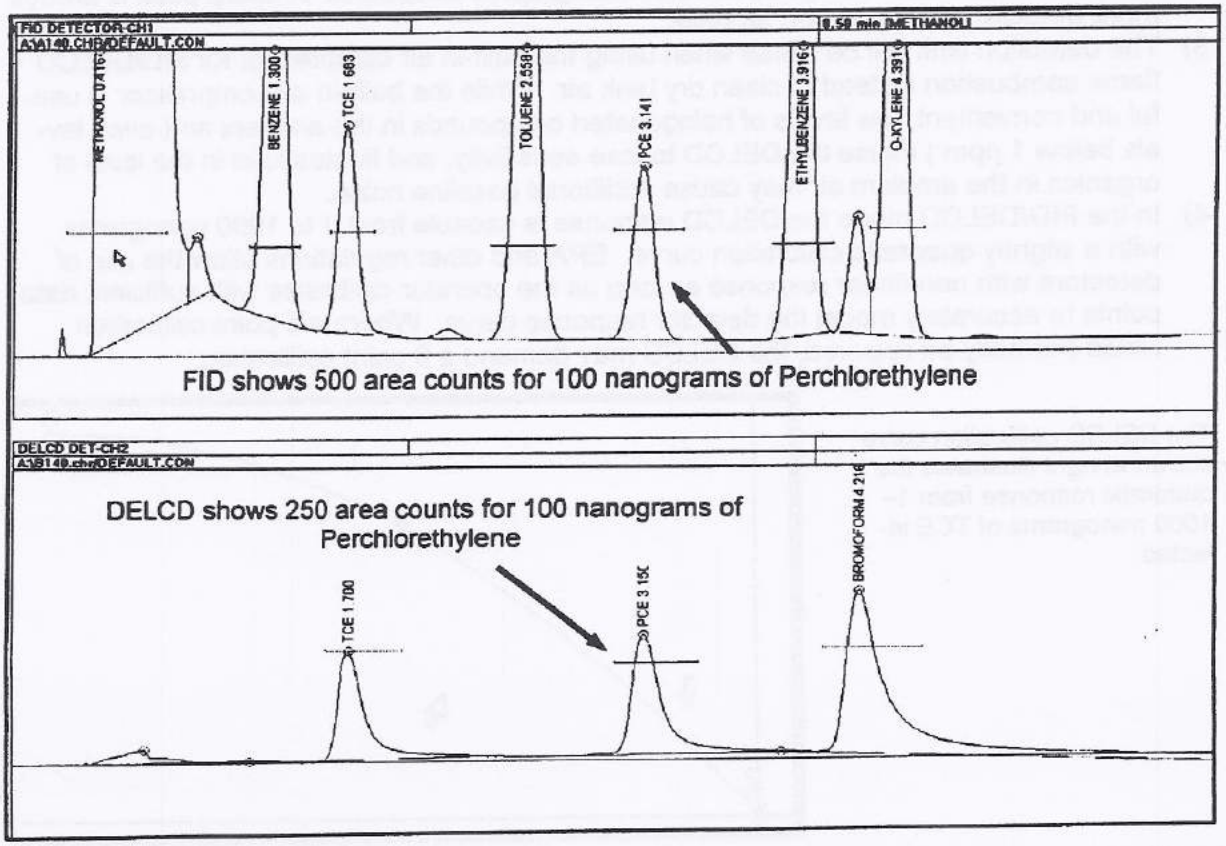
Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

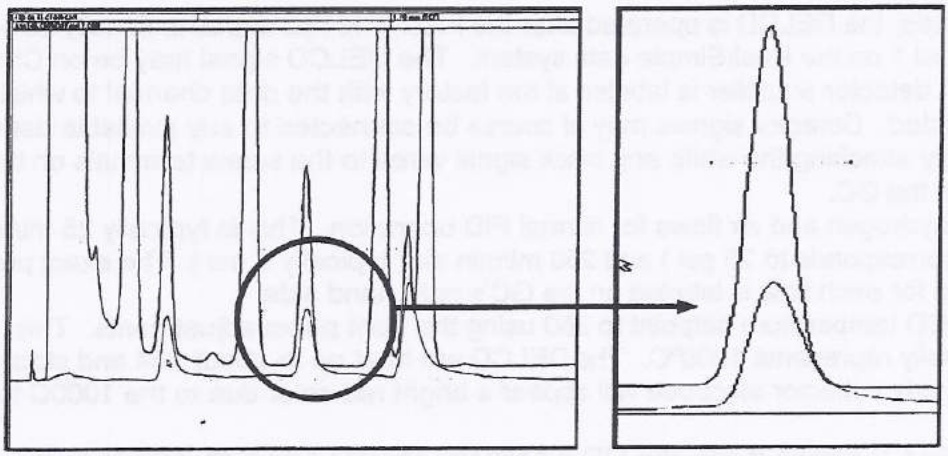
In the combo mode, the DELCD is operated after the FID. The FID signal is usually connected to Channel 1 on the PeakSimple data system. The DELCD signal may be on Channel 2 or 3. Each detector amplifier is labeled at the factory with the data channel to which it has been connected. Detector signals may 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).



Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

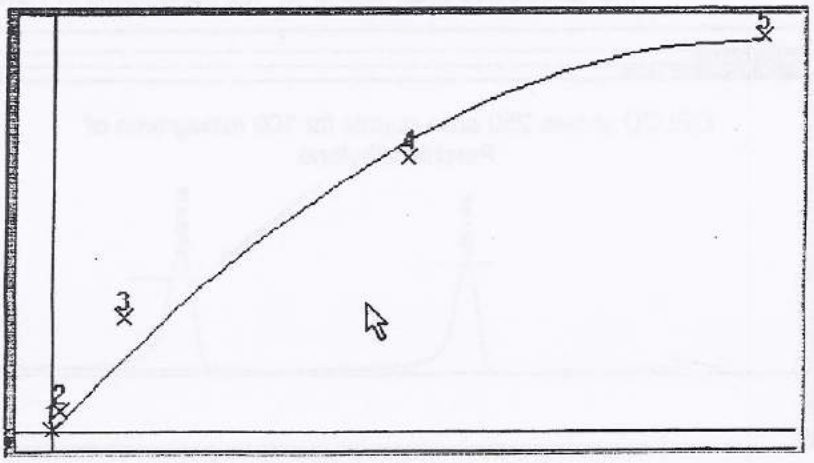


DELCD peak overlaid on FID peak for PCE, then expanded for clarity.

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



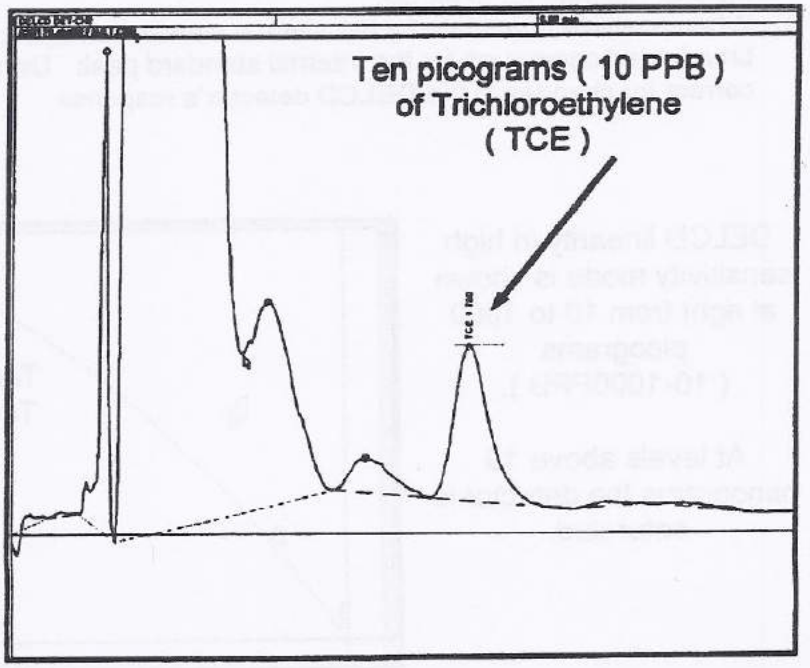
Chapter: Detectors

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.

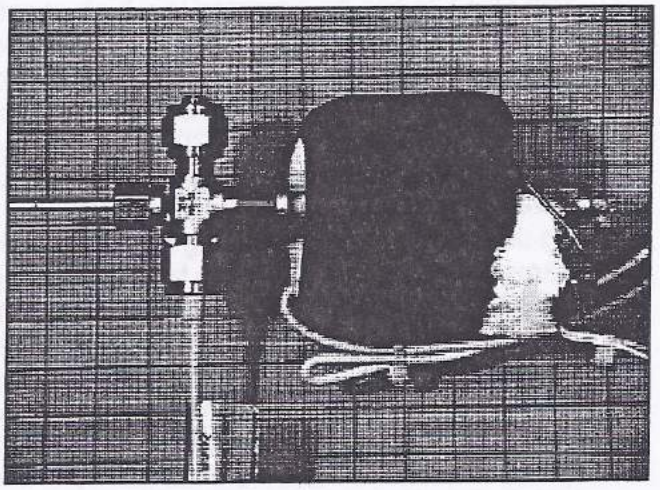


Chapter: Detectors

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.

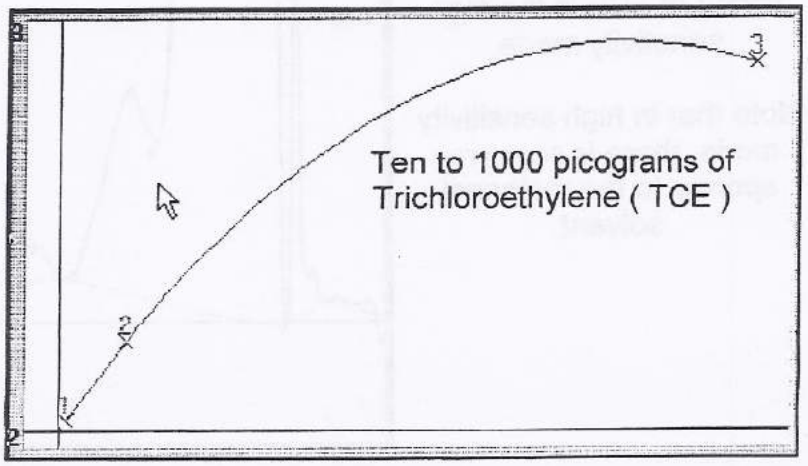
The collector electrode is removed and a 1/4" cap installed instead.



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).

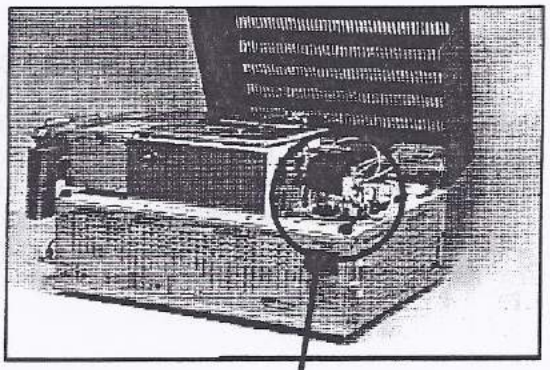
At levels above 10 nanograms the detector is saturated.



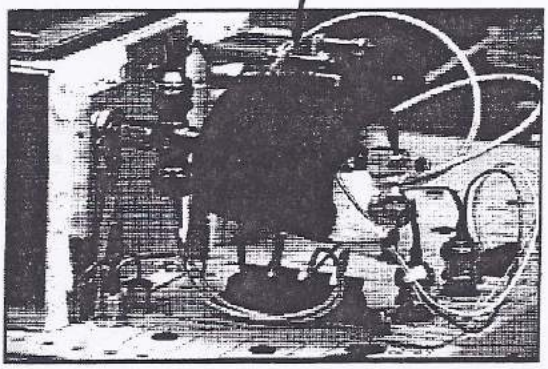
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

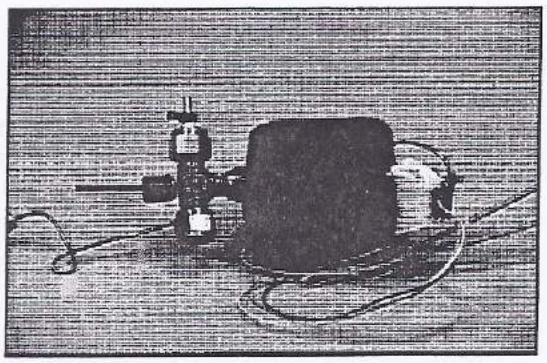
The DELCD detector is only available in combination with the FID detector.



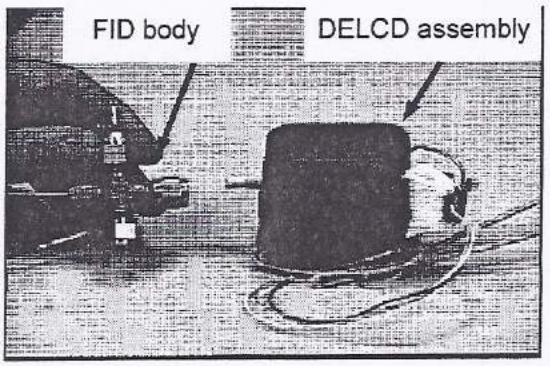
The FID/DELCD combo detector is mounted to a thermostatted aluminum heater block on the right hand side of the column oven.



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



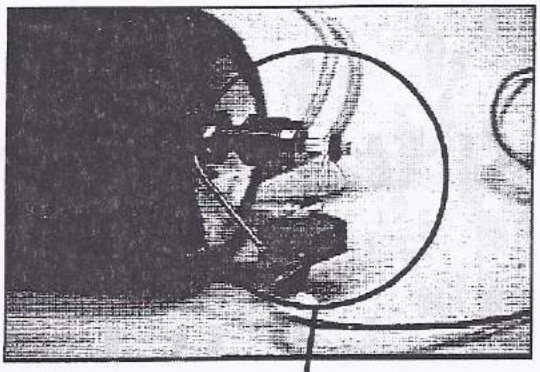
The DELCD part of the detector is the large black cylinder which mounts into the right hand port of the FID detector body. It can be separated from the FID body by loosening the 1/4" Swagelok nut and graphite ferrule which secures it in place.



