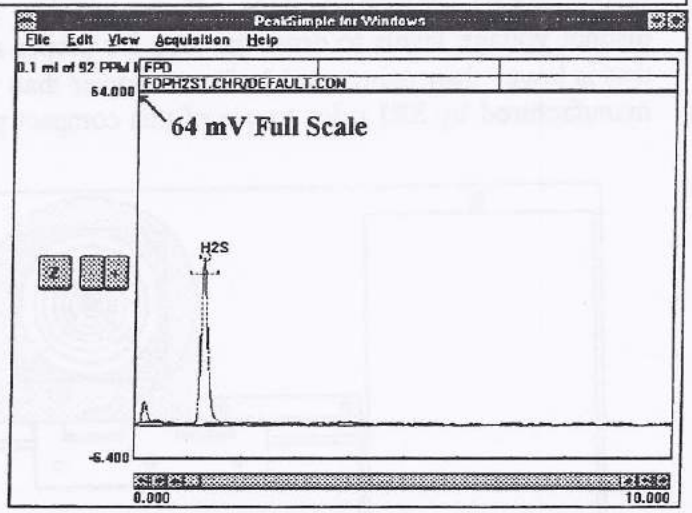
Chapter: DETECTORS

Topic: FPD Nonlinear Sulfur Response

The FPD sulfur response curve is extremely nonlinear even over very small ranges (see diagram below). Thorough calibrations must be developed with multiple data points and limited range in order to accurately quantitate desired components.

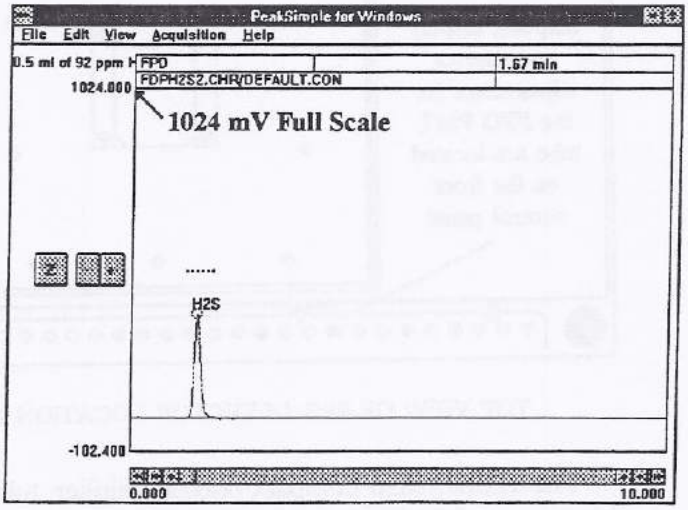
FPD 0.1 ml of 92 ppm H_2S Standard Response

1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 0.1 ml of 92 ppm H_2S
9) Expected area counts a minimum of 150 mVsec



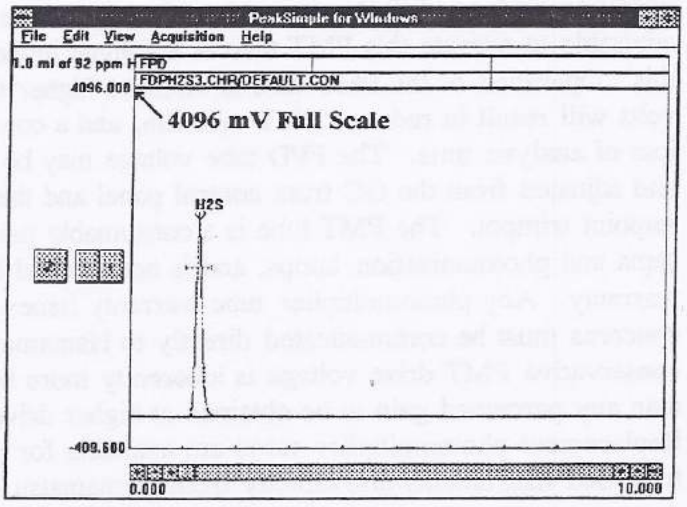
FPD 0.5 ml of 92 ppm H_2S Standard Response

1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 0.5 ml of 92 ppm H_2S
9) Expected area counts a minimum of 1500 mVsec



FPD 1.0 ml of 92 ppm H_2S Standard Response

1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 1.0 ml of 92 ppm H_2S
9) Expected area counts a minimum of 10000 mVsec



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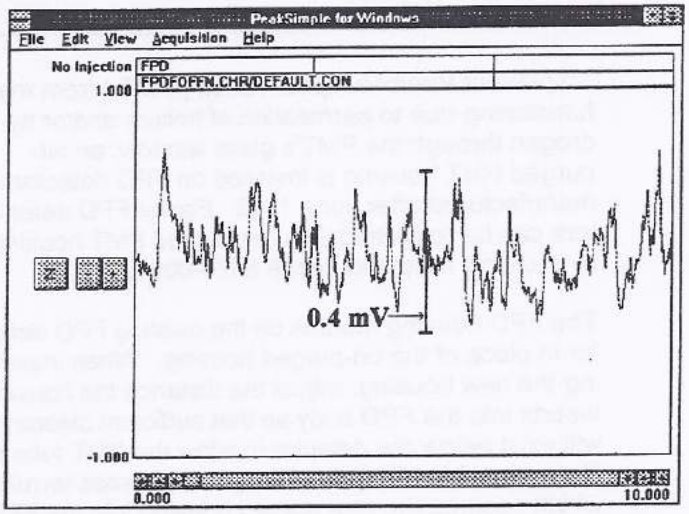
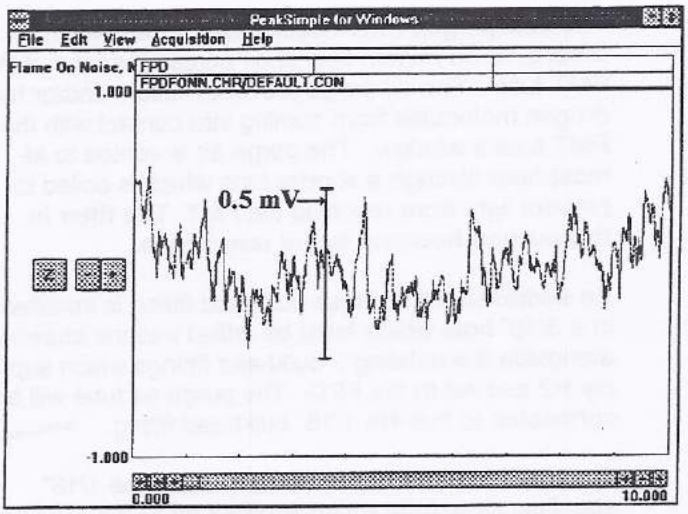
FPD Expected Performance



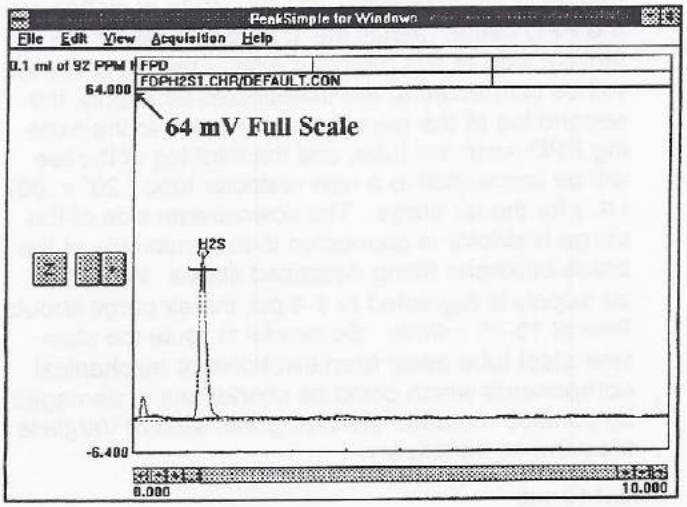
FPD Flame Off Noise

1) Air supply turned off
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Gain on high
5) FPD set at -400 V
6) 10 minute, 60 degrees Celsius isothermal temperature program
7) No injection



FPD Flame on Noise

1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Gain on high
5) FPD set at -400 V
6) 10 minute, 60 degrees Celsius isothermal temperature program
7) No injection



FPD 0.1 ml of 92 ppm H_2S Standard Response

1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 0.1 ml of 92 ppm H_2S
9) Expected area counts a minimum of 150 mVsec

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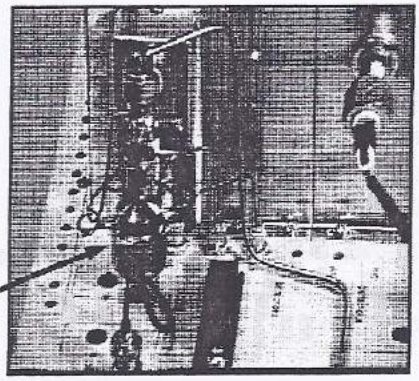
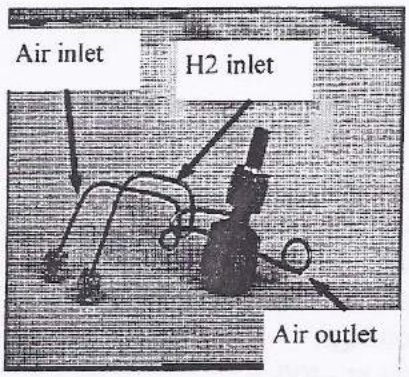
Chapter: FPD DETECTOR

Topic: Retrofit of air purged PMT tube housing

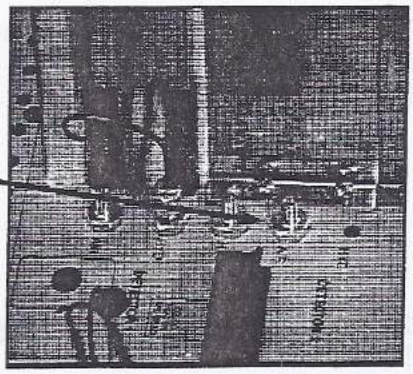
To prevent PhotoMultiplier Tubes (PMT) from malfunctioning due to permeation of helium and/or hydrogen through the PMT's glass window, an air-purged PMT housing is installed on FPD detectors manufactured after June 1998. Earlier FPD detectors can be retrofitted with the purged PMT housing by ordering retrofit kit part # 8670-0084.



The FPD housing mounts on the existing FPD detector in place of the un-purged housing. When installing the new housing, adjust the distance the housing inserts into the FPD body so that sufficient clearance will exist below the detector to allow the PMT tube to fit comfortably. Only then snug up the brass ferrule which secures the new purged housing into the FPD body.

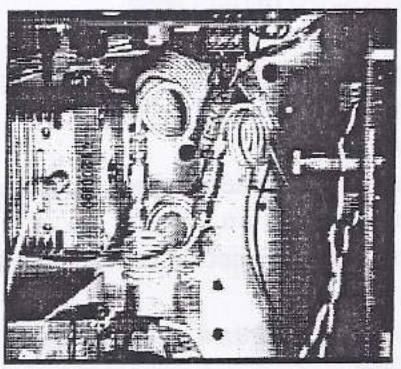


The new purged PMT housing has a tube which directs a 10-30 ml/min flow of air across the face of the PMT tube. The air purge prevents helium and/or hydrogen molecules from coming into contact with the PMT tube's window. The purge air is vented to atmosphere through a shorter tube which is coiled to prevent light from reaching the PMT. **The filter in the purged housing is not removable.**



An additional 1/16" brass bulkhead fitting is installed in a 3/16" hole which must be drilled into the chassis alongside the existing 3 bulkhead fittings which supply H2 and Air to the FPD. The purge air tube will be connected to this 4th 1/16" bulkhead fitting.

On the inside of the GC chassis, locate the 1/16" stainless steel tube which supplies air to the FPD detector. This tube acts as a restrictor so that 3-4 PSI results in approximately 100 ml/minute of air flow to the FPD flame. Install the 1/16" brass tee on the upstream side of this restrictor tube. One leg of the tee will be connected to the unrestricted air supply, the second leg of the tee will be connected to the existing FPD restrictor tube, and the third leg of the tee will be connected to a new restrictor tube (20" x .007 i.d.) for the air purge. The downstream side of the purge restrictor is connected to the underside of the brass bulkhead fitting described above. When the air supply is regulated to 3-4 psi, the air purge should flow at 10-30 ml/min. Be careful to route the stainless steel tube away from electronic or mechanical components which could be shorted out or damaged by contact. Insulate the tubing with tape or Varglass sleeving as necessary.



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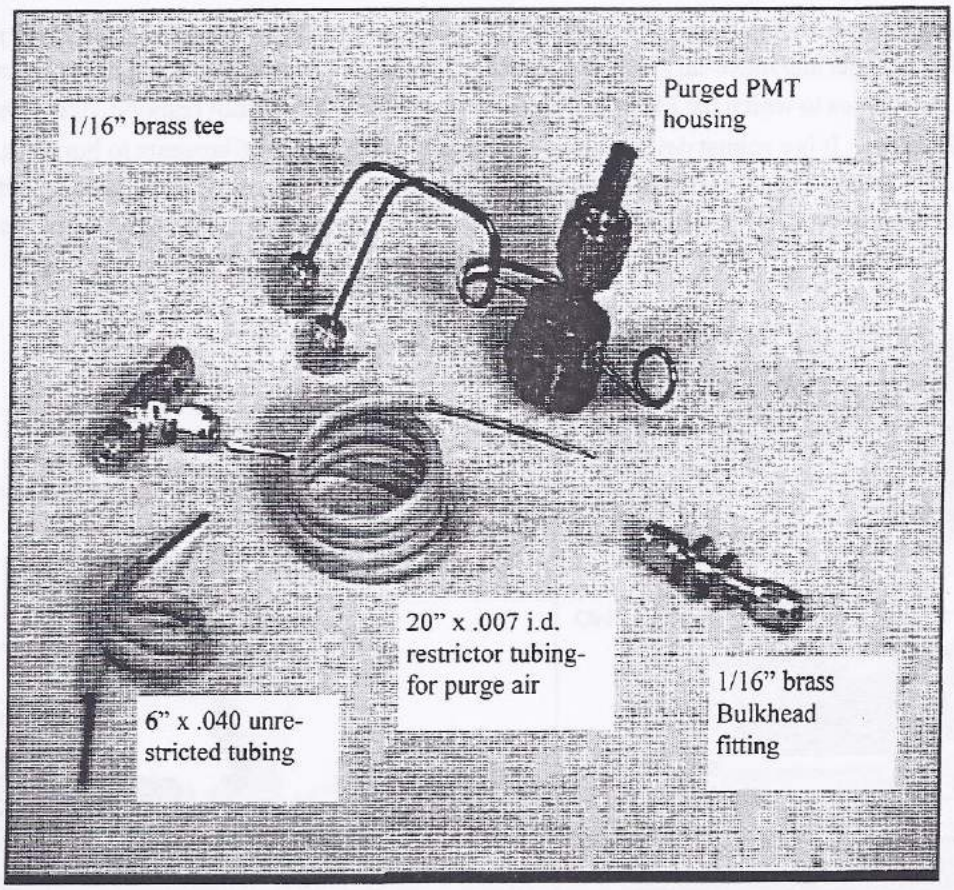
Chapter: FPD DETECTOR

Topic: Retrofit of air purged PMT tube housing



Contents of air purged PMT housing retrofit kit for FPD detector. SRI Part# 8670-0084.

1) Purged PMT housing (specify sulfur or phosphorus filter) filter is not removable.

2) 1/16" brass tee

3) 1/16" brass bulkhead fitting

4) 20" x .007 i.d. restrictor tubing with varglass insulation

5) 6" x .040 i.d. unrestricted tubing with varglass insulation for connecting the tee to the air supply

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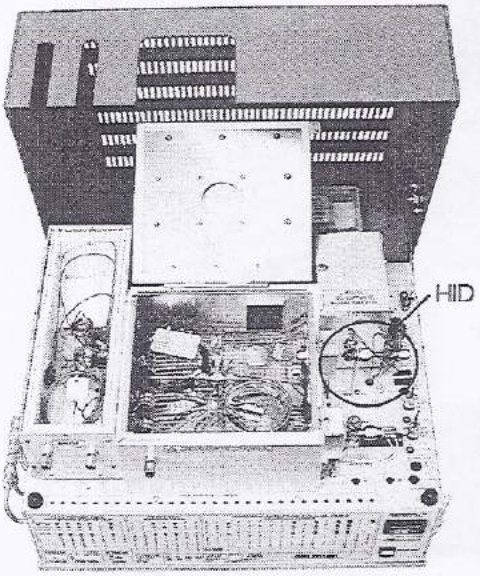
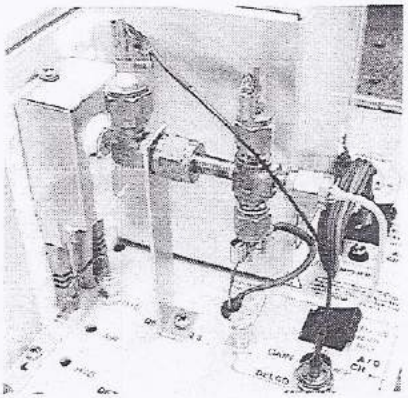
Overview

The Helium Ionization Detector is a universal detector, responding to all molecules except neon. It requires only helium carrier and make-up gas, and is sensitive to the low ppm range. The HID is particularly useful for volatile inorganics to which the FID and other selective detectors will not respond, like NOx, CO, CO_2, O_2, N_2, H_2S and H_2. It is a robust detector that, unlike the TCD, has no filaments to burn out. The SRI HID consists of a detector body, a collector electrode, an arc electrode assembly, and a thermostatted heater block which can be heated to 375°C. In SRI GCs, the HID is mounted on the right-hand side of the Column Oven.

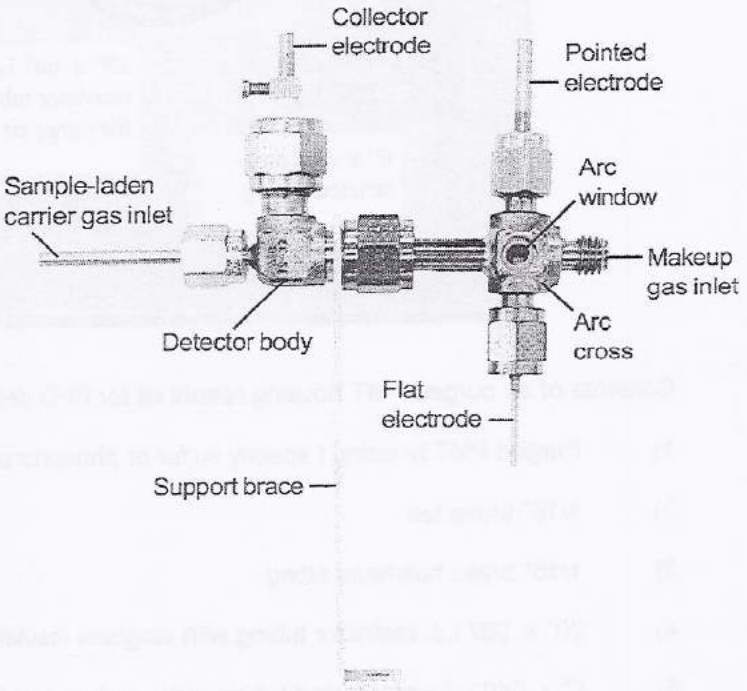


HID detector between TCD and FID detectors on an SRI GC

Close-up of the same HID detector



HID detector removed from GC and heater block



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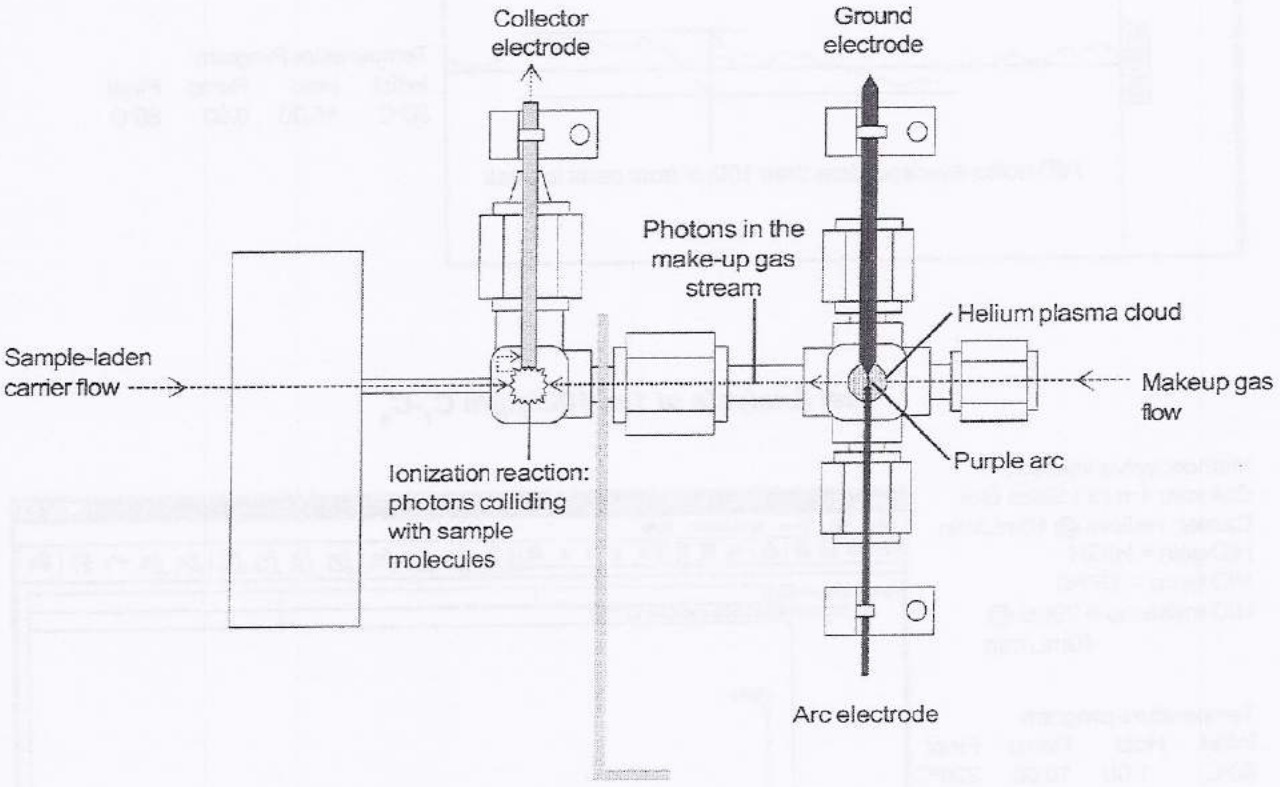
Theory of Operation

The SRI HID detector uses two electrodes which support a low current arc through the helium make-up gas flow. The helium molecules between the electrodes are elevated from ground state to form a helium plasma cloud. As the helium molecules collapse back to ground state, they give off a photon. The sample molecules are ionized when they collide with these photons. All compounds having an ionization potential lower than 17.7eV are ionized upon contact with photons from the helium cloud. The ionized component molecules are then attracted to a collector electrode, amplified, and output to the PeakSimple data system.



NOTE: If the arc electrode is covered with Teflon™ (translucent) insulation, it should leave 1mm of its tip exposed. If the flat electrode is covered with ceramic (white) insulation, then the tip should be flush with the edge of the insulation sleeve. There should be a 1-2mm gap between the arc electrodes, and this gap should be centered in the arc cross.

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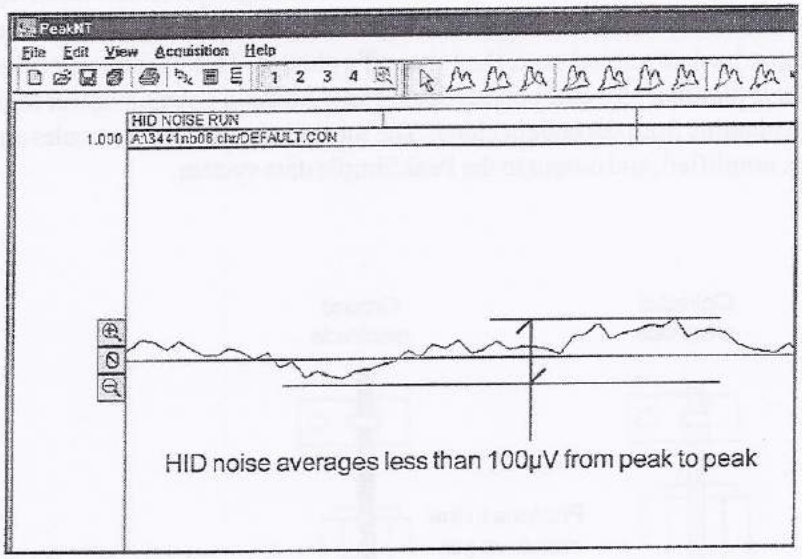
Expected Performance



HID noise run

Columns: 1m Mol. Sieve, 2m Hayesep-D
Carrier: Helium @ 10mL/min
HID gain = HIGH
HID current = 70
HID temp = 200°C

Temperature Program:

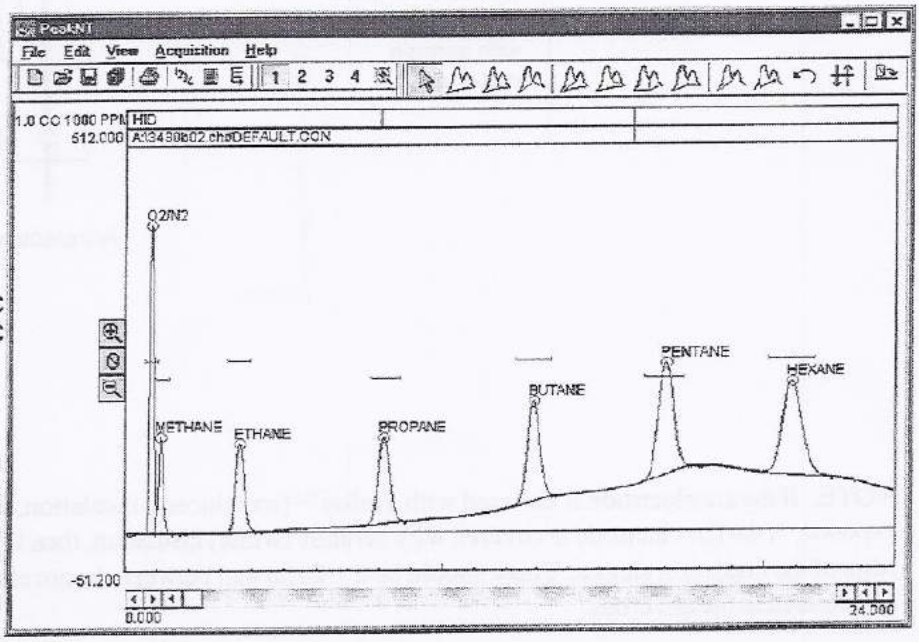
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

Test Analysis of 1cc 1000ppm C_1-C_6

Method: valve injection
Column: 1m (3') Silica Gel
Carrier: Helium @ 10mL/min
HID gain = HIGH
HID temp = 150°C
HID make-up = 29psi @ 40mL/min

Temperature program:

Initial	Hold	Ramp	Final
50°C	1.00	10.00	220°C
220°C	10.00	0.00	220°C



Results:

Component	Retention	Area
O2/N2	0.766	3350.0970
Methane	1.066	1163.1965
Ethane	3.550	2161.0940
Propane	8.083	3001.6200
Butane	12.850	3958.3250
Pentane	16.950	4849.9755
Hexane	20.800	5023.0105
	total	23507.3185

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General Operating Procedure

1. Set the HID amplifier gain switch to HIGH for most applications from the ppm level to 1%. Use the MEDIUM gain setting for slightly more concentrated samples.

2. Set the helium make-up gas flow to 40mL/min, and the helium carrier gas flow to 10mL/min. Clean, high purity helium is best; moisture, air, and other contaminants can cause problems.

3. Set the HID temperature to 200°C. This temperature will help prevent moisture accumulation in the detector's arc assembly.

4. Zero the data system signal, then switch ON the HID current; the switch is located on the GC's front control panel under "DETECTOR PARAMETERS." Set the HID current at 100 using the trimpot setpoint on the top edge of the front control panel.

5. When the HID is OFF and the signal zeroed, and the HID is then turned ON, the milliVolt offset at HIGH gain setting should be 200-800mV. A higher offset means more sensitivity, but less dynamic range. If the offset is less than 200, the arc and ground electrodes are probably too close.

6. Observe the arc window; if you can see the purple arc between the ground and arc electrodes, proceed to step 7. If the arc goes sideways to the detector body instead of down to the ground electrode, then the gap between the electrodes is too large. If you cannot see the arc,

   A. Use a multimeter to check the voltage between the arc and ground electrodes. With the HID current at 100, the voltage reading should be greater than 200VDC (our readings average around 240VDC).

   B. Look through the arc window at the arc and ground electrodes. If they appear to be touching, disconnect the red electrode lead wire then check the continuity between the electrodes using a multimeter; the reading should be open or infinite.

   C. If the continuity between the electrodes is not open, re-gap the electrodes.

7. Let the milliVolt reading stabilize, then begin the analytical run.

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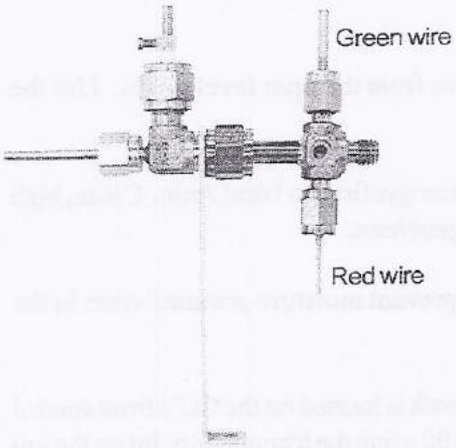
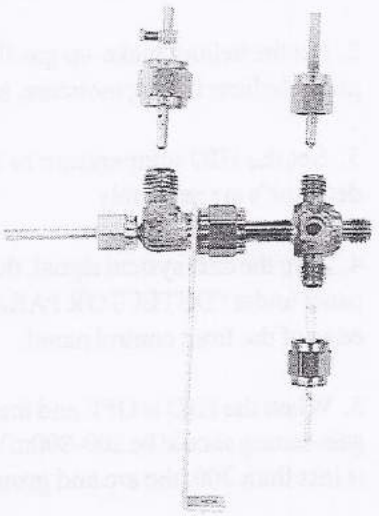
Cleaning the HID

If your HID baseline seems noisy, try cleaning the electrodes following the steps below. Over time, the HID electrodes can develop a coating of soot, which can cause the arc to flicker or change position, resulting in sudden baseline jumps.



1. Unclip the amplifier lead and slide it off the collector electrode. Unclip and remove the leads from the pointed and flat electrodes (note that the green wire is connected to the pointed electrode, and the red wire is connected to the flat electrode).

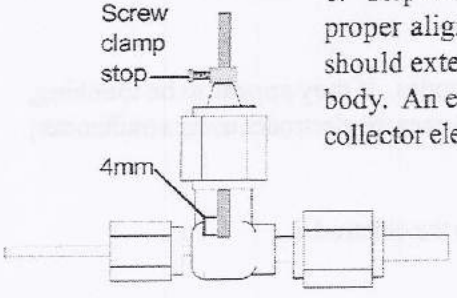


2. Remove the the arc and ground electrodes by loosening the 1/8" fittings that hold the electrodes in the arc cross.

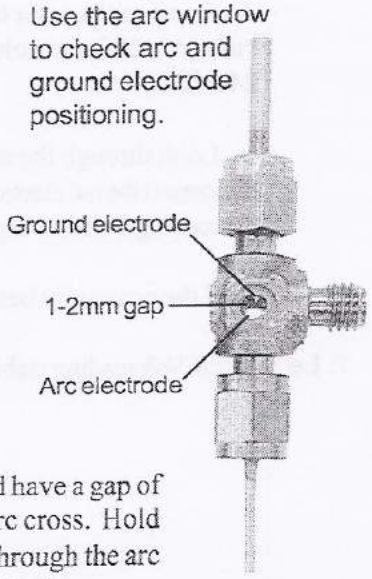
3. Remove the collector electrode by loosening the 1/4" fitting that secures it in the detector body.

4. Use a piece of 100-400 grain sandpaper to clean the surface of the collector electrode and the point of the ground electrode. Sand the tip of the arc electrode so that it is flush against the ceramic insulation, and to remove any residue. While handling the electrodes, try to minimize hand contact by holding them with a clean paper towel.

5. Remove any sanding residue from the electrodes using a paper towel optionally moistened with methanol or another quick-evaporating solvent.



6. Replace the electrodes and check for proper alignment. The collector electrode should extend about 4mm into the detector body. An existing screw clamp stop on the collector electrode should allow replacement without readjustment. Should adjustment be required, loosen the screw clamp to position the electrode, then tighten it to hold the position. To position the arc and ground electrodes, remove the arc cross from the detector body by loosening the 1/4" fitting connecting the two parts of the detector (this fitting also secures the support brace). The ground and arc electrodes should have a gap of about 1-2mm (0.040-0.080") between them, with the gap centered in the arc cross. Hold the arc cross up to the light and verify the electrodes' positions by looking through the arc window. Once the electrodes are positioned, tighten them securely with a wrench.



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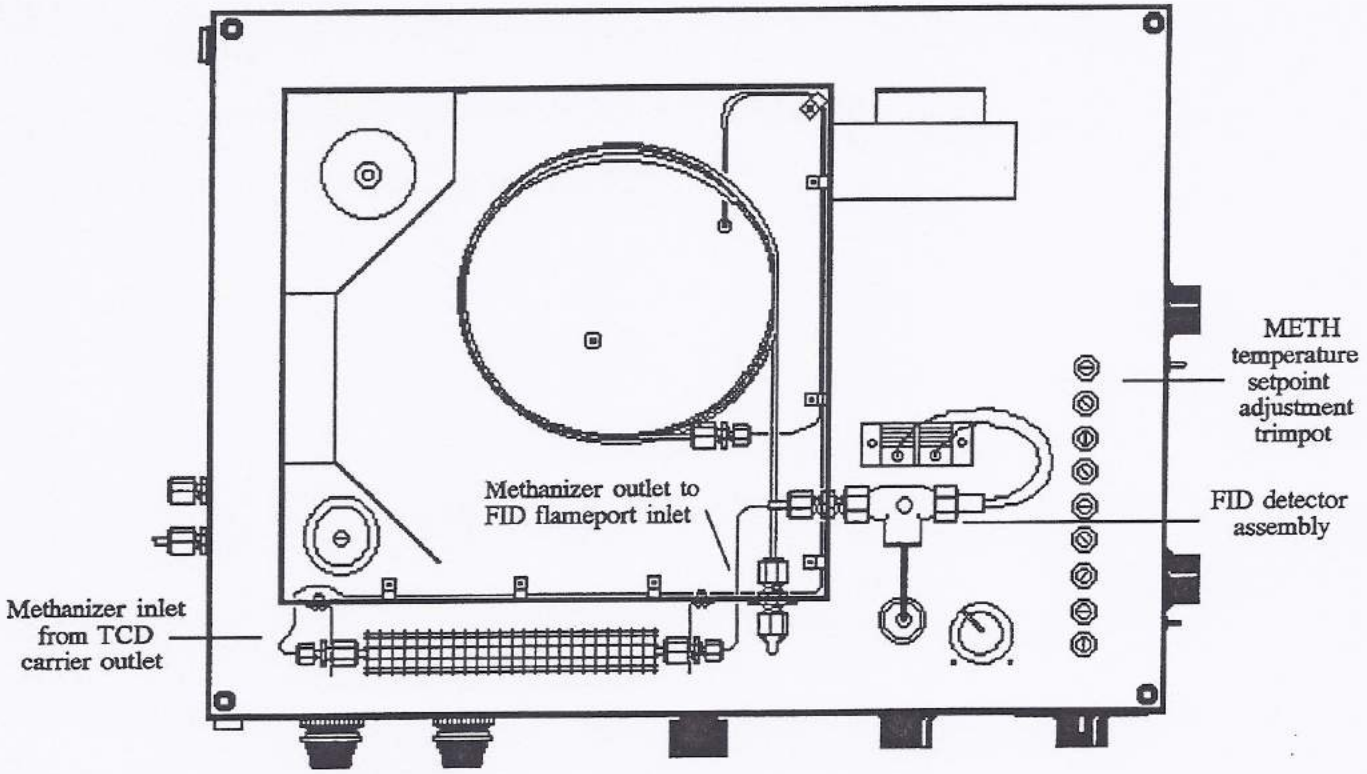
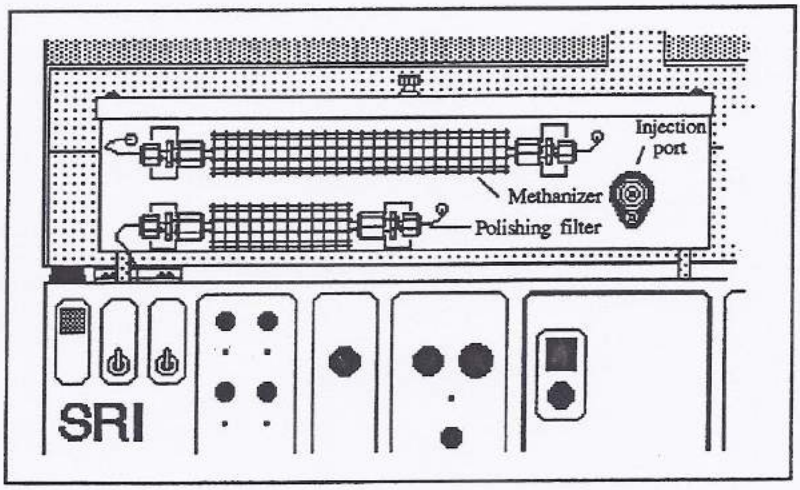
Chapter: DETECTORS

Topic: Operation of The Methanizer Accessory



TOP VIEW OF METHANIZER-EQUIPPED TCD/FID CHROMATOGRAPH



DETAIL - LOCATION OF METHANIZER ON FRONT OF COLUMN OVEN ABOVE CARRIER GAS POLISHING FILTER

Carbon dioxide and carbon monoxide can be catalytically reduced to methane if passed through a nickel-packed trap heated to 375° C with the use of hydrogen either as carrier or make-up gas. Methane can be detected to 1ppm using the FID detector, permitting lower detection limts than obtainable with unmethanized CO and CO2 using the TCD detector. With the SRI design, the methanizer is placed in series between the TCD and the FID. This enables the user to quantitate the sample first through the TCD as CO and CO2 and then through the FID after methanization. In this manner, high concentrations (1% and greater) are quantitated by the TCD and low concentrations by the FID.

The methanizer is held in place by 1/8" Swagelok® nuts. A metal ferrule is at the left end of the methanizer tube. A graphite ferrule is installed on the right end (Alltech # SF-200-G) so that the tube may be removed from the insulated heating sleeve for maintenance. The methanizer temperature is set to 375° C using the METH trimmer potentiometer located to the right of the FID assembly under the protective red oven cover and may be displayed on the digital temperature readout. A hydrogen make-up "T" fitting must be inserted prior to the methanizer if hydrogen gas is not used as carrier. Replacement nickel powder is available (Baseline # Y-CP-01-001, phone (800) 321-4665).