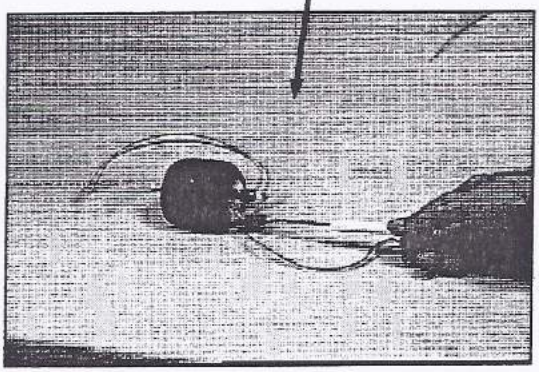
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

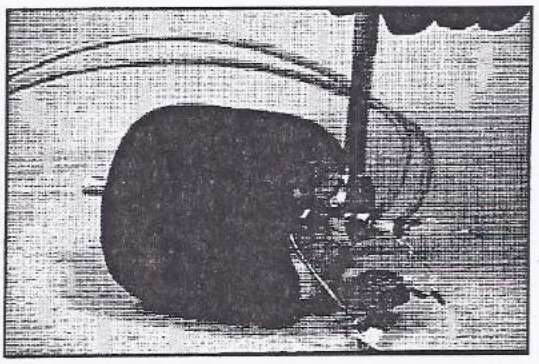
The DELCD collector electrode (part# 8670-1028) can be removed from the heater. Because the heater operates at close to 1000°C, it will fail eventually. A new heater (part # 8670-1027) is less expensive than the complete heater/collector assembly (part# 8670-1029), so it may make sense to remove the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



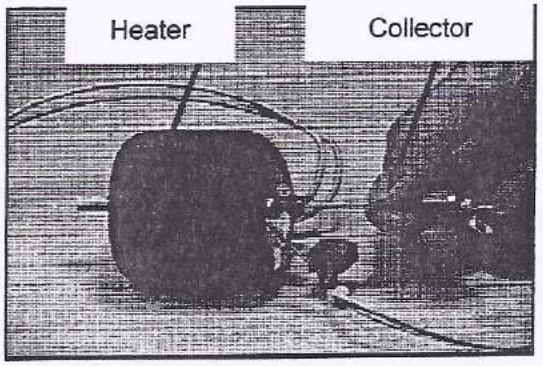
Dis-connect the three small platinum wires from the screw terminals and gently move them aside.



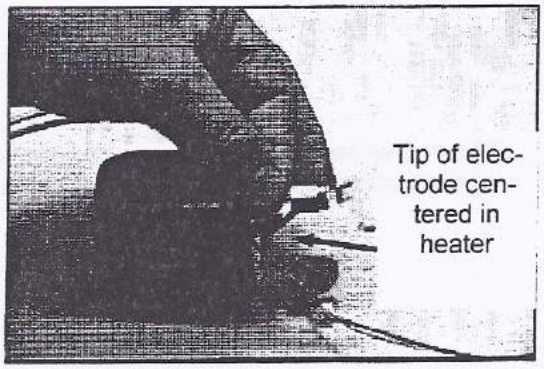
Using two wrenches to avoid rotating the fitting, loosen the 1/8" Swagelok nut and graphite ferrule which secures the collector electrode into the heater.



The collector can then be withdrawn from the heater.

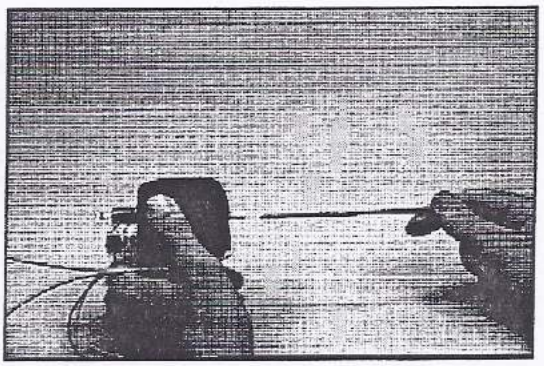


When the collector electrode is re-installed in the new heater, it is important that the tip of the electrode is positioned in the center of the heater.

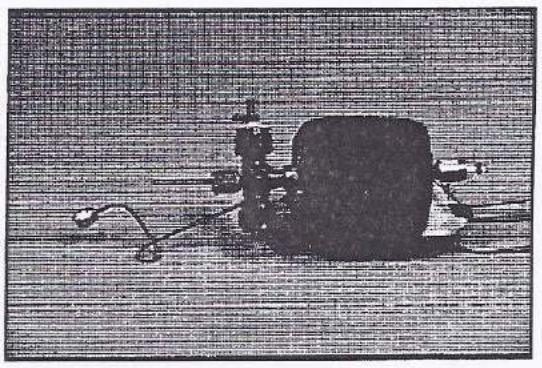


Tip of electrode centered in heater

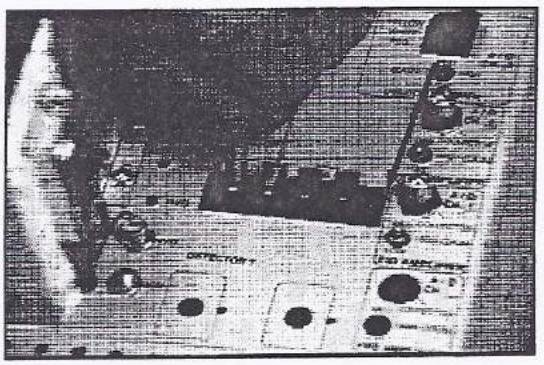
Use a file, rod, screwdriver or other long thin object as a gauge to verify that the electrode tip is centered in the new heater body. Gently re-position the electrode by sliding it through the graphite ferrule to get the proper adjustment. Finally, look down the bore of the heater and check to make 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.



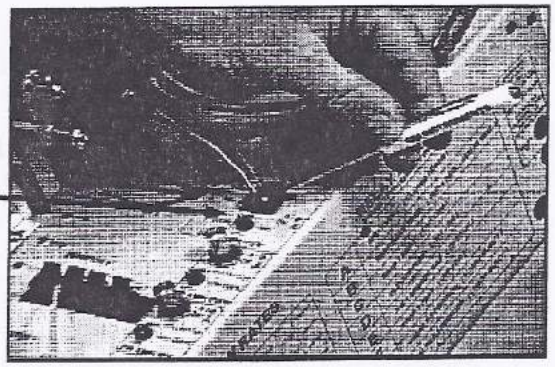
The two DELCD heater wires are connected to the push terminals on the deck of the GC which are labeled DELCD heater.



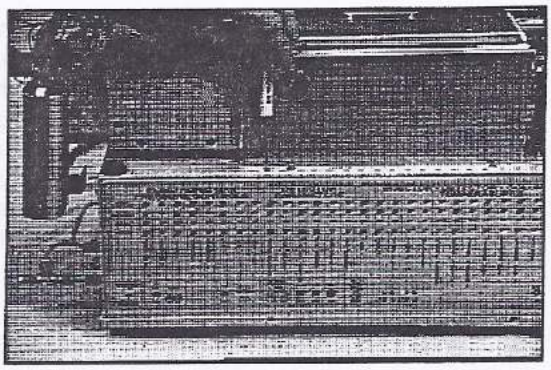
Chapter: Detectors

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.

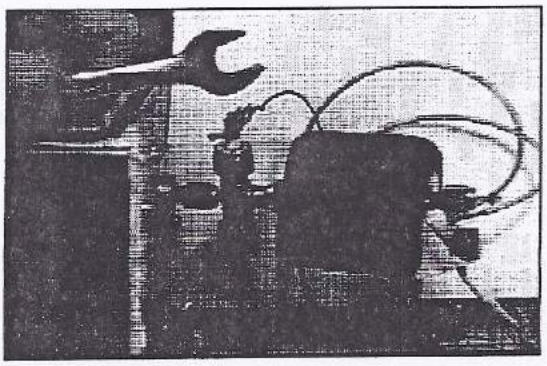


Set the DELCD heater setpoint temperature to 250. This is an arbitrary number which actually corresponds to a temperature of 1000°C. Better sensitivity can be obtained by raising the setpoint to 260 or 270, but at the cost of reduced heater lifetimes.



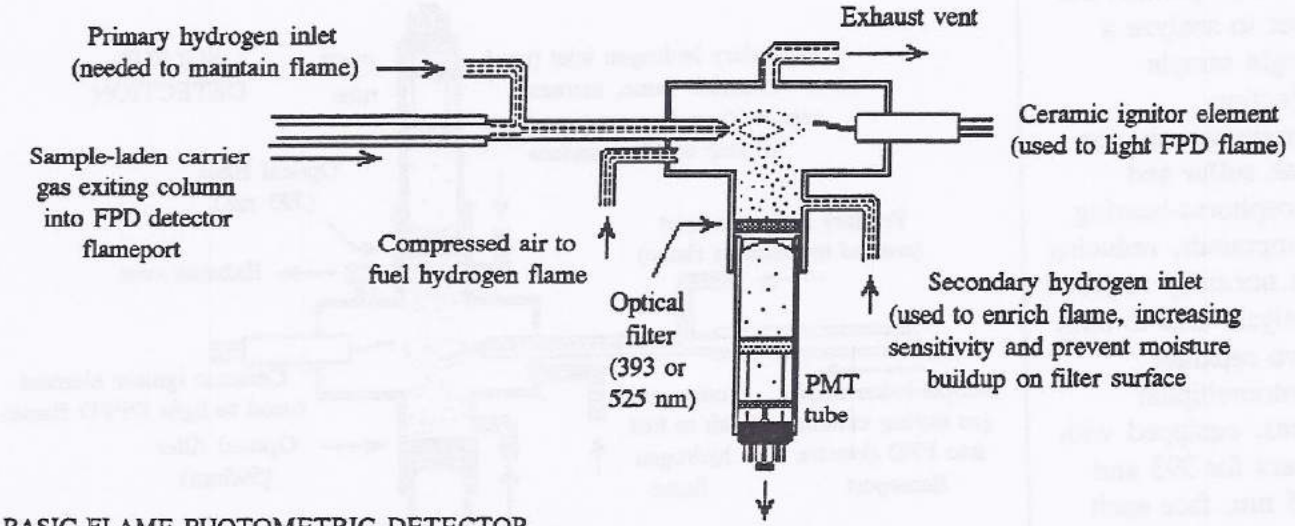
The actual temperature of the DELCD heater will equilibrate to about 7 degrees less than the setpoint within 10 minutes

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.



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.

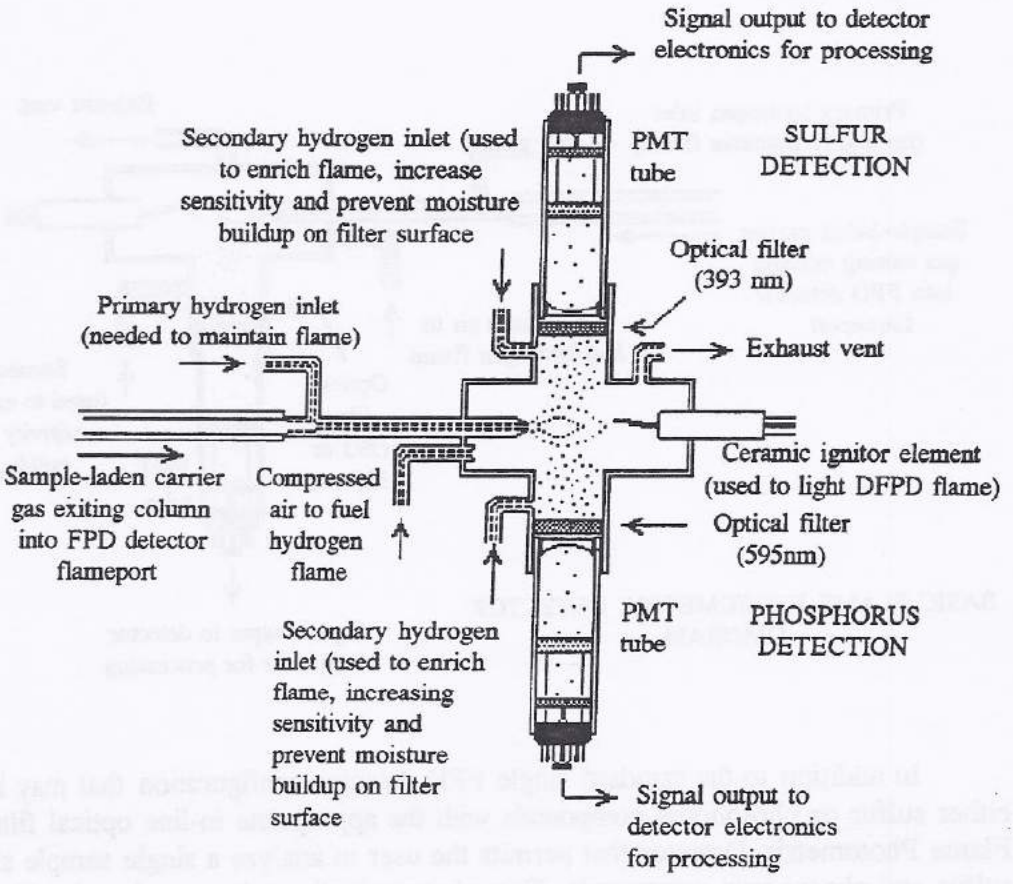
Chapter: DETECTORS

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.

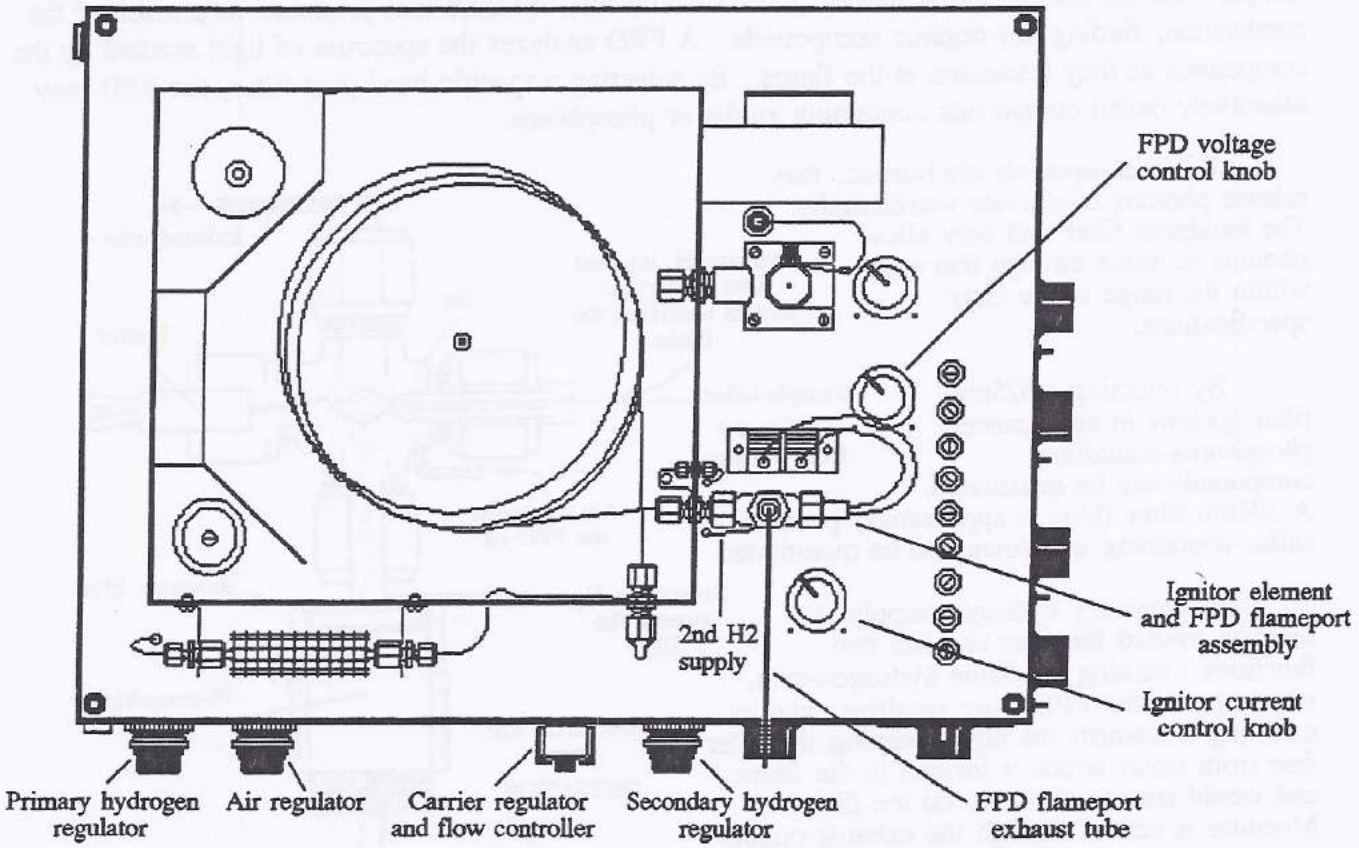
The SRI Dual Flame Photometric Detector (DFPD) permits the user to analyze a single sample injection simultaneously for both sulfur and phosphorus-bearing compounds, reducing the normally required analysis time to half. Two separate photomultiplier tubes, equipped with filters for 393 and 525 nm, face 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

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.

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.

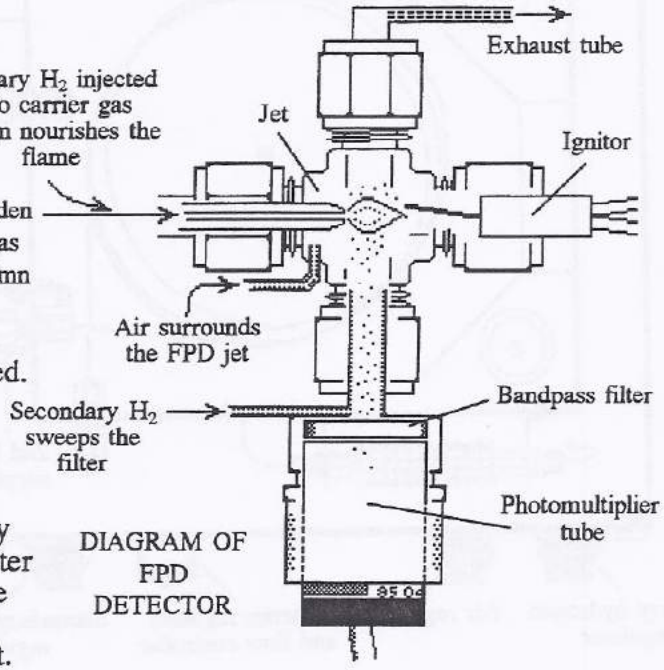


DIAGRAM OF FPD DETECTOR

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.

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.

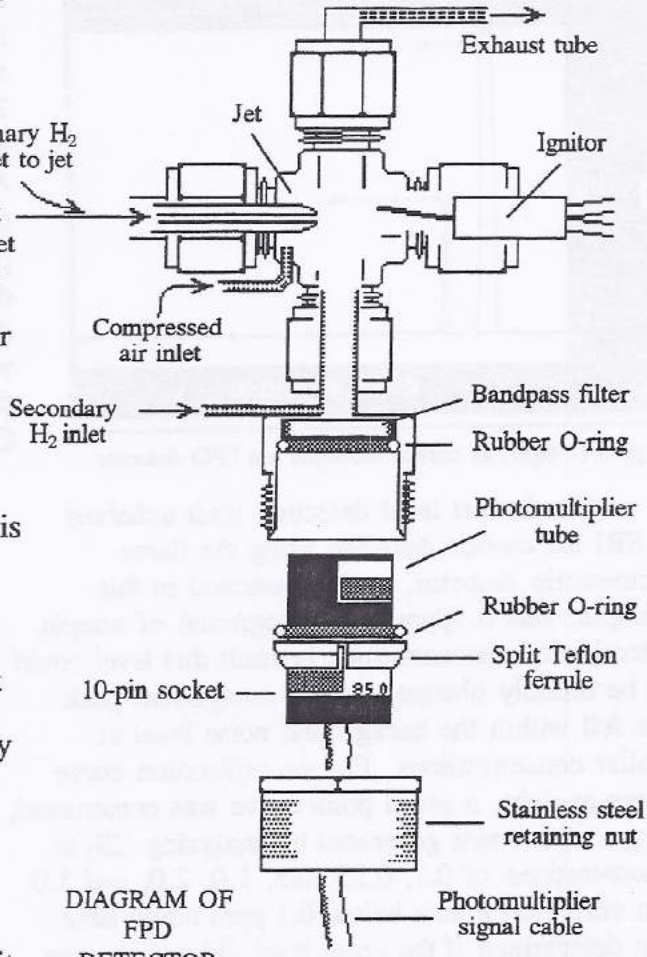


DIAGRAM OF FPD DETECTOR

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.

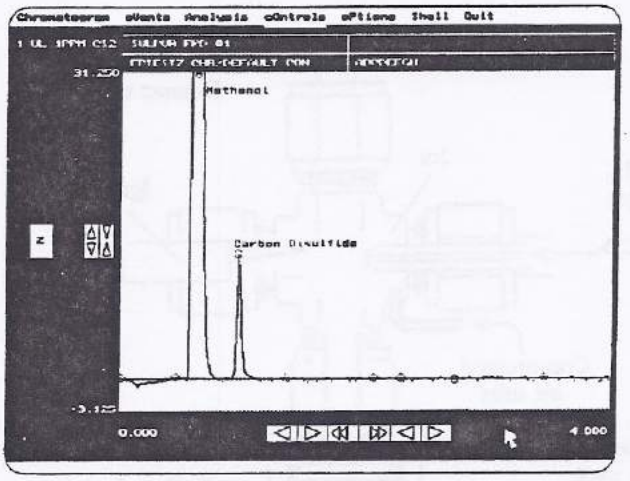
The tube / socket assembly is then inserted into the lower FPD assembly body and held in place by the knurled stainless steel retaining nut. When the FPD detector assembly is fully reassembled, pack any excess signal cable from the socket back into the FPD chassis orifice.

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.

Chapter: DETECTORS

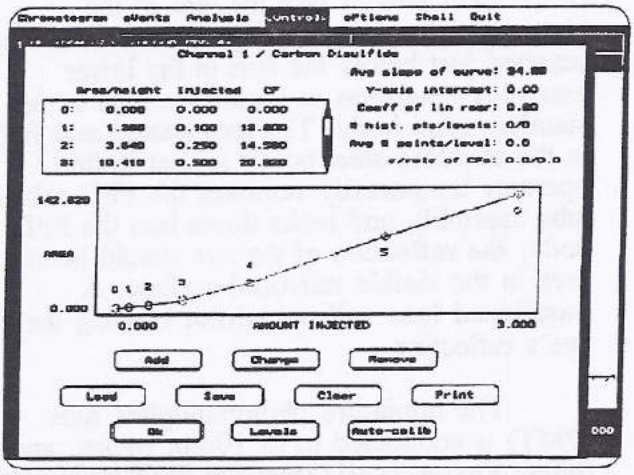
Topic: Demonstration of The Detection Limit of The FPD Detector

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.

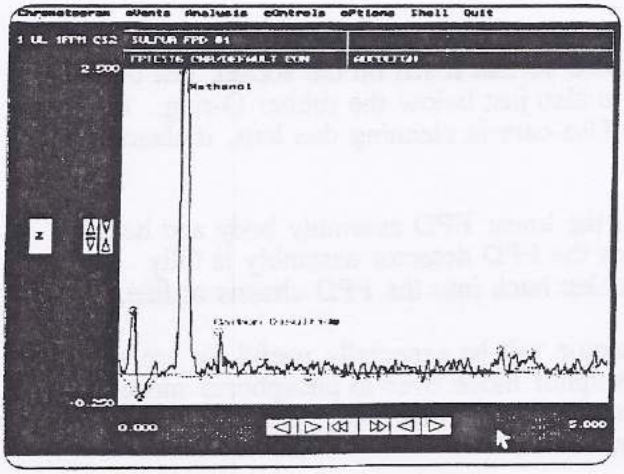


1 ppm (1 ngm) of carbon disulfide via FPD detector

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

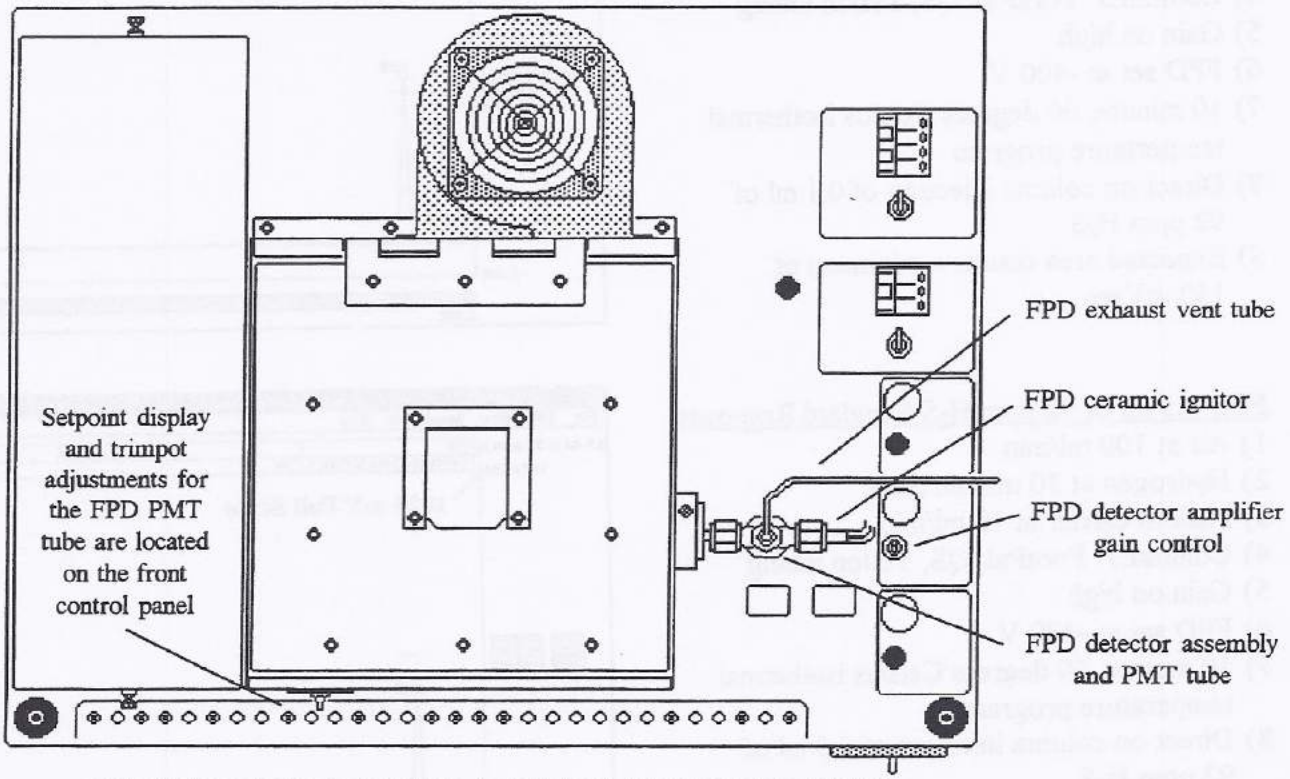


0.1 ppm of CS_2 detectable well above background

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

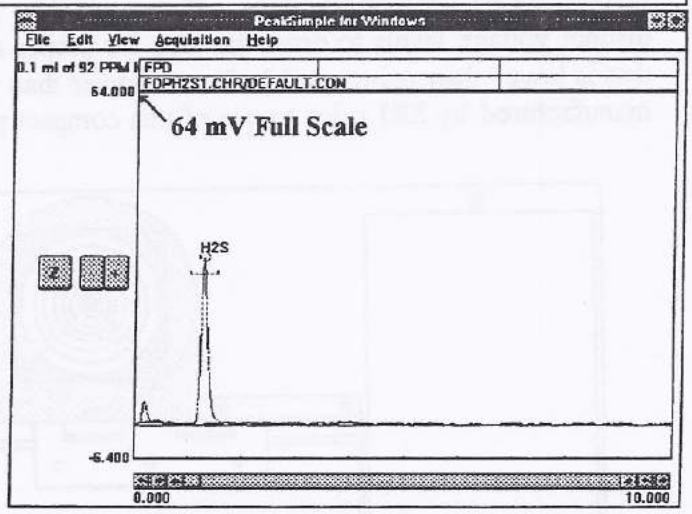
Chapter: DETECTORS

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.