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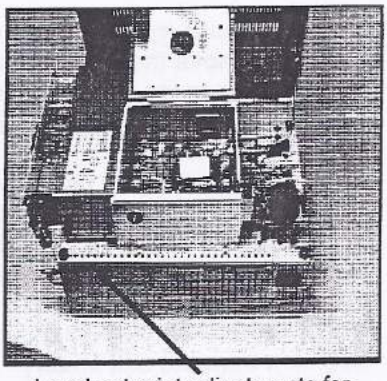
Chapter: Injectors

Topic: EPC (electronic pressure control) operation

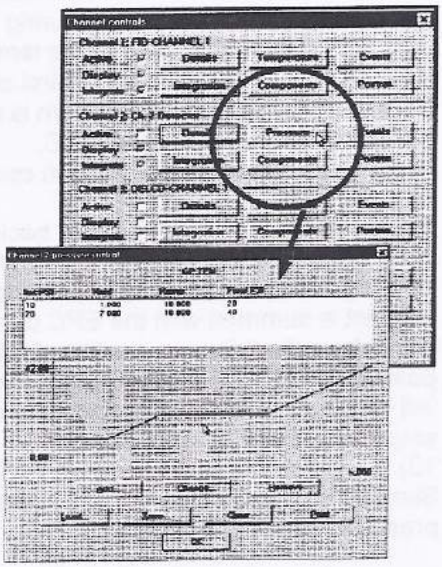
SRI GCs are equipped with electronic pressure control of all system gases. Detector support gases such as hydrogen and air are controlled by the screwdriver adjustable local setpoint on the GC, and once set are seldom altered. The carrier gas pressure may be controlled by either the local setpoint screwdriver adjustment or by the channel two pressure program in the PeakSimple data system software. **The main benefit of carrier gas pressure programming (ramping) is to speed up the flow rate through the column at the end of the run in order to elute high boiling peaks more quickly.**



Local setpoint adjustments for temperatures and pressures using small screwdriver

Most chromatographers choose to set the carrier gas pressure using the screwdriver local setpoint adjustment rather than the channel two pressure program for the following reasons:

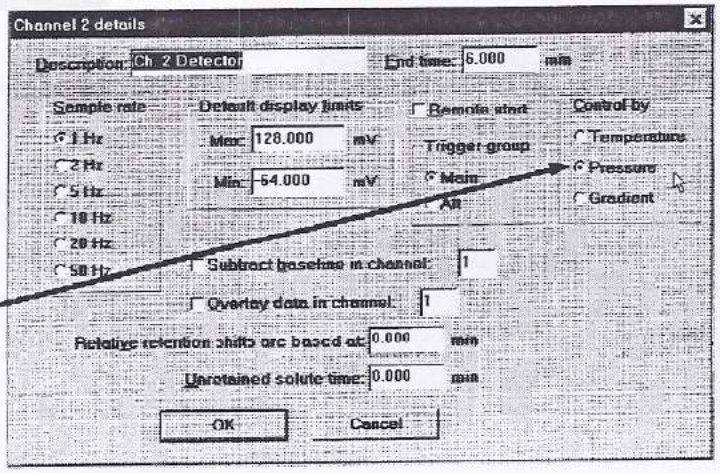
1) The screwdriver adjustment is simpler, and once set is not likely to be altered unintentionally.
2) The benefits of ramping the carrier gas pressure are often not worth the extra operational complexity.



Because very few users choose to utilize the pressure programming features, all SRI GCs are shipped with the EPC control disabled. Instructions for enabling the EPC are shown on the following page. Once the EPC is enabled, the carrier gas pressure will follow the pressure program loaded into channel two of the PeakSimple data system software. Channel two must be activated and a pressure program entered even if only a single detector signal is being acquired on channel one. The pressure program end time must be coordinated with the temperature program for the column oven which is loaded into channel one.

<u>Once you make the changes, don't forget to save your control file (default.con) so PeakSimple will remember your changes the next time you boot up.</u>

Set the "Control By" radio button in the Channel 2 Details screen to Pressure. Then enter the desired pressure program into Channel 2 by selecting the Pressure screen from the Edit/Channel menu



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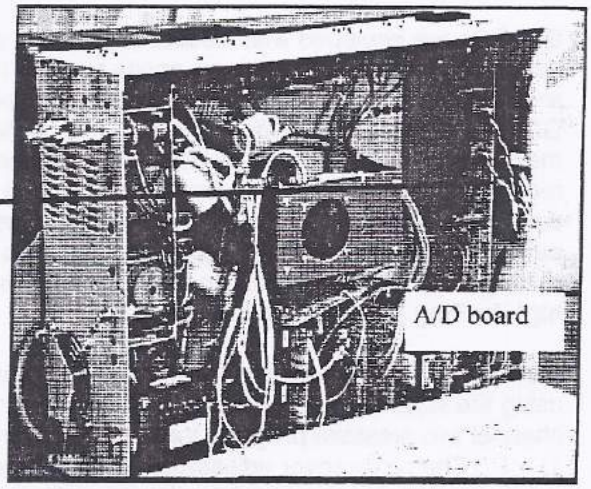
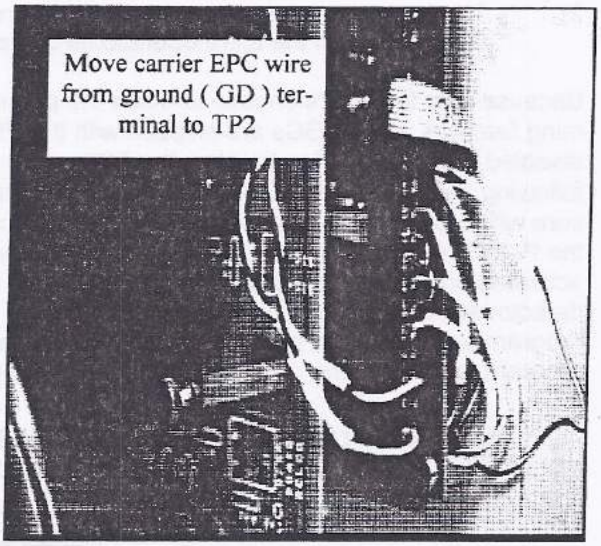
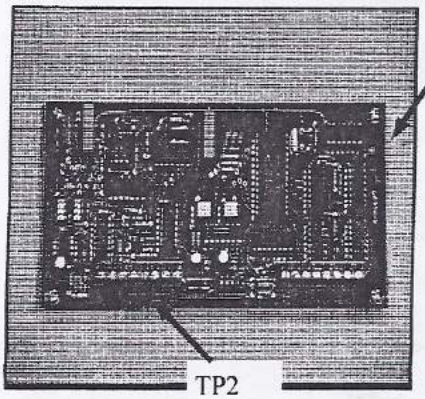
Chapter: Injectors

Topic: Enabling the carrier gas EPC

1) Un-plug the GC power cord.
2) Remove the six screw holding the bottom cover to the GC chassis.
3) Tilt the GC on its back and expose the interior.
4) Locate the A/D board which is mounted on the right hand interior wall.
5) Locate the carrier gas EPC wire (green with white stripe and labelled carrier EPC) This wire is attached to a Ground (GD) terminal on the A/D board before shipment from the SRI factory. Attaching this wire to Ground disables the computer control of the EPC.
6) Use the screwdriver provided with the GC to loosen the screw securing the wire and re-attach the wire to the terminal labelled TP2. The pressure control signal from the PeakSimple data system is now connected to the carrier gas EPC.
7) Re-assemble the botttom cover and screws.
8) Plug the GC power cord back in.
9) Use the screwdriver to adjust the carrier gas local setpoint to 0.00. The local setpoint is summed with the EPC control signal from PeakSimple, so if the local setpoint is not set to 0.00, the carrier pressure will be the sum of the local and computer setpoints.
10) Enter a pressure program in PeakSimple's channel 2, and verify that the GC pressure follows the program.



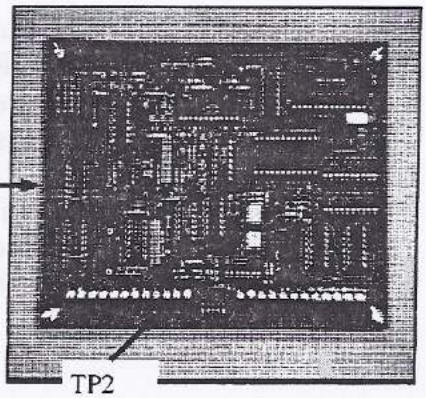




Some GCs will be equipped with the single channel Model 203 A/D board.

Other GCs will be equipped with the 4 channel Model 202 A/D board.

The procedure is identical on either board.



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Chapter: Injectors

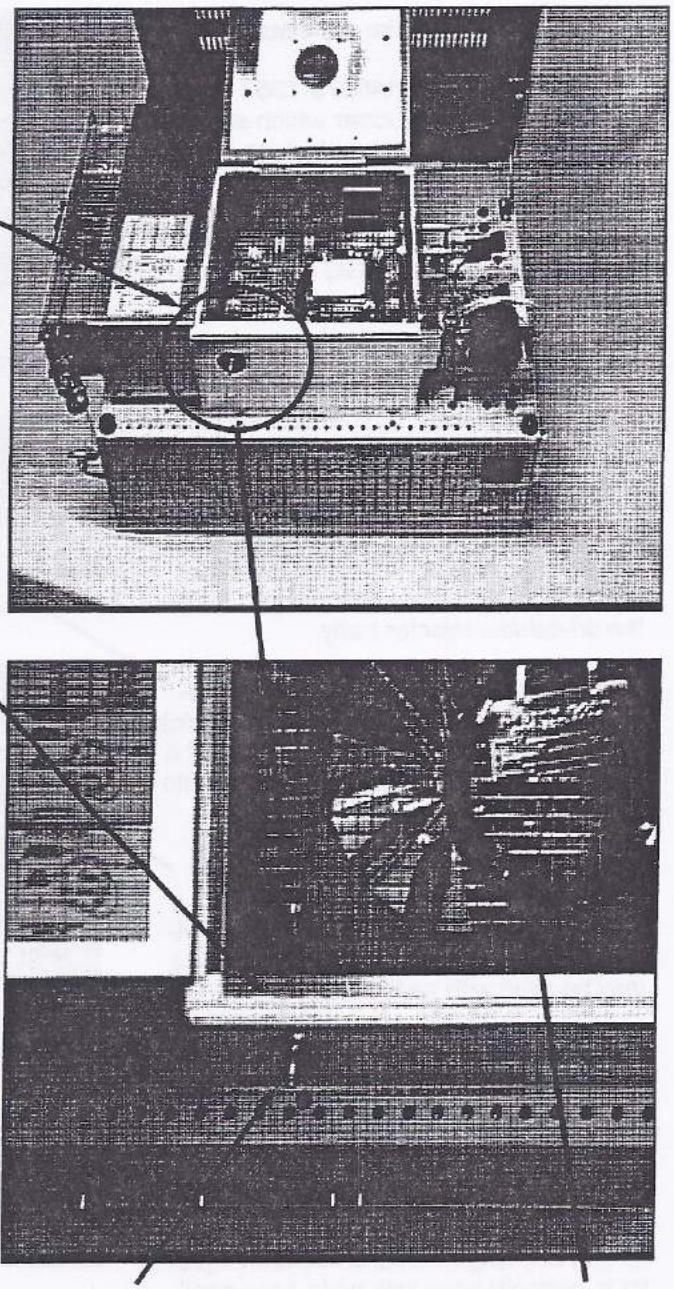
Topic: On-column Injector Operation

The On-column injector is designed for .53mm (wide-bore) capillary columns and 1/8" packed columns. One or two on-column injectors can be mounted on the 8610C GC, while a single on-column injector can fit on the Model 310 GC. The photo at right shows a single on-column injector mounted on the 8610C GC.

The on-column injector is not separately thermostatted because it closely follows the temperature of the column oven due to its low mass design and mounting location on the wall of the column oven.

Because the insulated oven wall on SRI GCs is only .75" thick, sample is injected onto the column well inside the column oven, so no cold spots can trap the sample, even if the sample consists of high boiling analytes.

For most applications, the on-column injector is the best way to inject a liquid sample because the syringe deposits the sample into the bore of the column itself. The column is usually the most inert surface available (more inert than glass injector liners), and unlike heated injectors, the sample does not undergo a flash vaporization which can broaden peaks and result in peak tailing. Also, because the entire sample is deposited on-column, boiling point discrimination can not occur as it can with split/splitless injection techniques.



Septum nut with silicone rubber septum seals carrier gas in, but allows syringe to penetrate into column

60 meter .53mm metal capillary column shown connected to on-column injector

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Chapter: Injectors

Topic: On-column Injector Operation

The On-column injector consists of:

1) The septum, (part#8670-1353) which is a plug of silicone rubber which allows the syringe to penetrate but which prevents the carrier gas from escaping. The septum used on SRI GCs is sometimes called a "shimadzu plug" type septum and is widely available from GC supply catalogs

2) The special septum nut (part#8670-9090) for 26-27 gauge syringe needles. The extended snout on the septum nut helps guide the syringe needle straight onto the column.

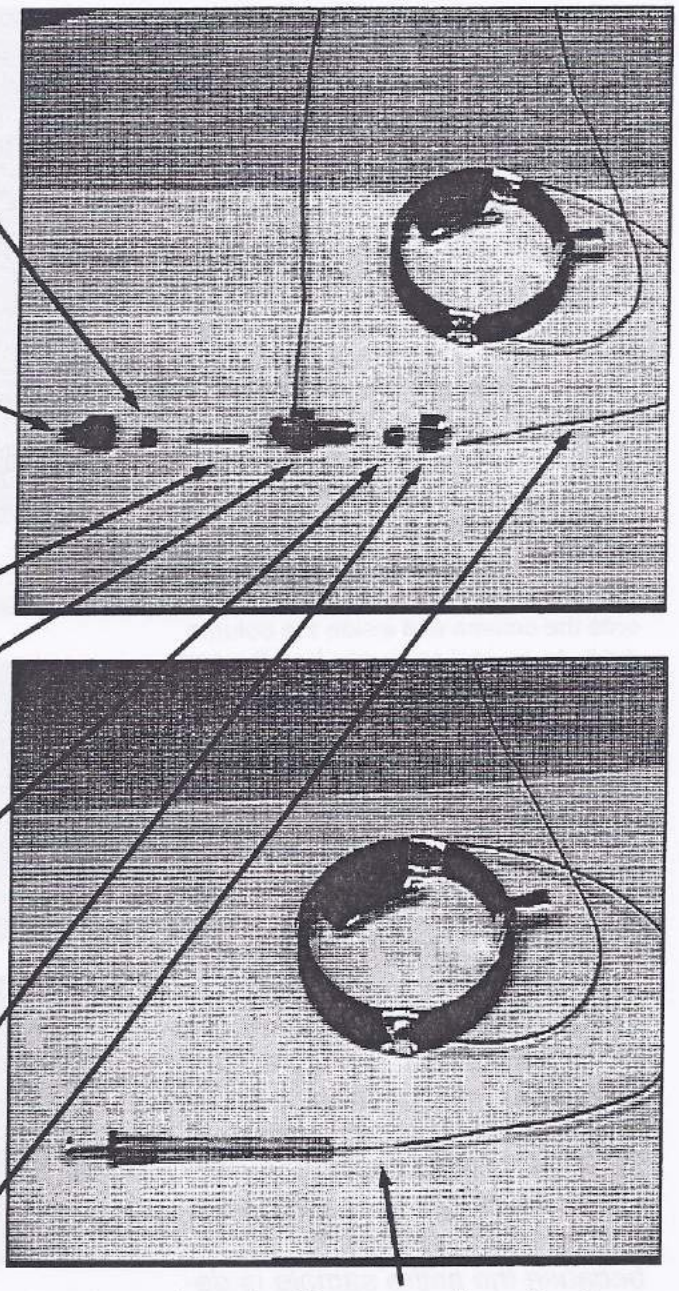
3) The wide-bore capillary column adapter (part#8690-9093) which aligns the syringe needle and the column inside the on-column injector body.

4) The injector body fitting (part#8670-9094). This is a stainless steel swagelok type fitting modified with the addition of a carrier gas inlet tube which is welded into the side.

5) A 1/8" to 0.8 mm graphite reducing ferrule secures the wide-bore (.53mm) capillary column into the injector body fitting. Either soft or hard graphite ferrules may be used with capillary columns.

6) A 1/8" swagelok type nut (stainless or brass) is used to compress the graphite ferrule around the column. Stainless is recommended for oven temperatures above 200°C.

7) A wide-bore capillary column (.53mm i.d.) of any length. The on-column injector is normally used with wide-bore capillary or 1/8" packed columns, not with columns whose inside diameter is less than .53mm since that is the smallest diameter into which a standard 26 gauge syringe will fit.



As shown above, the 26 gauge needle on the standard 10 ul GC syringe fits perfectly into the bore of a .53mm wide-bore capillary column

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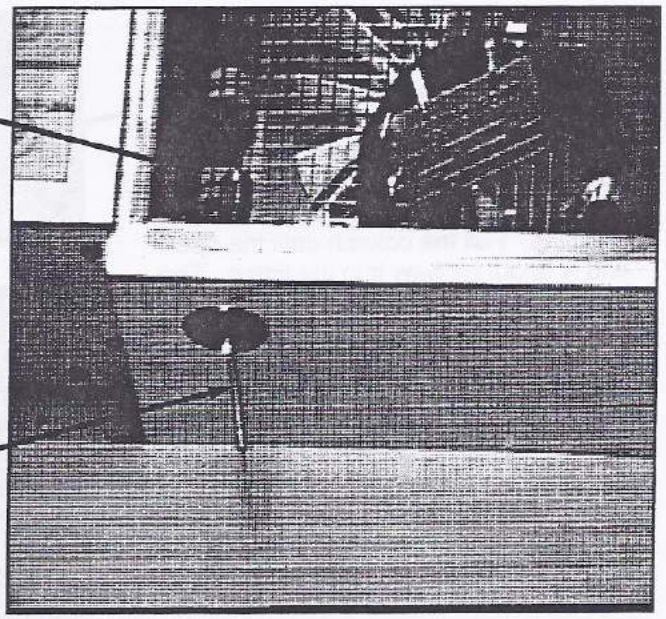
Chapter: Injectors

Topic: On-column Injector Operation

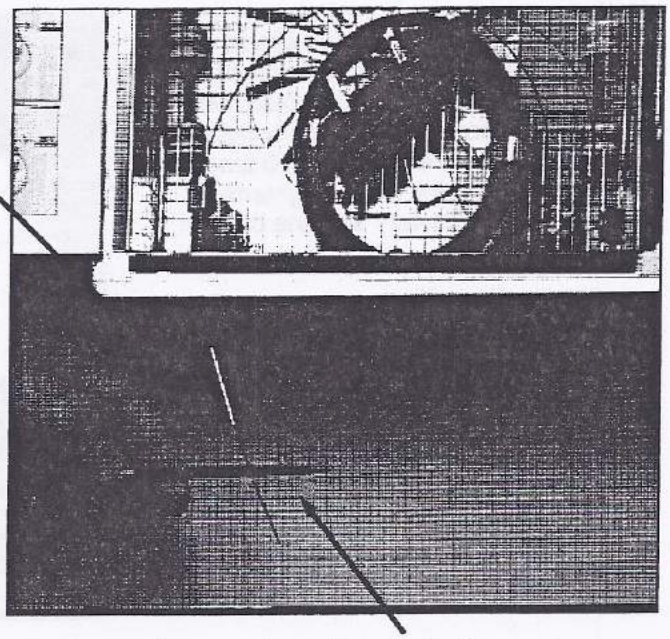
To install the column in the injector:

1) Feed the column end through the 1/8" swagelok type nut and graphite reducing ferrule. If the ferrule has been previously used, inspect it carefully to make sure it is still intact. Sometimes used ferrules will break inside the nut and a part of the ferrule will fall out. What's left inside the nut may not seal correctly. Try to avoid shaving bits of graphite from the ferrule into the bore of the column as this can cause peak tailing and absorption.



2) Push the column all the way through the injector fitting and out the front. Then slip the wide-bore adapter over the end of the column. Be sure that the conical end of the adapter is facing out towards the operator. The gash in the adapter allows carrier gas to enter the column even if the end of the adapter is plugged off.

3) If you are using a metal capillary as shown in the photo, use a sharp file make a score mark an inch or two from the end of the column. Holding your thumbnail under the score mark, snap the column end off to make a clean break. If you are using a polyimide coated fused silica capillary column, a razor or sharp knife edge is used to make the score mark. The end of the column is removed to ensure that no graphite particles or other debris which may have entered the column bore during the installation process remains in the column.



As shown above, a sharp triangle file is used to score the metal capillary column a few inches from the end which may have picked up graphite or other debris during the installation process.

HINT: Some chromatographers use a small reamer (Dremel tool bit) to clean up and smooth the end of the metal capillary column bore hole. The smoother hole allows the syringe to enter the column with less chance of snagging on the lip of the column. The syringe itself should be in good condition with no burrs or kinks. SRI supplies a syringe with a conical needle tip (part#8670-9550) in your choice of 5, 6, or 7 cm needle lengths

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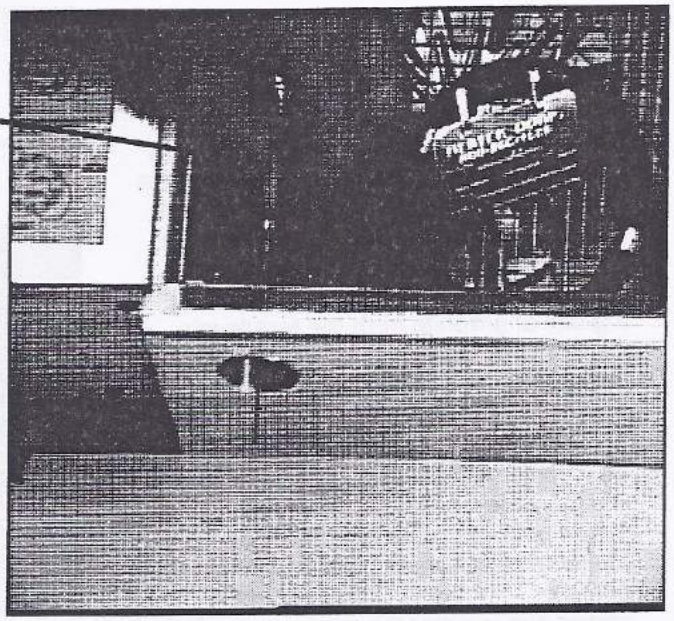
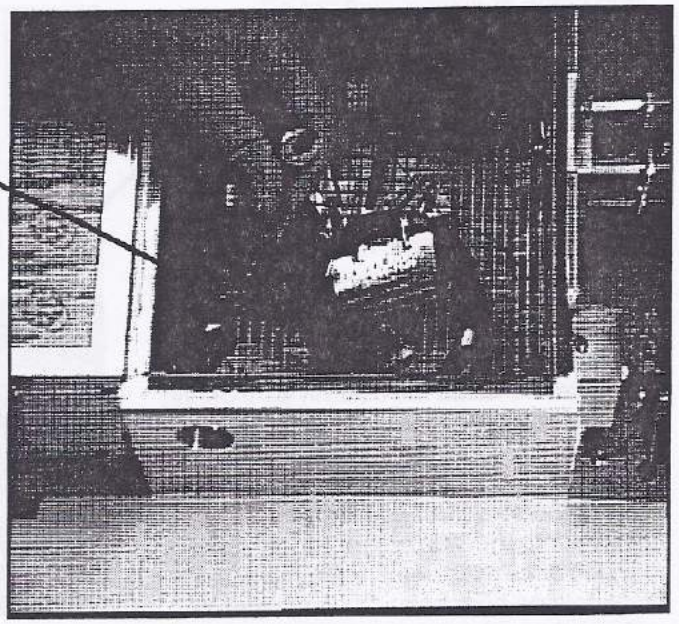
4) Pull the column and wide-bore adapter back into the injector fitting through the partially tightened nut and graphite ferrule. As you pull, the column will gradually disappear from view inside the injector fitting. Pull the column until the open end is about halfway into the fitting. The exact distance is not critical so long as the syringe needle ends up depositing the liquid sample in the bore of the column itself. If the column is pulled too far towards the oven, the syringe needle may deposit the sample in the adapter where it will gradually diffuse into the column causing wide or tailing peaks. If the column is positioned too far out towards the operator, the syringe needle may snag on the lip of the column as it is inserted.



With the column positioned, tighten the nut and graphite ferrule. You should feel the ferrule squish slightly as you tighten the ferrule, and the column should feel snug and immovable. A properly tightened ferrule can be re-used 5-10 times, while a ferrule which is over-tightened must be replaced every time the column is changed.

NOTE:

Metal capillary columns are easier to install than polyimide coated fused silica columns because as the syringe enters the column entrance it can chip away bits of the fused silica unless it is perfectly positioned. The metal columns are more forgiving since the column will not fracture when in contact with the syringe needle.



A 7/16" wrench is used to snug up the nut and graphite ferrule securing the column to the injector.

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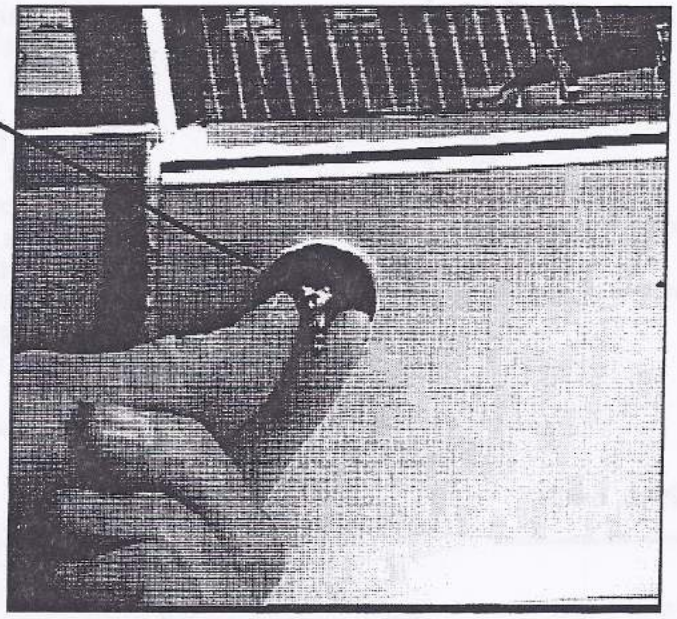
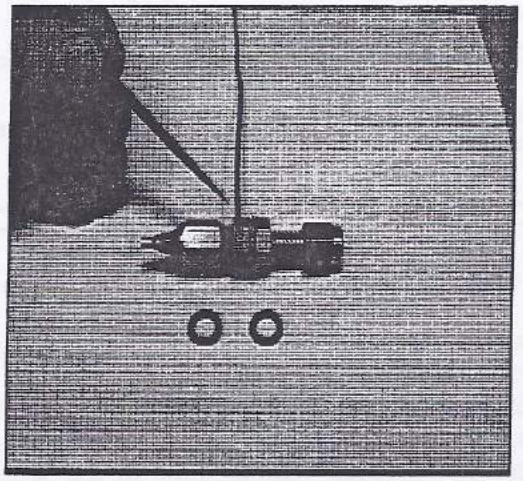
Chapter: Injectors

Topic: On-column Injector Operation

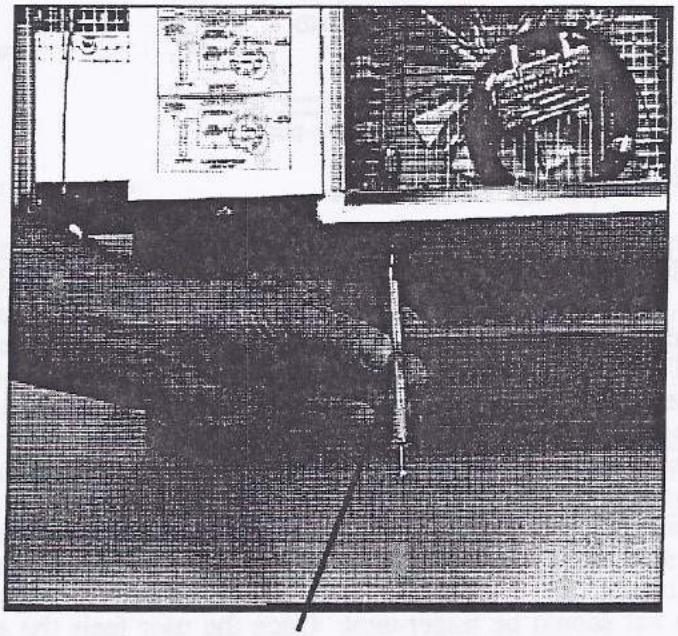


5) Tighten the septum nut until it contacts the one or two rubber o-rings on the injector body. The o-rings act as a helpful guide to avoid over-tightening the septum. When the soft silicone rubber of the septum is over-compressed, the syringe has to fight its way through often plugging with septum material in the process. A properly tightened septum cleaves easily to let the syringe needle pass, then self-heals itself when the syringe is withdrawn. Properly tightened, a plug type septum as used on the SRI GC will last up to 300 injections, while an over-tightened one will leak after 10-20 injections.



The photo above shows the injector fitting with two o-rings installed on it, and the septum nut tightened up so it just contacts the o-rings.

If the syringe snags on the edge of the column as it is inserted, loosen the swagelok nut and ferrule and pull the column another few millimeters further towards the inside of the column oven. Tighten the nut and retest by inserting the syringe.



Test your installation by inserting a syringe into the column as far as it will go. The syringe should glide into the column bore smoothly without snagging or feeling rough.

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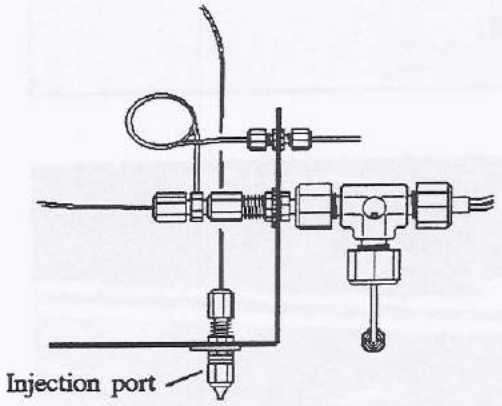
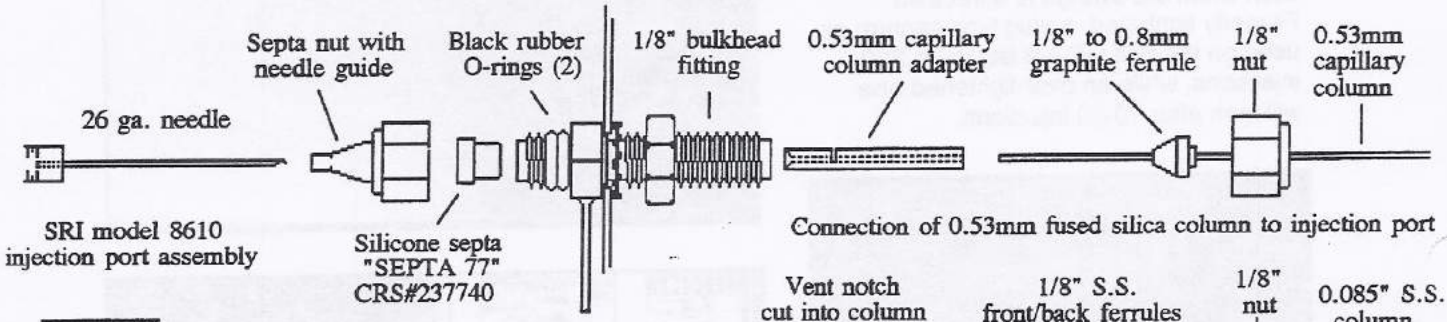
Chapter: INJECTORS AND GAS VALVES

Topic: INJECTION PORT

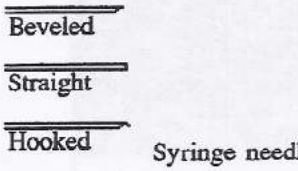


Location of injection port in typical FID system

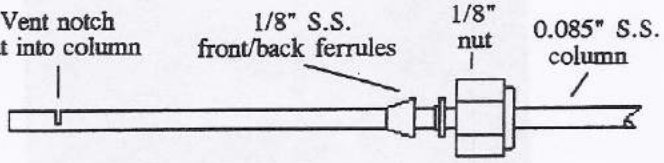
The 8610 gas chromatograph is shipped equipped with a direct injection-type injection port. This port permits on-column manual injections with traditional chromatography syringes. The injection port is simple and highly efficient by design. Swagelok stainless steel hardware is used in the assembly of the injection port. Injection of gas and liquid samples is performed using standard syringes equipped with a 26 ga. needle. Beveled (medical-style), straight, and hooked tips are available from many suppliers in this needle size. For larger needles, such as a side-port, blunt-tipped needle, a 1/8" Swagelok stainless steel nut is used in place of the supplied septa nut. Although several needle tips are available, hooked-tip needles promote septa life by slicing through the septa without "coring" the silicone, as do medical and straight-tipped needles.



Connection of 0.53mm fused silica column to injection port



Syringe needle tip styles



Connection of 0.085" I.D. stainless steel packed column to injection port

Therefore, they are recommended. "Coring" punches shards of septum into the injection port and may result in plugging of the syringe needle and failure to deliver sample. Over an extended period of time, these shards could migrate into the column. In a packed column, this accumulation of septum shards increases the exposed surface of silicone available to produce silicone or "septa" bleed. In a capillary column, these shards could plug the column completely. Routine maintenance of the septum prevents this from occuring. A bad septum may bleed excessively or permit carrier gas to leak out of the system, affecting retention times. It may visibly bulge or show numerous slices or shards of silicone protuding in toward the injection port. This usually occurs when the septum nut has been over-tightened and the physical characteristics of the septum have been altered due to compression of the silicone. If a septum is extremely bad, the user might see a puff of smoke blow out from the injection port after injection. This is the volatized sample blowing back out through the leak on a continuous stream of carrier gas. Septa may become tacky and unusable after extended service. The septa nut should be finger-tight. Once the user feels the septum seat snugly against the bulkhead fitting, the septa nut has been tightened sufficiently. Use the two black rubber O-rings on the injection port as a guide - the nut should barely make contact with the outer O-ring when the nut is properly tightened. NEVER use a wrench to tighten the septa nut. An over-tightened septum will have a markedly decreased lifetime. Larger side-delivery needles also tend to reduce septa life due to the size of the puncture created during injection. This requires more frequent servicing of the septum. Please note that when septum replacement is required during use of the thermal conductivity detector (TCD), the filament current should be turned off at the electrometer located on the right side control panel of the chromatograph, prior to removing the septa nut. Failure to do so could result in the destruction of the detector filaments due to lack of carrier gas flow through the column and into the detector.

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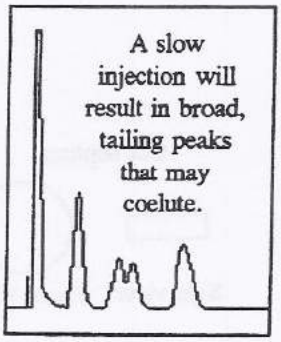
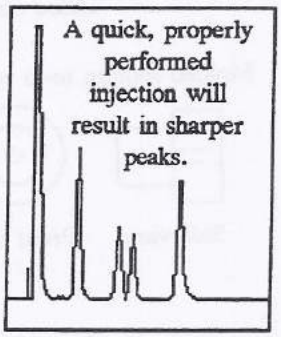
Chapter: INJECTORS AND GAS VALVES

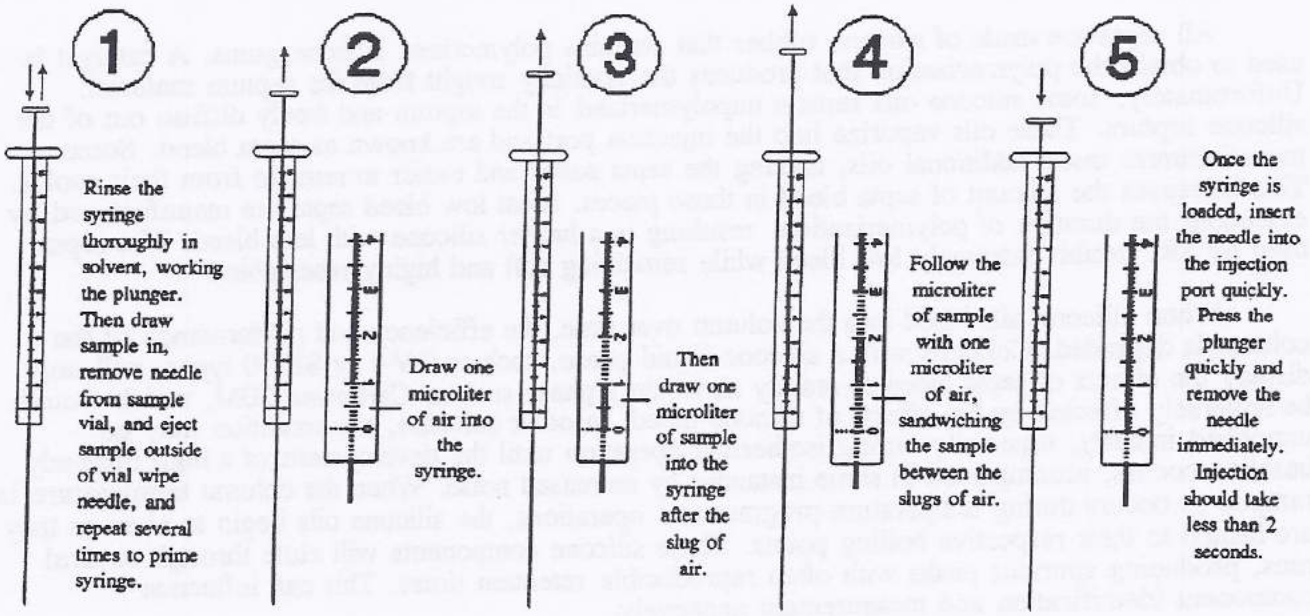
Topic: Manual Direct Injection Technique



When performing analyses using manual direct injection, the method or technique used to prepare the syringe and perform the injection can mean the difference between obtaining chromatograms that are either poorly resolved or clean and sharp. Reproducibility can also be affected if the amount injected varies from injection to injection. This is why it is imperative that a consistent, reproducible method or technique of manual injection be used when performing direct injection.



Sample volume affects the quality of data produced by the gas chromatograph. If too much sample is injected, the column becomes overloaded and the peaks produced will be broad and tailing. Insufficient sample will likely result in quantitation inaccuracies. If the syringe is not properly primed and loaded (or the sample slug contains air bubbles) when injecting liquid samples, or the syringe has not been properly evacuated, purged and loaded when injecting gas samples, the sample amount actually injected will vary, as will the results obtained. The procedure indicated below is just one of many in use today by chromatographers performing direct injection of liquid samples. The syringe and plunger are cleaned. The plunger should not be bent. Then the syringe is flushed thoroughly, primed, and loaded with precision.