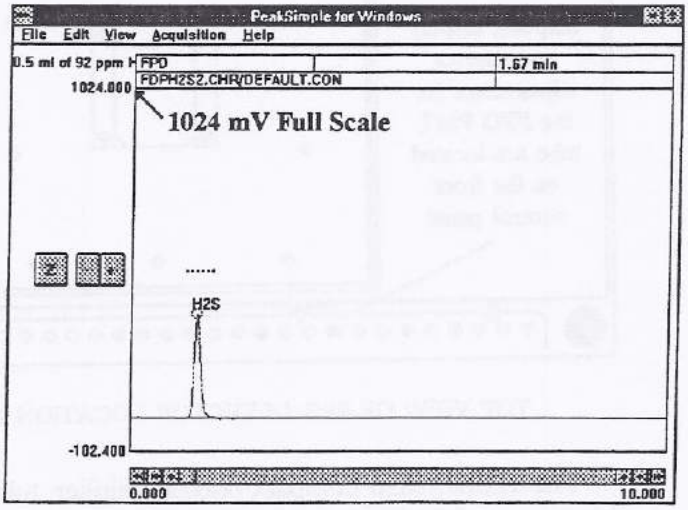
FPD 0.1 ml of 92 ppm H_2S Standard Response

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



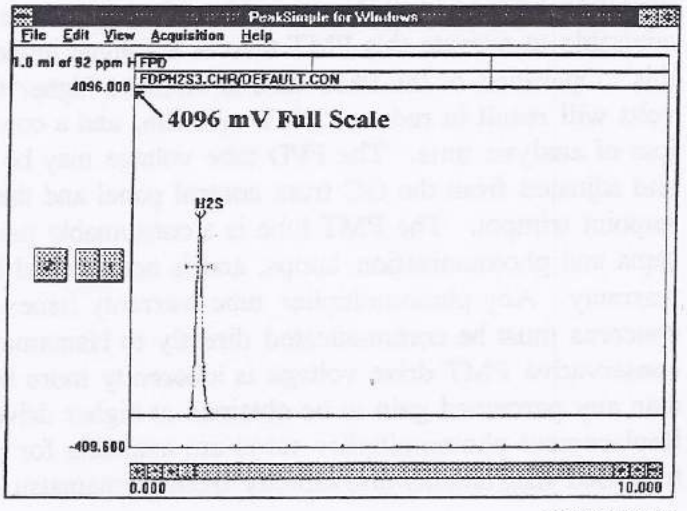
FPD 0.5 ml of 92 ppm H_2S Standard Response

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

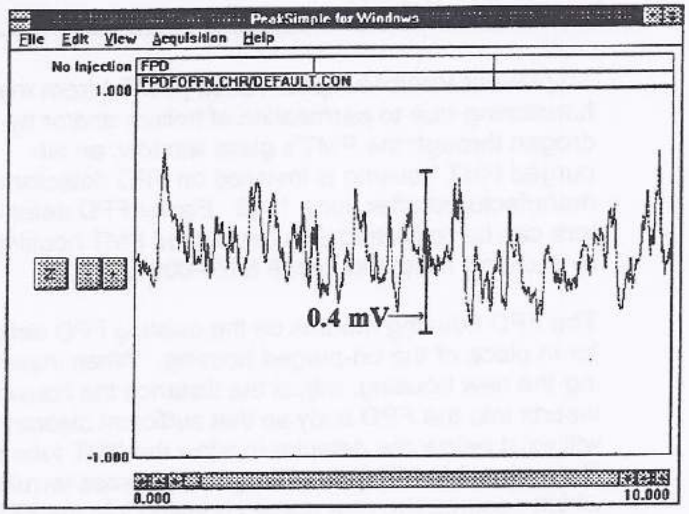


FPD 1.0 ml of 92 ppm H_2S Standard Response

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

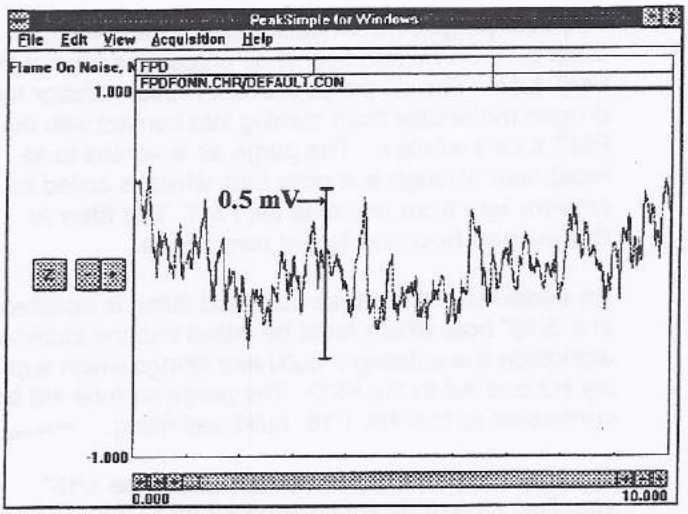


FPD Expected Performance



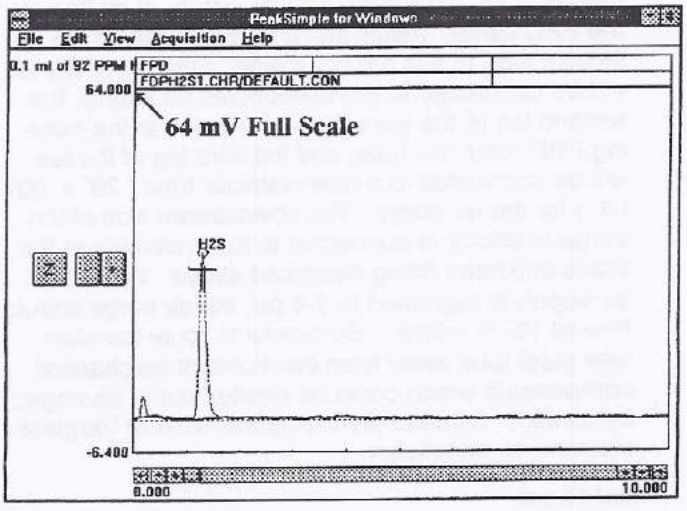
FPD Flame Off Noise

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



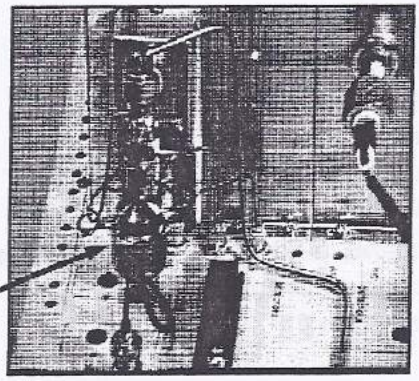
FPD 0.1 ml of 92 ppm H_2S Standard Response

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

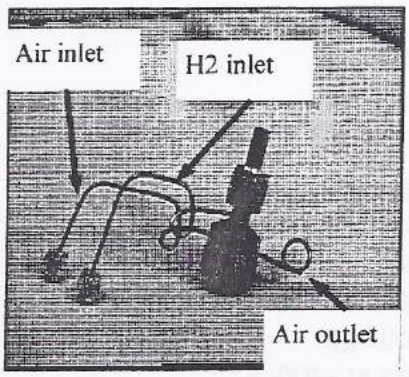
Chapter: FPD DETECTOR

Topic: Retrofit of air purged PMT tube housing

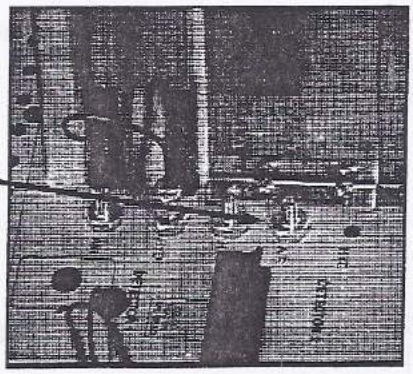
To prevent PhotoMultiplier Tubes (PMT) from malfunctioning due to permeation of helium and/or hydrogen through the PMT's glass window, an air-purged PMT housing is installed on FPD detectors manufactured after June 1998. Earlier FPD detectors can be retrofitted with the purged PMT housing by ordering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

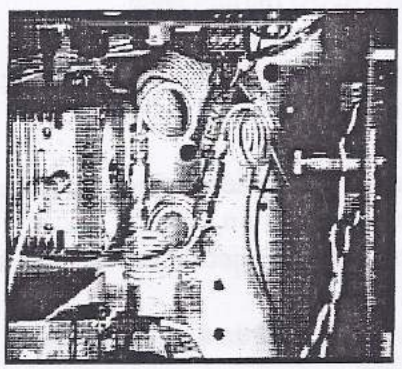


The new purged PMT housing has a tube which directs a 10-30 ml/min flow of air across the face of the PMT tube. The air purge prevents helium and/or hydrogen molecules from coming into contact with the PMT tube's window. The purge air is vented to atmosphere through a shorter tube which is coiled to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



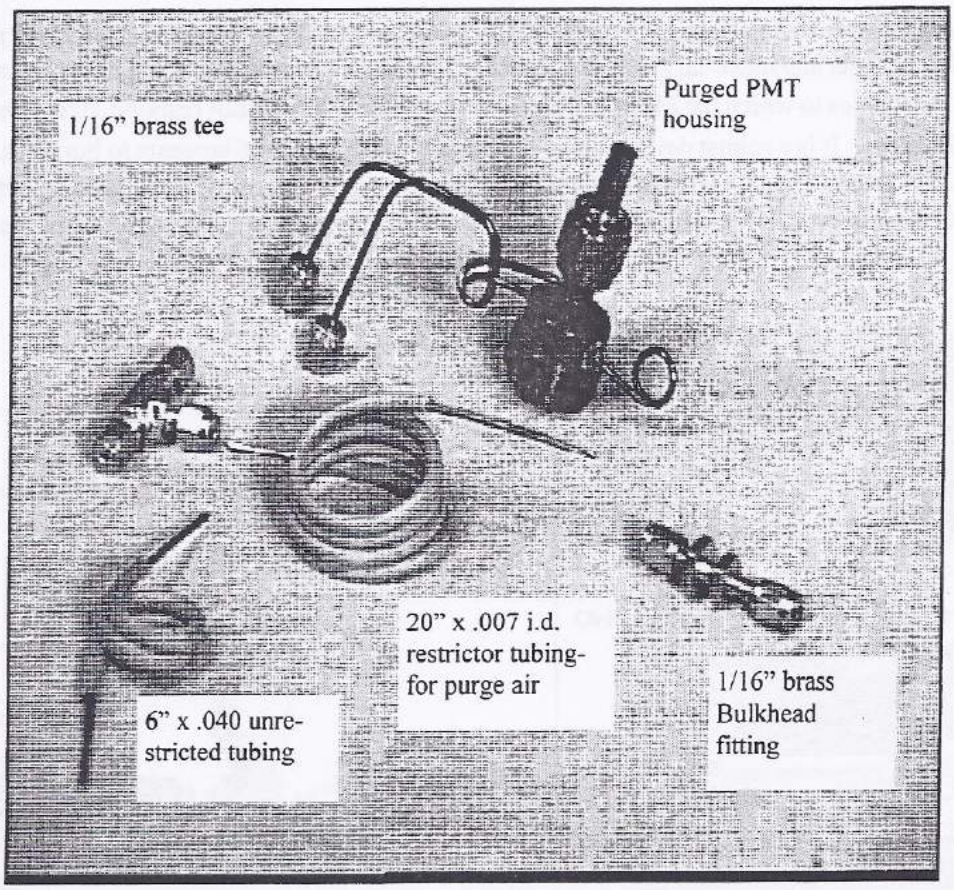
An additional 1/16" brass bulkhead fitting is installed in a 3/16" hole which must be drilled into the chassis alongside the existing 3 bulkhead fittings which supply H2 and Air to the FPD. The purge air tube will be connected to this 4th 1/16" bulkhead fitting.

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.



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

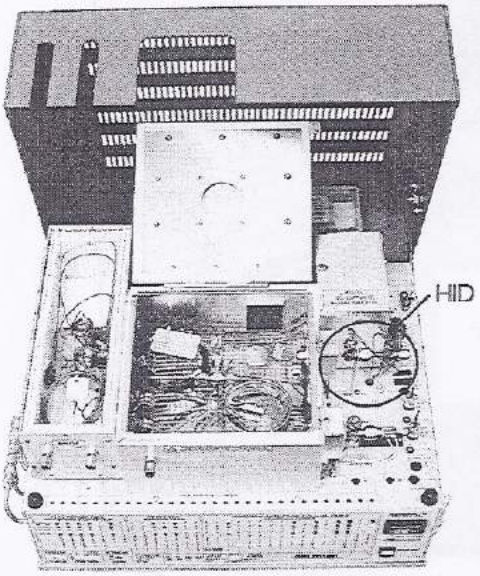
3) 1/16" brass bulkhead fitting

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

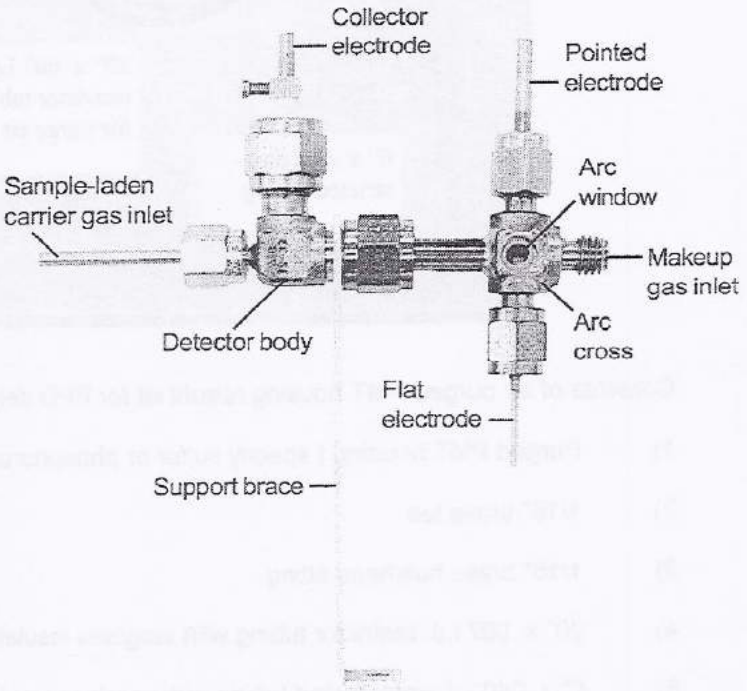
Overview

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.