Properly prepared, the syringe needle is inserted completely into the injection port in one smooth, quick motion. Then the plunger is driven home immediately. As soon as the plunger tip hits the end of the sample chamber, the syringe needle is withdrawn from the injection port in a quick, smooth motion. This will prevent any sample remaining in the needle from having time to vaporize into the injection port before or during withdrawal (if this were allowed to occur, it would result in peak broadening and tailing). You may currently be using a different technique for direct injection. As long as the method being used is consistent and reproducible, you will obtain reliable, consistent reproducibility from your direct injection analyses of gases and/or liquids.

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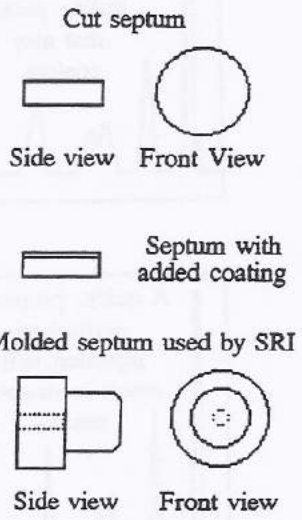
Chapter: INJECTOR & GAS VALVES

Topic: SRI Septa Overview

In order to place a sample into the column of a gas chromatograph without de-pressurizing the injection port and column or interrupting the carrier gas flow, some type of penetrable, resealable membrane must be used. The membrane must be penetrable to permit the introduction of the syringe needle into the injection port, but must also have the ability to re-seal itself. If it could not re-seal itself, each injection would leave a leak that would permit carrier gas to escape from the system. Each subsequent injection would worsen the condition, adversely affecting retention times and sensitivity. Silicone rubber is commonly used to produce injection port septa. Silicone, due to its formulation, is soft yet maintains the ability to seal puncture wounds created by syringe needles. Although septa differ in formulation, proper care will prolong the life of any septum. A silicone septa (CRS 800-327-3800, part number 237740) is installed in all SRI injection ports when shipped. This septum is very soft and resealable. It demonstrates low silicone bleed and does not affect sample component elution times. Additionally, this septum exhibits negligible "coring" for better durability and performance. This septum seals well in the tapered interior of the 1/8" modified Swagelok injection port. The example at right illustrates the difference in physical appearance between this septum and the standard cut septa machine-stamped from silicone sheets. Coated septa are manufactured this way. The coating is intended to reduce septum bleed and increase resealability.



All septa are made of silicone rubber that contains polymerized silicone gums. A catalyst is used to obtain the polymerization that produces the elasticity sought from the septum material. Unfortunately, some silicone oils remain unpolymerized in the septum and freely diffuse out of the silicone septum. These oils vaporize into the injection port and are known as septa bleed. Some manufacturers insert additional oils, making the septa softer and easier to remove from their molds. This increases the amount of septa bleed in those pieces. Most low bleed septa are manufactured by extending the duration of polymerization, resulting in a harder silicone with less bleed. The septa used by SRI exhibit extremely low bleed while remaining soft and highly resealable.

When silicone oils bleed into the column over time, the efficiency and performance of the column is degraded. Columns with a silicone liquid phase, such as OV-1 or SE-30 types, will not display the effects of septa bleed as readily as would a phase such as Carbowax 20M, which would be adversely affected by the effects of silicone bleed. In other columns, the condition may go unnoticed initially, especially during isothermal operation until the development of a high unsteady baseline occurs, accompanied in some instances by increased noise. When the column temperature is ramped as occurs during temperature-programmed operations, the silicone oils begin to elute as they are heated to their respective boiling points. These silicone components will elute through several runs, producing spurious peaks with often reproducible retention times. This can influence component identification and measurement negatively.

In some work, where sensitivity is not great, septa bleed is not a concern. To identify septa bleed, especially where temperature programming is employed, cool the unit to ambient temperature and hold for ten to fifteen minutes. Then ramp the temperature up to the maximum running temperature normally used, with the sensitivity set to high. Any peaks or baseline drift can be attributed to septa bleed. One method to minimize bleed is that of baking septa in an oven prior to insertion into the injection port in order to volatize the silicone oils. The septa may also be baked in the injection port overnight, as long as the column oven is maintained at the same temperature as the injection port to avoid the accumulation of bleed products. Regardless of septum type, septa should never be handled except with tools. Finger oils may appear on chromatograms as additional peaks.

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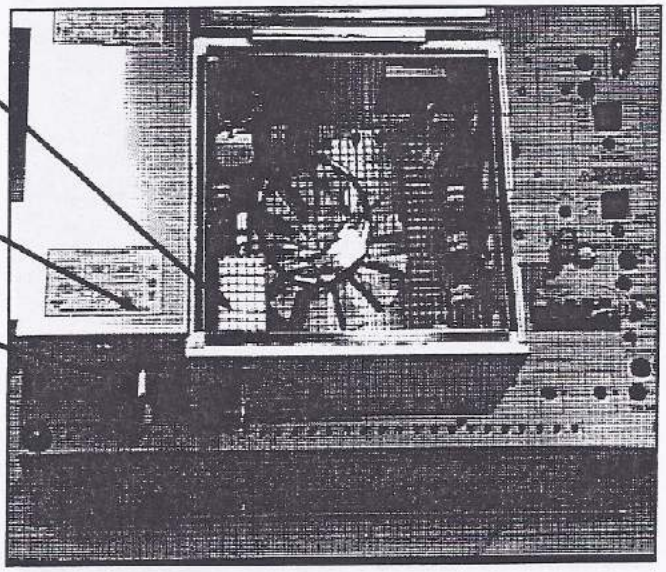
Chapter: Injectors

Topic: Heated Split/Splitless Injector

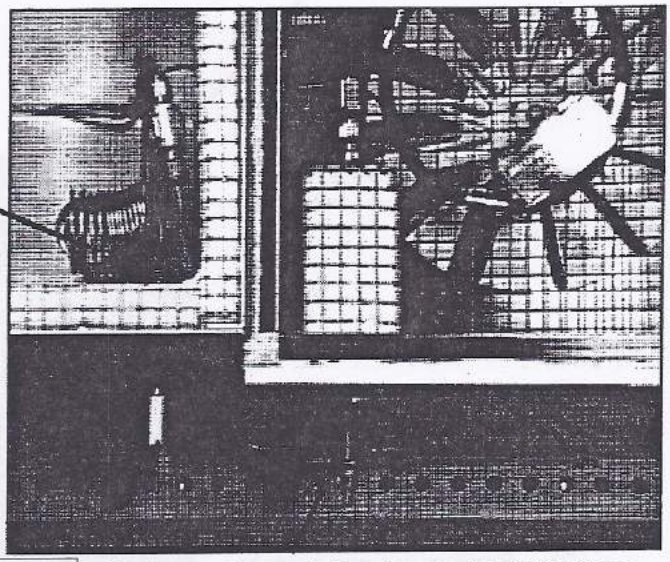
The Heated Split/Splitless Injector can be mounted on the 8610C or 310 GC. It is shown installed on the 8610C GC at right.

When mounted on the 8610C GC chassis, the precision needle valve which adjusts the split flow rate is mounted in the heated valve oven alongside the column oven.

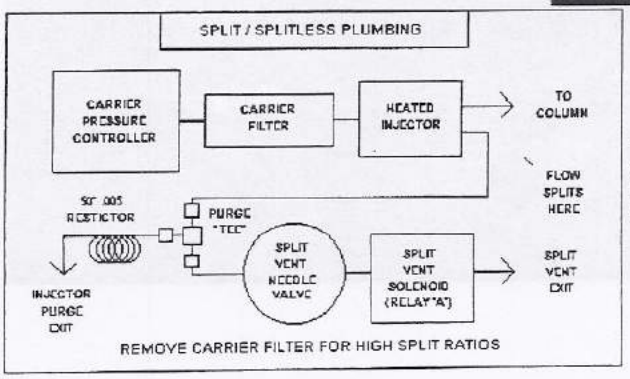
The split flow is adjusted by rotating this knob.



The lid on the valve oven has been removed to expose the Split/Splitless hardware which is installed in the valve oven.



Septum nut mounted on front of Split/Splitless Injector.



The plumbing schematic shown at left illustrates the hardware comprising the Heated Split/Splitless Injector

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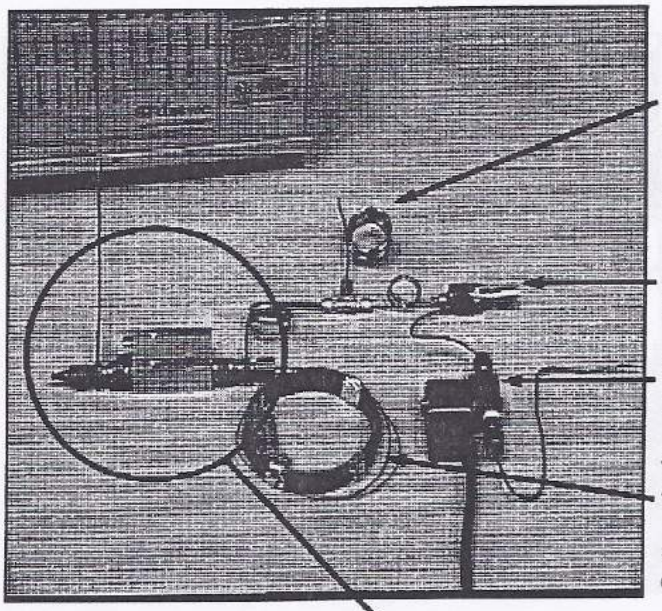
Chapter: Injectors

Topic: Heated Split/Splitless Injector



The Heated Split/Splitless Injector parts are shown at left removed from the GC for clarity.

1) Injector purge restrictor. A few ml/min of carrier gas continuously exit the injector through this restrictor tubing to prevent high boiling point analytes from diffusing back into the injector.

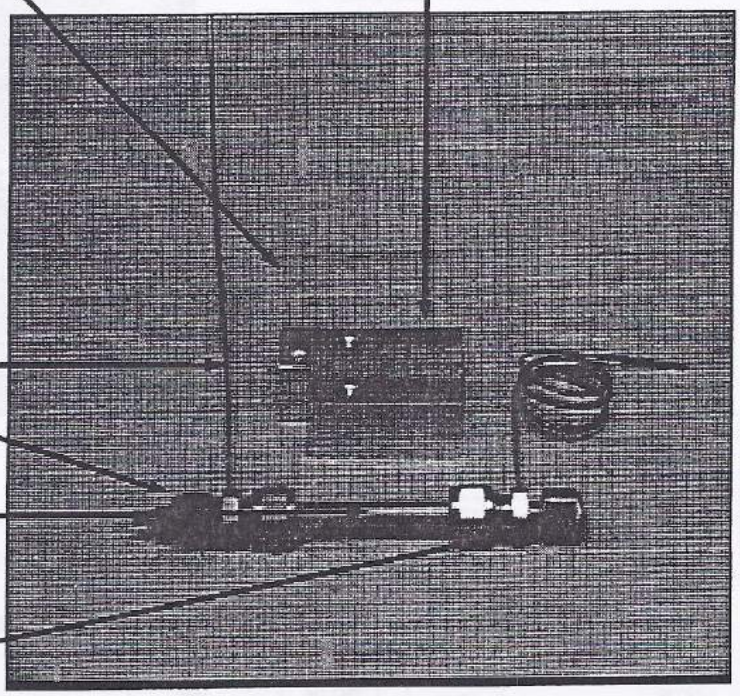
2) Precision needle valve for adjustment of split flow rate.

3) Split flow solenoid turns split on/off under control of the PeakSimple data system.

4) Column is secured into injector using nut and graphite ferrule.

5) Aluminum heater block contains heater cartridge and Type K thermocouple.

The injector liner is shown at right removed from the aluminum heater block for clarity.



1) Carrier gas inlet tubing .

2) Septum nut and septum.

3) SRI stainless steel injector liner.

4) End fitting where column connects and split flow exits to purge vent and needle valve

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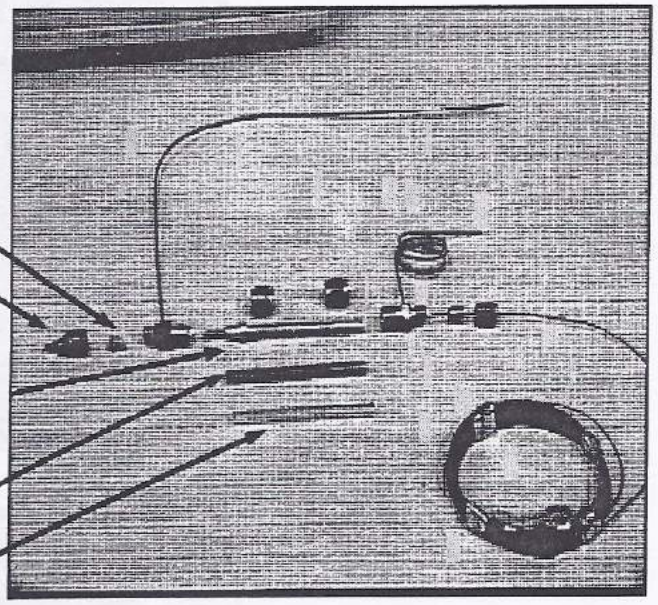
Chapter: Injectors

Topic: Heated Split/Splitless Injector

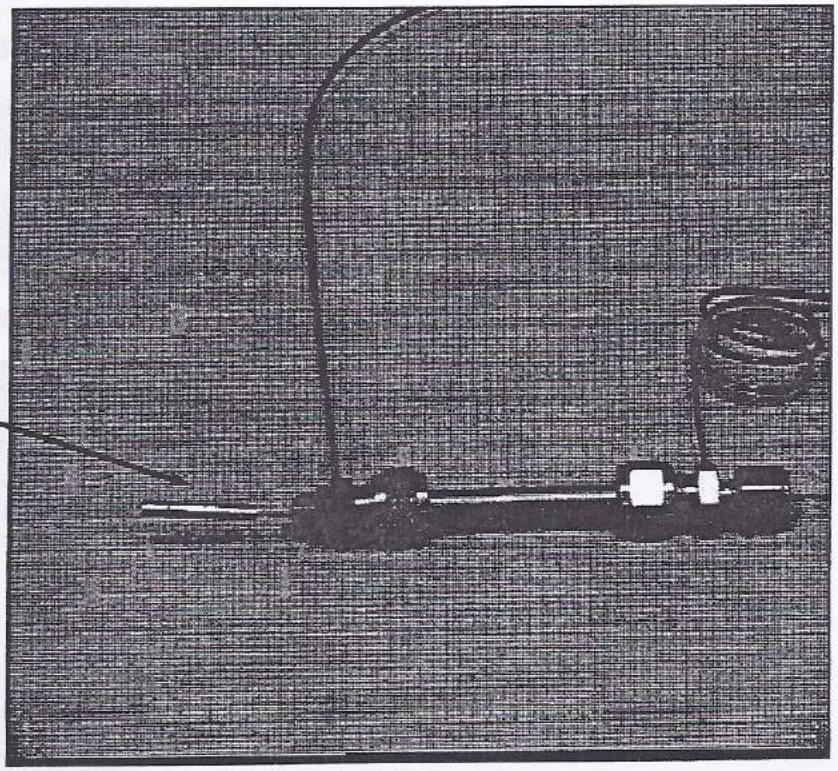
A variety of injector liners can be used with the Split/ Splitless injector depending on the column and application.



SRI designed the Split/Splitless injector to use the same size liner as Hewlett-Packard 5890/6890 series GCs. A huge variety and selection of suitable injector liner types can be purchased from chromatography catalogs such as Alltech, Restek, Supelco and others. The liner supplied with the SRI GC is an unbreakable stainless steel type which also adapts for on-column injection onto wide-bore capillary columns.

The SRI stainless steel injector liner supplied with the GC as standard equipment is shown at right with the wide-bore column adapter slipped over the column in preparation for final adjustment for on-column injection (see on-column injector instructions).

Wide-bore column (.53mm) adapter identical to that used in on-column injector fits perfectly into recess in stainless steel injector liner.



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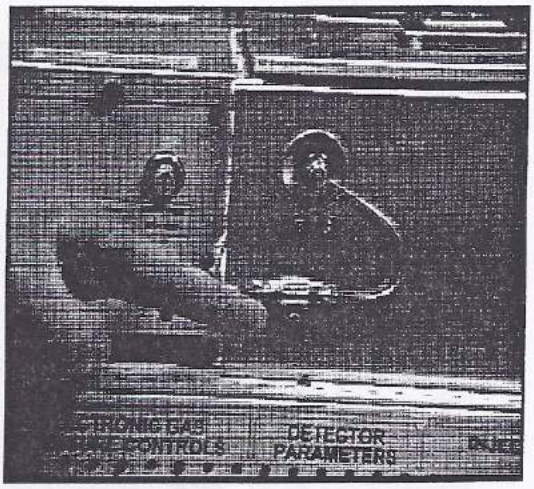
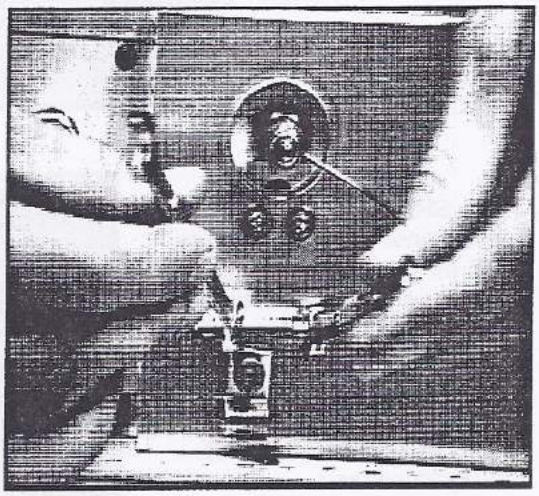
Chapter: Injectors

Topic: Heated Split/Splitless Injector

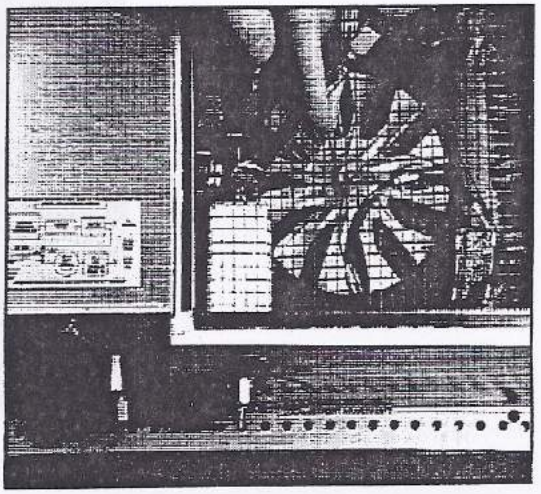
To remove the injector liner from the Split/Splitless injector:



1) Loosen the brass thumbscrew holding the 1/16" stainless union in the carrier gas supply line.



2) Using two 1/4" wrenches, loosen the nut and ferrule on the downstream side of the union and disconnect the tubing leading to the injector.



3) Using a 7/16" and 1/2" wrench, loosen the nut and graphite ferrule securing the column to the oven side of the injector.

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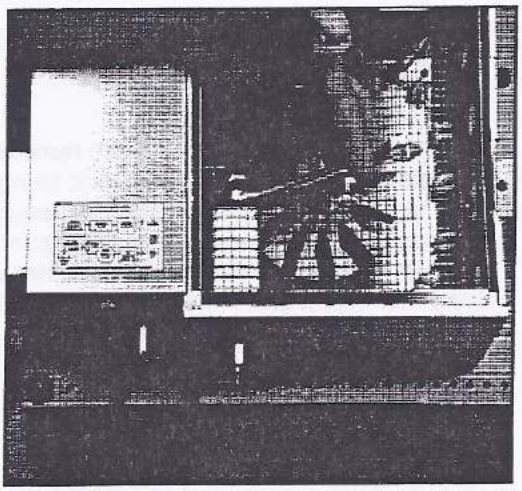
Chapter: Injectors

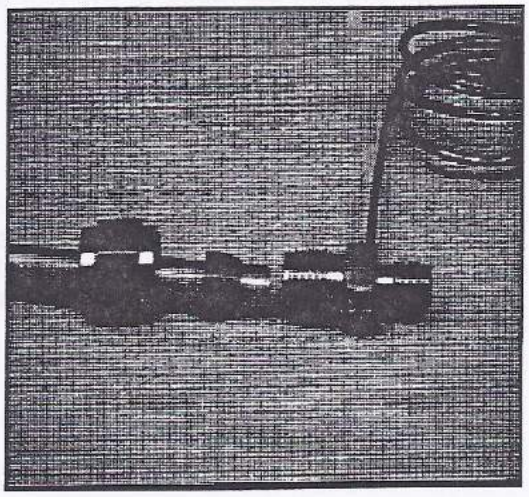
Topic: Heated Split/Splitless Injector

To remove the injector liner from the Split/Splitless injector:

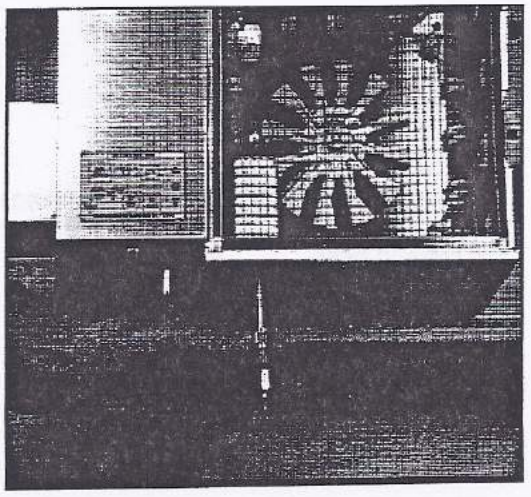
4) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting.



5) The end fitting is shown here removed from the GC for clarity. Notice the hard 1/4" hard graphite (mixture of graphite and vespel) ferrule on this end of the liner. If you are using a glass liner instead of stainless, a soft graphite (100% graphite) ferrule may be a better choice. A graphite ferrule is used on this end of the liner so the nut can slide off the liner.



6) The injector liner and carrier inlet fitting can then be removed from the GC by pulling straight out towards the operator.



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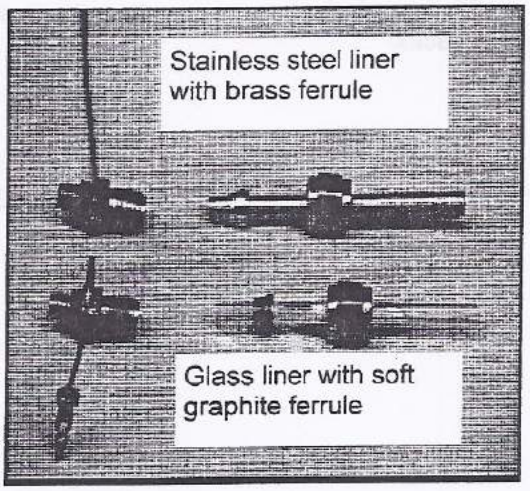
Chapter: Injectors

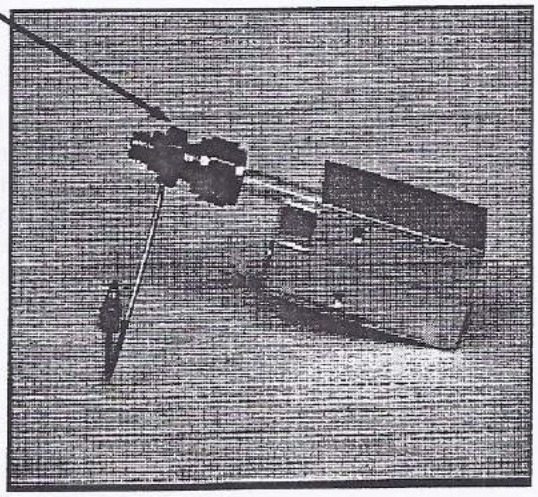
Topic: Heated Split/Splitless Injector

To replace the injection liner:

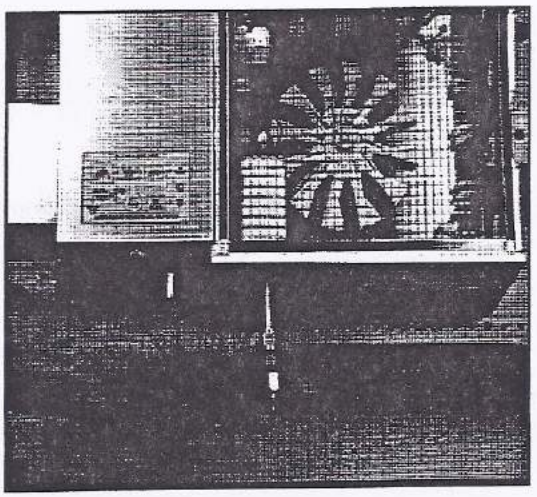


7) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting. The stainless steel liner provided as standard equipment with the split/splitless injector uses a brass ferrule on the septum end of the liner, but if you replace the stainless liner with a glass liner, you will need to use a 1/4" soft graphite ferrule instead.

Align the flat surfaces of the nut and fitting



8) The glass liner and end fitting is shown here partially inserted into the heater block and removed from the GC for clarity. Be sure to align the flats on the nut and the fitting so that the carrier gas inlet tube will adopt the same orientation once the liner is fully inserted into the heater block.



9) The injector liner and carrier inlet fitting can then be installed into the GC by sliding straight in towards the column oven

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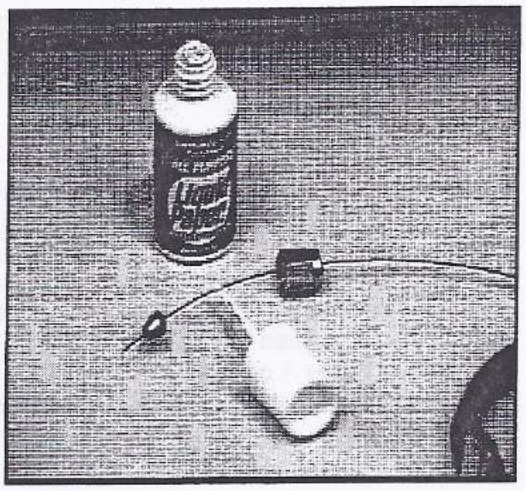
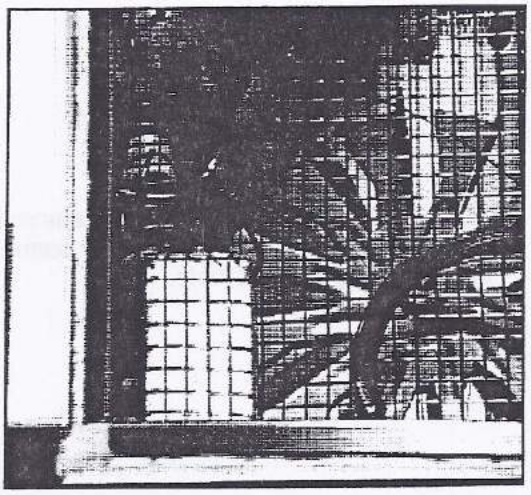
Chapter: Injectors

Topic: Heated Split/Splitless Injector

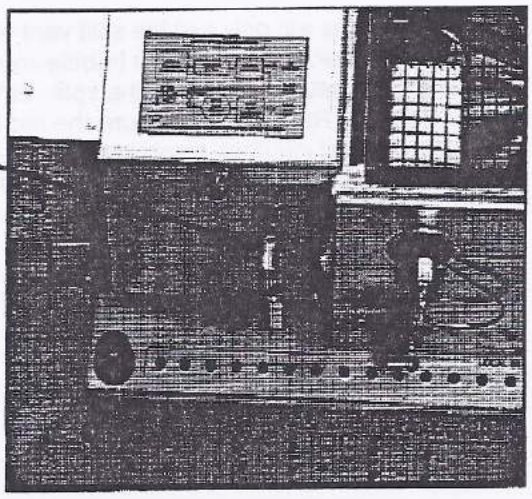
To install a narrow bore (.25mm) capillary column:



10) Use some white correction fluid to mark the column approximately 1.5" (4cm) from the end. Slip a 1/8" swagelok type nut and 1/8" to .5mm graphite reducing ferrule over the column. You can use soft or hard graphite ferrules.



11) Using a 7/16" and 1/2" wrench secure the column into the injection liner so that the white mark on the column is just visible. The intent is to position the end of the column upstream of the split vent exit tube which is welded into the side of the end fitting.



12) Adjust the split flow rate using the needle valve located on the front of the valve oven.

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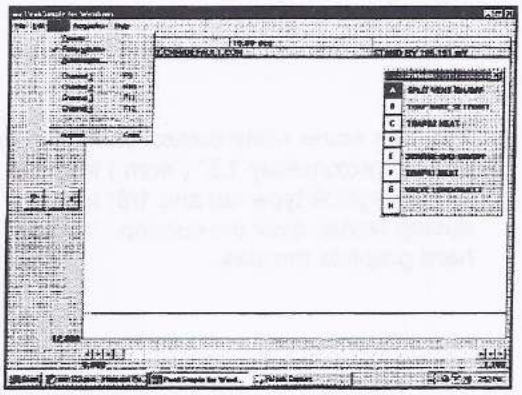
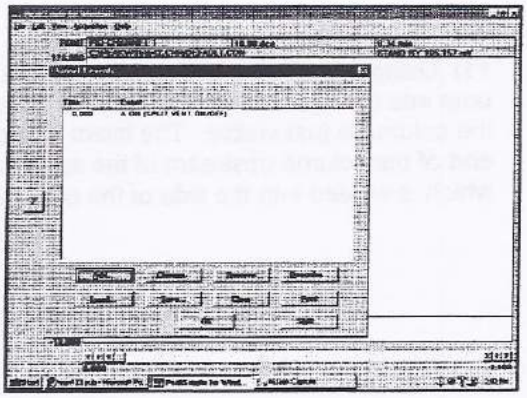
Chapter: Injectors

Topic: Heated Split/Splitless Injector

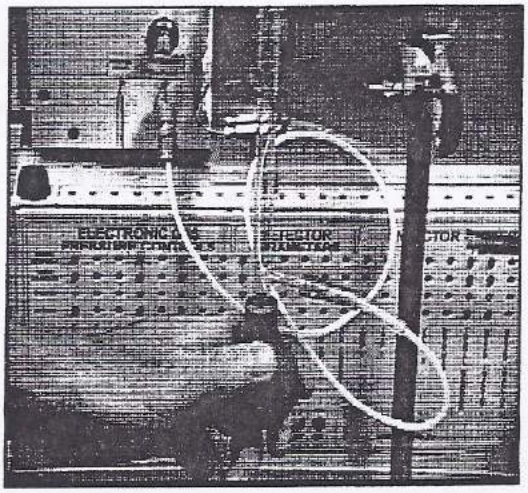
To install a narrow bore (.25mm) capillary column:



13) The split vent must be opened by activating one of the relay outputs from the PeakSimple data system. Typically Relay A is used to activate the split vent solenoid. If another relay has been allocated to this function, it will be annotated in the relay assignment chart located on the right hand side panel of the GC. Relay A can be turned on/off by displaying the relay window and then using the mouse to click on the letter A.



14) The relay can be turned on/off automatically during an analysis by entering the relay commands in the PeakSimple event table.



15) Carrier gas will only exit the split vent when Relay A is activated. Connect your bubble-meter or other flow measuring device to the split vent exit tube. Activate Relay A. Make sure the red lid of the GC is down (lid interlock disables solenoid function). Adjust the needle valve to obtain desired flow.

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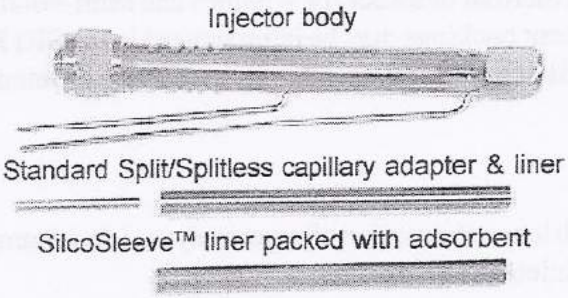
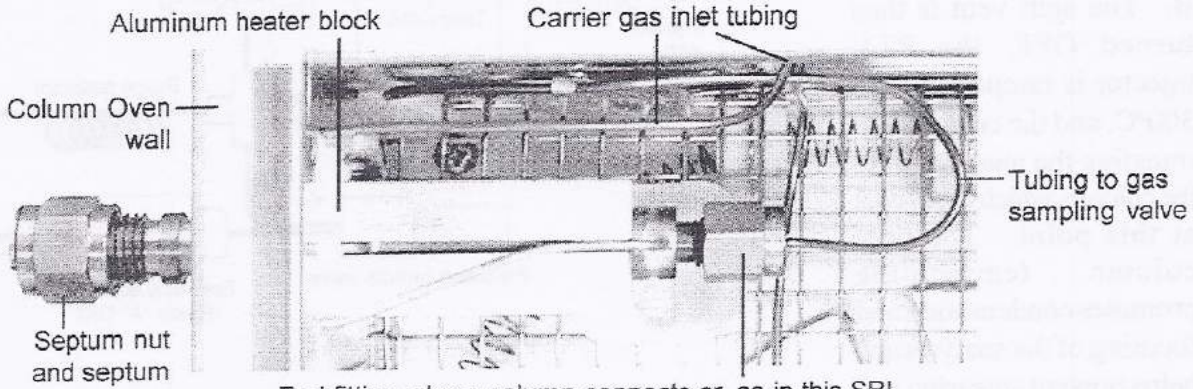
Overview

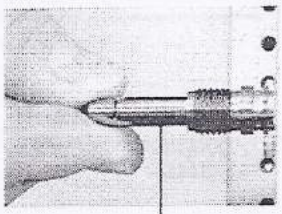


PTV and Split/Splitless injector components

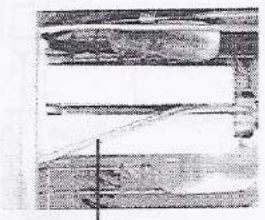
The Programmed Temperature Vaporization (PTV) injector is composed of the same parts as the Heated Split/Splitless injector: the injector body, a SilcoSleeve™ liner, an injector purge restrictor, a precision needle valve for adjustment of split flow rate, a split flow solenoid that turns on & off from the PeakSimple data system, and an aluminum heater block containing a heater cartridge and Type K thermocouple. Contrasted with the Split/Splitless injector, the PTV injector has a removable insulating sleeve, a larger (250 watts) heater cartridge with ballistic heating capability, and carrier flow ON/OFF control. The SilcoSleeve™ liner can be packed with a variety of optional adsorbents, depending on the application. The SRI PTV injector has three modes of operation: **1)** large volume liquid injector, **2)** an offline thermal desorber, or **3)** an online thermal desorber in conjunction with a gas sampling valve.



End fitting where column connects or, as in this SRI Multiple Gas Analyzer #1, tubing to a gas sampling valve

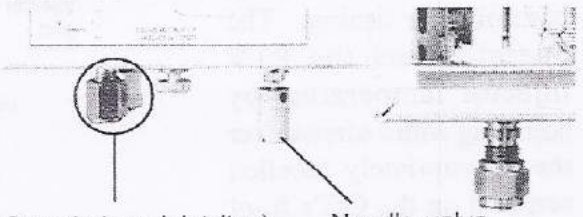


Tenax-GR™ packed SilcoSleeve™ liner, partially slid out for visibility



Split flow exits to purge vent and needle valve

Front of Valve Oven and Column Oven - top view



Sample loop inlet (top) and purge vent (bottom)

Needle valve precision control

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Theory of Operation

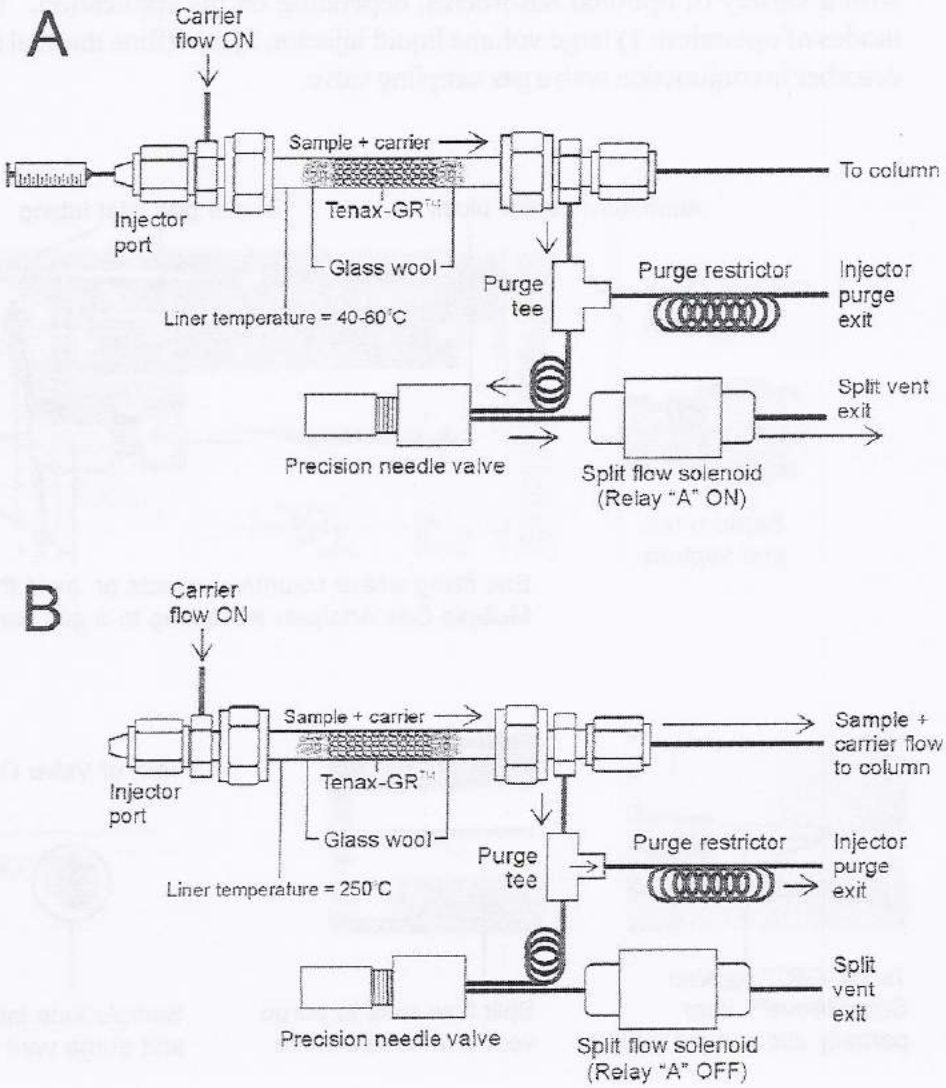
The Programmed Temperature Vaporization injector is basically a Heated Split/Splitless injector with the ability to rapidly heat to 300°C. This ballistic heating capability enables large volume liquid sample injections. The PTV injector can be used as a thermal desorber for volatiles and semi-volatiles, online or offline. Multiple liners with different adsorbent packings may be interchanged in the SRI PTV injector. The adsorbent used depends on the compounds of interest, as each has its own selective retention properties.

1) Large Volume Liquid Injector

Large volume injections allow analysis of samples with low concentration of target analytes. Liquid samples from 1µL to 200µL may be injected using the SRI PTV injector.