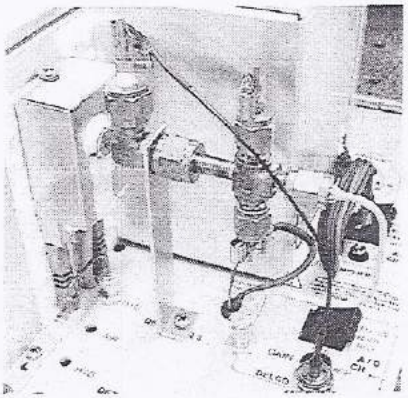


HID detector between TCD and FID detectors on an SRI GC

HID detector removed from GC and heater block

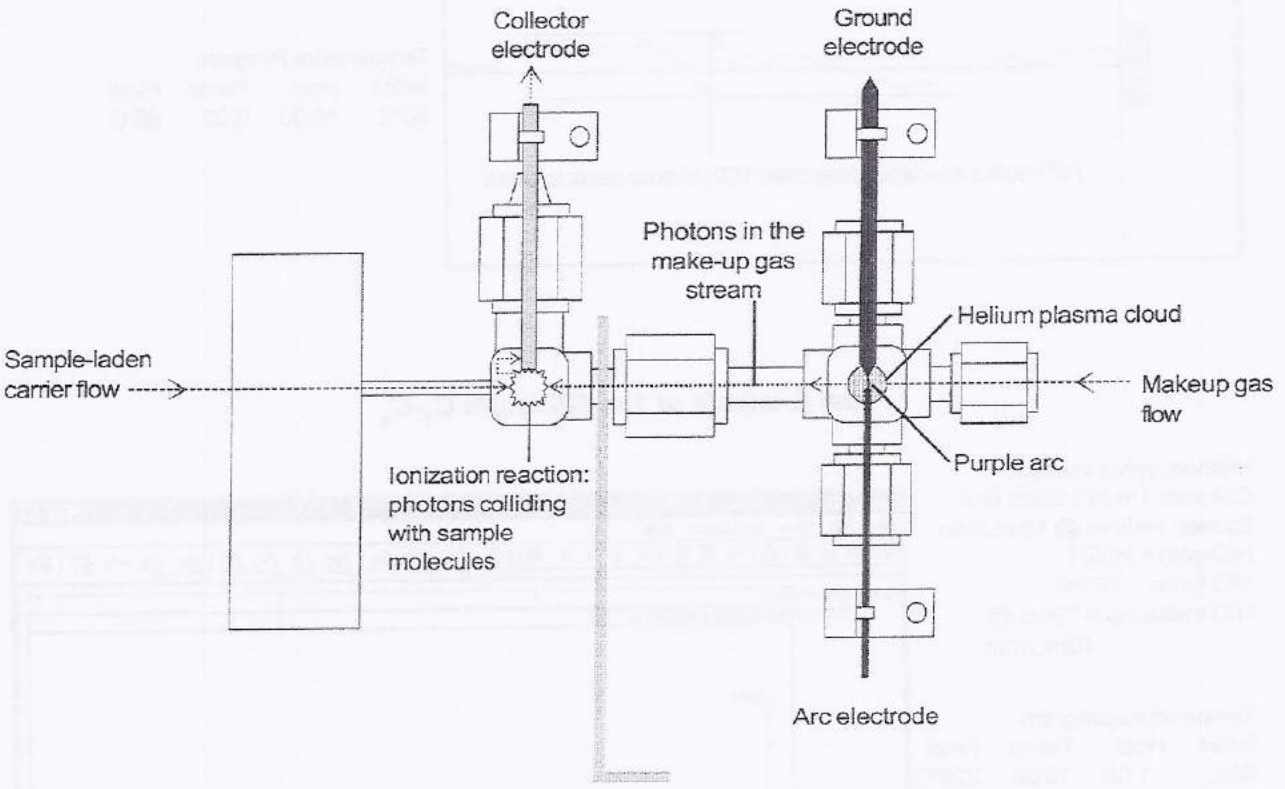


Close-up of the same HID detector



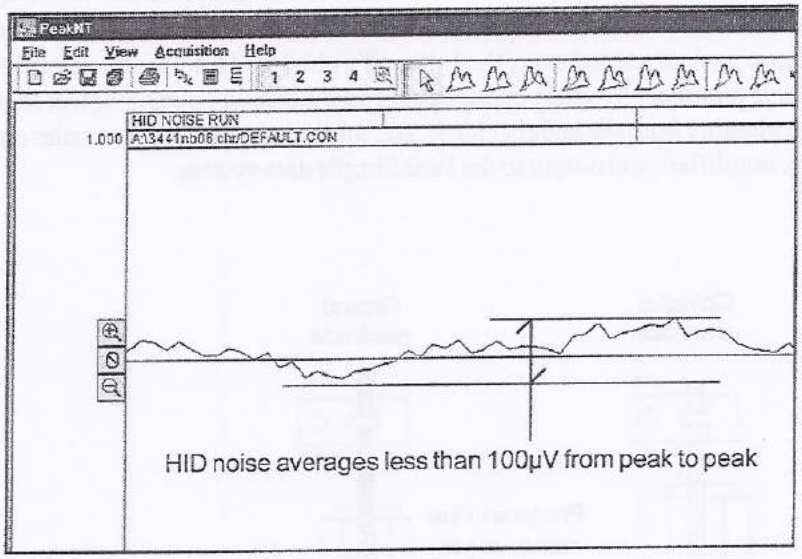
Theory of Operation

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.

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:

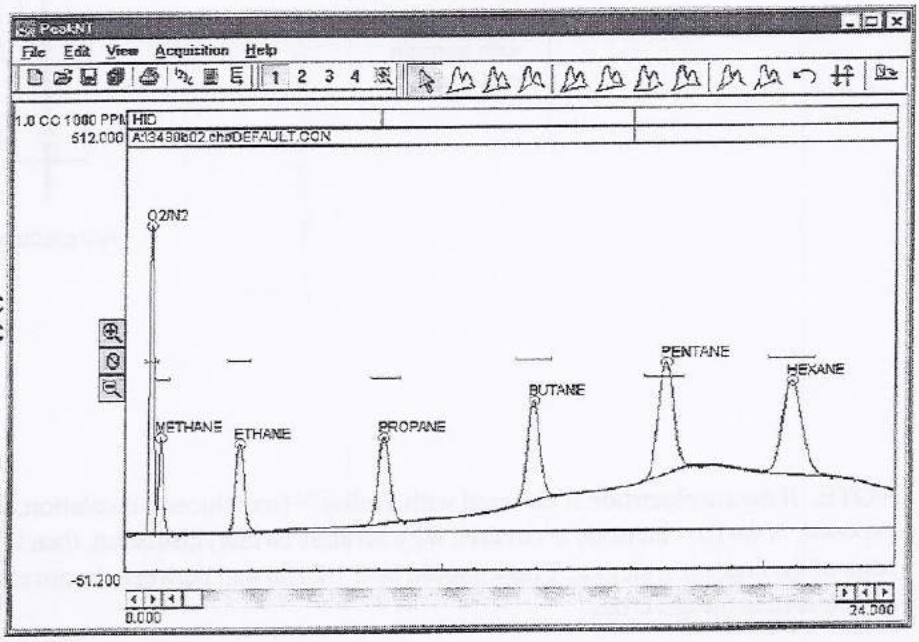
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

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

DETECTORS
HID - Helium Ionization Detector

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).

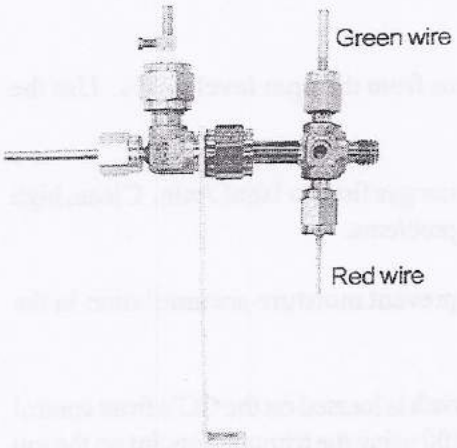
   B. Look through the arc window at the arc and ground electrodes. If they appear to be touching, disconnect the red electrode lead wire then check the continuity between the electrodes using a multimeter; the reading should be open or infinite.

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

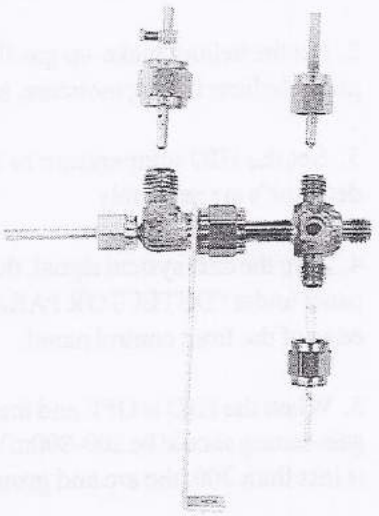
7. Let the milliVolt reading stabilize, then begin the analytical run.

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).

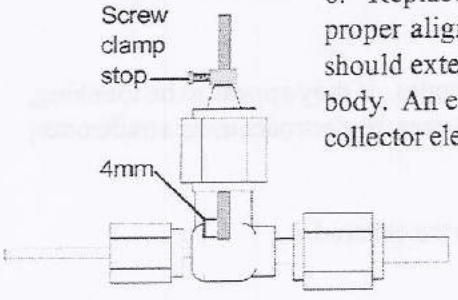


2. Remove the the arc and ground electrodes by loosening the 1/8" fittings that hold the electrodes in the arc cross.

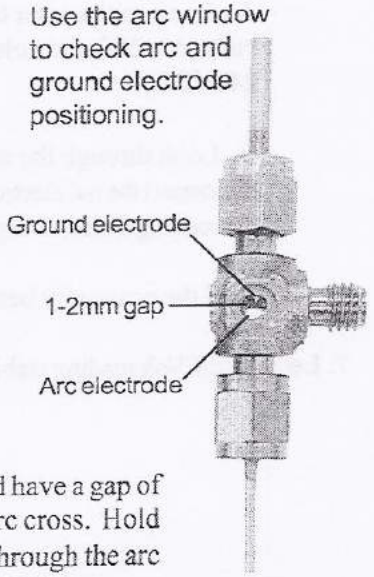
3. Remove the collector electrode by loosening the 1/4" fitting that secures it in the detector body.

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.

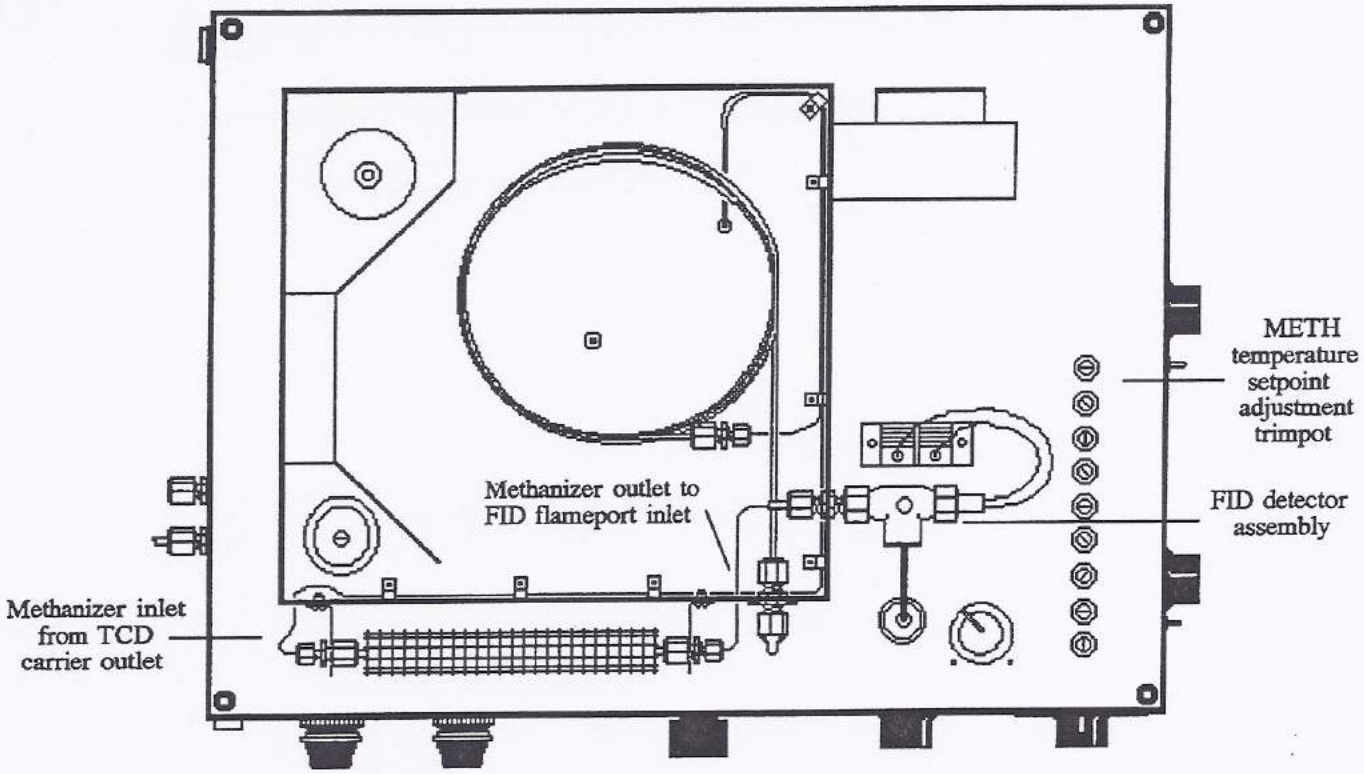


6. Replace the electrodes and check for proper alignment. The collector electrode should extend about 4mm into the detector body. An existing screw clamp stop on the collector electrode should allow replacement without readjustment. Should adjustment be required, loosen the screw clamp to position the 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.

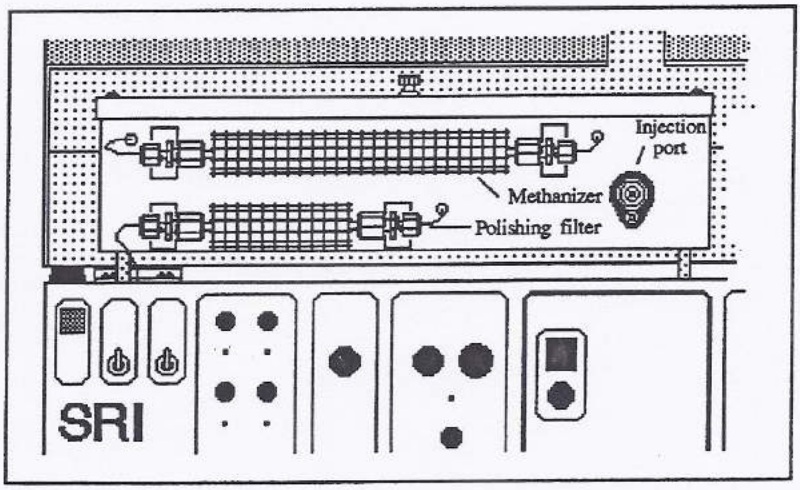


Chapter: DETECTORS

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

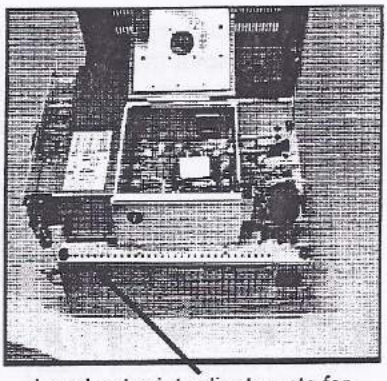
Carbon dioxide and carbon monoxide can be catalytically reduced to methane if passed through a nickel-packed trap heated to 375° C with the use of hydrogen either as carrier or make-up gas. Methane can be detected to 1ppm using the FID detector, permitting lower detection limts than obtainable with unmethanized 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).