A. To begin, both the Column Oven and the PTV injector are held at 40-60°C. Prior to injection, the split vent is opened. Thus, the large volume liquid sample is injected into the PTV injector at 40-60°C with the split wide open. Introducing the sample at a low temperature allows the solvent to vent while the injector liner packing retains higher boiling point analytes.

B. The split vent is then turned OFF, the PTV injector is ramped to 200-300°C, and the carrier flow transfers the analytes onto the column, which is still cool at this point. The cool column temperature promotes condensation and focusing of the analytes and helps prevent smearing and excessive tailing. Each of these events is automatically controlled through the PeakSimple data system, so operators can precisely control their timing. The operator sets the PTV injector temperature by adjusting with a screwdriver the appropriately labelled setpoint on the GC's front panel.



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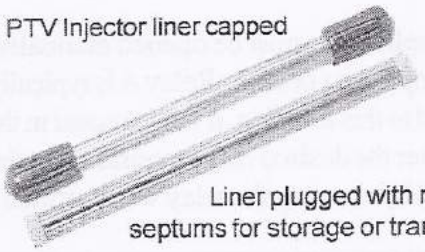
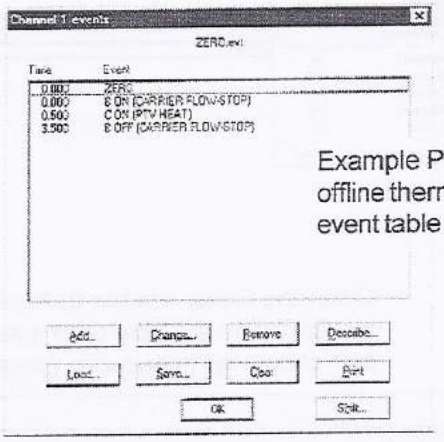
Theory of Operation continued

2) Offline Thermal Desorber

For offline thermal desorption, the SilcoSleeve liner packed with adsorbent such as Tenax-GR™ is loaded with sample outside of and separate from the GC. Although the best analysis is obtained from a fresh sample, the ends of the liner may be plugged after loading sample with rubber septa or capped with rubber end caps for storage or transportation. Turn off the flow before removing the injector liner by activating relay B, which stops the carrier gas flow. Leave the EPC flow off until the beginning of the analytical run (see the event table at right). To replace the liner, unscrew the septum nut and septum protruding from the front of the Column Oven wall. Remove the rubber septa or caps from the liner and slide it in with the gash toward the operator. Replace and close the septum and nut. With the carrier flow still turned OFF, start the run. When the PTV injector reaches temperature, the carrier flow is turned ON and the analytes are swept onto the column.





Example PTV as offline thermal desorber event table

3) Online Thermal Desorber

For online thermal desorption, the PTV can be plumbed with a gas sampling valve. In this mode of operation, the PTV functions as a sample loop, trapping and concentrating compounds for analysis.

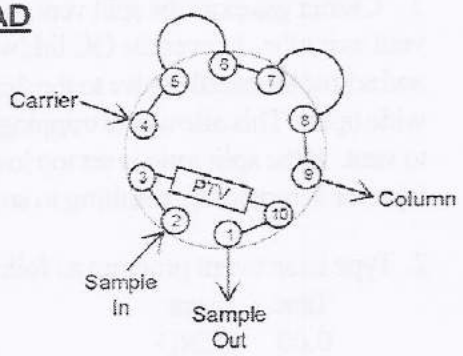
LOAD Position:
(Relay "G" OFF)

When the gas sampling valve is in LOAD position, the PTV injector can be loaded with sample through the sample inlet and outlet. The PTV injector is at 40-60°C. Analytes are trapped in the injector's liner packing.

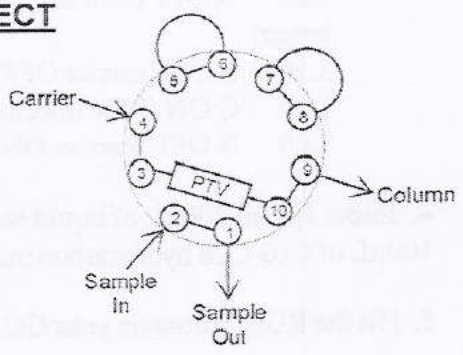
INJECT Position:
(Relay "G" ON)

In the INJECT position, the PTV injector ramps to 300°C, vaporizing the sample. The carrier gas flow then flushes the desorbed components onto the column(s). The valve should be rotated back to the LOAD position after the components are transferred to the column to avoid smearing and peak tailing.

LOAD



INJECT



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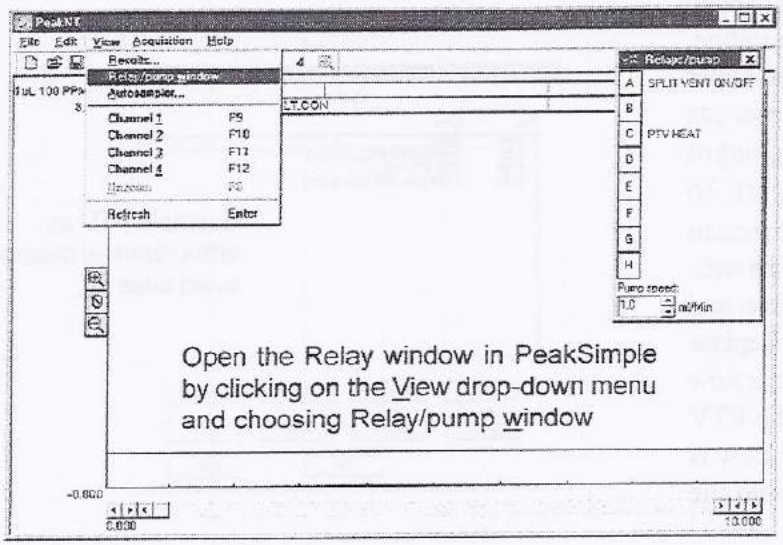
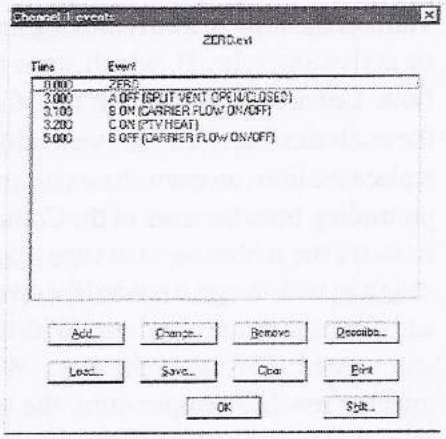
General Operating Procedure
Large Volume Liquid Injection Steps

1. The split vent must be opened manually prior to the run by activating one of the relay outputs from the PeakSimple data system. Relay A is typically used to activate the split vent solenoid. If another relay has been allocated to this function, it will be noted in the relay assignment chart located on the right hand side panel of the GC. Enter the desired relay commands in the PeakSimple Events table. The split vent can also be turned ON (or OFF) by opening the relay window then clicking on the letter A.





Example PeakSimple PTV event table

3. Carrier gas exits the split vent only when Relay A is activated. Connect a flow measuring device to the split vent exit tube. Lower the GC lid (when open, lid interlock disables the solenoid function), activate Relay A, and adjust the needle valve to the desired flow. For most liquid injections using a PTV, the split vent should be wide open. This allows the trapping material to retain the compounds of interest and quickly flush the solvent to vent. If the split ratio is set too low, some of the solvent and analytes may enter the column before the PTV injector is heated up, resulting in smeared or double peaks.

2. Type in an event program as follows:

Time	Event
0.00	ZERO
3.00	A OFF (split vent closed; if you get too large a solvent peak, keep the split vent open longer)
3.10	B ON (carrier OFF)
3.20	C ON (PTV injector heat)
5.00	B OFF (carrier ON)

4. Inject 1µL to 200µL of liquid sample into the PTV injector. In the "Expected Performance" example, 100µL of C10-C28 hydrocarbon mixture was injected.

5. Hit the RUN button on your GC or press the spacebar on your computer keyboard.

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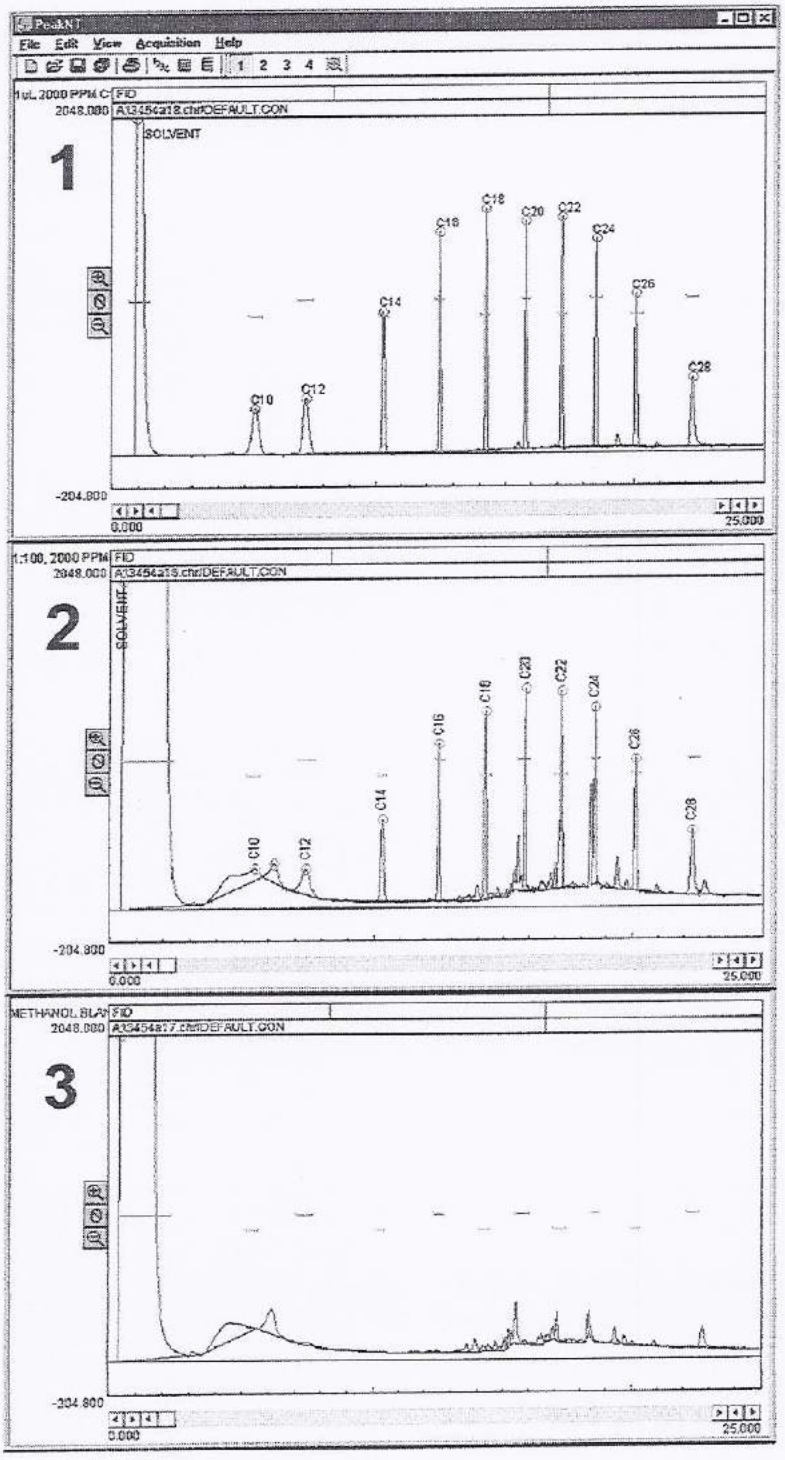
Expected Performance

The following three chromatograms are from the FID in a SRI GC with a PTV injector upgrade. The liner was packed with 0.1 grams of Tenax-GR™ adsorbent. All three 25 minute runs utilized the same temperature and event programs. In the first one, a 1µL 2000ppm C_{10}-C_{28} sample was injected through the PTV injector. In the second chromatogram, the same sample was diluted 1:100, then 100µl injected, achieving results consistent with the first run, and demonstrating the high volume liquid injection capability of the PTV injector. In the third chromatogram, 100µL of methanol was injected as a blank, resulting in a small hump between the 4 and 7 minute marks and miniscule peaks which correspond to contaminants in the methanol blank and bleed from the Tenax-GR™.



Chromatogram 1 Results:

Component	Retention	Area
Solvent	0.866	84953.1370
C10	5.366	5299.9150
C12	7.300	5034.0980
C14	10.233	4814.2000
C16	12.450	4500.0300
C18	14.216	4436.1780
C20	15.750	4528.2890
C22	17.150	4570.0975
C24	18.483	4778.9380
C26	20.033	4863.4290
C28	22.216	4135.4760
	Total	132013.7875

Chromatogram 2 Results:

Component	Retention	Area
Solvent	0.450	499472.8740
C10	5.433	2258.5340
C12	7.366	2614.0540
C14	10.266	3813.8985
C16	12.483	3924.8340
C18	14.266	3939.9080
C20	15.800	3933.0400
C22	17.200	4660.5860
C24	18.516	4737.3130
C26	20.083	4174.2920
C28	22.266	3260.1120
	Total	536789.4455

Temperature programs & events for all 3 runs:

PTV = 110°C (3min) to 275°C

Events: (A = split vent)

Time	Event
0.00	ZERO
3.00	A OFF
3.10	B ON
3.20	C ON
5.00	B OFF

Temperature program:

Initial	Hold	Ramp	Final
110°C	7.00	15.00	270°C
270°C	20.00	0.00	270°C

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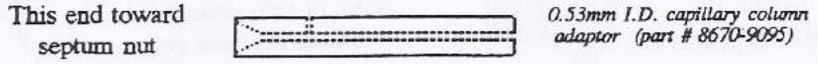
CONVERTING TO COLD ON-COLUMN MODE:

To set up the unit into a cold on-column mode, raise the red lid and adjust the injector temperature setpoint to 20° C. This will ensure that the injector does not heat itself but will be at the oven temperature. This temperature setpoint can be displayed on the digital display by turning the readout selector switch to INJECTOR SET.

When the injector and oven are cool, remove the oven lid. Remove the injector nut and 9.5mm septum (Alltech # 15428). Use a 7/16 inch wrench to loosen the nut that secures the column in place while holding the split vent fitting with a 1/2 inch wrench. Slide the column all the way through the injector until it is protruding out the front of the injector. Remove the 0.53mm I.D. capillary column adaptor from the holder in the oven lid and slip the column through the adaptor. Insert the 0.53mm I.D. capillary column adaptor all the way into the injector sleeve. The column should be inserted midway into the adapter. The adapter is then inserted into the injector so that the "funneled" end of the adaptor facing the needle is near the septum. The adaptor is vented so that carrier gas will flow to the column even if the adaptor is installed against the septum.



Tighten the nut to secure the column in place while holding the split vent fitting with a 1/2 inch wrench. Replace the injector nut and 9.5mm septum making it finger tight.

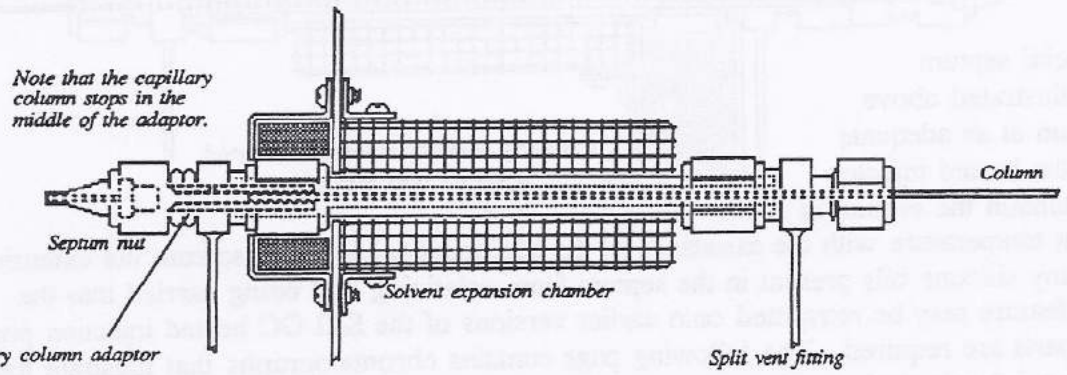
Use the toggle switch to select the flow controller to regulate carrier gas.

COLD ON-COLUMN OPERATION:

The GC is shipped configured in a cold on-column mode. This mode is the simplest to operate. The heated injector temperature setpoint is set to 20 degrees C. The flow controller is used to regulate the flow of carrier gas. A packed column can be directly connected to the back of the injector by removing the solvent expansion chamber. If a 0.53mm capillary column is used, it will be mounted in a capillary column adaptor (part # 8670-9095) which will allow direct injections onto the column. The adaptor is stored in a special holder in the back right corner of the red lid when not in use.

Unlike the split methods where much of the sample is lost, a cold on-column injection places all of the sample directly onto the column, therfore no sample is lost. Cold on-column injection method is ideal for samples of low concentration and gives the best sensitivity and sharpest peaks. The split vent is never opened for the cold on-column method.

CORRECT PLACEMENT OF COLUMN AND CAPILLARY ADAPTOR FOR COLD ON-COLUMN MODE:



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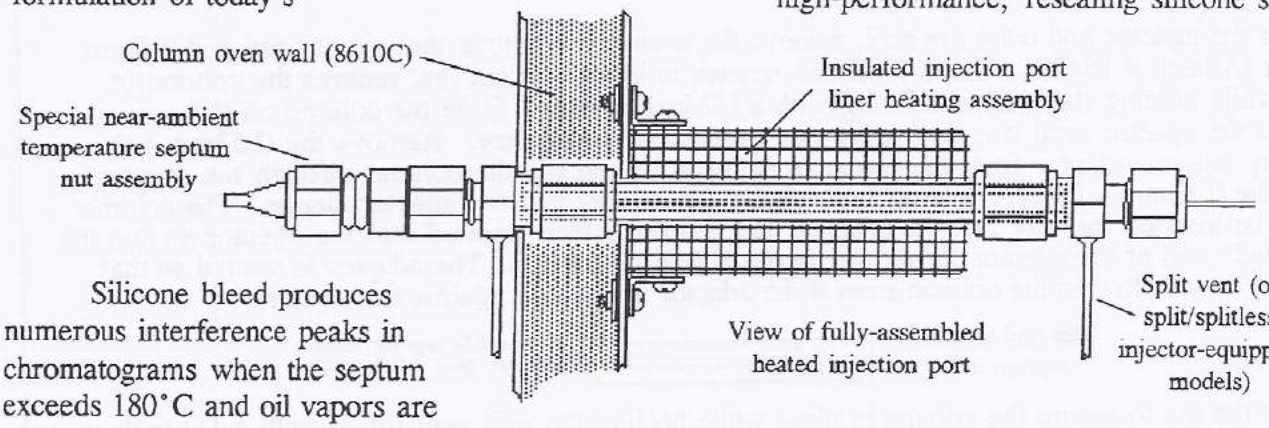
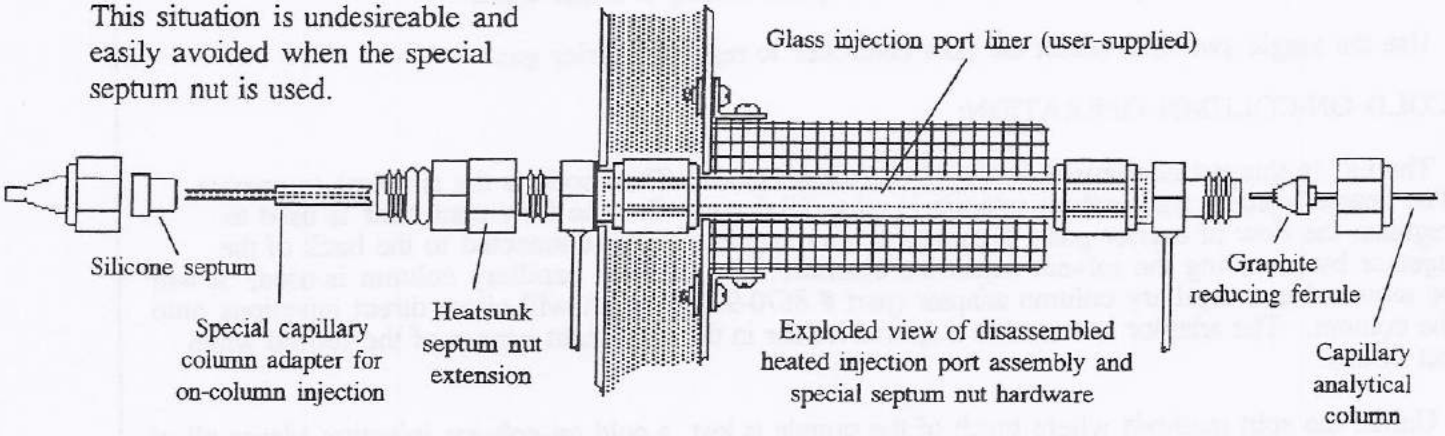
Chapter: INJECTORS AND GAS VALVES

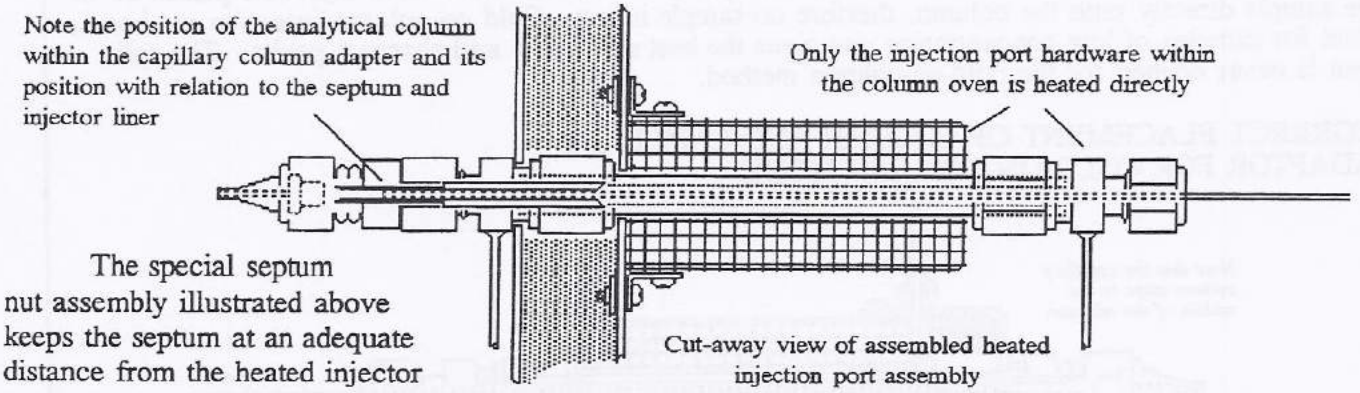
Topic: Near-Ambient Temperature Septum Nut Assembly For Heated Injectors

All SRI Instruments heated injection ports are equipped with a specially-designed septum nut which dissipates any heat that could be transfered from the heated injection port body (including split-splitless configurations), to the septum nut and septum by contact. Experience indicates that when injection ports are permitted to transfer heat to the silicone septum, that septum bleed can and does occur. Septum bleed is the volatization under heat of silicone oils used in the manufacture and formulation of today's high-performance, resealing silicone septa.



Silicone bleed produces numerous interference peaks in chromatograms when the septum exceeds 180°C and oil vapors are swept into the column and quantitated. This situation is undesireable and easily avoided when the special septum nut is used.





The special septum nut assembly illustrated above keeps the septum at an adequate distance from the heated injector assembly to maintain the septum at or near ambient temperature with the assistance of the additional mass of the septum nut extension. This prevents any silicone oils present in the septum from volatizing and being carried into the column. This feature may be retrofitted onto earlier versions of the SRI GC heated injection port, as only two new parts are required. The following page contains chromatographs that illustrate the effect of this new injector design

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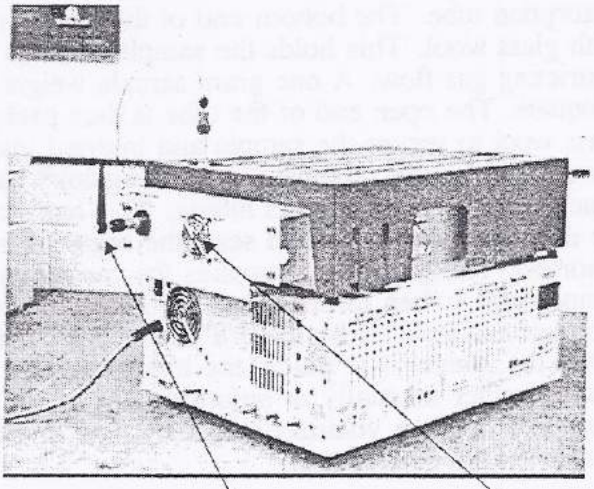
Chapter: INJECTORS

Topic: THERMAL DESORBER OPERATION

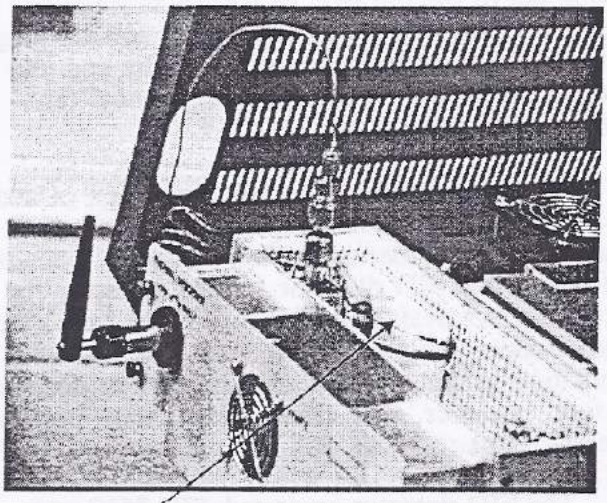
The SRI Thermal Soil Desorber accessory is useful for the analysis of volatile and especially semi-volatile compounds in soil or other granular solids. Because the analyte can be extracted from the soil by heat alone, with little or no sample preparation, field analyses can be performed without liquid solvent extraction. In addition, very high sensitivity for semi-volatile compounds such as diesel fuel can be obtained because essentially all the analyte is extracted from up to a gram of soil and deposited on column.



Valco Valve handle rotates to inject sample

Heated Valve Oven contains Thermal Desorber

The SRI Thermal Soil Desorber accessory is mounted in a heated valve oven on the left hand side of the 8610C Gas Chromatograph. The glass tube which contains up to a gram of soil is inserted into the hot (250 C) desorber cell through an opening in the top of the GC's red lid, and then secured by tightening the nut and 3/8" graphite ferrule. The handle of the manually operated Valco 10 port valve exits from the left rear of the heated valve oven, and is rotated to direct the carrier gas flow down and through the hot soil, transporting any hydrocarbons with boiling points below 300 C onto the GC column. The stainless steel tubing leading from the Valco valve to the column is routed and insulated to maintain a high temperature all along the path to the column oven to prevent high boiling compounds from condensing or tailing.



Transfer line from valve to column must be kept as hot as possible to avoid sample condensation. Arrange insulation to create "hot pocket" in this area.

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Chapter: INJECTORS

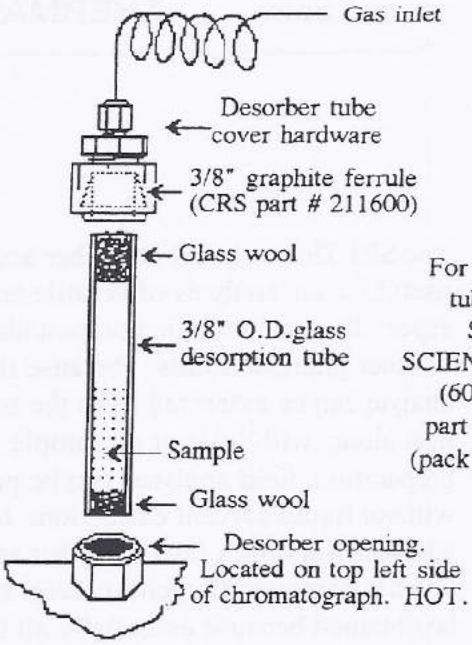
Topic: THERMAL DESORPTION

With the SRI 8610 Thermal Desorption unit, samples of soil or other solids can be analyzed for organic compounds without any extraction or other special sample preparation. The sample being tested is placed directly into the 3/8" O.D. machine glass desorption tube. The bottom end of the tube is plugged with glass wool. This holds the sample in place without restricting gas flow. A one gram sample weight is adequate. The open end of the tube is then packed with glass wool to secure the sample and inserted into the opening of the 3/8" stainless steel Swagelok® hardware attached to the pigtailed gas tubing. This hardware is the desorber tube cover and seals the organics in until desorbed. The gas tubing supplies the carrier gas. The sample tube is then inserted into the heated desorption chamber and secured by the 3/8" Swagelok® nut. When the sample is in place, the injection valve is rotated (either manually or automatically, if so equipped), and the volatized organics flow into the column on the carrier gas.

Historically, samples in soil have required solvent extraction with methlyene chloride, hexane, carbon disulfide or others prior to injection into a gas chromatograph. Unfortunately, solvent extraction often dilutes the sample and adversely affects detection limits. The detection limit for diesel fuel in soil by extraction is typically 10 ppm. When thermal desorption is employed, 10 ppb is attainable. With the phasing out of the use of CFCs such as freon and the ever-increasing scrutiny of laboratory solvent usage, the stripping of analytes from the soil by and into the column by thermal desorption is a practical (and sensible) alternative.

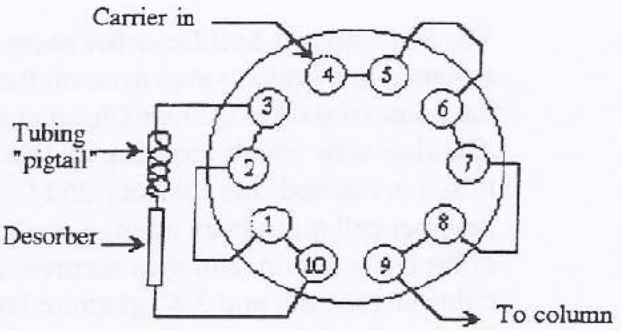
In the past, direct thermal desorption of average soil samples had been difficult due to the massive amounts of water liberated. This tended to extinguish the flame of the FID detector (typical detector for hydrocarbon analysis). Water elutes along with the early gasoline components and may interfere with the quantitation of benzene and toluene. Water does not interfere, however, with diesel quantitation because the diesel components elute well after the water.

The FID detector-equipped SRI 8610 gas chromatograph is supplied with an advanced design ceramic ignitor which can be run hot continuously, thus re-igniting the FID flame should it momentarily be affected by the passing water vapor. This minimizes the water interference and flame-out difficulties normally experienced with high moisture content samples analyzed with an FID detector.

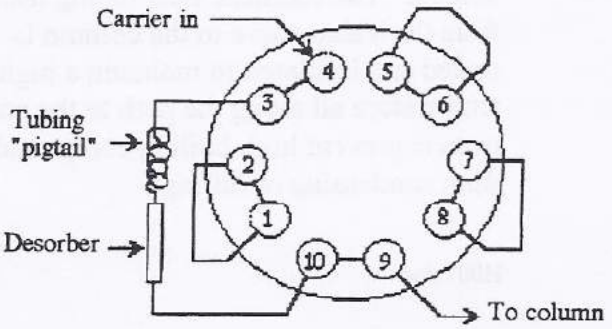


THERMAL DESORBER DETAIL

For replacement tubes, contact: SANDFIRE SCIENTIFIC LTD. (604) 826-5355 part no. S-9100 (pack of 10 tubes)



10 PORT VALVE DIAGRAM "LOAD" POSITION



10 PORT VALVE DIAGRAM "INJECT" POSITION

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Chapter: INJECTORS

Topic: OPERATION OF THE THERMAL DESORBER

To operate the SRI model 8610 thermal desorption unit, the following steps are required:

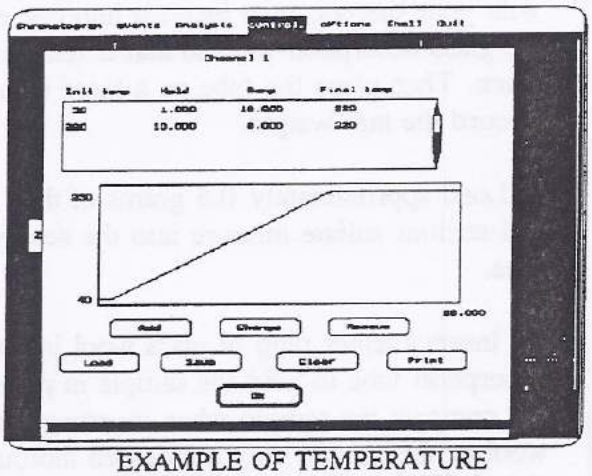
1. Place a clean desorption tube with a glass wool plug at one end on a scale of known accuracy. The tare weight is obtained. This is done by either weighing the clean, empty tube and recording the weight, or by placing the tube on the weighing platform and zeroing the balance.
2. Load the sample into the desorption tube and place the tube back on the balance. The gross or sample weight is recorded. The actual sample weight is obtained by subtracting the tare weight from the gross weight. A sample of solid weighing between 0.1 and 1.0 gm is recommended for best results. It is preferable to use a small sample due to the moisture that average samples contain. A small sample is less likely to interfere with the FID detector flame. A larger sample will permit the user to attain lower detection limits, but water content must be considered.
3. The tube containing the weighed sample is plugged with glass wool to hold the sample inside and the tube is inserted into the 3/8" opening of the Swagelok® hardware comprising the desortion tube cover. The end of the plugged tube is slid into the opening with the nut loosened. Once the tube has been inserted, the nut is tightened to seal the sample in the assembly.
4. Verify that the injection valve is in the "LOAD" position. Insert the sealed desorption tube assembly into the desorption chamber opening on top of the chromatograph and quickly secure it in place by tightening the Swagelok® nut at the opening. Care should be exercised when performing this step, as the desorption chamber is typically maintained at 350 degrees C and a burn potential exists.
5. Initiate the chromatogram either by keyboard or foot switch.
6. As soon as the desorption tube assembly has been secured into the desorption chamber, the injection valve is rotated to the "INJECT" position, and the sample is allowed to flow into the column. After the sample has desorbed completely, the valve is returned to the "LOAD" position. The tube may then be removed from the desorption chamber and cleaned. The contents of the tube should be removed and discarded. Once the tube has been thoroughly cleaned, it may be returned to service. If in doubt, a blank run should be used to verify that the tube has been cleaned adequately. Once the blank chromatogram is acceptable, the tube may be re-used for a subsequent sample.

Users may make their own tubes if so desired.

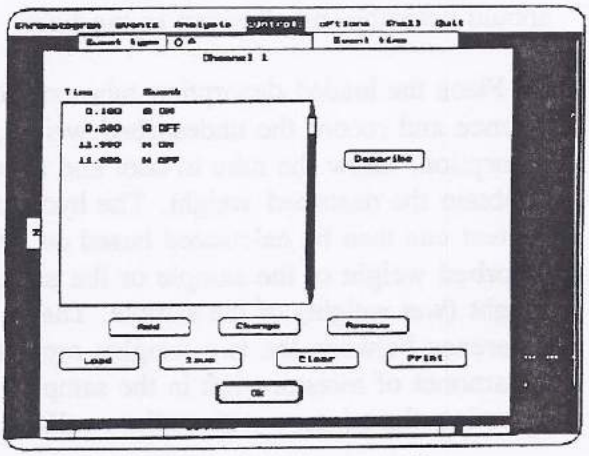
The column is connected to the injection valve inside the valve oven using a 1/16" to 1/8" adapter and 0.040" I.D. stainless steel tubing (1/16" O.D.). This ensures a uniform sample temperature while en route to the column and eliminates any possible cold spots.

The ignitor element may be set to 600°C (a dull red glow) for the duration of the run in order to avoid any possibility of FID flame-out should the sample have a high moisture content. The ignitor element can operate continuously at this high temperature without affecting its normal life expectancy.

Replacement desorber tubes may be ordered directly from Sandfire Scientific Ltd. in Mission, B.C., Canada at phone (604) 826-5355 (part no. S-9100).



EXAMPLE OF TEMPERATURE PROGRAM FOR DESORPTION



EXAMPLE OF TIMED EVENT TABLE FOR CONTROL OF AUTOMATED INJECTION

Chapter: INJECTORS & GAS VALVES

Topic: Thermal Desorber Soil Sample Preparation

1) To ensure that the soil sample analyzed is representative of the site sample, mix the soil in the sample container completely. Then weigh 10 grams of soil from the sample container into a 150 ml beaker.

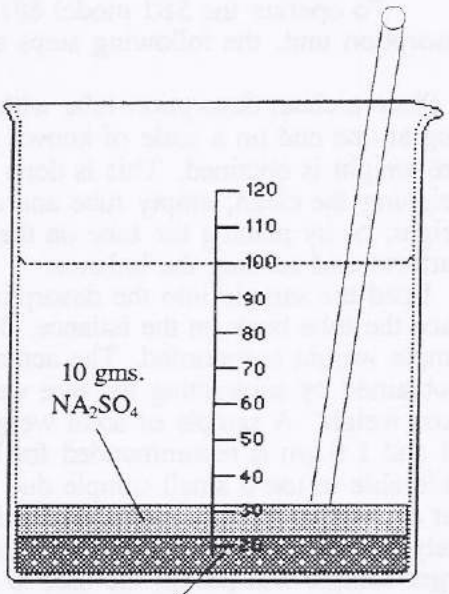
2) Add 10 grams of granular sodium sulfate (Na_2SO_4) to the beaker and mix with a stirring rod or spatula. The granular sodium sulfate, when mixed with the soil, absorbs most of the moisture from the soil, allowing clay soils to be ground into smaller particles. This is important because dense clay will not fully desorb. The mixture of soil and NA_2SO_4 should be of a granular consistency with small uniform particles.

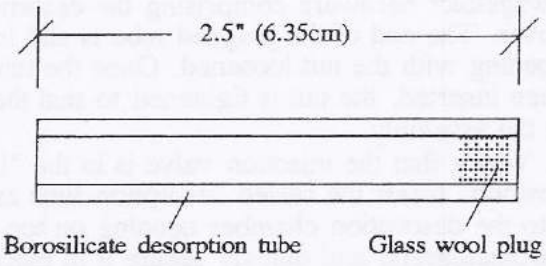
3) Roll a small amount of glass wool into a ball with your fingers, then insert it into one end of the glass desorption tube so that it remains in place. Then place the tube on a tared balance. Record the tare weight.

4) Load approximately 0.5 grams of the soil-sodium sulfate mixture into the desorption tube.

5) Insert another plug of glass wool into the desorption tube to hold the sample in place. Do not compact the sample when inserting the glass wool or the sample may not desorb thoroughly. When properly loaded and plugged, the tube should resemble the diagram to the right.