Chapter: Injectors

Topic: EPC (electronic pressure control) operation

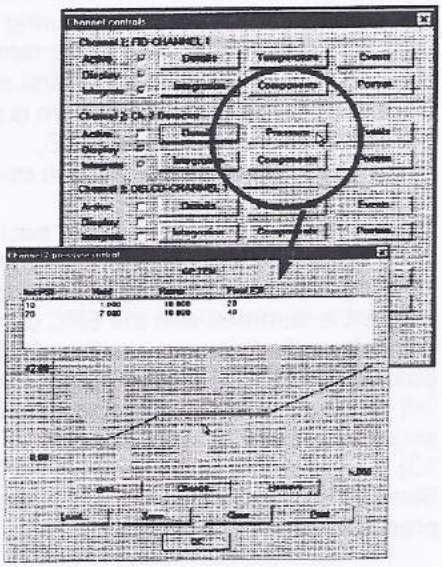
SRI GCs are equipped with electronic pressure control of all system gases. Detector support gases such as hydrogen and air are controlled by the screwdriver adjustable local setpoint on the GC, and once set are seldom altered. The carrier gas pressure may be controlled by either the local setpoint screwdriver 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:

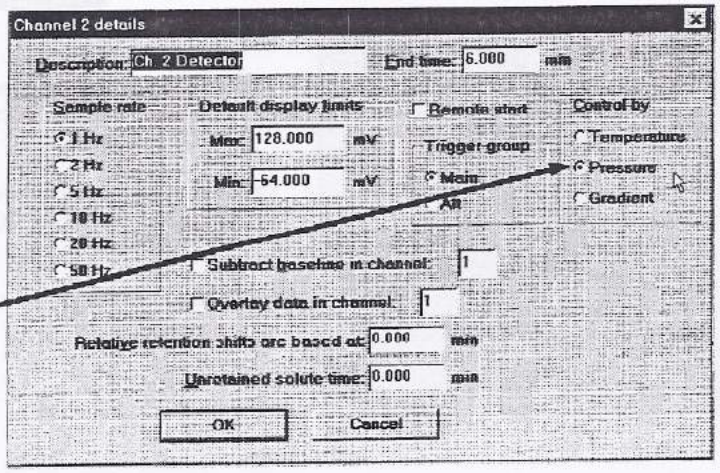
1) The screwdriver adjustment is simpler, and once set is not likely to be altered unintentionally.
2) The benefits of ramping the carrier gas pressure are often not worth the extra operational complexity.



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>

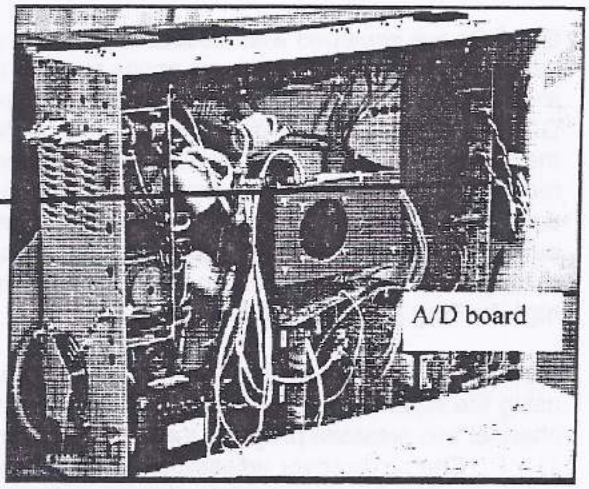
Set the "Control By" radio button in the Channel 2 Details screen to Pressure. Then enter the desired pressure program into Channel 2 by selecting the Pressure screen from the Edit/Channel menu

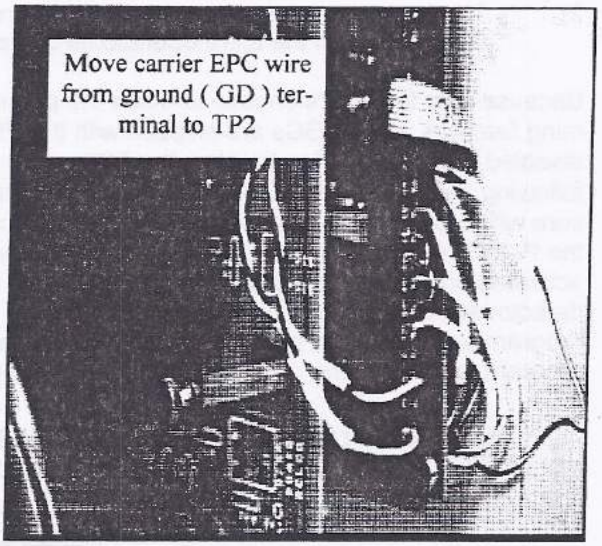


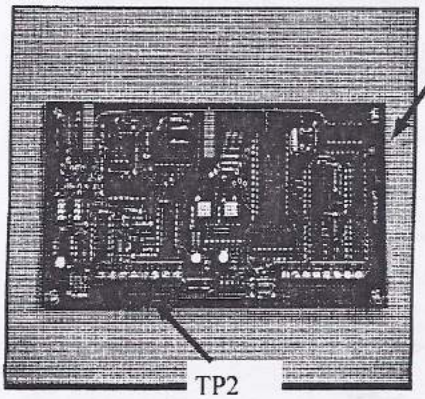
Chapter: Injectors

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.



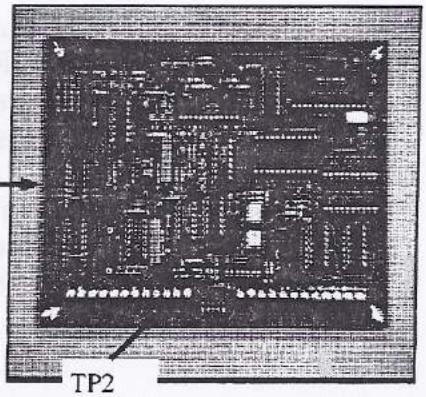




Some GCs will be equipped with the single channel Model 203 A/D board.

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

The procedure is identical on either board.



Chapter: Injectors

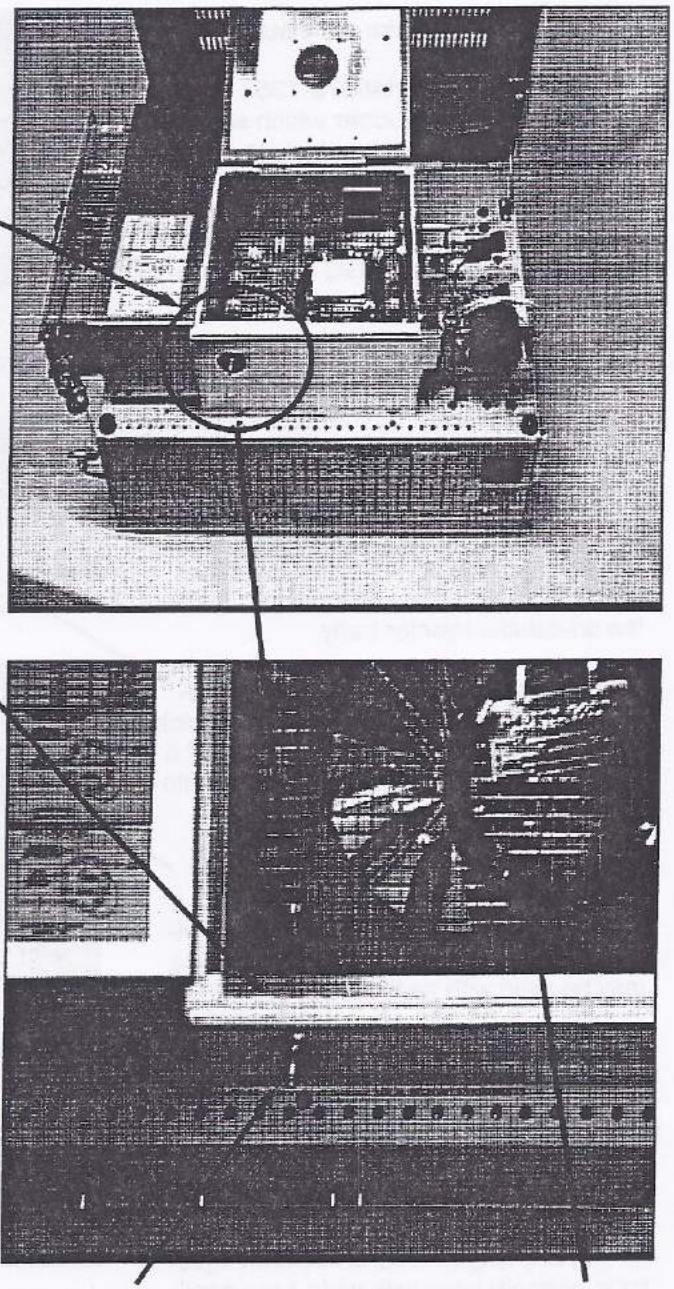
Topic: On-column Injector Operation

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

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.

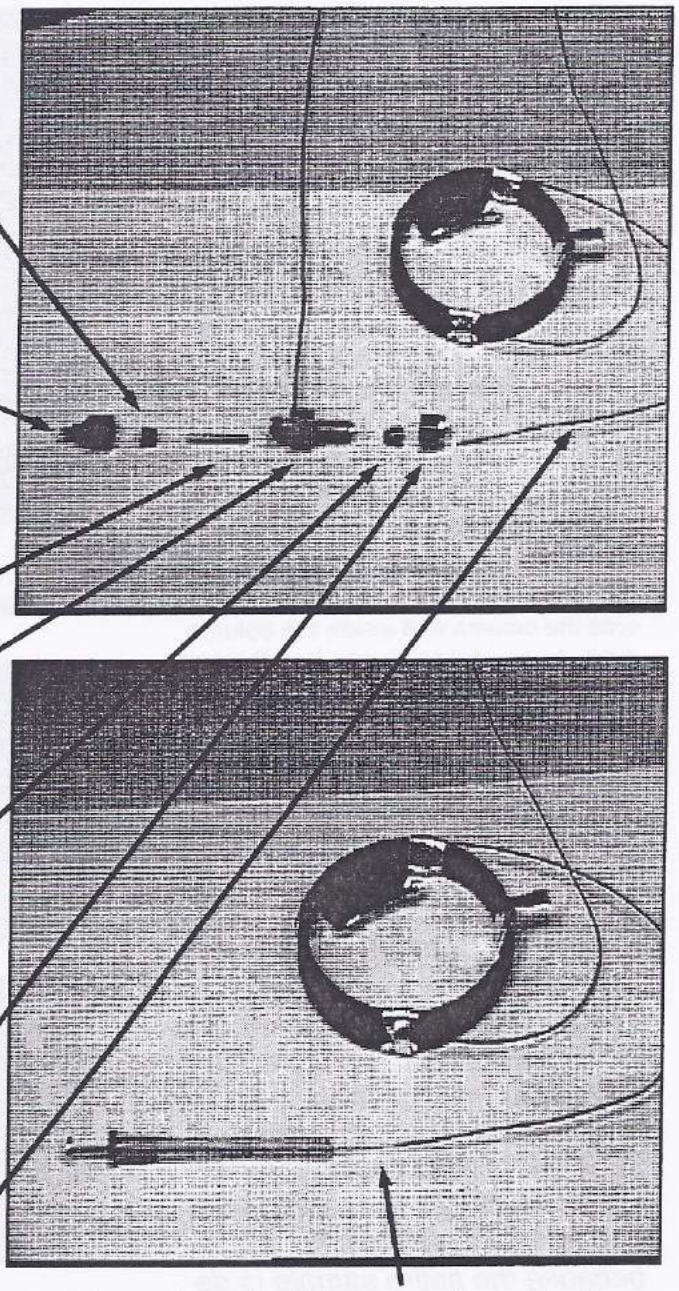
3) The wide-bore capillary column adapter (part#8690-9093) which aligns the syringe needle and the column inside the on-column injector body.

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

Chapter: Injectors

Topic: On-column Injector Operation

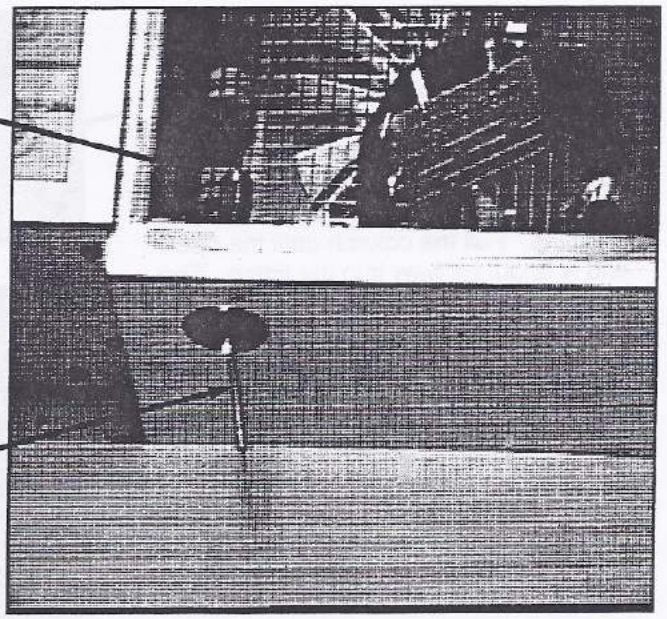
To install the column in the injector:

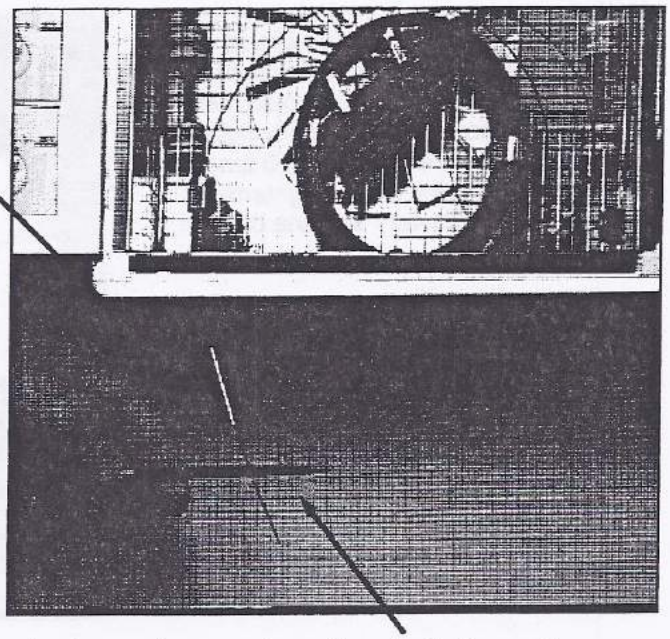
1) Feed the column end through the 1/8" swagelok type nut and graphite reducing ferrule. If the ferrule has been previously used, inspect it carefully to make sure it is still intact. Sometimes used ferrules will break inside the nut and a part of the ferrule will fall out. What's left inside the nut 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.

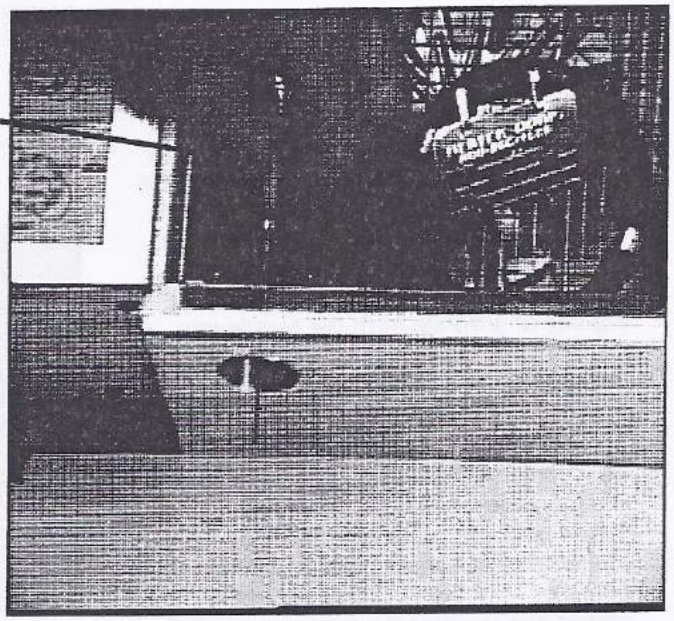
HINT: Some chromatographers use a small reamer (Dremel tool bit) to clean up and smooth the end of the metal capillary column bore hole. The smoother hole allows the syringe to enter the column with less chance of snagging on the lip of the column. The syringe itself should be in good condition with 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





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.

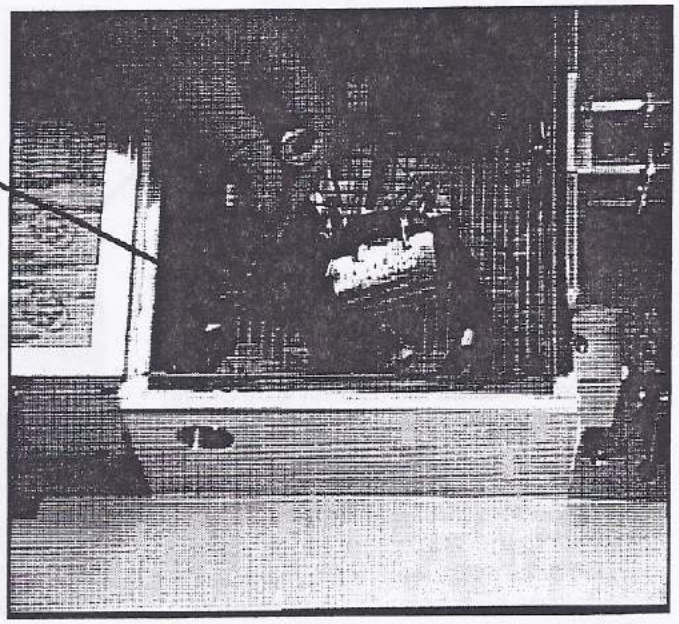
4) Pull the column and wide-bore adapter back into the injector fitting through the partially tightened nut and graphite ferrule. As you pull, the column will gradually disappear from view inside the injector fitting. Pull the column until the open end is about halfway into the fitting. The exact distance 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.

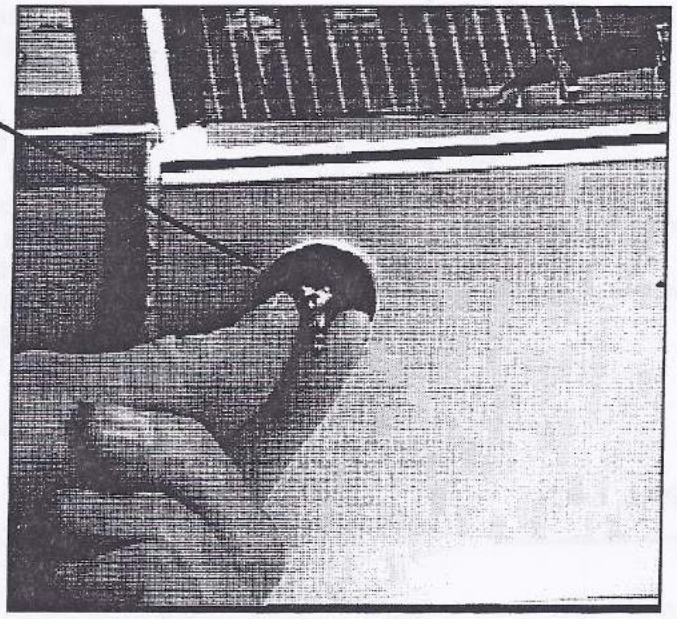


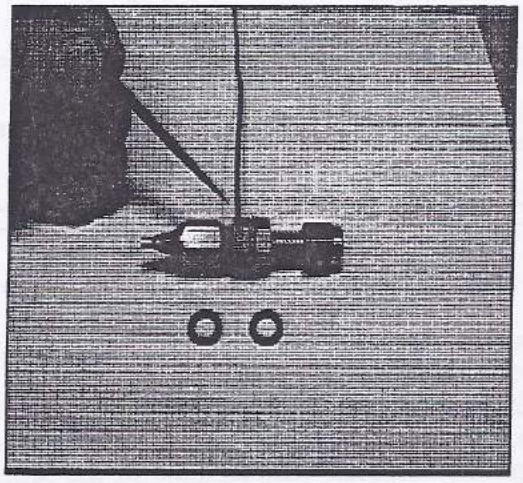
A 7/16" wrench is used to snug up the nut and graphite ferrule securing the column to the injector.

Chapter: Injectors

Topic: On-column Injector Operation

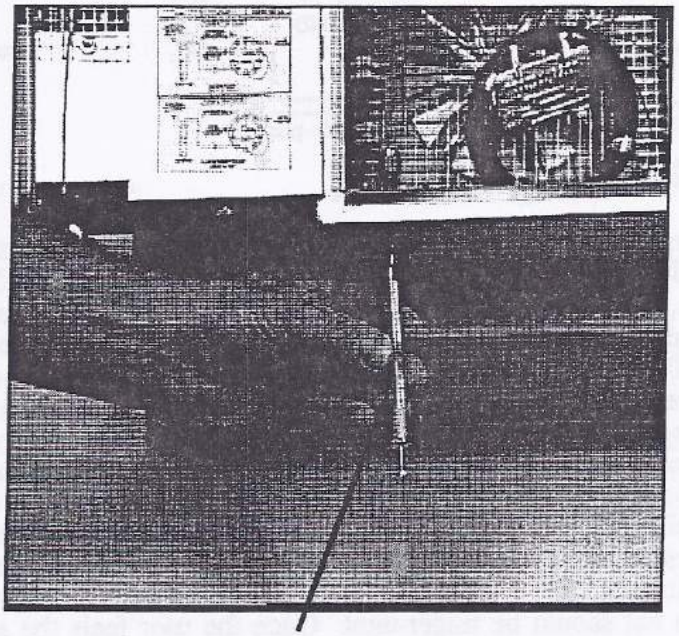
5) Tighten the septum nut until it contacts the one or two rubber o-rings on the injector body. The o-rings act as a helpful guide to avoid over-tightening the septum. When the soft silicone rubber of the septum is over-compressed, the syringe has to fight its way through often plugging with septum material 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.

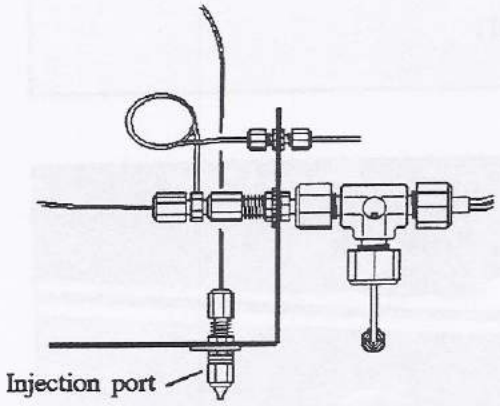
If the syringe snags on the edge of the column as it is inserted, loosen the swagelok nut and ferrule and pull the column another few millimeters further towards the inside of the column oven. Tighten the nut and retest by inserting the syringe.



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.

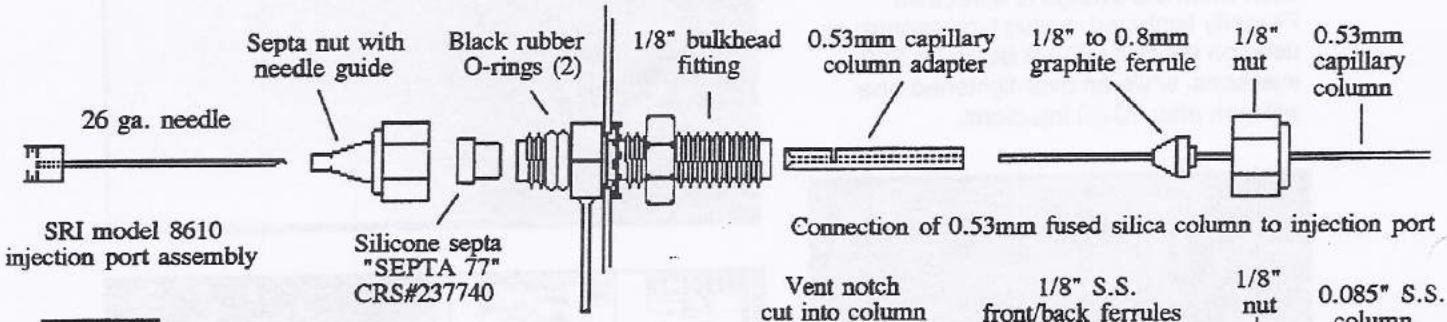
Chapter: INJECTORS AND GAS VALVES

Topic: INJECTION PORT

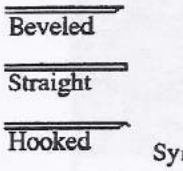


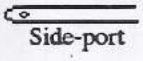
Location of injection port in typical FID system

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.

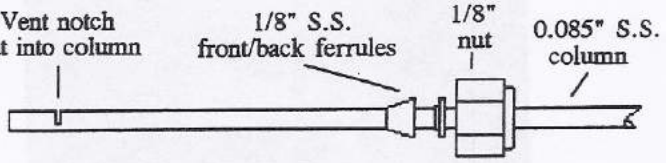


Connection of 0.53mm fused silica column to injection port





Syringe needle tip styles

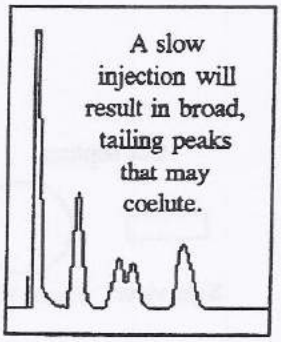


Connection of 0.085" I.D. stainless steel packed column to injection port

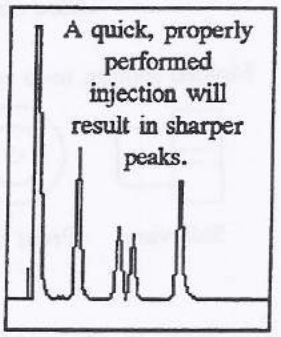
Therefore, they are recommended. "Coring" punches shards of septum into the injection port and may result in plugging of the syringe needle and failure to deliver sample. Over an extended period of time, these shards could migrate into the column. In a packed column, this accumulation of septum shards 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.

Chapter: INJECTORS AND GAS VALVES

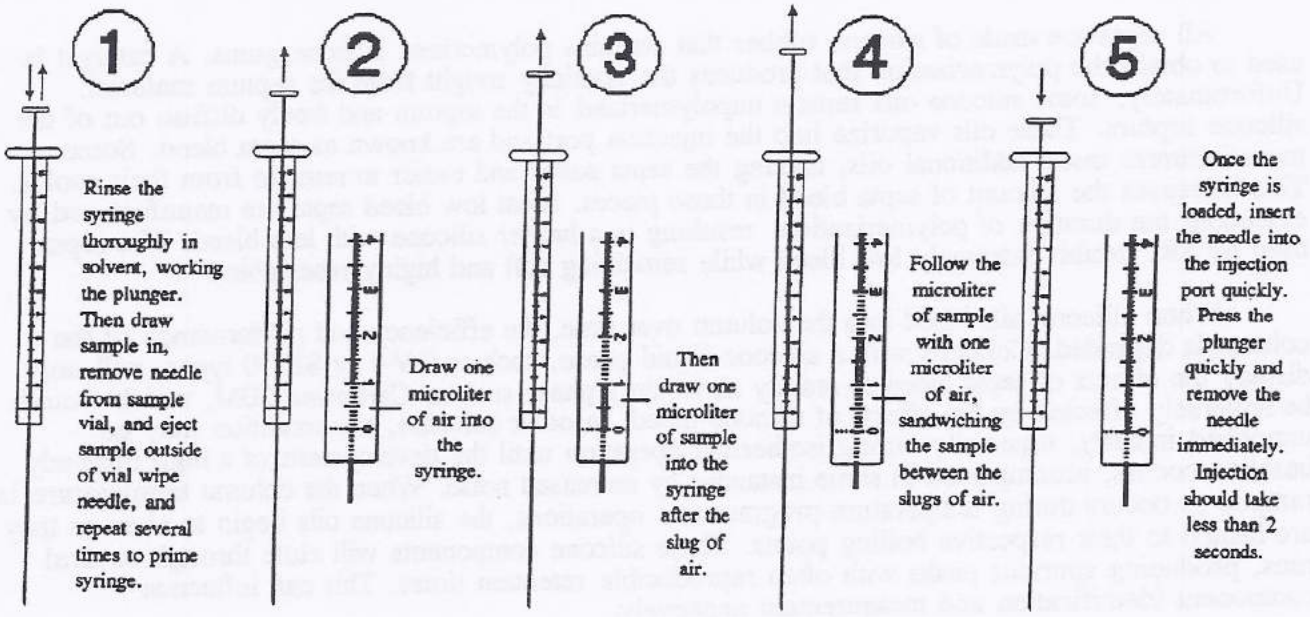
Topic: Manual Direct Injection Technique



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.