6) Place the loaded desorption tube on the balance and record the undesorbed weight. After desorption, allow the tube to cool and re-weigh to obtain the desorbed weight. The hydrocarbon content can then be calculated based on either the desorbed weight of the sample or the undesorbed weight (wet weight) of the sample. The difference between the two weights represents the amount of moisture left in the sample following the mixture with sodium sulfate.





Placement of glass wool in desorption tube prior to sample insertion

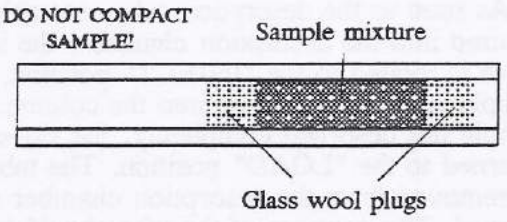


Diagram of assembled sample desorption tube containing 0.5 gms of soil - Na_2SO_4 sample mixture

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Chapter: CUSTOM MODIFICATIONS

Topic: Custom Valve Configuration Diagram

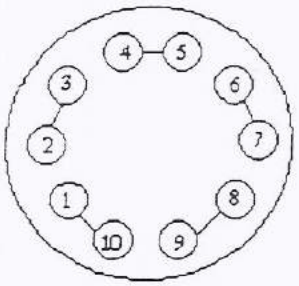
Most of the gas chromatographs manufactured by SRI that employ multi-port gas valves follow a standard gas line connection and flow path scheme that is specific to the user's application and/or dictated by the analytical test method. The majority of these gas valve schemes have been diagrammed and are included in the Injector and Gas Valves section of the unit's manual. The page header information will quickly identify the different application diagrams for the user's reference. In certain cases, the ten-port valve must be plumbed differently in order to perform a unique function as required by the user of the instrument. If manual entries have been made on this diagram page, the SRI gas chromatograph that accompanies this manual has been equipped with a ten port valve that has been custom-configured to the specifications of the user.

All custom plumbing of this ten-port valve will be documented on this page by the builder for the user's reference. Please note that there are TWO diagrams shown on this page. The first diagram represents the relationship between port connections and flow scheme when the valve is in the LOAD position (rotated counter-clockwise). The second diagram represents the relationship between port connections and flow scheme when the valve is in the INJECT position (rotated clockwise). These diagrams are applicable to both manually-operated valves and automated valves built into this chromatograph.

APPLICATION OF VALVE: _____

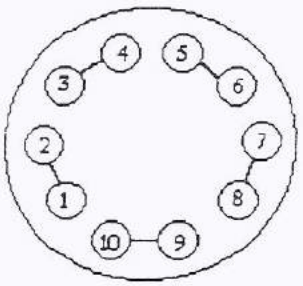
_____

10 PORT GAS VALVE



Valve in LOAD position

10 PORT GAS VALVE



Valve in INJECT position

DESCRIPTION OF CONNECTIONS MADE AT EACH PORT OF GAS VALVE

1 _____ 6 _____

2 _____ 7 _____

3 _____ 8 _____

4 _____ 9 _____

5 _____ 10 _____

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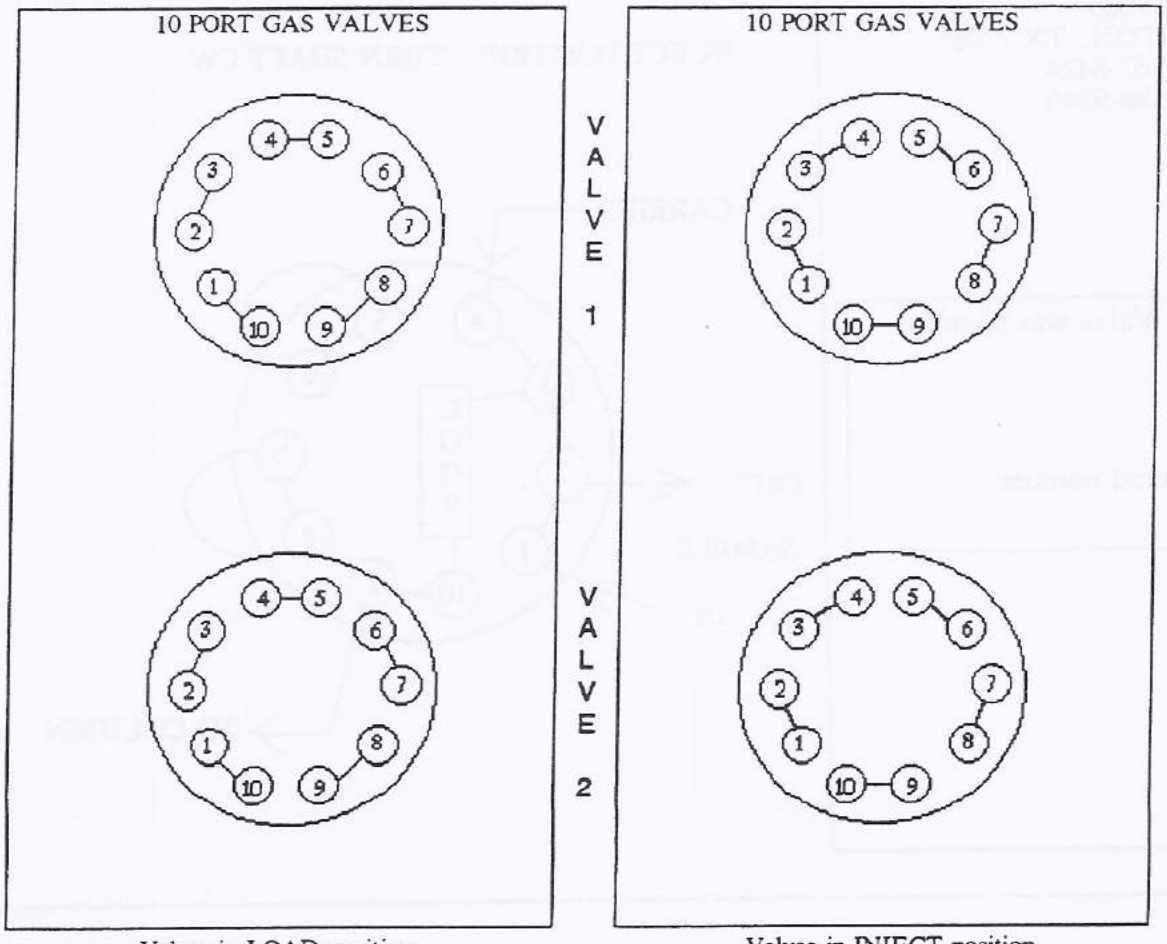
Chapter: INJECTORS AND GAS VALVES

Topic: Custom Dual Valve Configuration Diagram

Most of the gas chromatographs manufactured by SRI that employ multi-port gas valves follow a standard gas line connection and flow path scheme that is specific to the user's application and/or dictated by the analytical test method. The majority of these gas valve schemes have been diagrammed and are included in this section of the unit's manual. The page header information will quickly identify the different application diagrams for the user's reference. In certain cases, the ten-port valve must be plumbed differently in order to perform a unique function as required by the user of the instrument. In some applications, dual valves are required and utilized. If manual entries have been made on this diagram page, the SRI gas chromatograph that accompanies this manual has been equipped with dual ten port valves that have been custom-configured to the specifications of the user.

All custom plumbing of these ten-port valves will be documented on this page by the builder for the user's reference. Please note that there are TWO diagrams shown on this page. The first diagram represents the relationship between port connections and flow scheme when the valves are in the LOAD position (rotated counter-clockwise). The second diagram represents the relationship between port connections and flow scheme when the valves are in the INJECT position (rotated clockwise). These diagrams apply to both manually-operated valves and automated valves built into this chromatograph.

APPLICATION OF VALVES: _____



Valves in LOAD position

Valves in INJECT position

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SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

Carrier gas flows onto the column while sample gas flows through the sample loop.

In the INJECT position:

Carrier gas flows through the sample loop and then onto the column.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

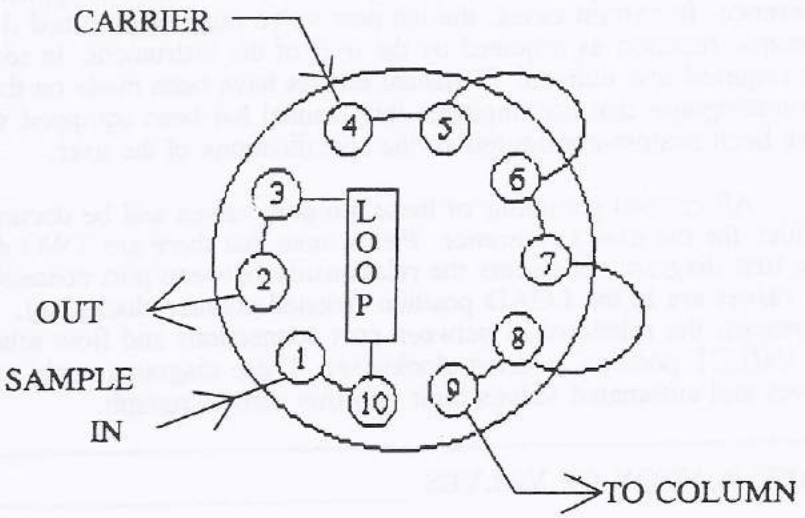
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

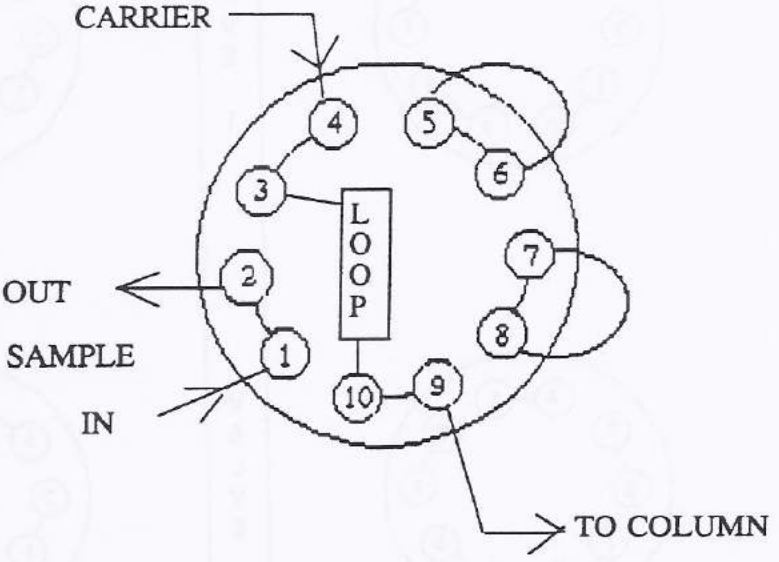
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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Chapter: INJECTORS & GAS VALVES
Topic: ALTERNATE LOOP SAMPLING OF TWO DIFFERENT STREAMS

REV. 9-7-91

SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

Sample loop A is in position to be loaded while sample loop B has carrier gas flowing through it onto the column.

In the INJECT position:

Sample loop B is in position to be loaded while sample loop A has carrier gas flowing through it onto the column.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

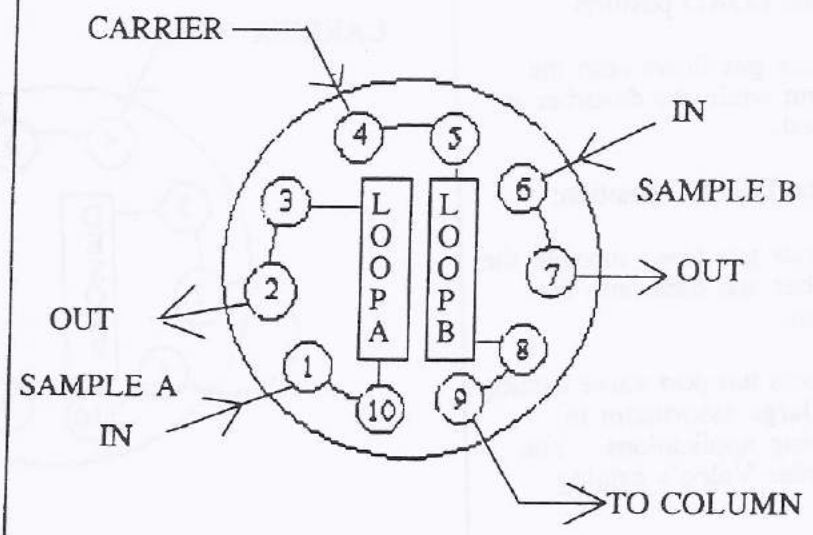
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

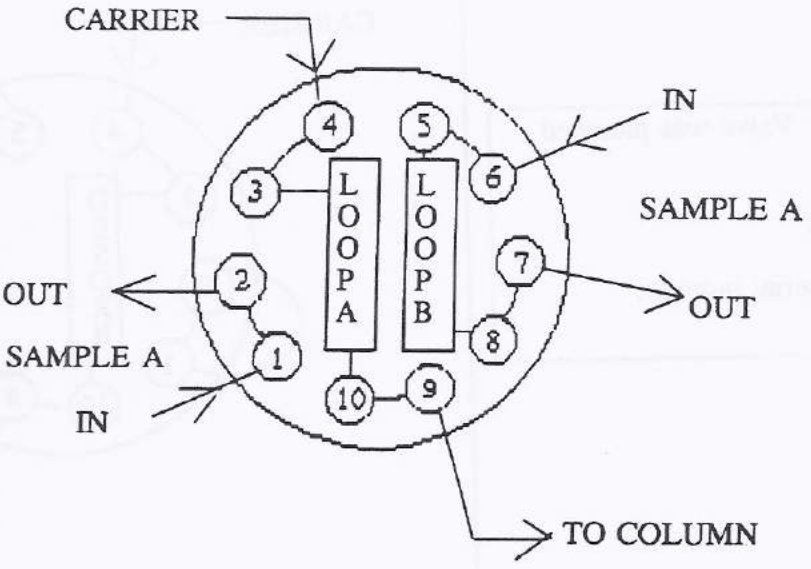
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

Carrier gas flows onto the column while the desorber is isolated.

In the INJECT position:

Carrier gas flows through the desorber and then onto the column.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

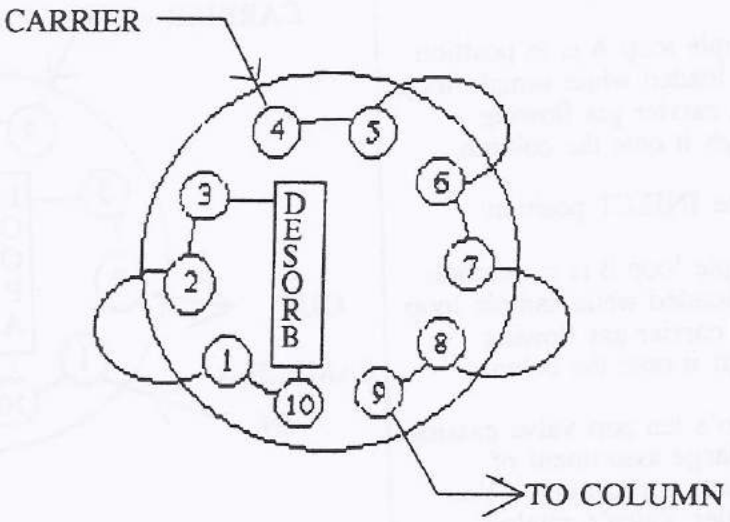
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

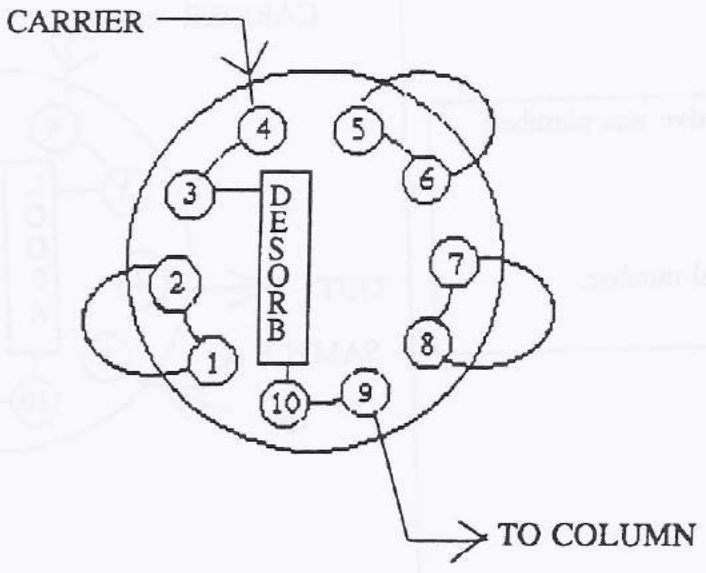
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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Chapter: INJECTORS & GAS VALVES
Topic: SIMULTANEOUS INJECTION OF THE SAME SAMPLE INTO TWO SEPARATE COLUMNS

REV. 9-7-91

SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

Both sample loops are in the load position while carrier A flows onto column A and carrier B flows onto column B.

In the INJECT position:

Carrier A flows through loop A onto column A while carrier B flows through loop B onto column B.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

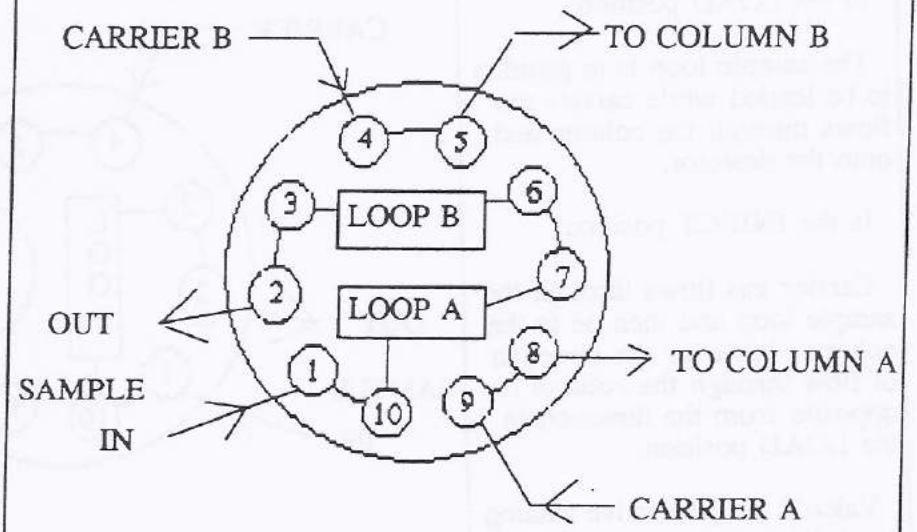
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

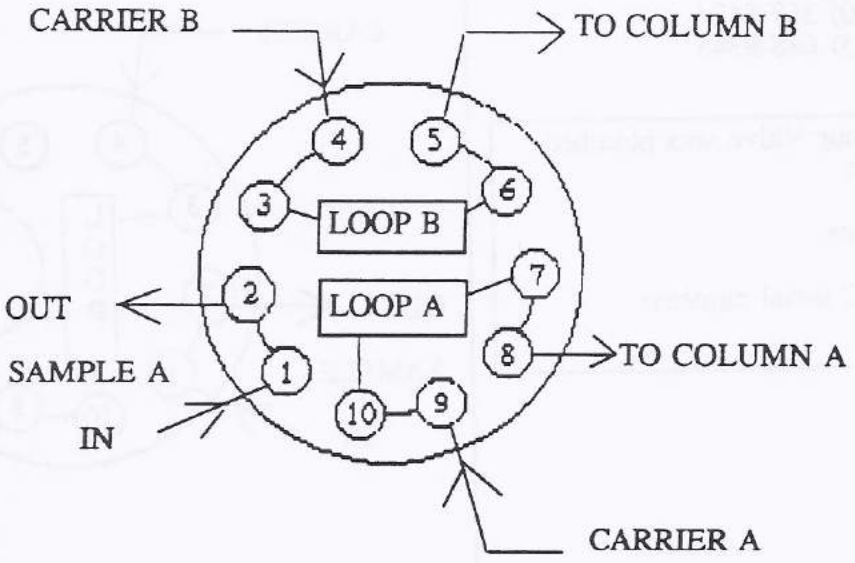
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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Chapter: INJECTORS & GAS VALVES
Topic: LOOP SAMPLING WITH BACKFLUSH TO DETECTOR

REV. 9-7-91

SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

The sample loop is in position to be loaded while carrier gas flows through the column and onto the detector.

In the INJECT position:

Carrier gas flows through the sample loop and then on to the column, however the direction of flow through the column is opposite from the direction in the LOAD position.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

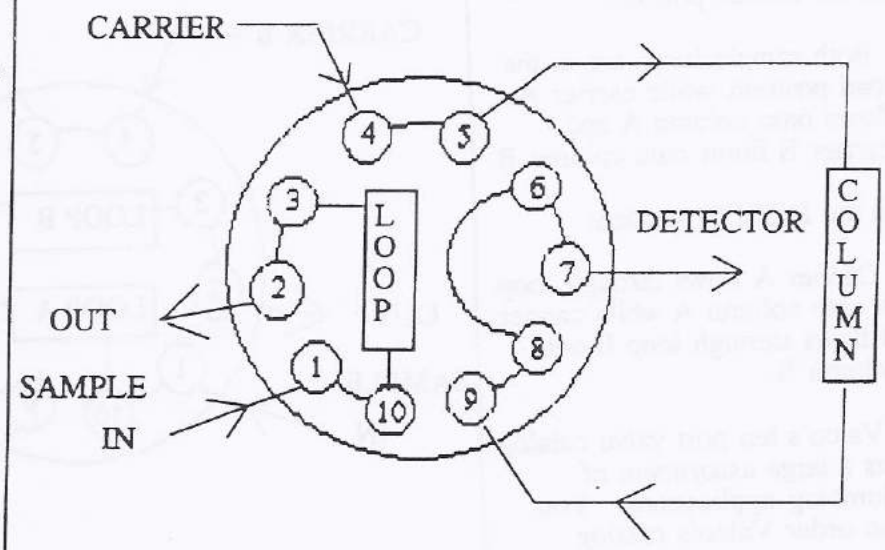
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

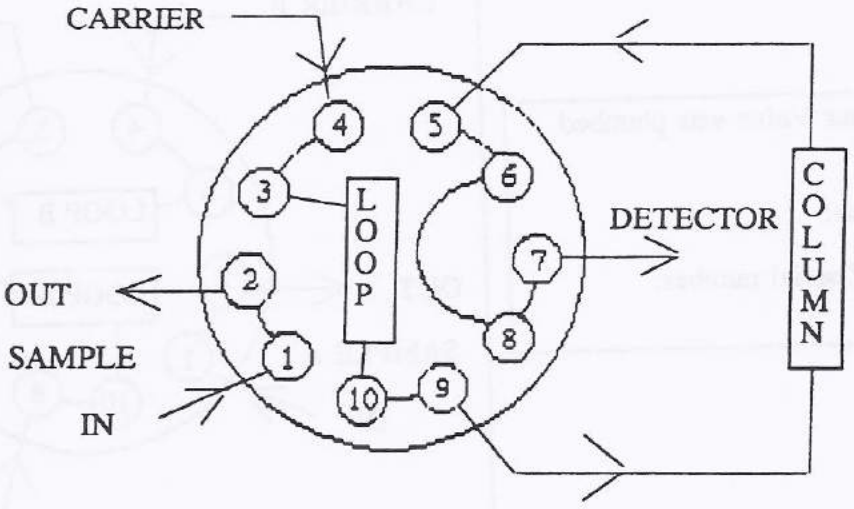
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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Chapter: INJECTORS & GAS VALVES
Topic: LOOP SAMPLING WITH BACKFLUSH OF PRE-COLUMN TO VENT

REV. 9-7-91

SRI has plumbed the valve in your GC according to the accompanying schematic.

In the LOAD position:

The sample loop is in position for loading. Column 1 has carrier flowing through and out the vent. Column 2 has flow from carrier 2.

In the INJECT position:

Carrier 2 is venting while carrier 1 flows through column 1 and 2. The direction of flow through column 1 in the INJECT position is reversed from the LOAD position.

Valco's ten port valve catalog has a large assortment of plumbing applications. You can order Valco's catalog from:

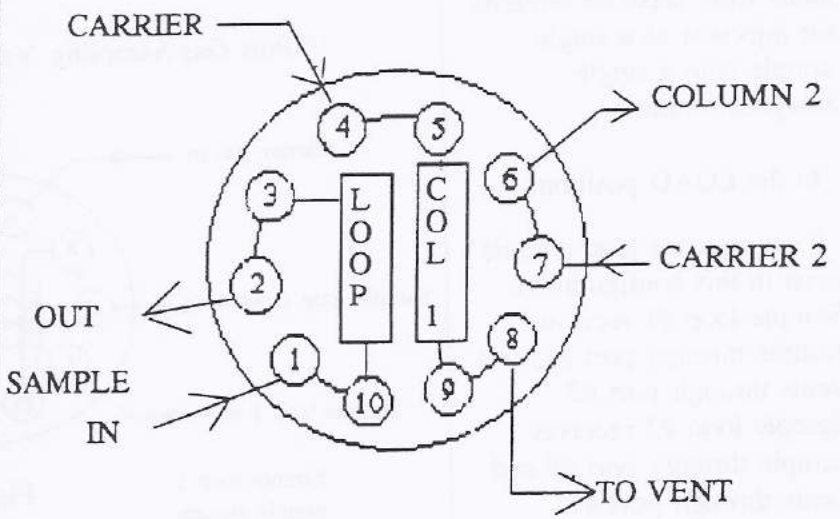
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

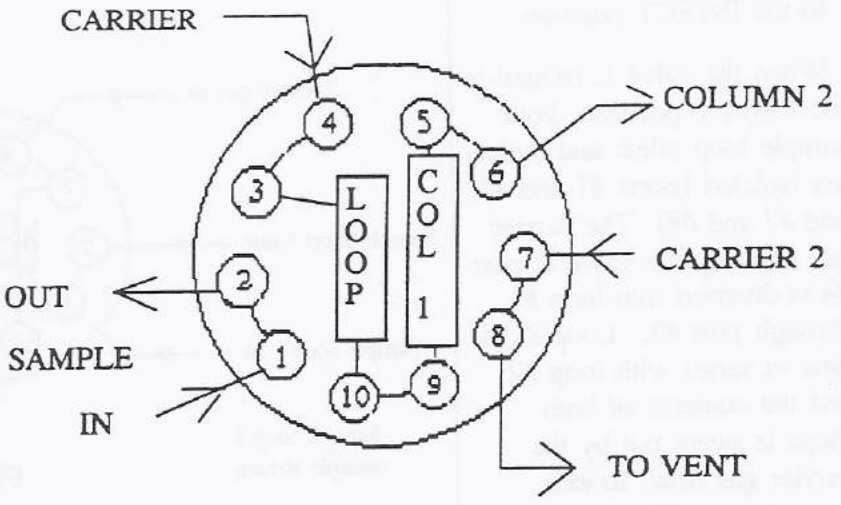
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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Chapter: INJECTORS & GAS VALVES

Topic: Dual Loop Injection of Two Separate Streams Onto One Column

The following is a description of the 10 port gas sampling valve plumbed to permit the loading of dual loops from separate streams for injection as a single sample onto a single analytical column.

In the LOAD position:

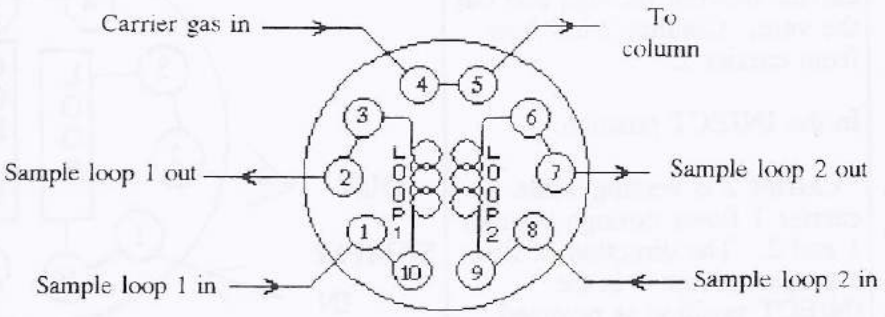
Two separate loop circuits exist in this configuration. Sample loop #1 receives sample through port #1, and vents through port #2. Sample loop #2 receives sample through port #8 and vents through port #7. Meanwhile the carrier gas is routed into port #4 from the injector, through the valve, and out through port #5 to the analytical column and detector.

In the INJECT position:

When the valve is rotated to the INJECT position, both sample loop inlets and outlets are isolated (ports #1 and #2, and #7 and #8). The carrier gas entering the valve at port #4 is diverted into loop #1 through port #3. Loop #1 is now in series with loop #2 and the contents of both loops is swept out by the carrier gas flow, to exit through port #5 to the analytical column for analysis. At no time is the carrier gas flow to the column interrupted, protecting both the column and the detector.

This gas sampling valve configuration permits two separate loops to be loaded simultaneously from two streams and injected together onto the analytical column.

10 Port Gas Sampling Valve in LOAD Position

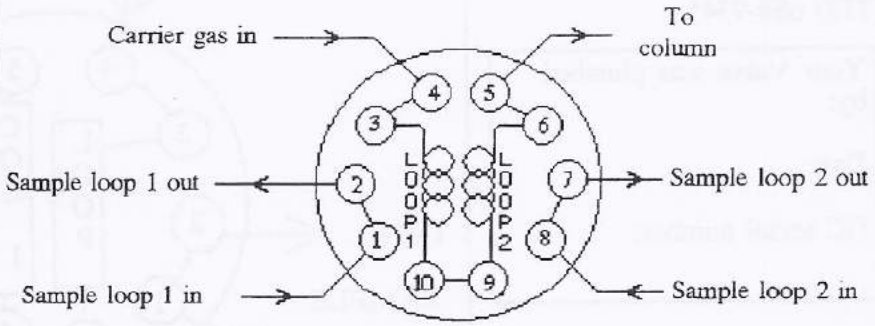


Sample loop 1 sample stream

Fig. 1

Sample loop 2 internal standard

10 Port Gas Sampling Valve in INJECT Position



Sample loop 1 sample stream

Fig. 2

Sample loop 2 internal standard

This configuration is convenient for applications where an internal standard must be inserted into the sample prior to analysis. Both samples are then merged and deposited on-column for analysis when the sampling valve is rotated to the INJECT position.

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Chapter: INJECTORS & VALVES
Topic: Liquid and Loop Sampling with Backflush of Pre-column to Vent (Using External Liquid Sample Valve)

SRI has plumbed the valves in your GC according to the accompanying schematic.

In the LOAD position:

The liquid sample valve (LSV) in in position for loading while the gas sample valve is in inject position.

In the INJECT position:

Carrier gas flows through the liquid sample slot in LSV and the 10-port valve then on to the column.

The 10-port valve remains in the inject position throughout the injection procedure and then switches to load position for vent.

Valco's ten port valve catalogue has a large assortment of plumbing applications. You can order Valco's catalogue from:

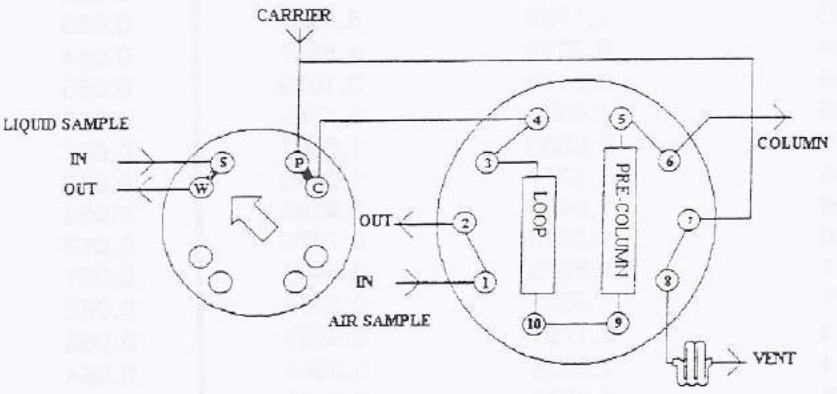
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

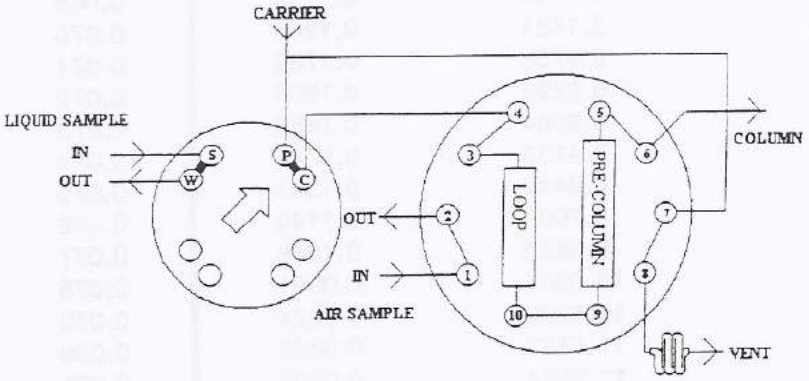
Date:

GC serial number:

LOAD POSITION



INJECT POSITION



TUBE VOLUME SELECTION GUIDE

INTERNAL DIAMETER	MICROLITERS PER INCH	INCHES PER MICROLITER
0.001	0.0129	77.6979
0.002	0.0515	19.4245
0.003	0.1158	8.6331
0.004	0.2059	4.8561
0.005	0.3218	3.1079
0.006	0.4633	2.1583
0.007	0.6306	1.5857
0.008	0.8237	1.2140
0.009	1.0425	0.9592
0.010	1.2870	0.7770
0.011	1.5573	0.6421
0.012	1.8533	0.5396
0.013	2.1751	0.4598
0.014	2.5226	0.3964
0.015	2.8958	0.3453
0.016	3.2948	0.3035
0.017	3.7195	0.2689
0.018	4.1700	0.2398
0.019	4.6462	0.2152
0.020	5.1481	0.1942
0.021	5.6758	0.1762
0.022	6.2292	0.1605
0.023	6.8084	0.1469
0.024	7.4133	0.1349
0.025	8.0440	0.1243
0.026	8.7003	0.1149
0.027	9.3825	0.1066
0.028	10.0903	0.0991
0.029	10.8239	0.0924
0.030	11.5833	0.0863
0.031	12.3684	0.0809
0.032	13.1792	0.0759
0.033	14.0158	0.0713
0.034	14.8781	0.0672
0.035	15.7662	0.0634
0.036	16.6799	0.0600
0.037	17.6195	0.0568
0.038	18.5847	0.0538
0.039	19.5758	0.0511
0.040	20.5925	0.0486
0.041	21.6350	0.0462
0.042	22.7032	0.0440
0.043	23.7972	0.0420
0.044	24.9169	0.0401
0.045	26.0624	0.0384
0.046	27.2336	0.0367
0.047	28.4306	0.0352
0.048	29.6532	0.0337
0.049	30.9017	0.0324
0.050	32.1758	0.0311

INTERNAL DIAMETER	MICROLITERS PER INCH	INCHES PER MICROLITER
0.051	33.4757	0.0299
0.052	34.8014	0.0287
0.053	36.1527	0.0277
0.054	37.5299	0.0266
0.055	38.9327	0.0257
0.056	40.3613	0.0248
0.057	41.8157	0.0239
0.058	43.2958	0.0231
0.059	44.8016	0.0223
0.060	46.3332	0.0216
0.061	47.8905	0.0209
0.062	49.4735	0.0202
0.063	51.0822	0.0196
0.064	52.7167	0.0190
0.065	54.3770	0.0184
0.066	56.0630	0.0178
0.067	57.7747	0.0173
0.068	59.5122	0.0168
0.069	61.2754	0.0163
0.070	63.0643	0.0159
0.071	64.8790	0.0154
0.072	66.7195	0.0150
0.073	68.5856	0.0146
0.074	70.4775	0.0142
0.075	72.3952	0.0138
0.076	74.3386	0.0135
0.077	76.3077	0.0131
0.078	78.3026	0.0128
0.079	80.3232	0.0124
0.080	82.3696	0.0121
0.081	84.4417	0.0118
0.082	86.5395	0.0116
0.083	88.6631	0.0113
0.084	90.8124	0.0110
0.085	92.9875	0.0108
0.086	95.1882	0.0105
0.087	97.4148	0.0103
0.088	99.6670	0.0100
0.089	101.9450	0.0098
0.090	104.2488	0.0096
0.091	106.5783	0.0094
0.092	108.9335	0.0092
0.093	111.3145	0.0090
0.094	113.7212	0.0088
0.095	116.1537	0.0086
0.096	118.6119	0.0084
0.097	121.0958	0.0083
0.098	123.6055	0.0081
0.099	126.1409	0.0079
0.100	128.7020	0.0078

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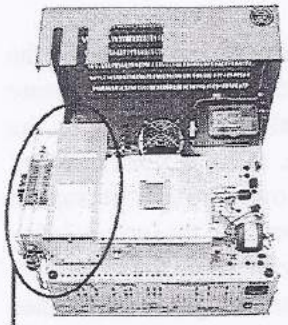
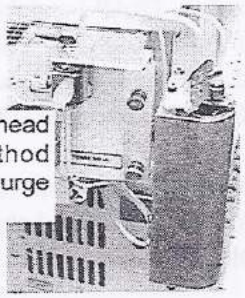
Overview

Built-in to the 8610C gas chromatograph, the SRI Purge & Trap is designed for compliance with EPA Methods 5030 and/or 5035 for the extraction of volatile organic compounds from water or soil samples. The purge and trap technique is applicable to a range of molecules from C_3 to C_{12}. The Purge & Trap hardware consists of a 10 port valve in a heated, ducted valve oven, two traps, a cooling fan, and the purge head(s). The unique dual trap design enables the simultaneous trapping of compounds with different boiling points. Each trap has its own heater, and the ends of the traps are enclosed in the valve oven ducts to prevent cold spots. The cooling fan maintains the adsorption temperature and rapidly lowers trap heat after desorption. The trap in the lower position (TRAP 1) is usually packed with Tenax™-GR at the factory, while the upper trap (TRAP 2) is left empty for the user to pack with the desired adsorbent. A Carbosieve™ packed trap is also shipped with the GC for optional installation in the TRAP 2 position. The Carbosieve trap is used only when the analysis includes light gaseous VOC's, the most common being vinyl chloride. The Method 5030 Purge & Trap is the standard model with a fixed purge head that uses disposable 16mm test tubes for ambient temperature purging. There is a built-in septum port on this purge head through which gas standards may be spiked. The Method 5030/5035 Purge & Trap features interchangeable purge heads. The 5035 purge head is a thermostatted heater body (from ambient to 50°C) which accepts standard 40mL VOA vials. Inside the heater body are two needles which puncture the septum: the longer one bubbles helium purge gas through the sample, while the shorter needle exhausts sample-laden gas to the adsorbent traps. In compliance with EPA Method 5035, the purge head is mechanically agitated while the sample is being purged. There is a syringe port on the Method 5030/5035 Purge & Trap that allows water and internal standard to be added to the sample in the vial without puncturing the septum again. Operation of the Purge & Trap is automated by the PeakSimple data system.

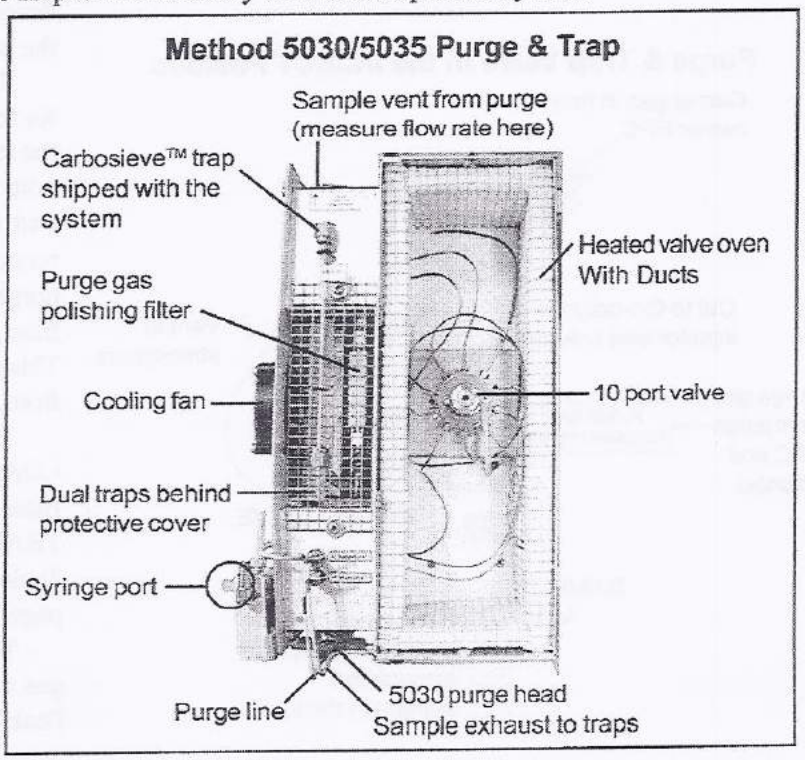


SRI GC equipped with Method 5030 Purge & Trap

Purge head on a Method 5030 Purge & Trap



5035 purge head on a Method 5030/5035 Purge & Trap



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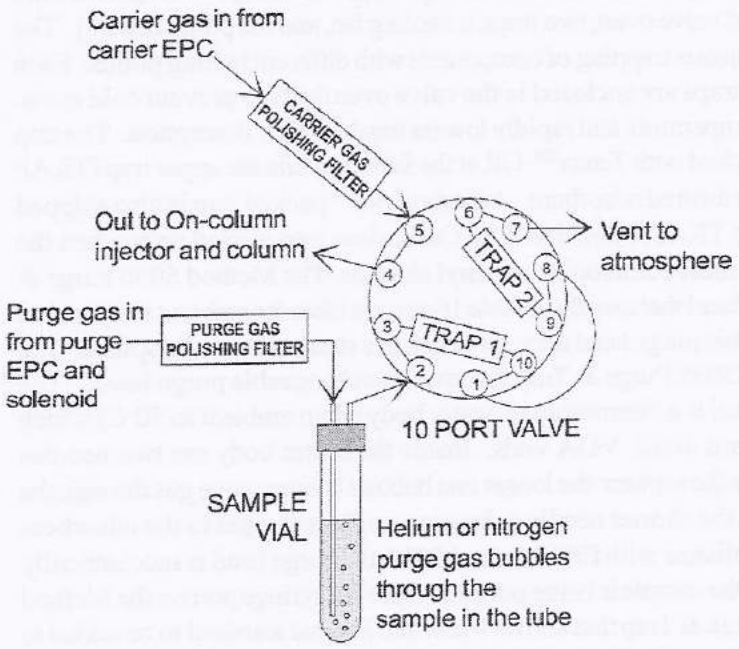
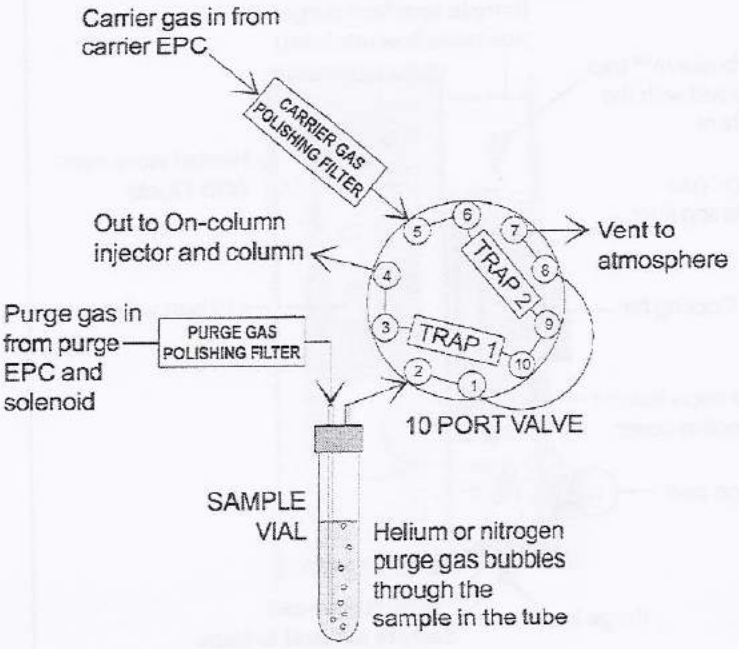
Theory of Operation

Purge & Trap Valve in the LOAD Position



Purge & Trap Valve in the INJECT Position



The SRI Purge & Trap uses a 10 port gas sampling valve and dual adsorbent traps. Each trap has independent adsorption and desorption setpoints to optimize the analyte trapping and releasing from each adsorbent.

When the valve is in the LOAD position, the sample-laden purge gas from the test tube or VOA vial is directed through the two traps, then out to vent, loading the traps with sample at the adsorption temperature (30-40°C). In this position, the carrier gas merely enters and exits the valve.

After a period of time sufficient for the traps to reach desorption temperature (200°C), the valve is actuated to the INJECT position. In the INJECT position, the carrier gas flows through the traps in the direction opposite to the sample-laden purge gas flow with which the traps were loaded. The carrier gas backflushes desorbed analytes into the column, while the purge gas flows out to vent.

The valve remains the INJECT position for the optional bake cycle, during which the respective desorption temperatures of both traps are raised an additional 50°C, and the purge gas polishing filter is reconditioned. A relatively high flow of purge gas sweeps through the hot polishing filter, which heats whenever TRAP 1 heats. This purge gas flow sweeps contaminants from the polishing filter and out to vent.

The valve is then acutated back into the LOAD position, TRAP 1 and the polishing filter heat are turned OFF, followed by TRAP 2, then the purge gas (see the Event Table on the ***General Operating Procedures*** page.)

Trap heating, valve rotation, and purge gas control are automated through the PeakSimple data system.

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Sample Preparation

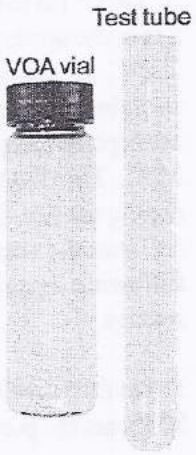
Sample preparation depends on the sample type, concentration, amount, etc. The third edition of SW-846 from the EPA is accessible on the Internet. Go to **http://www.epa.gov/epaoswer/hazwaste/test/main.htm** and click on the **5000 Series** link to download Methods 5030 and 5035.

Method 5030

Method 5030 style purge and trap is for the analysis of VOCs in aqueous samples. This purge and trap technique is limited to analytes that purge efficiently from water. 10mL of the sample is placed in a clean test tube. The test tube headspace will contain ambient air, so if your laboratory or work area is not free of solvent fumes, they will show up in your chromatogram.

For aqueous samples:

1. Insert a 10mL aliquot of the aqueous sample into a clean test tube.
2. Plug the test tube opening with your thumb and shake it until the contents are evenly dispersed.
3. Quickly slide the test tube over the purge gas tubing and into the purge head, and tighten it in place with the knurled retaining nut.
4. Immediately begin the analysis by pressing the RUN button on the front of the GC or by pressing the spacebar on your computer keyboard.



Always use clean sample containers

For medium concentration soil samples, do a quick methanol extraction:

1. Place 10g of sample into a clean glass container. Add 20mL of methanol and shake it for 1-3 minutes.
2. Allow the soil to settle, then pull 100µL of the liquid solution into a glass syringe and inject it into the test tube containing 10mL of organic free reagent water.
3. Plug the test tube opening with your thumb and shake it until the contents are evenly dispersed.
4. Begin the analysis. You may need to dilute the sample more or less, depending on the concentration.

Method 5035

Method 5035 style purge and trap is for the analysis of VOCs that are purgeable from soil at 40°C. This method does not allow the VOC's to escape the VOA vial until it is punctured by the 5035 purge head needles. Approximately 5g of soil, weighed in the field at the time of collection, is sealed in a pre-weighed, septum-sealed, screw-top VOA vial containing a preservative solution. There is no need to insert a magnetic stirring bar since the SRI purge and trap mechanically agitates the VOA vial during the analysis. Organic-free reagent water, surrogates, and internal standards (if applicable) are added through the syringe port immediately before beginning the analysis.

1. Insert the VOA vial containing 5g of soil and 5mL of reagent water into the Method 5035 purge head.
2. Using the syringe port, inject 5mL of organic free reagent water, internal standards, and surogate compounds into the VOA vial.
3. Begin the analysis by pressing the RUN button or the computer keyboard spacebar.

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General Operating Procedures

The following are generalized operating guidelines for the SRI Purge & Trap system.

1. The purge gas flow is controlled with an Electronic Pressure Controller (EPC). Set the purge flow (measurable at the trap vent at the rear of the purge and trap system). 40mL/min is a typical purge flow. The pressure required for 40mL/min through a single Tenax trap is printed on the right panel of the GC. If you install the optional Carbosieve trap or another adsorbent trap in the TRAP 2 position, you will need to raise the pressure to maintain the flow. ***NEVER use hydrogen as a purge gas.*** SRI recommends helium purge gas.

Typical 5030/5035 Event Table

EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
0.200	(D "ON")	Shaker "ON"
5.000	(D "OFF")	Shaker "OFF"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.050	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
12.900	B "ON"	Trap set "ON" (+50°C)
13.000	G "OFF"	Valve in "LOAD"
14.900	F "OFF"	Trap 1 "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.300	E "OFF"	Purge "OFF"
15.500	B "OFF"	Trap set "OFF" (+0)

2. TRAP 1 is in the lower position in the Purge & Trap, and TRAP 2 is in the upper position. The trap temperatures are factory set at 200°C for desorption and may be adjusted using the trimpot setpoints on the top edge of the GC's front control panel. For adsorption temperatures, trap 1 is set at 30°C and trap 2 is set at 35°C. Trap heating will be controlled by the timed Event Table during the run. ***Note:*** the actual trap temperatures typically run 5°C over the setpoint. See the information and instructions on the following 2 pages for adjusting the trap adsorption temperature settings.

3. Set the valve oven temperature to 100°C or higher to avoid water condensation. If you're using Method 5035, set the purge head heater body temperature to 40°C. It is factory set to 40°C but is user adjustable.

4. Load or create an event table that is appropriate to the sample to be analyzed, or that is designed for compliance with a particular EPA Method. The valve oven in your Purge & Trap system is labeled with a typical Purge & Trap event table for a single Tenax trap. The event table shown above is an example for both methods; the only difference is that Method 5030 does not use Relay D (the sample vial shaker).

5. Load or create an appropriate temperature program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software. As a basic rule for good separation, the column oven should be kept at 40°C for 10-12 minutes: the first 8 minutes of the run plus 2-4 more minutes after the valve actuates to the INJECT position.

6. Activate and energize the detectors as necessary. For instance, if you had an Environmental GC system, you would turn on the PID lamp current, light the FID flame, and set the DELCD reactor temperature. Choose the detector gain settings according to the analysis. Consult the manual sections for your particular detector(s) operating procedures.

7. When the system is at temperature and displaying a stable signal, insert the sample test tube or VOA vial into the purge head and begin the analysis.

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General Operating Procedures Continued
Using Two Traps

The SRI dual trap design gives the Purge & Trap user many options to effectively trap and release analytes from a particular adsorbent. Due to its low affinity for water, Tenax™-GR is especially useful for the purging of VOCs from aqueous samples, making it a good general purpose trap for EPA style purge and trap techniques. The Carbosieve™ packed trap is very retentive. Because it tends to retain a large water peak and smear the other peaks, it should only be used when vinyl chloride is among the target analytes. This tendency to smear may be reduced by manipulating the desorption times for the two traps. If the Carbosieve™ trap (TRAP 2) is desorbed while the Tenax™-GR trap (TRAP 1) is still cold, the components will refocus on the Tenax™-GR. The Tenax™-GR trap is then heated to desorb the components in a more narrow band, which results in sharper peaks on the chromatogram.

Tenax™-GR Properties
Composite of Tenax TA and 30% graphite
Low water affinity
350°C temperature limit
200nm average pore size
60/80 mesh size
Available from Alltech
2051 Waukegan Road
Deerfield, IL 60015 USA
908-788-5550
www.alltechweb.com

Carbosieve™ S111 Properties
Carbon molecular sieve
Moderate water affinity
400°C temperature limit
15-40 angstrom average pore size
60/80 mesh size
Available from Supelco
Supleco Park
Bellefonte PA 16823
800-247-6628
www.sigma-aldrich.com

Dual Trap Event Table (Epap&t2c.evt)		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (Carbosieve) heat "ON"
7.000	G "ON"	Valve in "INJECT"
8.000	G "OFF"	Valve in "LOAD"
8.100	F "ON"	Trap 1 (Tenax-GR) heat "ON"
10.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
13.000	G "OFF"	Valve in "LOAD"
13.100	B "ON"	Trap set "ON" (+50°C)
14.900	F "OFF"	Trap 1 "OFF"
15.000	E "OFF"	Purge "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.200	B "OFF"	Trap set "OFF"

Version 2.66 of the PeakSimple software includes **Epap&t1c.evt** for a single trap, and **Epap&t2c.evt** for two traps.

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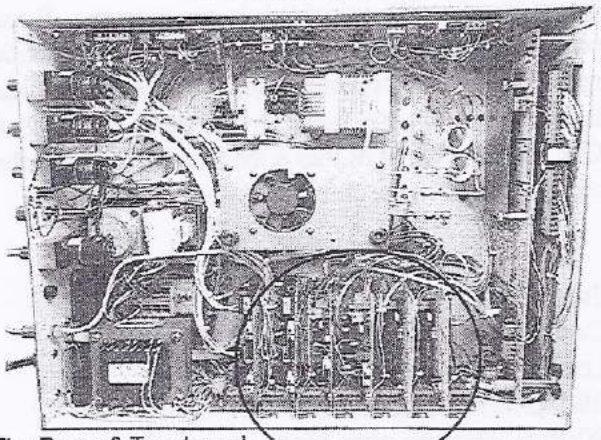
General Operating Procedures continued

Adjusting Trap Adsorption Temperatures

During the purge and trap process, the purge gas carries significant amounts of water into the traps. The Tenax™ trap is unaffected, due to its low affinity for water. The Carbosieve™ packing tends to retain the water, resulting in a large water peak at desorption. Adsorption settings can be adjusted by the user to set the Carbosieve trap at a high enough temperature to avoid water retention. However, this temperature may be too hot to trap target analytes. Therefore, experiment to find the adsorption temperatures that work best for your analyses. Once pinpointed, they usually require no further adjustment.

1. Remove the 6 screws that secure the bottom panel to the rest of the GC chassis. Support the panel while you gently rock the GC onto its back, then lower the panel to your working surface to access the chassis interior.

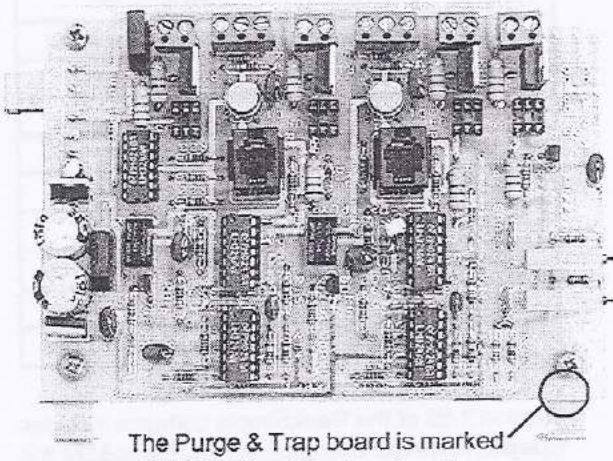
GC Chassis Interior



The Purge & Trap board is installed in this area inside the GC chassis

2. Locate the Purge & Trap board; it is one of a group of similar-looking boards installed along the back and top walls of the GC interior. The Purge & Trap board has two trimpots right next to each other, and it is marked with an upside-down "P&T" on the lower outer corner.

The Purge & Trap Board



Trap 2 trimpot setpoint

Trap 1 trimpot setpoint

The Purge & Trap board is marked with "P&T" (upside down) on the lower right corner

3. The two trap trimpot setpoints are on the outer edge of the board. The trimpot for Trap 1 is on the bottom, and the top trimpot is for Trap 2. Turn the trimpot while pressing the TOTAL button and observing the bright red LED display to set the trap adsorption temperature.

4. When you are finished adjusting both trap adsorption temperatures, place the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its base. Secure the base with its 6 screws.

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Switching / Replacing Traps

Three traps are included with your SRI Purge & Trap: a Tenax™-GR trap and a Carbosieve™ trap, both permanently packed, and a blank trap. The blank trap may be packed with an adsorbent of the user's choice or left blank, depending on the analytical situation. Follow the instructions below to access the traps for switching or replacement.

1. With the red protective GC cover raised, remove the Purge & Trap cover plate by loosening the four brass thumbscrews at its corners.

2. Carefully remove the two squares of white insulation from each valve oven duct to expose the fittings that secure the traps ends to the 1/16" O.D. tubing leading to the 10 port valve.

3. Gently slide the trap assembly out of the slots in the valve oven ducts (there is enough slack in the heater and thermocouple wires to pull either trap about 1 inch outside the duct).

4. Use two wrenches to loosen the 1/8" Swagelok type nuts that secure the traps ends to stainless steel 1/8"-1/16" reducing unions.

5. The trap heater is a clamshell design, consisting of two halves. To remove the heater from the trap, loosen but do not remove the two securing hex head screws. The two halves of the clamshell heater will open enough to let the trap drop out.

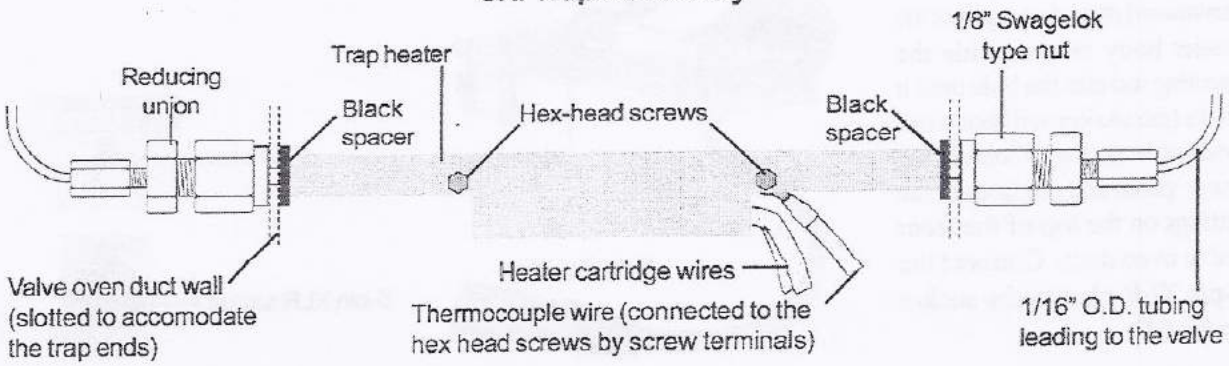
6. Attach the replacement trap to the reducing unions with the trap's two 1/8" nuts. Use stainless steel nuts and brass ferrules when replacing traps. DO NOT use graphite ferrules, as graphite has some adsorption properties and may interfere with your analysis.

7. Slip the trap into the clamshell heater and tighten the two hex head screws.

8. Gently push the trap ends back into the slots in the two interior duct walls, making sure that the black spacers are between the duct walls and the trap heater. TO AVOID DAMAGE, ARRANGE THE TRAPS SO THAT ONE TRAP'S HEATER WIRES DO NOT LAY ACROSS THE OTHER TRAP'S HEATER.

9. Repeat the process with the other trap if necessary. Replace the white duct insulation squares, then replace and secure the Purge & Trap cover plate.

SRI Trap Assembly



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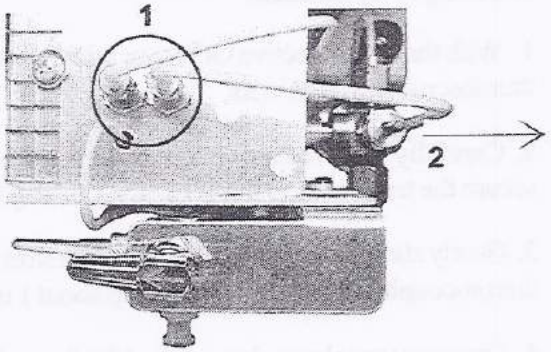
Method 5030/5035 Purge & Trap:
Changing the Purge Heads

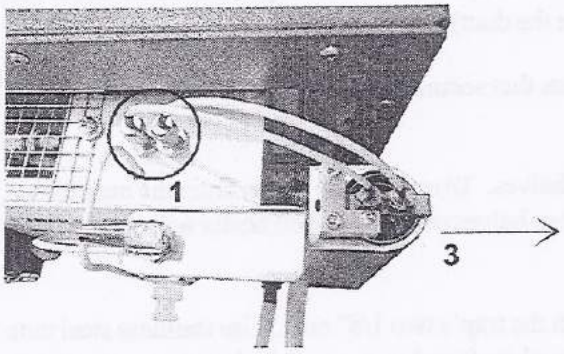
1. To change the purge heads, first disconnect the two purge gas lines at their fittings on the top of the front valve oven duct.

5030 Purge Head

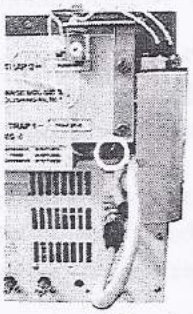


2. If you are removing the 5030 purge head, pull it out toward the front of the GC, and unplug the 5-pin XLR dummy plug.

5035 Purge Head



3. If you are removing the 5035 purge head, squeeze the protruding tail of the spring toward the heater body with your thumb as you pull the purge head out toward the front of the GC. Unplug the cord from the socket on the GC.

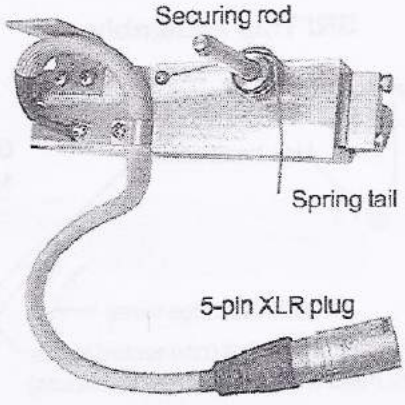


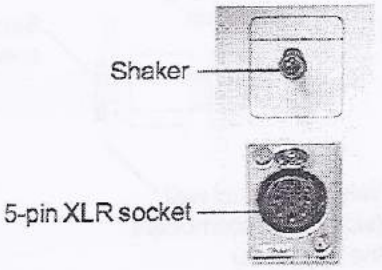
The spring tail (encircled in white) protrudes from the bottom of the front valve oven duct.

4. To install the 5030 purge head, line up the securing rod with the hole, and gently but firmly push it in until it locks into place. Connect the purge gas in and out lines to the fittings on the top of the front valve oven duct. Connect the dummy 5-pin XLR plug to the socket on the GC.

5. To install the 5035 purge head, hold the spring tail in a downward direction against the heater body as you slide the securing rod into the hole until it locks (the shaker will not work without the spring). Connect the purge gas in and out lines to the fittings on the top of the front valve oven duct. Connect the 5-pin XLR plug to the socket on the GC.

5035 Purge Head





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Expected Performance

The following two sets of chromatograms are from an Environmental GC system equipped with a Method 5030 compliant Purge & Trap, a PID detector, and a FID/DELCD combination detector. First, a 10ppb BTEX Plus sample was analyzed using the 5030 event table on the ***General Operating Procedures*** page and the **Epap&t.tem** temperature file. Second, a water blank was run through the system under identical conditions to show the component carry-over level of the Purge & Trap system. Toluene is used as a representative of the carryover in the Purge & Trap system; if the carryover level of Toluene is below 0.5ppb, then it will not affect subsequent analyses. NOTE: The TCE ghost peaks in the water blank chromatograms are augmented or caused by our factory test laboratory contamination.

Sample: 1µL 100ppm BTEX Plus dissolved in 10mL of water to yield 10ppb BTEX Plus

FID Results:

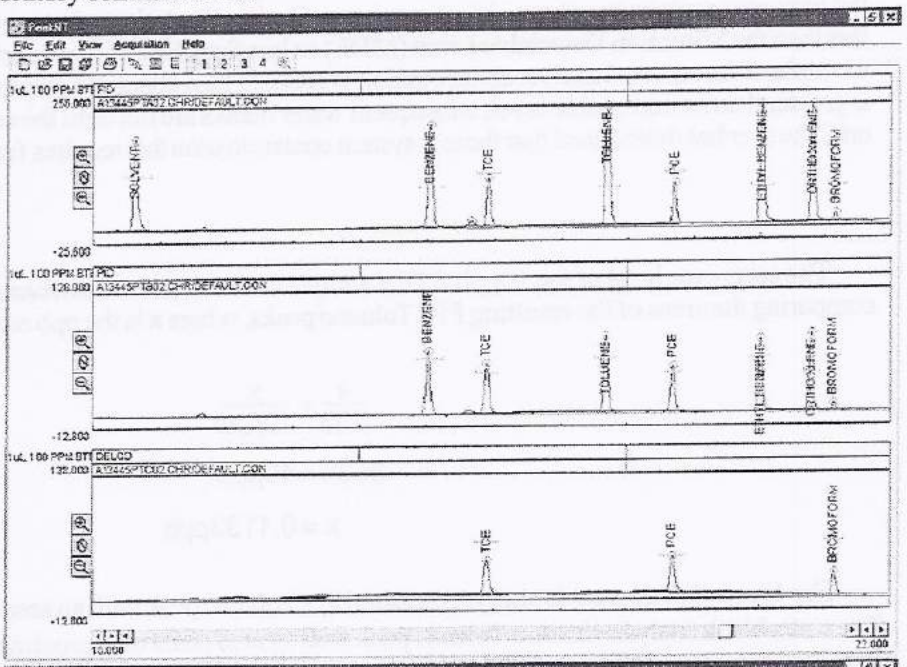
Component	Retention	Area
Solvent	10.616	921.0990
Benzene	15.033	1019.9260
TCE	15.883	441.8700
Toluene	17.683	1195.3320
PCE	18.700	383.3770
Ethyl Benzene	20.016	1247.3420
Ortho Xylene	20.800	1258.9260
Bromoform	21.166	78.9360
	Total	6546.8080

PID Results:

Component	Retention	Area
Benzene	15.016	311.1630
TCE	15.866	258.4360
Toluene	17.666	353.2160
PCE	18.683	233.4780
Ethyl Benzene	20.000	343.9640
Ortho Xylene	20.783	350.7040
Bromoform	21.133	32.3470
	Total	1883.3080

DELCD Results:

Component	Retention	Area
TCE	15.883	192.1020
PCE	18.683	209.2260
Bromoform	21.150	126.2820
	Total	527.6100



Sample: clean water blank (NOTE: the chromatograms are magnified for carryover visibility)

FID Results:

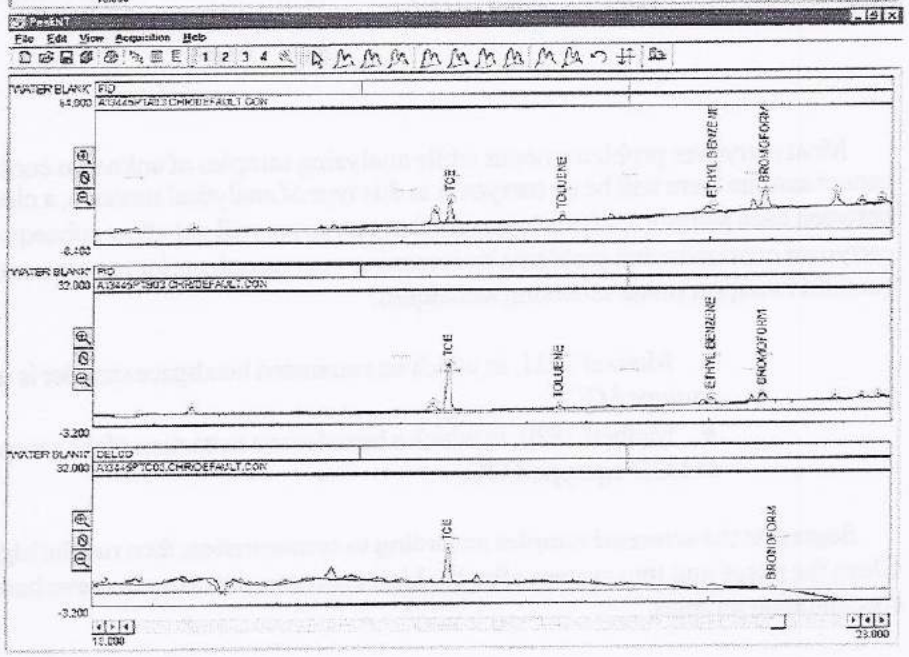
Component	Retention	Area
TCE	15.883	441.8700
Toluene	17.566	17.4000
Ethyl Benzene	20.016	1247.3420
Ortho Xylene	20.800	1258.9260
	Total	6546.8080

PID Results:

Component	Retention	Area
TCE	15.866	258.4360
Toluene	17.533	4.340
Ortho Xylene	20.783	350.7040
	Total	609.1300

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340



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Troubleshooting and Maintenance

Carryover

Carryover is a slight contamination of the purge and trap system by analytes (especially high boiling components), and is a normal condition of operation. All purge and trap systems exhibit some carryover. An organic free reagent water blank is analyzed after sample runs to determine the carryover level, as shown on the ***Expected performance*** page. Most regulatory Quality Control requirements allow carryover that is either less than the Minimum Detectable Limit (MDL) or less than 10% of the reported analyte concentration. For example, if the reported analyte concentration is 100ppb, then 10ppb is acceptable carryover. If the carryover is greater than an acceptable level, subsequent water blanks are run until the carryover is sufficiently low, or until the user has determined that there is system contamination that requires further cleaning.

The carryover level of the 10ppb BTEX sample on the ***Expected performance*** page was determined by comparing the areas of the resulting PID Toluene peaks, where **x** is the ppb concentration of the carryover:

$$\frac{4}{353} = \frac{x}{10ppb}$$

$$353x = 40ppb$$

$$x = 0.1133ppb$$

The 10ppb BTEX sample analysis resulted in a Toluene peak with an area count of approximately 353. The water blank analysis shows a Toluene peak with an area count of approximately 4. Since the carryover of Toluene is less than 10% or 0.5ppb, subsequent analyses may be resumed.

Most carryover problems occur while analyzing samples of unknown concentration. Because the user cannot assume there will be no carryover in this type of analytical situation, a clean water blank should be run between each sample analysis to ensure that carryover will not affect subsequent sample analyses. Avoid carryover contamination problems by screening your samples prior to purge and trap GC analysis. SW-846 contains two appropriate screening techniques:

- Method 5021, in which an automated headspace sampler is used with a PID and DELCD equipped GC
- Method 3820, in which a hexadecane extraction of the sample is analyzed by a FID and/or ECD equipped GC.

Segregate the screened samples according to concentration, then run the highly concentrated ones first. Clean the purge and trap system after the high concentration samples have been run, then analyze the low concentration samples.

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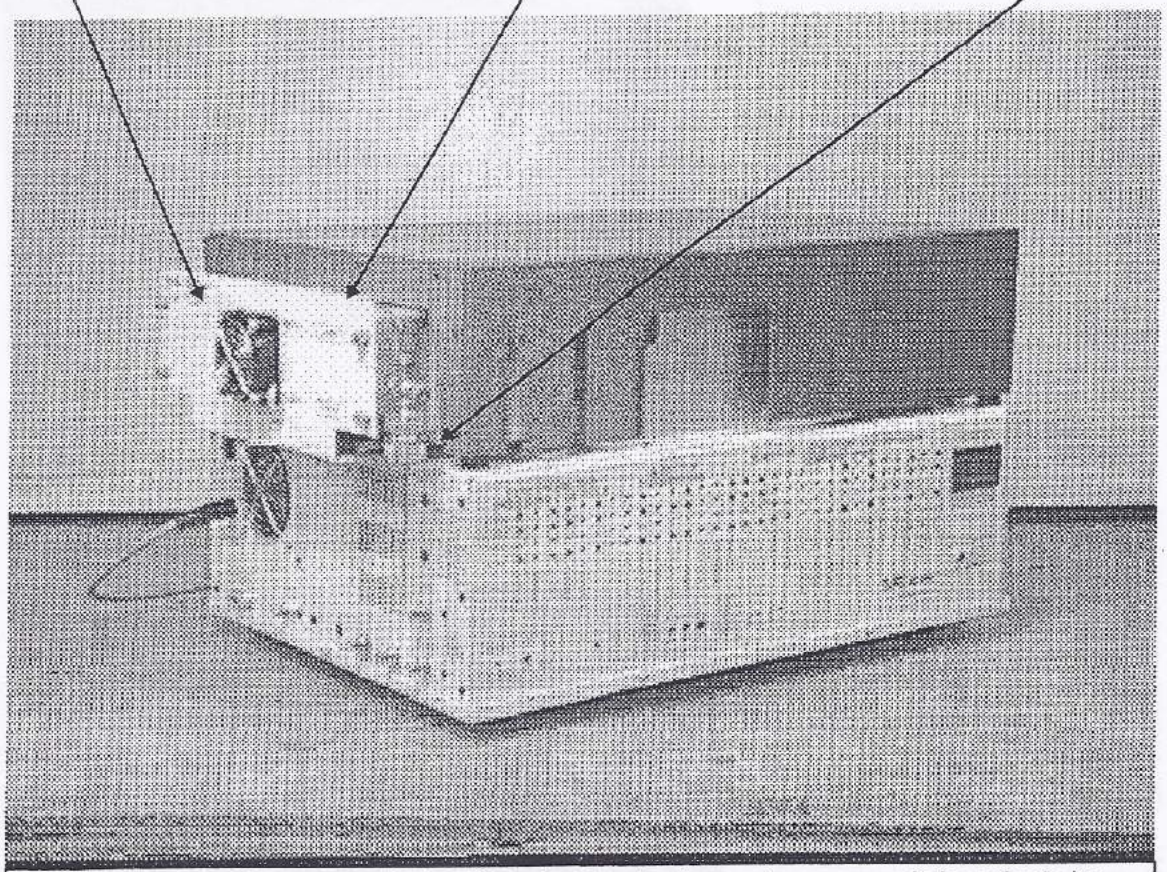
Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

Cooling fan for traps maintains selected adsorbtion temperature and rapidly lowers trap temperature from desorbtion temperature (typically 200 C)

Purge and Trap Sample Concentrator Option is mounted in the special ducted heated valve oven located on the left side of the 8610C Gas Chromatograph. The Purge and Trap option is not available on the Model 310 GC.

Purge vessel uses disposable 16mm test tubes and rugged needle sparging tube. Sparge head allows gas standards to be spiked in through built-in septum port.



The SRI Purge and Trap concentrator allows low levels of organic compounds in water to be automatically extracted from the water matrix and collected on one or two series mounted adsorbent traps. The purge and trap technique is applicable to a broad range of molecules from about C3 to C12. Molecules heavier than C12 do not purge well from water nor do polar molecules which resist purging due to their solubility. The SRI Purge and Trap is unique because of the dual trap design which allows two different adsorbent trapping materials to be used, and each material can be adsorbed and desorbed at individual temperatures and different times. This flexibility allows for tighter desorbtion bandwidths, and greater water rejection than other Purge and trap designs which have only a single mixed adsorbent bed trap. Additionally, the disposable test tubes which hold the water sample are inexpensive (5 cents each U.S) so they can be thrown away in the event of contamination. A 10 position autosampler can be easily added to the Purge and trap for un-attended operation.

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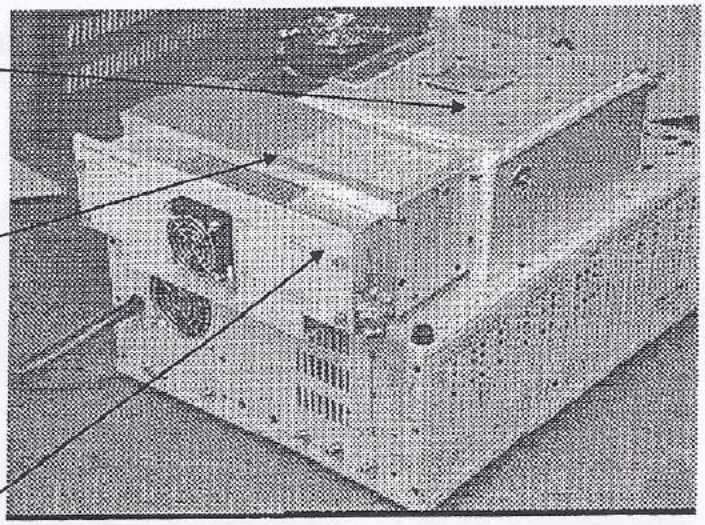
Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

Column oven

The Purge and Trap option is mounted on the GC chassis in a special ducted heated valve oven just to the left of the column oven

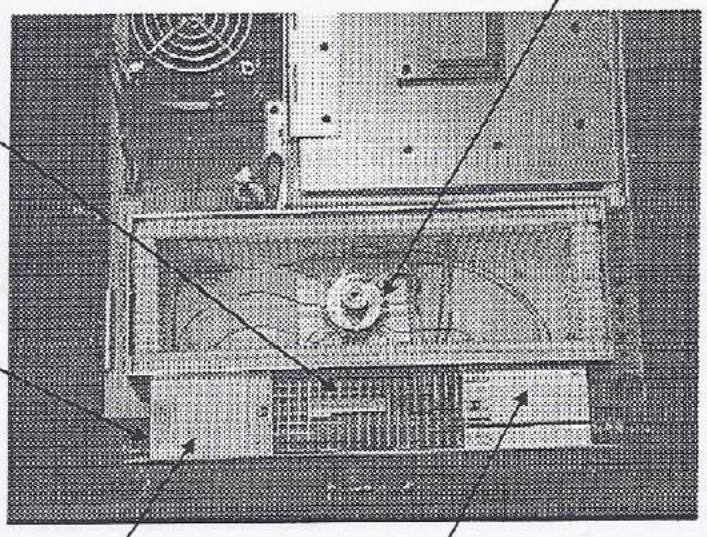
To change traps this cover plate must be removed by loosening the four brass thumbscrews located at the corners.



10 port electrically operated Valco valve mounted in the heated valve oven is the heart of the purge and trap hardware. The valve oven is typically set to 150 degrees C so water will not condense.

Traps are located between ducts so that the ends of the traps are enclosed within the heated duct area while the body of the traps are suspended in the trap heaters between the ducts. A protective grill keeps fingers and tools out of the trap heat zone while allowing hot air to escape.

A vent tube is located at the back of the P&T valve oven. The sparge gas exits from this vent tube after passing through the traps.



Ducts enclose ends of traps within heated valve oven to prevent cold spots.

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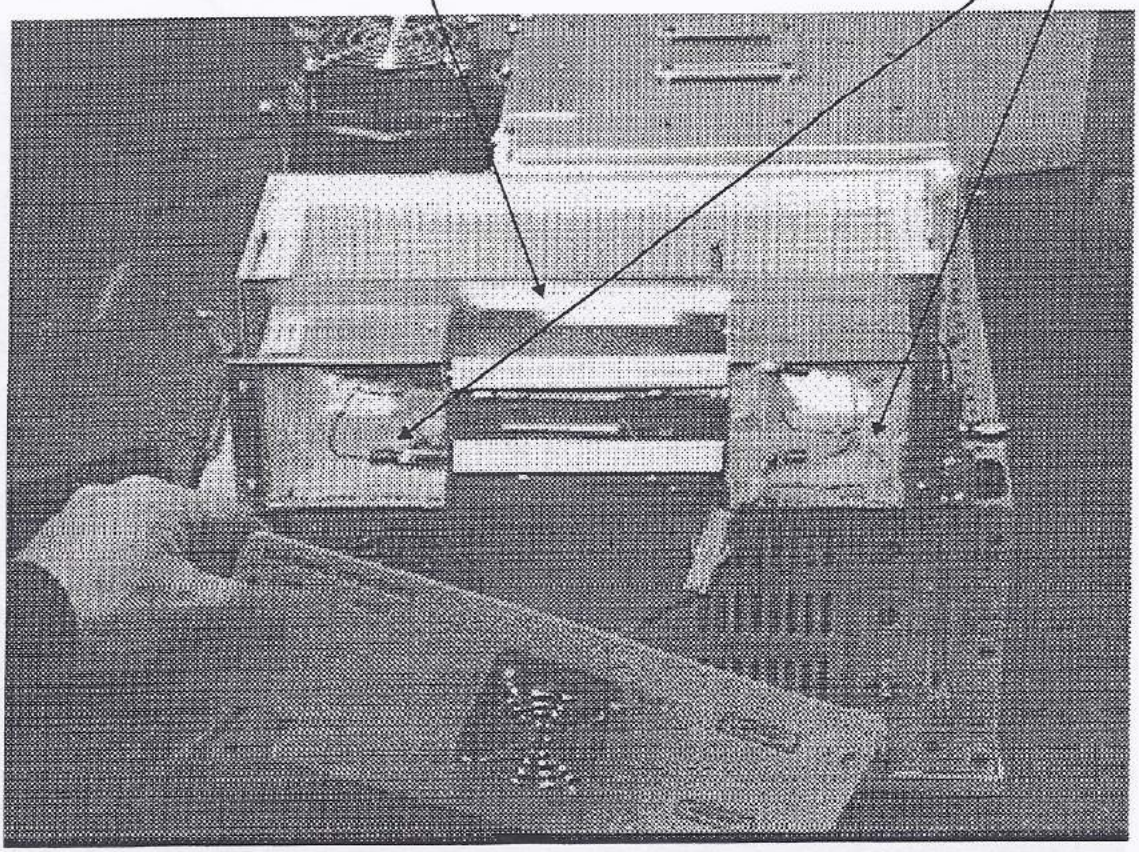
Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

Remove the protective wire grill from the top of the valve oven for better access to the traps

Remove the two squares of white insulation from each duct to expose the fittings which secure the trap ends to the 1/16th inch O.D. tubing leading to the Valco valve.



To access the traps for maintenence or replacement:

1) Remove the left side plate from the purge and trap valve oven by loosening the 4 brass thumbscrews at the corners.
2) Remove the protective grill from the top of the valve oven by loosening the two screws.
3) Carefully remove the two squares of white insulation from the ducts at the ends of the traps.

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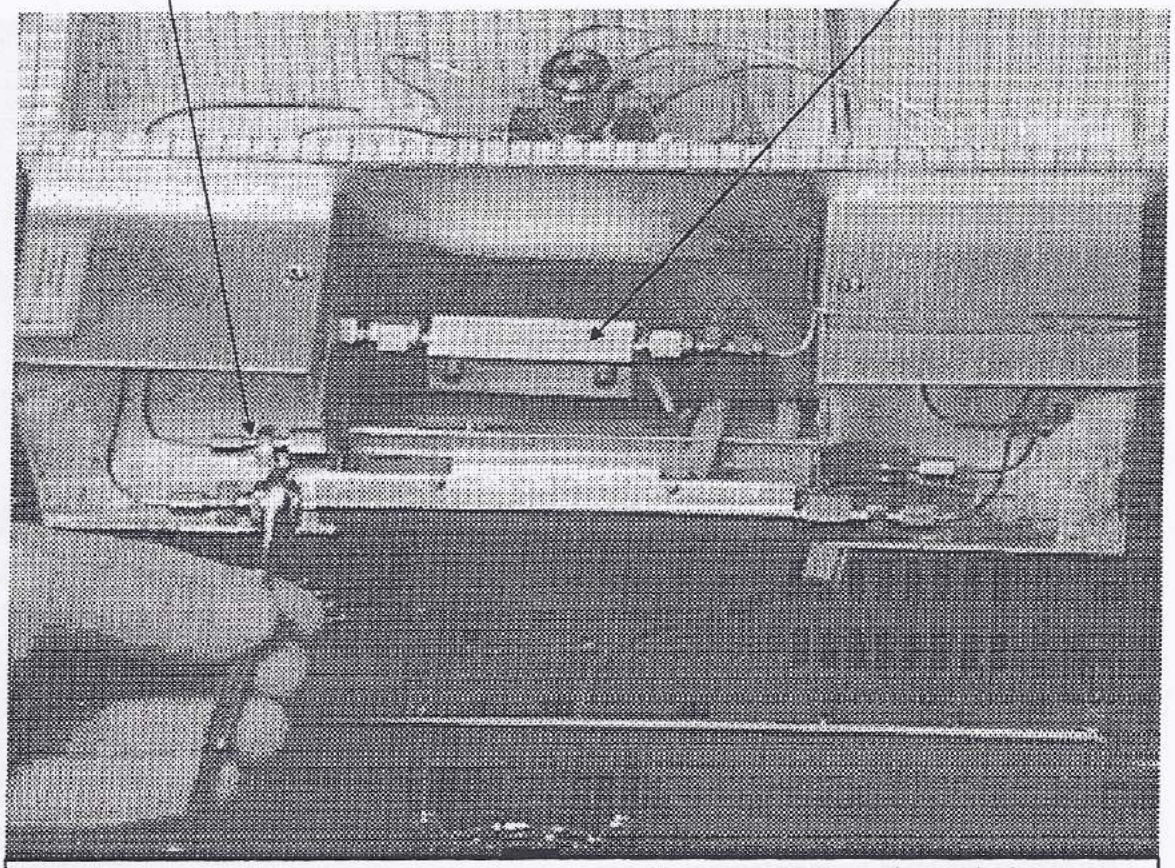
Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

1/8th to 1/16th reducing fitting at end of trap

Clamshell type trap heater halves can be separated by loosening two hex head type screws.



There is enough slack in the heater/thermo-couple wires to pull the trap about 1 inch beyond the duct . To remove the trap :

1) Loosen the 1/8th inch swagelok type nuts which secure the trap ends to stainless steel 1/8 to 1/16th reducing unions using two wrenches.
2) The trap heater is a clamshell design which will separate when the two hex head screws holding the heater together are loosened. With the trap heater apart the trap itself can be removed.

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Chapter: PURGE-AND-TRAP

Topic: Troubleshooting Purge and Trap Contamination

Use this troubleshooting flowchart if you are experiencing difficulty with contamination when performing purge and trap analyses.

Is the 10-port gas sampling valve oven temperature set and operating above 150°C?

NO → Set valve oven temperature above 150°C and perform 2 blank runs. Does the contamination persist?

NO → Contamination problem has been resolved.

YES → Determine the concentration of the contamination. Compare its area to the area obtained from a 10 ppb standard analysis. Is the concentration of contamination *significant* (>1/20 of 10 ppb [0.5 ppb])?

NO → The concentration is below 0.5 ppb (1/20 of the area obtained from a 10 ppb standard). This level of background is acceptable and insignificant and will not adversely affect your analysis.

YES → Turn off the sparge gas pressure (set to 0 psi). After performing two blank analyses, are the contamination peaks still a problem?

YES → **Proceed to page 2 of the diagnostic flowchart.**

NO → Turn the sparge gas pressure back to normal setting (4-5 psi). Bake out the polishing filter. Then run 2 clean water blanks. Is contamination still a problem?

NO → The contamination, now reduced to an acceptable level, appears to have been accumulated on the polishing filter. The polishing filter should be baked-out periodically as part of routine preventitive maintenance.

YES → Clean the sparge head, backflow trap and all gas lines in between. Then make two blank runs. Is the contamination still a problem?

NO → The contamination, now *insignificant*, was located in the area of the sparge/ backflow heads and/or related tubing.

YES → Using a heat gun, heat the entire sparge head assembly with the sparge head's injection port nut removed while sparge gas is flowing. After performing a blank analysis, is the contamination still a problem?

NO → Localized heat cleaning resolved the problem.

YES → Replace the entire sparging apparatus with a section of clean tubing. After performing two more blank runs, is the contamination still a problem?

NO → Repeat the cleaning of the complete sparging apparatus, including the water trap. This should eliminate the problem.

YES → Contact SRI Instruments for technical assistance in diagnosing this condition.

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From page 1

Use this troubleshooting flowchart if you are experiencing difficulty with contamination when performing purge and trap analyses. Begin on page 1.

Remove trap heating events (relays C and F on) from event table. Perform two more blank runs. Is the concentration of contamination still a problem?

YES

Replace the graphite ferrules securing the traps and the column. Clean the fittings. Look for graphite shavings in trap and column ends. Remove shaving if found. Then perform a blank analysis. Are the contamination peaks still a problem?

YES

Remove* and clean the gas sampling valve rotor. Clean the rotor seat. Reinstall the rotor. Is contamination still a problem?

YES

Replace the stainless steel tubing around the gas sampling valve. Is the contamination still a problem?

YES

Call SRI for tech. support

NO

Dirty tubing. New tubing was the fix.

NO

The sampling valve rotor and / or rotor seat appear to have been contaminated. Cleaning the area eliminated the contamination problem.

NO

Graphite is adsorbent. It is possible that the ferrules could adsorb analyte if exposed to a high concentration. New ferrules eliminated the high contamination level.

NO

With a clean test tube, sparge and carrier gas flowing and the gas sampling valve in the INJECT position, bake out the traps for 15 minutes. Activate relays C and F with trap setpoints of 300° C. In the INJECT position, the traps will exhaust to the column (raise the column temperature). Perform 2 blanks. Is the contamination still a problem?

YES

Set the trap temperature to 250° C. Is the contamination still a problem?

YES

Replace the traps with blank tubes. Perform 2 more blank runs. Is the contamination still a problem?

YES

Remove* and clean the sampling valve rotor. Clean the rotor seat. Perform 2 blank runs. Is contamination still a problem?

YES

Replace all 1/16" stainless steel purge and trap tubing around the gas sampling valve. Is contamination still a problem?

YES

Call SRI for further assistance.

NO

Dirty tubing. New tubing was the fix.

NO

It appears that the traps that were in use are defective and retaining contaminants. Replace the traps to eliminate the problem.

NO

The sampling valve rotor and / or seat was contaminated. Cleaning the rotor area surfaces eliminated the problem. Further cleaning may be necessary as contamination works through the valve body.

NO

The traps were retaining analyte. Spent traps may tend to retain analyte. Replacement may be indicated.

NO

Graphite is adsorbent. It is possible that the ferrules could adsorb analyte if exposed to a high concentration. New ferrules eliminated the high contamination level.

* prior to removing the rotor, note the position of a letter stamped on one end of the metal fin protruding from the top. This letter indicates the rotor temperature rating, but also assists in orienting the fin properly during reinstallation of the rotor after cleaning. Much like a automotive distributor, the rotor can be reinserted 180° wrong, resulting in valve failure.

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Anal. Chem. 1996, 68, 3431−3433

Effectiveness of Purge-and-Trap for Measurement of Volatile Organic Compounds in Aged Soils

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The U.S. EPA-recommended method for measurement of trace levels of volatile organic compounds (VOCs) in soil, purge-and-trap, measures the readily desorbable organic contaminants from soil pore spaces and external soil surfaces. It does not, however, measure contamination that has diffused into internal micropores of soil matrix. Thus, the purge-and-trap method measures only a small fraction of total soil contaminants, especially in long-contaminated soils, where ~90−99% of contamination may be in the interior of the soil matrix. We compared three methods for determination of VOCs in aged field samples: purge-and-trap, methanol immersion, and hot solvent extraction. Hot solvent extraction proved to be much more effective than the U.S. EPA-approved purge-and-trap technique. For three long-contaminated soils containing such VOCs as trichloroethene, benzene, toluene, chloroform, methylene chloride, and *cis*-1,1-dichloroethylene, recovery from purge-and-trap ranged between 1.5 and 41.3% that of hot solvent extraction. Our data show that purge-and-trap may not be the best methodology for measuring soil VOCs concentrations, particularly in aged soils. It is clear from this and previous studies that the best overall choice for soil VOCs measurements is hot solvent extraction. These results also indicate the inefficiency of purge-and-trap as a method for evaluating vapor extraction remediation technology. Our results suggest that the EPA should review the use of the purge-and-trap method for measuring VOCs concentrations in soils.

A critical requirement in the cleanup of contaminated soil sites is an accurate determination of the nature and extent of soil contamination. The primary U.S. EPA-recommended method (EPA/SW-846-5030A and 8260A) for measurement of volatile organic compounds (VOCs) in soils is purge-and-trap,[1,24,25] followed by gas chromatography/mass spectroscopy. Under this protocol, organic-free water containing internal standards and surrogates is mixed with a soil sample and heated to 40 °C. An inert gas is bubbled through the solution at ambient temperature, and the vapor is passed through a sorbent column, where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.[1] Use of purge-and-trap to measure VOCs in soil is based on the assumption that soil VOCs rapidly equilibrate with soil water. Recent studies,[2−5] however, strongly question this assumption and indicate that soil desorption is a biphasic process with an initial rapid surface desorption followed by a much slower, diffusion-limited, desorptive phase from the interior of the soil matrix.[2−4,6−11]

The biphasic nature of desorption casts doubt on the widely used, EPA-recommended purge-and-trap method. When soil has been in contact with VOCs for a long time period (aged soils), VOCs diffuse into soil micropores, where they are unavailable for purge-and-trap measurement. Except for a single study[8] involving 1,2-dibromoethane (EDB), the impact of soil aging on the effectiveness of VOCs measurement techniques is largely unexplored. The present study focuses on the effectiveness of three commonly used techniques for measuring VOCs concentrations in aged soils: purge-and-trap, methanol immersion, and hot methanol extraction. Since vapor extraction is a currently popular technique for removal of VOCs from soils, we subjected one soil sample to air stripping to evaluate the effect of vapor extraction on the extraction efficiency of purge-and-trap measurements.

EXPERIMENTAL PROCEDURE

Soils. Soil samples were obtained from three geographically distributed sites with a 10−20-year history of VOCs contamination. The Kentucky soil had high clay content, with 100 ppb of trichloroethylene. The Louisiana soil was a silty loam, with 3000 ppb of *cis*-1,1-dichloroethylene and 6000 ppb of trichloroethylene. The Florida soil was silty, fine to very fine sand, containing methylene chloride at 240 ppb, benzene at 2 ppb, toluene at 190 ppb, and chloroform at 2 ppb.

Sample Collection. Soil samples were extracted with a hollow-stem auger and split-spoon sampler. Undisturbed soil

[†] University of Tennessee.
[‡] Chemical and Analytical Sciences Division, ORNL.
[§] U.S. Department of Energy.
[⊥] Health Sciences Research Division, ORNL.

(1) Environmental Protection Agency. *Code of Federal Regulations*, Part 261; Title 40; U.S. Government Printing Office: Washington, DC, 1994; Chapter 1, App. III, p 67 (July 1, 1994).
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Table 1. Comparison of Three Methods for measuring VOCs in Soils

soil type, component	compounds	purge-and-trap[a] (mg/kg)	methanol immersion[a] (mg/kg)	hot methanol extraction[a] (mg/kg)
Kentucky (clay)	trichloroethylene	100 ± 57	140 ± 15	240 ± 31[b]
Louisiana (silty loam)	*cis*-1,1-dichloroethylene	3070 ± 351	26 000 ± 4359[b]	41 700 ± 2082[b]
	trichloroethylene	5900 ± 1210	85 000 ± 13 115[b]	121 700 ± 11 719[b]
Florida (silty, fine to very fine sand)	methylene chloride	240 ± 63	530 ± 31[b]	630 ± 58[b]
	benzene	2 ± 1	110 ± 5[b]	150 ± 25[b]
	toluene	190 ± 41	240 ± 11[c]	270 ± 37[b]
	chloroform	2 ± 0	110 ± 5[b]	130 ± 25[b]

[a] Average of three soil sample measurements. [b] Significantly higher mean than purge-and-trap at 95% level of confidence. [c] Significantly higher mean than purge-and-trap at 90% level of confidence.

Table 2. Comparison of Extraction Methods for Louisiana Soil after 1 Week of Air Stripping

compound	purge-and-trap[a] (mg/kg)	methanol immersion[a] (mg/kg)	hot methanol extraction[a] (mg/kg)
methylene chloride	94 ± 50	150 ± 43	160 ± 110
cis-1,1-dichloroethylene	86 ± 39	390 ± 122[b]	2110 ± 1688[c]
trichloroethylene	310 ± 60	5400 ± 916[b]	5500 ± 1081[b]

[a] Average of three soil sample measurements. [b] Significantly higher mean than purge-and-trap at 95% level of confidence. [c] Significantly higher mean than purge-and-trap at 90% level of confidence.

cores were sealed in glass jars with minimum headspace and stored at 4 °C on arrival. At the start of each experiment, the sample core was plugged and subdivided to obtain three 5-g subsamples. Subsamples were extruded directly into VOA vials (Dynatech, Baton Rouge, LA) and mixed with 5 mL of water containing internal standards and surrogates before capping.

Measurements of VOCs. Contaminants were extracted from soil samples by methods of purge-and-trap, methanol immersion, and hot methanol extraction. Contaminant concentrations were expressed as micrograms per kilogram of soil.

Purge-and-Trap. Sample aliquots were purged using a Dynatech PTA-30 autosampler and a Tekmar (Cincinnati, OH) LSC-2 purge-and-trap device. Soil samples of 2 g each were purged at 40 °C. The trap was desorbed, and measurement was performed with a Hewlett-Packard Model 5890/5971 GC/MS, using and EPA method 8260.[12,13] All quality assurance measures given in the method were followed.

Methanol Immersion. Five milliliters of purge-and-trap grade methanol was added to sample aliquots. The vial was capped as previously described and vigorously shaken for 30 s to facilitate wetting of the soil surface. A 50-μL aliquot of the methanol was then removed and added to 5 mL of water containing internal standards and surrogates. The water was then subjected to purge-and-trap and analyzed for the concentration of chemicals by GC.

Hot Methanol Extraction. Hot methanol extraction was performed in the same manner as methanol immersion except that, prior to withdrawal of the aliquot of methanol, the VOA vial was placed in a 40 °C ultrasonic water bath for 30 min. The methanol was drawn and analyzed as described above.

Extraction Following Air Stripping. The Louisiana sample was mixed in a 7:3 ratio with calcium oxide to prevent solidification during air stripping. The sample was then placed in a Buchner funnel with a vacuum running from the bottom of the funnel through a flask. Ambient air was passed through the sample using a vacuum of 450−675 mmHg for 1 week. Occasional mixing of the soil was performed throughout this time. The soil sample was divided into three subsamples, and each subsample was then subjected to one of the three methods as previously described.

All of the above extractions was performed as written in EPA/SW-846 methodology. Internal standards were added, and surrogate recovery was within the limits of the method for all extractions. All quality assurance procedures were applied.

RESULTS

Measurement results from the three different sites—Kentucky, Louisiana, and Florida—are presented in Table 1. Trichloroethylene was the only contaminant detected in the Kentucky soil (Table 1), with a purge-and-trap recovery only 42% as compared to hot methanol extraction. Both *cis*-1,1-dichloroethylene and trichloroethene were detected in the Louisiana soil. Purge-and-trap recovery of these two contaminants was only 7.4 and 4.8%, respectively, when compared to hot methanol extraction method. Four compounds were identified in the Florida soil: methylene chloride, benzene, toluene, and chloroform, with purge-and-trap recovery 38.0, 1.5, 71.2, and 1.5%, respectively, in comparison to hot methanol extraction.

Effect of Air Stripping. Contaminant concentrations for air-stripped Louisiana soil are presented in Table 2. Purge-and-trap detected levels of methylene chloride, *cis*-1,1-dichloroethylene, and TCE at 58, 4.1, and 5.6%, respectively, that of hot methanol extraction. Comparison of these results with those in Table 1 for Louisiana soils indicates that air stripping does not appear to affect the distribution of contaminants between the accessible and inaccessible phases.

DISCUSSION

Purge-and-trap is the EPA-recommended method (EPA/SW-846-5030A) for measurement of VOCs in soils. Under the

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protocol, organic-free water is mixed with a soil sample and heated to 40 °C. An inert gas is bubbled through the water, and the concentration of chemicals in the gas is measured with a gas chromatograph. This method is effective only if VOCs in soils rapidly desorb from the soil surface into surrounding water. Laboratory control samples using sea sand in place of soils indicate that, for nonadsorptive solids, purge-and-trap recovery is acceptable. Recent studies[2-4,8,14,15] on the physical inaccessibility of contamination in soils suggest that this may not be the case, particularly in long-contaminated soils. It has been hypothesized that aging involves diffusion into soil micropores, partitioning into soil organic matter, strong surface adsorption, or a combination of these processes.[2,14,16,17] Previous studies[6,18] indicate that 20–90% of contamination may be located in the interior of the soil matrix and thus inaccessible for purge-and-trap measurement. The purpose of the present study was to compare the effectiveness of three commonly used techniques for measuring VOCs in soils: purge-and-trap, methanol immersion, and hot methanol extraction. We found that purge-and-trap consistently underestimated the concentration of VOCs in aged soils by factors ranging from 2 to ~100. This consistent underestimation of soil concentrations undermines the EPA's attempt to remediate contaminated soils to levels consistent with health-based cleanup standards.

The soil desorption process is known to involve two distinct phases: a rapid desorption from the soil surface occurring within 24 h and a much slower diffusion-limited desorption from the interior of the soil matrix occurring over a period of days to years.[5,19] This biphasic desorption pattern is most pronounced in aged soils, where a significant fraction of contamination is located in the interior of the soil matrix. For example, despite its high volatility and degradability, 1,2-dibromoethane (EDB), a soil fumigant, was found[8] in agricultural topsoil 19 years after its last known application. The persistence of EDB was attributed to desorption half-times of 2–3 decades at 25 °C.[2] For trichloroethylene (TCE), a continuous desorption study[10] of long-contaminated soils revealed persistence of 18% of the initial TCE concentration after desorption with 24 000 pore volumes of water. In a subsequent study[3] on simultaneous desorption of TCE, tetrachloroethylene, toluene, and xylene, a substantial portion (48–94%) of the sorbed contaminant mass resisted desorption after 7 days of contact time. TCE soil concentrations at the Picatinny Arsenal were found to be 1–3 orders of magnitude greater than predicted using soil–gas concentrations and equilibrium conditions.[18] The present study found that the purge-and-trap method, as compared to hot solvent extraction, recovered only 42 and 4.8%, respectively, of TCE in long-contaminated clays and silty loam soils.

Even in freshly spiked soils, desorption rates of pollutants can be 1–3 orders of magnitude smaller than equilibrium-predicted rates.[4] Clean soils spiked with halogenated aliphatic hydrocarbons for 24–72 h resisted desorption after 16 extractions of 24–72 h each.[6] These observations bring into question the occurrence of desorption equilibrium necessary for validity of the purge-and-trap measurements in the freshly spiked and aged soils.

Previous analysis of EDB in long-contaminated soils has shown that purge-and-trap is less effective than extraction at 75 °C with organic solvents such as methanol, acetonitrile, and acetone,[2,6,8] recovering less than 11% of the total EDB found by hot solvent extraction.[8] Our purpose in the present study was to extend these results to a larger class of VOCs in aged field samples. Hot methanol extraction proved to be more effective than the EPA-approved purge-and-trap technique. For three long-contaminated soils containing such VOCs as trichloroethylene, benzene, toluene, chloroform, methylene chloride, and *cis*-1,1-dichloroethylene, recovery from purge-and-trap ranged from 1.5 up to 41% that of hot methanol extraction.

Slow desorption is recognized as a serious obstacle to soil remediation technologies.[11,14,20-23] For such technologies as pump-and-treat, vapor extraction, and bioremediation to be effective, soil contaminants must be accessible. To simulate the effect of vapor extraction on the efficiency of the purge-and-trap methodology, we subjected the Louisiana soil to a week of air stripping. Purge-and-trap recovered only 58% of the methylene chloride, 4.1% of the *cis*-1,1-dichloroethylene, and 5.6% of the TCE that hot methanol extraction was able to recover. These results indicate that the purge-and-trap method is not a reliable method for evaluating vapor extraction as a remediation technology.

It is clear from the results of this and previous studies that the best overall choice for measurement of soil VOCs is hot methanol extraction, since this method yields a more accurate analysis, regardless of the age of contaminated soil. The VOC data from three different soil types clearly demonstrate the limitations of the EPA-approved purge-and-trap method, which can bias analytical results by several orders of magnitude, depending on soil type and chemical properties. We suggest that the EPA review the use of purge-and-trap as a method for measuring VOCs in soils.

ACKNOWLEDGMENT

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[⊗] Abstract published in *Advance ACS Abstracts,* August 1, 1996.

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GC ACCESSORIES
10 Position Method 5030 Purge & Trap Autosampler Retrofit

The purge & trap autosampler retrofit kit contains everything you need to add a 10 Position Method 5030 Purge & Trap Autosampler to your existing SRI purge & trap equipped GC. The kit includes a new purge & trap cover plate (A), a transfer line to valve connection(B), and a purge gas line(C).

A. Purge & trap cover plate:

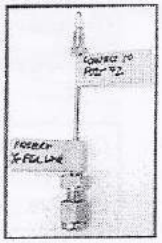
1. Remove the existing purge & trap cover plate from the GC by unscrewing the four thumbscrews that hold it in place.

2. Remove the fan from the existing purge & trap cover by unscrewing the four securing philips head screws and unplugging its white plastic 2-wire connector. Transfer the fan and its four screws to the new cover.

3. Attach the new purge & trap cover to the GC with the four screws from the old cover and re-connect the fan power.

4. Feed the autosampler's red heat transfer line through its cover plate hole, then secure it with the hose clamp included with the autosampler.

B. Transfer line to valve connection:



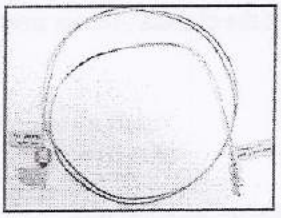
1. Remove the valve oven lid by unscrewing the brass thumbscrew on the front edge of the lid, tilting the front of the lid up, then sliding back slightly to free it from the screw in the back. Carefully remove the white insulation padding, and set it securely aside with the oven lid.

2. Connect the Valco fitting to PORT #2 of the gas sampling valve (the Valco fitting is labeled "CONNECT TO PORT #2").

3. Feed the transfer line completely through the Swagelok fitting and 1/16" tubing (this fitting is labeled "INSERT X-FER LINE").

4. Use a wrench to securely tighten the fitting until it is snug against the graphite ferrule; do not over-tighten.

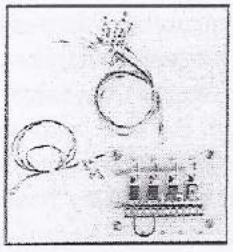
C. Purge gas line:



1. Remove the glass test tube from the purge & trap.

2. Connect the brass union on the purge gas line to the purge gas tube (formerly inside the glass test tube). The durable Teflon™ ferrule in the brass union allows this operation to be performed many times.

3. Connect the 1/8" nut on the purge gas line to "PURGE IN" on the autosampler.



See the SRI manual for relay board installation instructions: "AUTOSAMPLERS; Installation of the Relay Board for the 10 Position Method 5030 Purge & Trap Autosampler (and other Autosamplers)"

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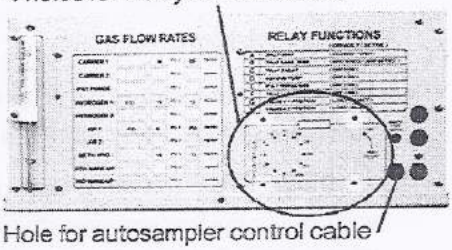
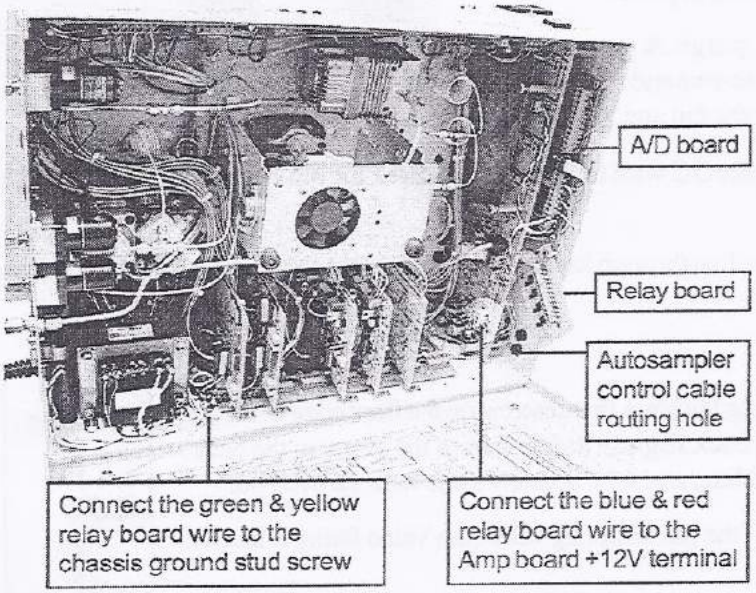
Installation of the Relay Board for the 10 Position Method 5030 Purge & Trap Autosampler (and other Autosamplers)

A relay board is provided with the autosampler for connecting it to an SRI 8610 GC. This relay board supplies the additional relays required to operate the autosampler, and must be installed inside the GC by the user. The relay board comes with the necessary wiring, and no soldering is required.

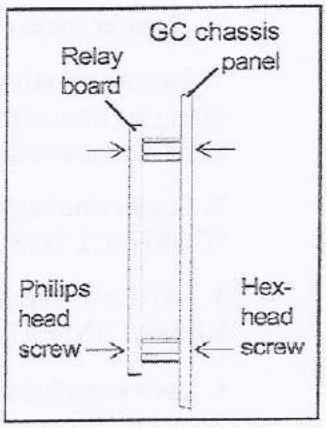
The four holes in the right side panel of the GC chassis, under the "Relay Functions" table, correspond with the relay board securing screws. The relay board is installed on the inside of this panel.



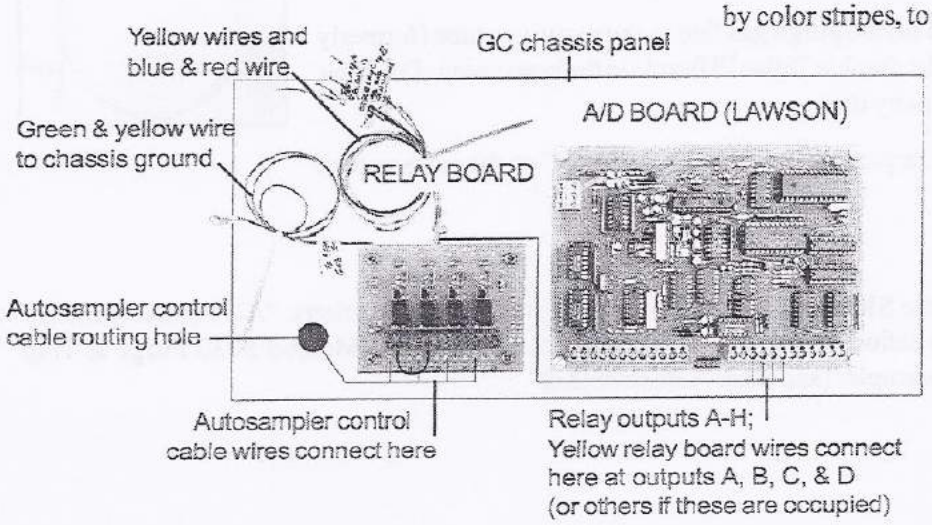


1. Remove the six screws holding the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its back, then lower the panel to your working surface to access the chassis interior.

2. Secure the four aluminum stand-offs in the relay board holes. Use the four hex-head screws provided, and secure the stand-offs from the outside of the GC panel. Insert the relay board into position so that the component side faces outward. Secure it in place on the aluminum stand-offs with the four philips head screws provided.



3. Connect the green and yellow wire to the chassis ground stud screw on the left rear of the chassis interior near the main power transformer.



4. Connect each of the four yellow wires, differentiated by color stripes, to the appropriate TTL relay outputs on the A/D (Lawson) board. All eight of the TTL outputs are identical; use any available ones. Connect the blue and red wire to the Amp board 12V terminal.

5. Remove one of the plastic hole plugs for the autosampler control cable. Route the control cable through the hole, and connect each wire to the appropriate relay circuit on the relay board (see autosampler control cable labeling).

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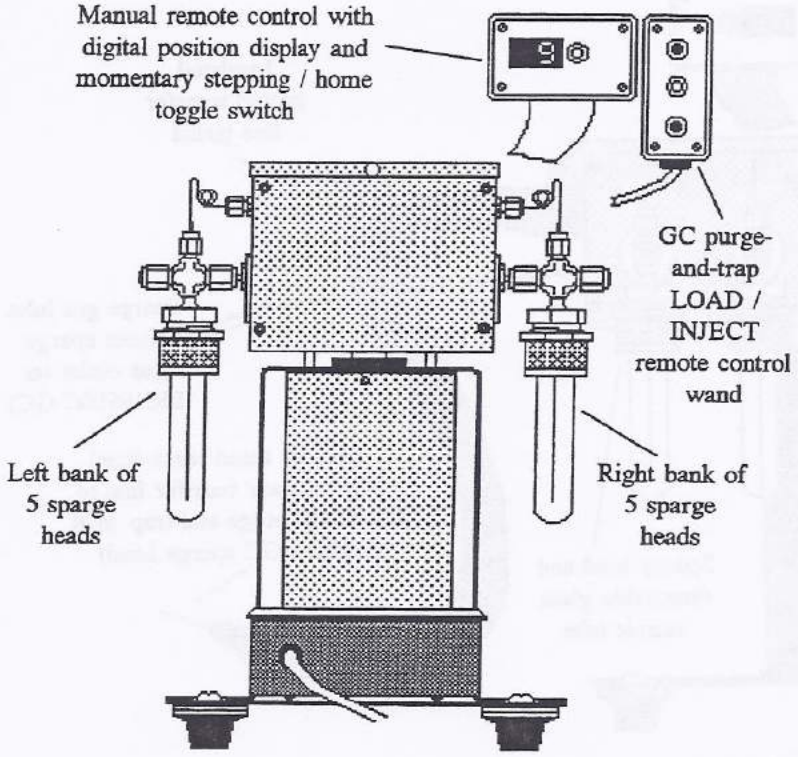
Chapter: PURGE-AND-TRAP

Topic: 10 Station Purge-and-Trap Autosampler

The SRI 10 Station Purge-and-Trap Autosampler permits the unattended sparge, concentration, and analysis of up to 10 separate water and / or soil-in-water samples, when used in conjunction with the SRI EPA-Style Automated Purge-and-Trap Sample Concentrator option available as a built-in option for all SRI 8610C gas chromatographs. The PeakSimple data system (or other data system offering timed event control of external events via relays) is required for automated operation of this system. Manual controls are also provided for direct, manual control by the analyst, including a remote cabled sample position stepping control that features a digital LED display of the sample position in use. The toggle switch on this control has two momentary-on positions. Pushing the toggle switch to the up momentary-on position causes the automated sample stream selection valve to step to the next sample in order. Pushing the toggle switch down causes the sampling valve to return to the home (sample sparge head 1) position. The sample sparge heads are numbered according to their sampling order. A stainless steel knurled fitting holds the disposable glass sample tubes in place. Teflon ferrules in the knurled fitting seal the sample tubes in place, preventing gas leaks.



10 STATION PURGE-AND-TRAP AUTOSAMPLER SIDE VIEW

Only two gas connections are needed to supply the autosampler with sparging gas, and to deliver the sample-laden sparge gas to the purge-and-trap sampler's dual trap concentration system. These two gas lines replace the sparge head assembly on the EPA-style purge-and-trap system. A remote control signal cable connects the autosampler valve control electronics to the data system external event control circuitry. This simple cable requires only three connections to the data system event control relays, common, step, and home. The data system must provide a momentary closure between the common wire and one of the two action wires to move the stream selection valve to the desired position.

Any of the 10 sample vessels may also be used to contain a clean water blank (or air) for use between analyses for blank runs. The stream selection valve must be stepped to this blank position, and then to the desired sample position for blank operation. A large volume headspace sample may also be introduced into the system for concentration onto the dual adsorbent traps, as each sparge head is equipped with a gas injection port for manual syringe injections. In this manner, a 50cc, 100cc, or larger volume headspace sample can be passed through the traps, in order to acheive low sample detection levels unattainable by regular headspace injection on-column using standard microliter to milliliter volumes. The sparge gas supply should be turned on to assist the injected sample to flow through the traps when this feature is used.

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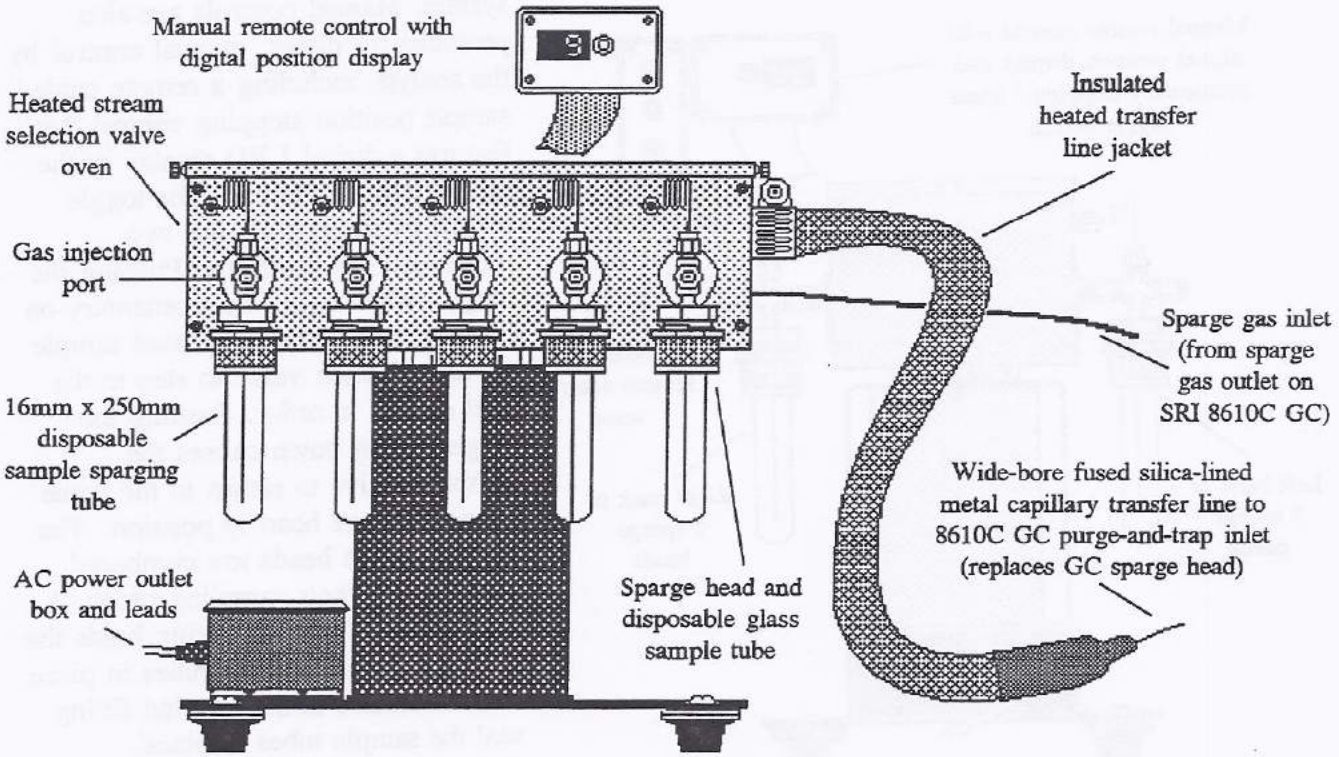
As illustrated below, the SRI 10 Station Purge-and-Trap Autosampler is configured in a symmetrical, space-saving bilateral design. Located along each side of the unit are 5 sparge heads with respective headspace injection ports. The autosampler should be located to the left side of the SRI 8610C GC for ease of operation and access to the sample tubes. The gas transfer lines provided with the autosampler permit separation between the autosampler and GC of up to 24 inches. This



SRI 10 STATION PURGE-AND-TRAP AUTOSAMPLER FRONT VIEW

allows the analyst to have access to each sample sparging head and injection port, and to move the autosampler around on the lab bench as needed, while maintaining all connections and operability. As the autosampler is a stand-alone unit, it does not require a hard attachment or bracket for GC mounting. A 3' length of 1/16" stainless steel tubing carries the sparge gas from the GC's EPA-Style Purge-and-Trap Sampler sparge gas outlet (previously supplying gas to the single sparge head), and an insulated, electrically-heated capillary transfer line returns the sample-laden gas to the purge-and-trap system's dual adsorbent traps via the GC-mounted purge-and-trap plumbing and sampling valve hardware. The glass sample tubes are low-cost disposable 16mm x 250mm (20cc) straight-mouthed test tubes available in bulk packs from SRI or any laboratory supplier. The sparge head assemblies are stainless steel hardware that is heated by the valve oven that they are mounted to, eliminating cold metal condensation of sparged analytes. The bilateral configuration of sparge heads with respect to the stream selection valve, located inside the heated, insulated valve oven, permits the use of the minimum amount of valve plumbing. This ensures efficient and complete transfer of sample-laden gas through the autosampler system, for delivery to the GC and purge-and-trap concentrator. The headspace gas injection ports use the same 1/8" molded silicon septa that are used in the GC's direct on-column injector, minimizing the need to maintain a variety of consumable replacement parts. Unlike the on-column injector, the headspace injection ports accept needle sizes larger than 26 gauge, such as those commonly found on large volume gas sampling syringes.

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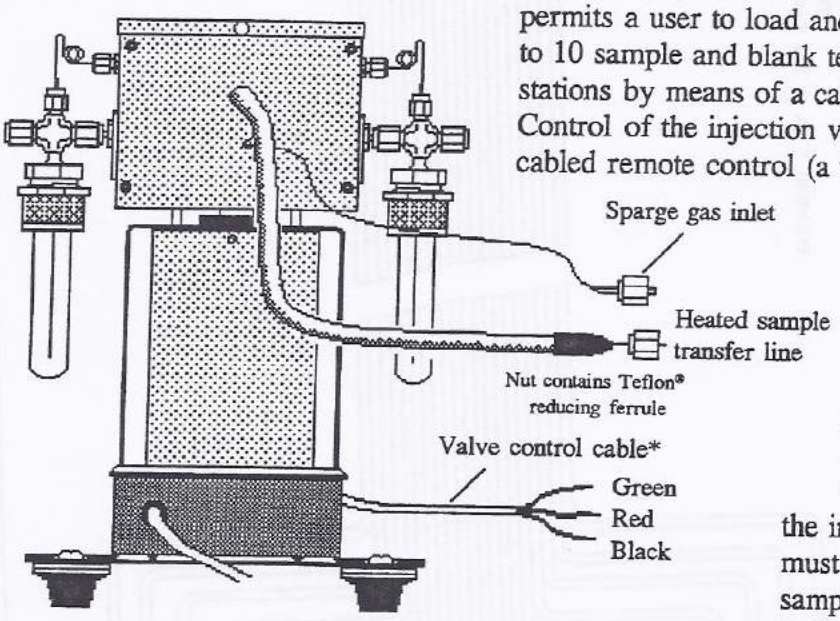
Chapter: PURGE AND TRAP

Topic: Connecting The Free-Standing 10-Station Purge and Trap Autosampler

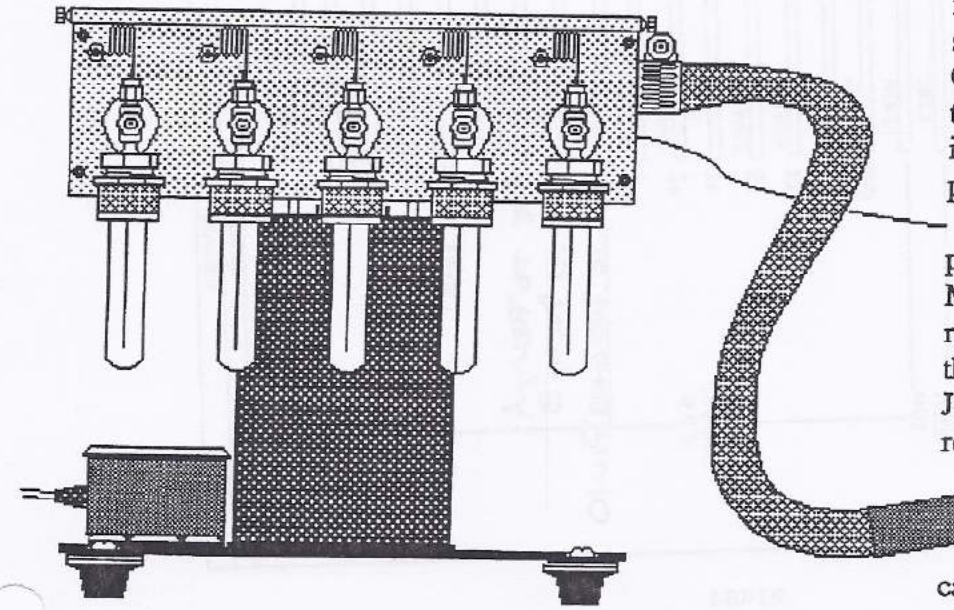
The physical appearance and configuration of the 10-station purge-and-trap is subject to change without notice due to continuing improvements in hardware design

The SRI 10-station purge and trap autosampler option for the SRI gas chromatograph is a free-standing purge and trap system that may be connected to any purge-and-trap concentrator-equipped brand of gas chromatograph in a matter of minutes. The system permits a user to load and sparge any combination of up to 10 sample and blank test tubes and select sampling stations by means of a cabled digital remote control. Control of the injection valve is performed by another cabled remote control (a three-wire cable is also provided to permit relay switching under data system or integrator control). The system includes a heated valve oven that prevents any condensation of analyte in the valves or adjacent plumbing. The sparging heads are also maintained warm to prevent adhesion of sample on the interior surfaces. A transfer line must be used in order to move the sample analytes from the valve oven to the injection port or sampling valve of the host chromatograph. A built-in heating element prevents sample analyte from condensing in the line, actually 36" of virtually indestructible, fused-silica lined stainless steel capillary tubing. Connections are as follows: connect the sparge gas supply to sparge gas inlet tubing. 5psi of helium is preferred. Connect the heated transfer line to the sparge head port of the purge-and-trap system. Momentary connection of green and red wire of three-wire cable rotates the sampling valve to LOAD. Joining the green and black wires rotates the sampling valve to the INJECT position. The green wire is the control cable's GROUND. These wires can be connected to data system relays or other switches. Plug the power cables into an AC outlet and allow the system to warm up. It is now ready for use.



SIDE VIEW SHOWING CONNECTIONS

* for information regarding rainbow ribbon cable used for remote control station selection, see INJECTOR & GAS VALVE section of this manual.



FRONT VIEW OF 10-STATION PURGE-AND-TRAP AUTOSAMPLER

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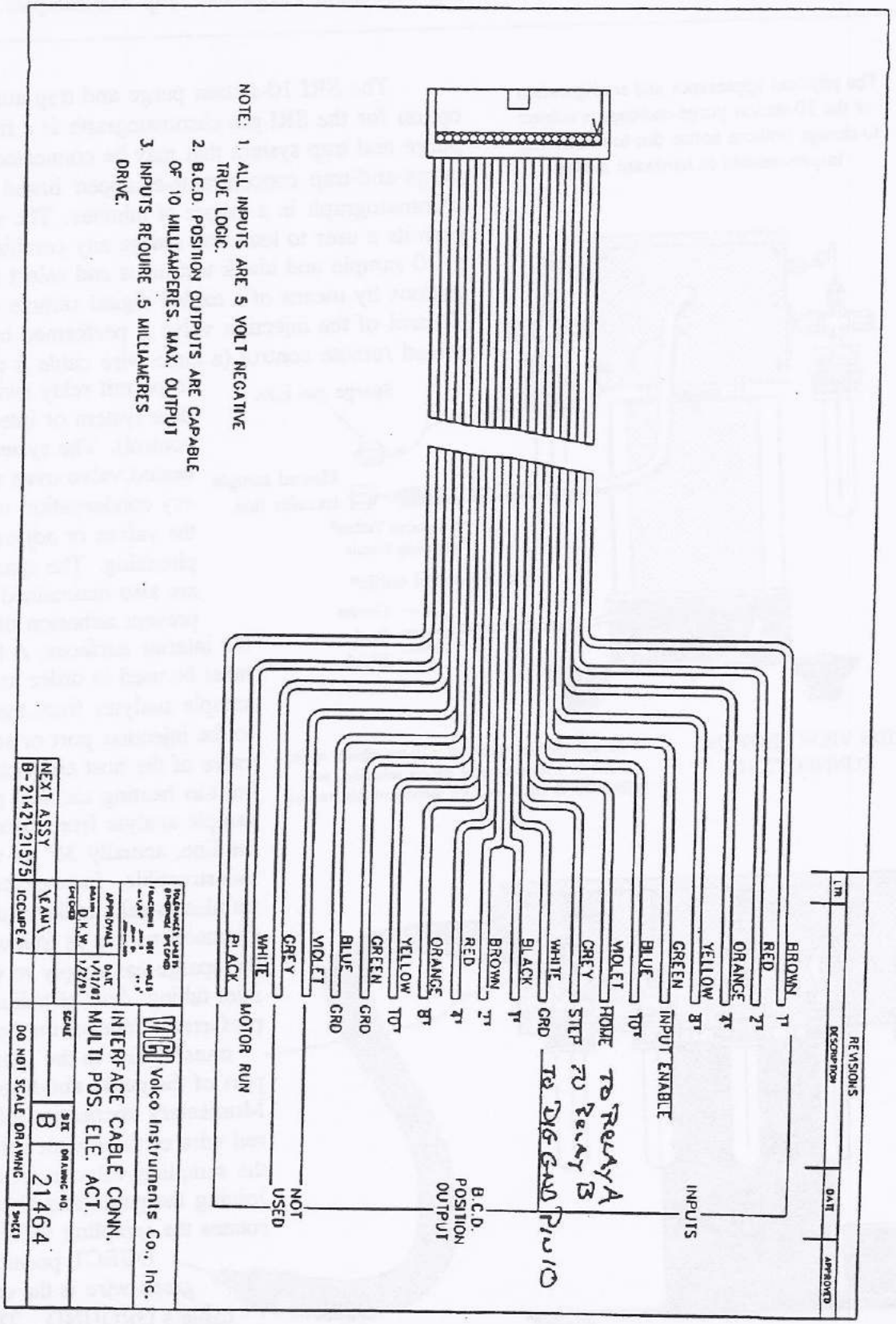

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Chapter: PURGE-AND-TRAP

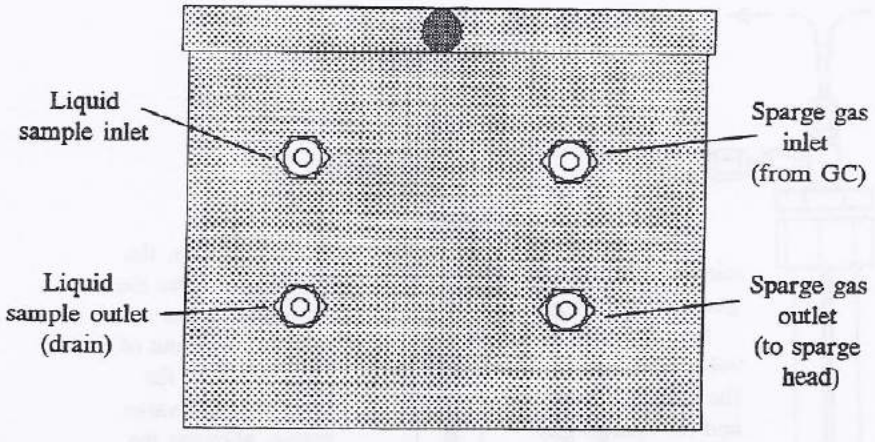
Topic: Connecting The On-Line Liquid Sampler To An Existing SRI Purge-and-Trap

The on-line liquid sampler accessory for the SRI purge-and-trap sampling system is an external unit designed to deliver, purge, and evacuate a liquid sample from the SRI purge-and-trap sample concentrator, on a repetitive basis, under data system automation. This permits the unattended monitoring of any fluid effluent or stream on a continuous basis. In order to operate this accessory, the following installation steps are required:

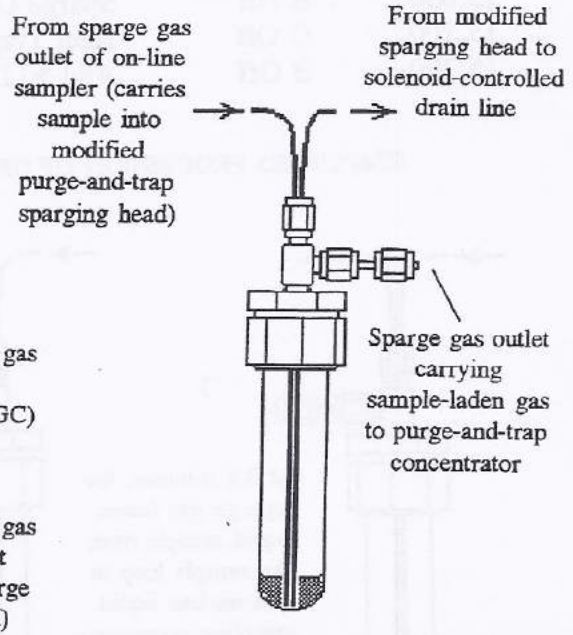
1. Locate the three cables exiting the rear of the on-line liquid sampler. Find and connect the AC supply cable to an available AC wall outlet. A second cable terminates in a remote control wand for the optional manual operation of the liquid sampling valve. Operation of this control is by means of the toggle switch provided. Make sure that the valve is in the LOAD position before proceding. The third cable is the control cable for the sampling valve actuator. Of the 5 wires in this cable, only three are used (green, black, and red) for control of the valve loading and injection. Connect these wires to an unused relay (or relays) in your data system (green is common).

2. Four ports are located on the front panel of the on-line liquid sampler. The upper left port is the liquid sample inlet, where the incoming sample flow is connected. The lower left port is the sample outlet for liquid that has passed through the 5cc sampling loop to the drain line. Route the drain away from the GC and any electrical devices and connections. The upper right port is the sparge gas inlet. Disconnect the sparge gas supply line from the GC to the original purge-and-trap sparge head, and connect it here. The lower right port is the sparge gas outlet from the internal liquid sampling valve. This outlet is directed to the inlet of the modified sparging head provided with this unit.

3. Remove the original purge-and-trap sparging head from the GC purge-and-trap sampler, and replace it with the sparging head provided with the on-line liquid sampler. Note that it is equipped with two lines that enter the sparge head through the top. One line delivers the liquid sample, propelled by the flow of sparge gas through the liquid sampling valve. The second line is the drain line that carries spent sample from the sparging head to the drain line, after passing through a solenoid-controlled valve that controls draining.



FRONT VIEW OF ON-LINE LIQUID SAMPLER ACCESSORY



MODIFIED SPARGE HEAD FOR PURGE-AND-TRAP SAMPLER

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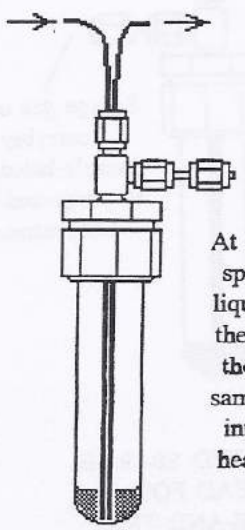
4. Once the on-line sampling accessory hardware has been connected to the SRI purge-and-trap sampling system, edit the purge-and-trap timed event table in order to control the on-line sampler as an integral part of the purge-and-trap system. The event table used should be similar to the timed event table that follows, making note that events H and D specifically control the liquid sampling valve rotation and the sparge head drain valve, respectively. Once the event table has been input and saved, the system is ready for operation.

TIMED EVENT TABLE FOR ON-LINE LIQUID SAMPLING ACCESSORY

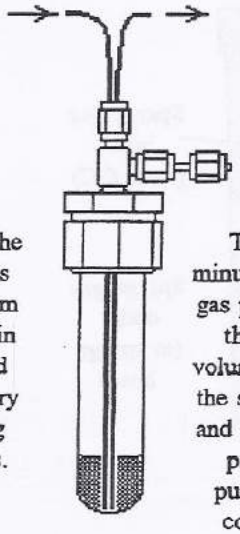
TIME	EVENT	DESCRIPTION
0.100	E On	Sparge Gas Activation (Gas On)
0.200	H On	Rotate Liquid Sampling Valve To INJECT Position
4.500	D On	Sparge Head Drain Valve Open To Drain
5.100	E Off	Sparge Gas Activation (Gas Off)
5.300	D Off	Sparge Head Drain Valve Closed
5.400	H Off	Rotate Liquid Sampling Valve To LOAD Position
6.000	C On	Heat Trap #2
6.100	F On	Heat Trap #1
8.000	G On	Rotate Purge-and-Trap Sampling Valve To INJECT Position
12.000	E On	Sparge Gas Activation (Gas On)
13.000	G Off	Rotate Purge-and-Trap Sampling Valve To LOAD Position
13.100	B On	Add 50 Degrees To Trap Temperature Setpoint (For Bakeout)
14.900	F Off	Heat Trap #1(Heat Off)
15.050	E Off	Sparge Gas Activation (Gas Off)
15.100	C Off	Heat Trap #2 (Heat Off)
15.200	B Off	Add 50 Degrees To Trap Temperature Setpoint (Back To Normal)

SIMPLIFIED PROCESSION OF OPERATION - ON-LINE LIQUID SAMPLING ACCESSORY



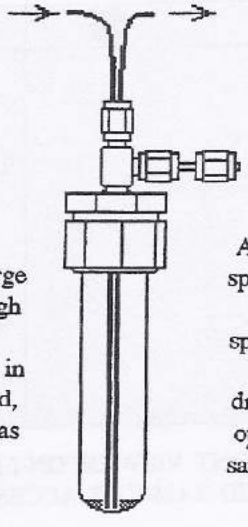
1

At 0.2 minutes, the sparge gas forces liquid sample from the sample loop in the on-line liquid sampling accessory into the sparging head for analysis.



2

Through 4.5 minutes, the sparge gas passes through the measured volume of liquid in the sparging head, and the sparge gas passes to the purge-and-trap concentrator.



3

At 4.5 minutes, the sparge gas forces the liquid in the sparging head out of the vessel as the drain solenoid valve opens, allowing the sample to be drained completely.

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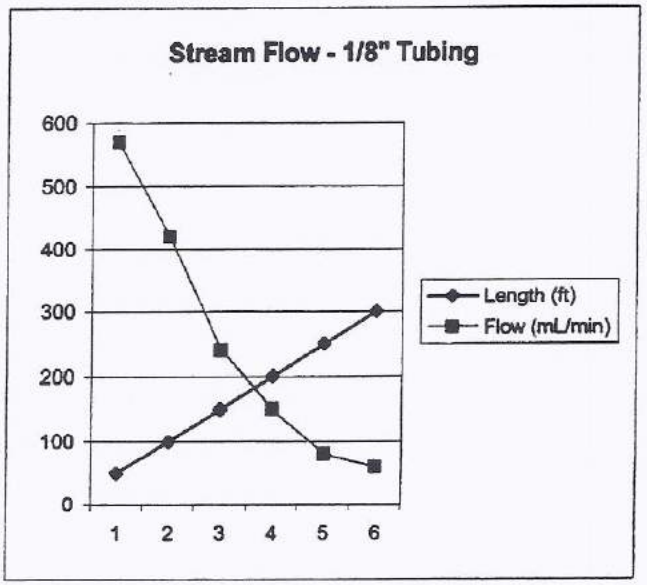
Stream Flow using Standard Vacuum Pump

3/16/03

1/8" Tubing Length (ft)	Flow (mL/min)
50	570
100	420
150	240
200	150
250	80
300	60

The flow was measured from the exit port of a standard Rena Vacuum Pump.



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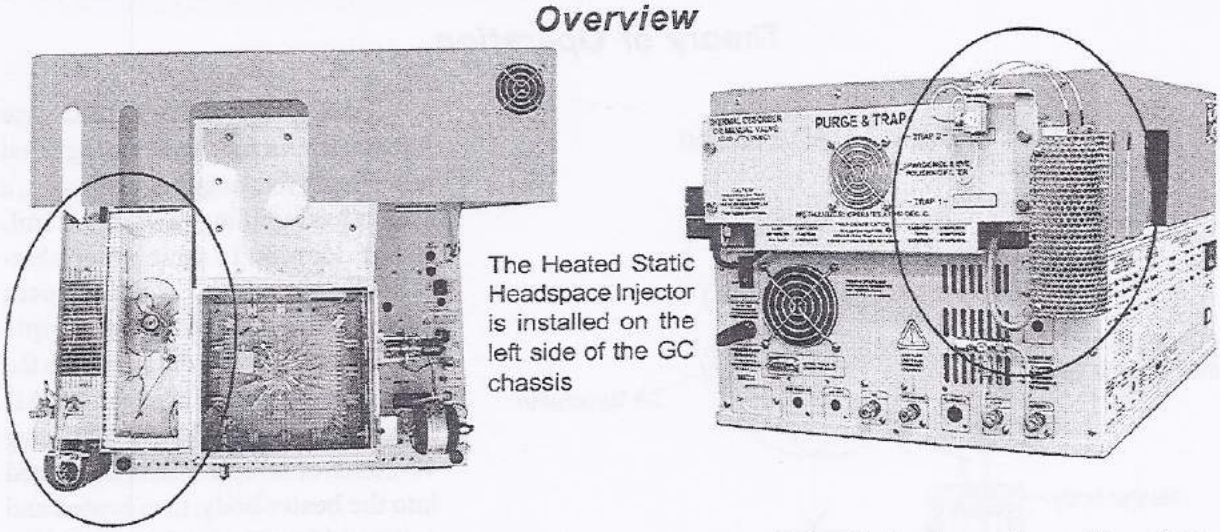
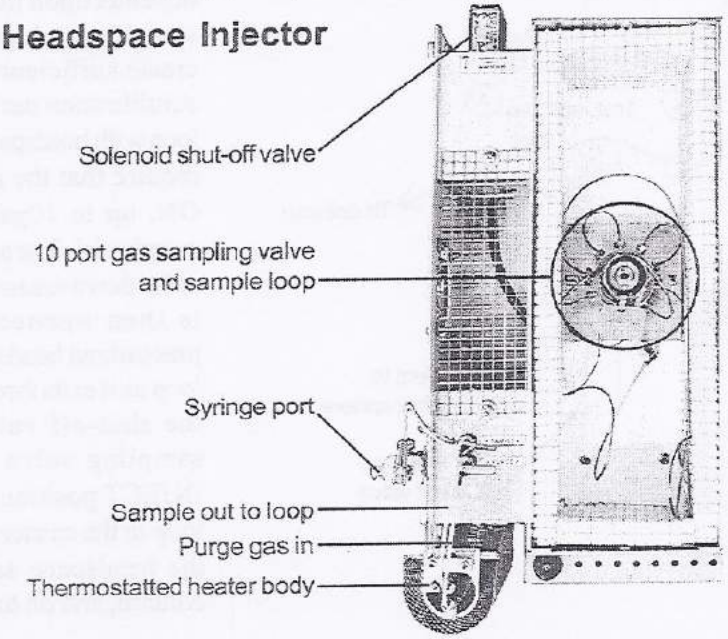
Overview



The Heated Static Headspace Injector is installed on the left side of the GC chassis

The Heated Static Headspace Injector is useful for anaylsis of the light volatiles that can be partitioned into the headspace of a vial containing either a liquid or a solid matrix sample. It can be used with dirty or complex samples, such as blood, urine, powders, foods and flavors. The SRI Heated Static Headspace Injector is built into the 8610C GC, on the left side of the chassis, eliminating the need for transfer lines and reducing dead volume. Thermostatted from ambient to 90°C, a heater body accepts a 40mL VOA vial containing 10-20mL of sample. Covered with a protective heat shield, the heater body is heated and mechanically agitated under control of the PeakSimple data system. Two needles puncture the vial's septum top upon insertion into the heater body. Purge gas to pressurize the vial is delivered through one needle. The other needle carries the headspace vapors to the sample loop on the 10 port gas sampling valve, located in the valve oven. On the downstream end of the loop is a solenoid shut-off valve, also controlled through the data system. This solenoid shut-off valve opens to fill the sample loop with the sample headspace from the pressurized vial. A syringe port allows the addition of internal standards, spikes, etc. into the vial without exposing the sample to ambient air.

Heated Static Headspace Injector



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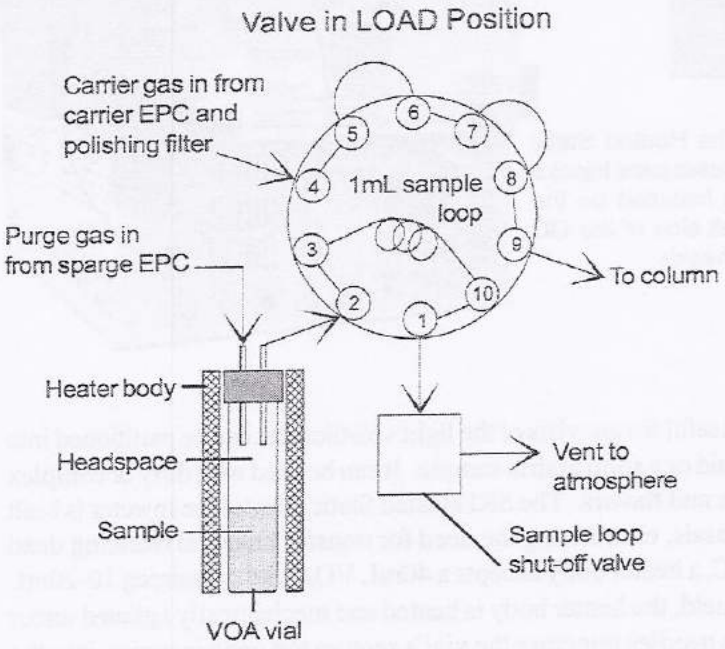
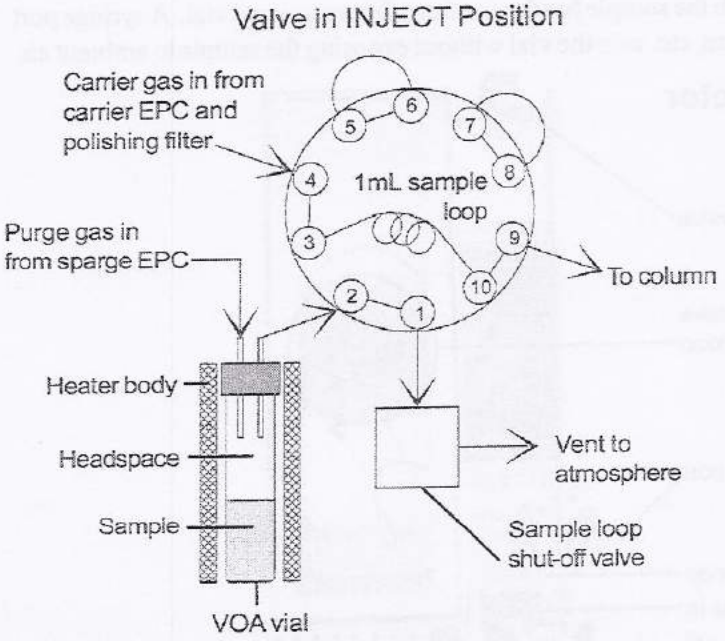
Theory of Operation





The SRI Heated Static Headspace Injector uses a mechanically agitated heater body for sample equilibration, a 10 port gas sampling valve with a 1mL sample loop, and a sample loop shut-off solenoid valve. The headspace analysis begins with the sample equilibration period, during which the gas sampling valve is in the LOAD position. A 40mL VOA vial containing 10-20mL of sample matrix is inserted into the heater body, then heated and agitated (the time it takes to achieve equilibration depends on the sample matrix and the target analytes). Two needles in the upper part of the heater body puncture the septum top of the vial in order to deliver purge gas into and route the sample out of the vial. After the equilibration period, the vial is pressurized so that the sample will escape when the solenoid shut-off valve is opened. The amount of purge gas needed to pressurize the VOA vial depends upon the type of sample. A sample with high liquid content can create sufficient pressure during the eqiulibration period to fill the sample loop with headspace. A dry sample will require that the purge gas be turned ON, up to 10psi, to pressurize the sample vial. The solenoid shut-off valve at the downstream end of the 1mL loop is then opened briefly, and the pressurized headspace sample fills the loop as it exits through the valve. After the shut-off valve closes, the gas sampling valve is actuated to the INJECT position, placing the sample loop in the carrier gas stream to sweep the headspace sample into the GC column, and on to the detector(s).

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General Operating Procedures

1. The Headspace injection technique requires little sample preparation, since just the sample headspace, not the sample itself, is run through the GC. Insert 10-20mL of sample into a clean VOA vial and seal it.

2. The purge gas pressure is controlled with an Electronic Pressure Controller (EPC). Set the purge gas to 0-10psi, depending on the liquid content of the sample. SRI recommends helium purge gas.

3. Using the trimpot on the top edge of the GC's front control panel, set the heater body temperature between ambient and 90°C. Pressure builds as the vial is heated. The temperature setting depends upon the target analytes and the liquid content of the sample.

4. Create or load an event table. **Hdspace.evt,** shown at right and on the ***Expected Performance*** page, is included in version 2.66 (and higher) of the PeakSimple software. A typical event table heats and agitates the vial for 20 minutes; it may take more or less time to achieve headspace equilibration.

5. Create or load a temperature program. The column oven is typically held at the initial temperature (ususally 40°C) for the duration of the sample equilibration period, plus 2-4 more minutes.

6. Set the valve oven temperature to 100°C or higher to avoid water condensation (120°C is a typical setting).

Hdspace.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	F "ON"	VOA Vial Heater
0.200	D "ON"	Shaker solenoid "ON"
19.400	D "OFF"	Shaker solenoid "OFF"
19.500	F "OFF"	VOA Vial Heater
19.600	E "ON"	Purge "ON"
19.700	E "OFF"	Purge "OFF"
19.800	A "ON"	Sample loop exit solenoid open
19.900	A "OFF"	Sample loop exit solenoid closed
20.000	G "ON"	Valve in INJECT
27.000	G "OFF"	Valve in LOAD

7. Activate and energize your detectors as necessary. Consult the manual sections for your particular detector(s).

8. Insert a VOA vial filled with 10-20mL of sample into the headspace heater body: slide the vial into the heater body from the bottom. You will feel some resistance as the needles meet the vial septum lid, and once the needles have penetrated the septum, the vial will stop against the top of the heater body interior. The needles will hold the vial in place. Begin the analysis by pressing the RUN button on the GC or the spacebar on your computer keyboard.

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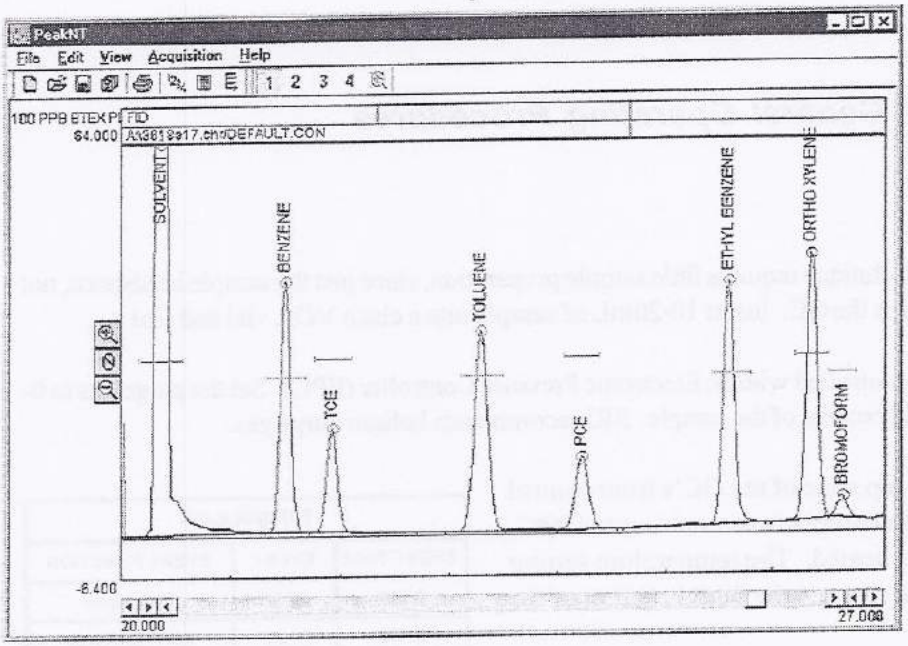
INJECTORS
Heated Static Headspace Injector

Expected Performance



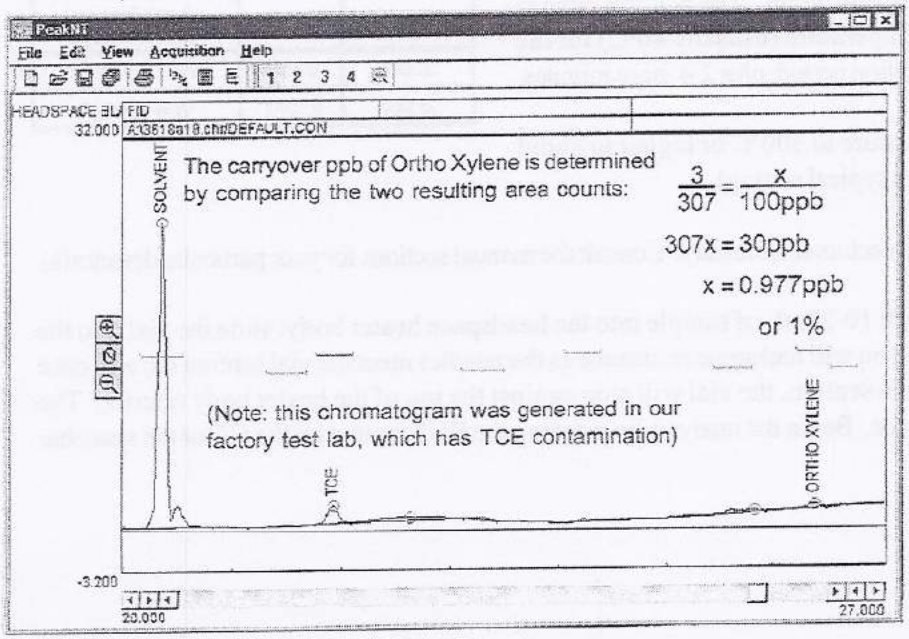
The following two chromatograms were produced in series by an SRI GC equipped with a static headspace injector and an FID detector. The first chromatogram is a 100ppb BTEX Plus sample, and the second is a water blank. Both were run under identical conditions. Magnified for visibility, the water blank shows the carryover level of the Headspace injection system.

Sample: 1µL 100ppb BTEX Plus (vial 25% full of sample solution with 10µL of 100ppb BTEX Plus) VOA vial set to heat from ambient temperature to 50°C
Column: 15m MXT-VOL
Carrier: helium @ 10mL/min

FID gain: HIGH
FID temp: 300°C
FID ignitor: -400
Valve temp: 120°C

Column oven temperature program (**Hdspace.tem**):

Initial	Hold	Ramp	Final
50°C	23.00	10.00	160°C



The carryover ppb of Ortho Xylene is determined by comparing the two resulting area counts:

$$\frac{3}{307} = \frac{x}{100ppb}$$

$$307x = 30ppb$$

$$x = 0.977ppb$$

or 1%

(Note: this chromatogram was generated in our factory test lab, which has TCE contamination)

Events:

Time	Event
0.100	F ON (VOA vial heater)
0.200	D ON (shaker solenoid)
19.400	D OFF
19.500	F OFF
19.600	E ON (purge gas)
19.700	E OFF
19.800	A ON (sample loop exit solenoid)
19.900	A OFF
20.000	G ON (valve actuator)
27.000	G OFF

BTEX sample results:

Component	Retention	Area
Solvent	20.350	7332.0805
Benzene	21.483	266.2780
TCE	21.916	119.6645
Toluene	23.266	285.2310
PCE	24.216	98.6710
Ethyl Benzene	25.583	298.6540
Ortho Xylene	26.333	306.6115
Bromoform	26.616	24.4815
	Total	8731.6720

Water blank results:

Component	Retention	Area
Solvent	20.350	137.0385
TCE	21.916	10.0770
Ortho Xylene	26.350	3.1305
	Total	150.2460

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