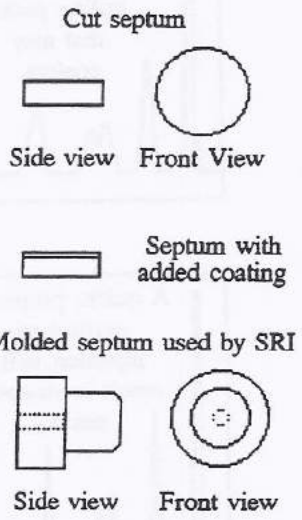


Properly prepared, the syringe needle is inserted completely into the injection port in one smooth, quick motion. Then the plunger is driven home immediately. As soon as the plunger tip hits the end of the sample chamber, the syringe needle is withdrawn from the injection port in a quick, smooth motion. 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.

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.

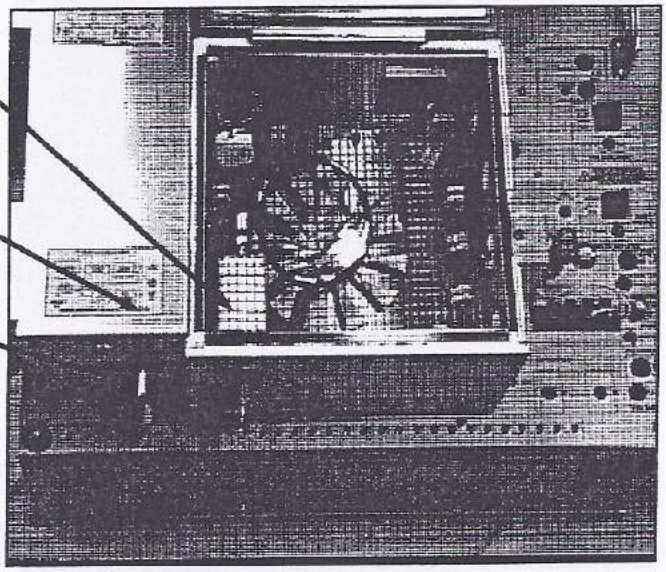
Chapter: Injectors

Topic: Heated Split/Splitless Injector

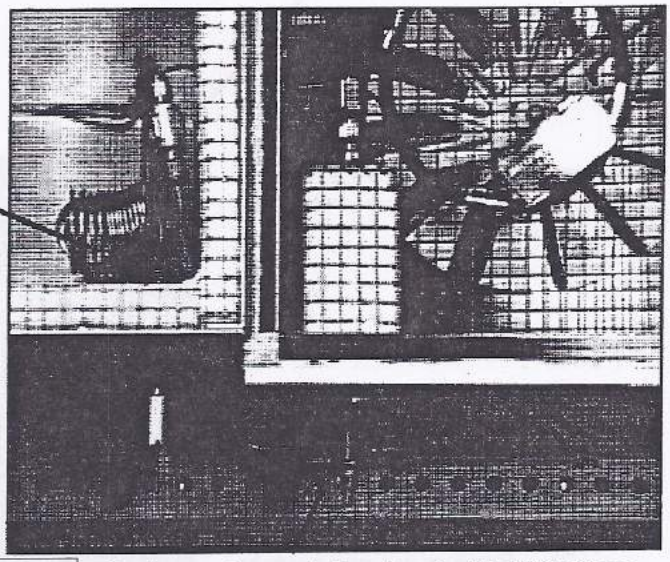
The Heated Split/Splitless Injector can be mounted on the 8610C or 310 GC. It is shown installed on the 8610C GC at right.

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.

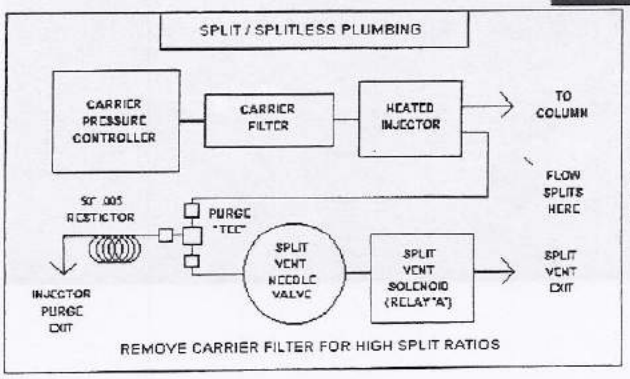
The split flow is adjusted by rotating this knob.



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



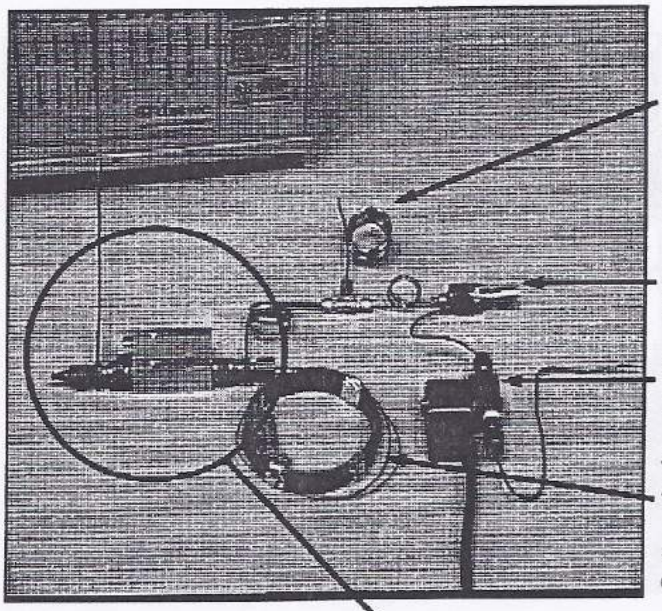
Septum nut mounted on front of Split/Splitless Injector.



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

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.

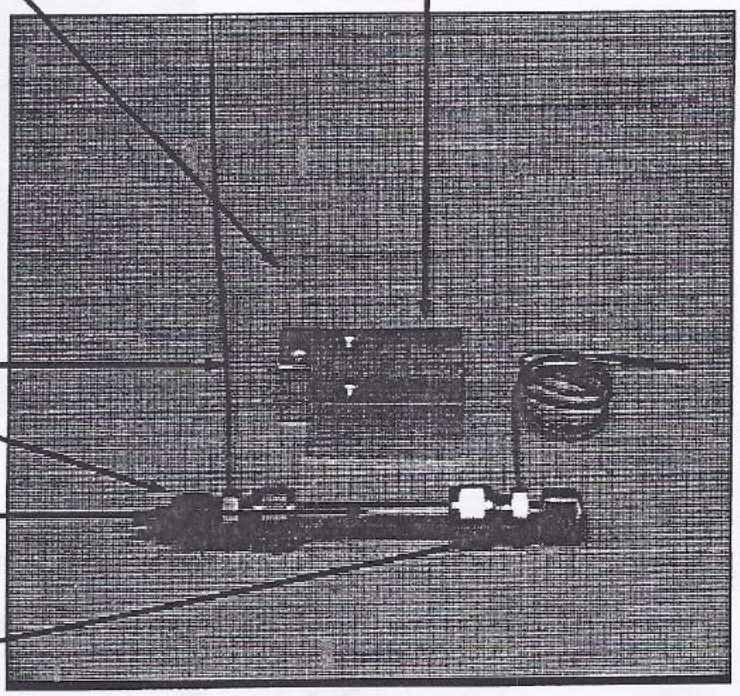
2) Precision needle valve for adjustment of split flow rate.

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.

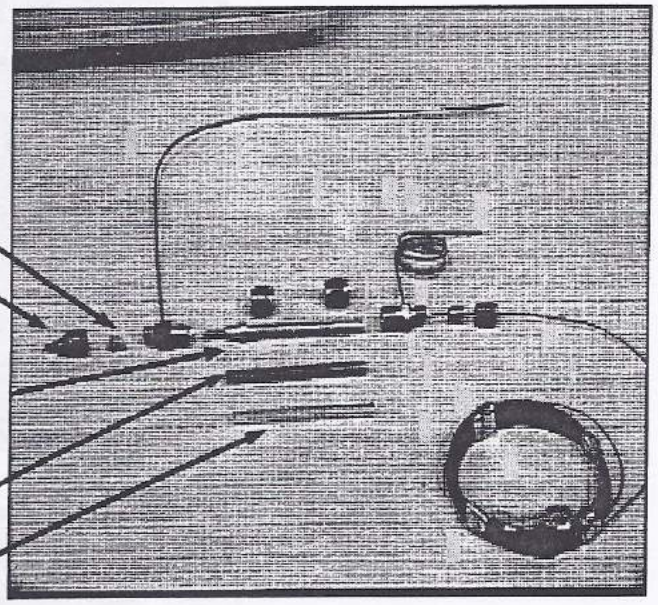
3) SRI stainless steel injector liner.

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

Chapter: Injectors

Topic: Heated Split/Splitless Injector

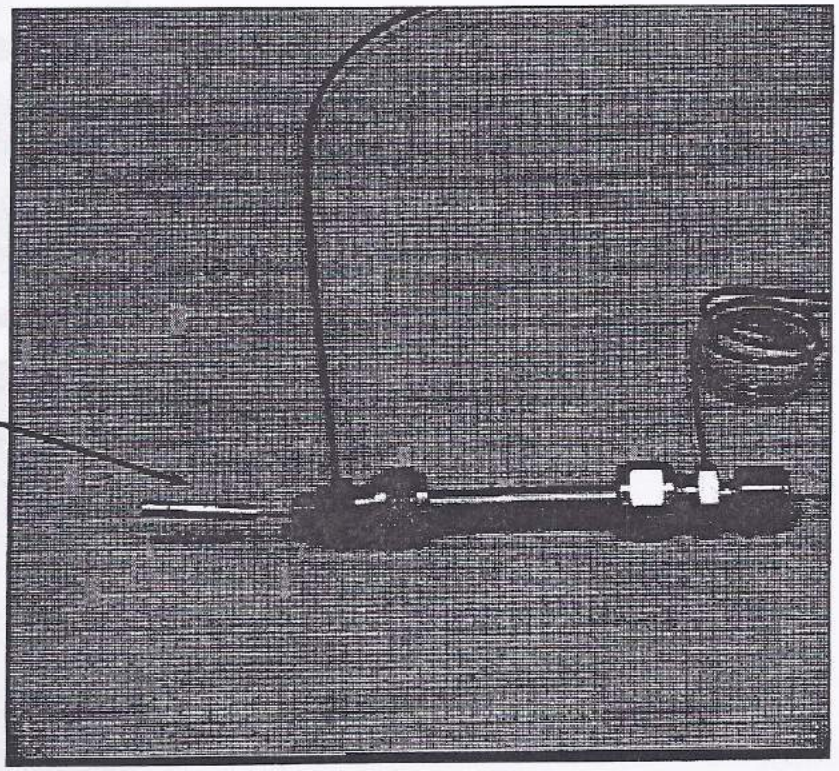
A variety of injector liners can be used with the Split/ Splitless injector depending on the column and application.



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).

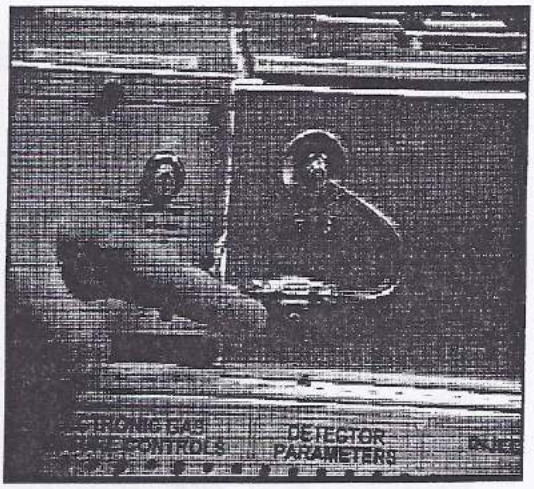
Wide-bore column (.53mm) adapter identical to that used in on-column injector fits perfectly into recess in stainless steel injector liner.



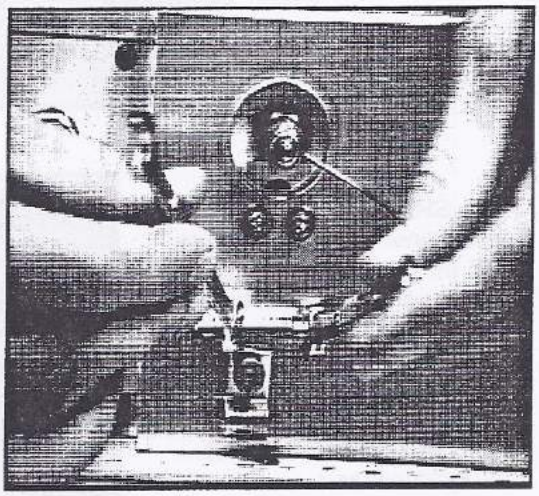
Chapter: Injectors

Topic: Heated Split/Splitless Injector

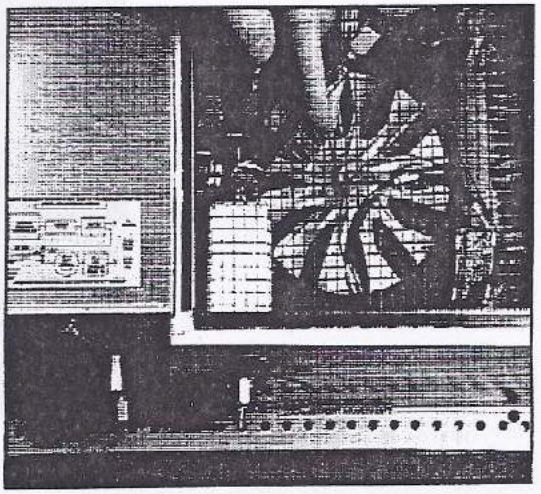
To remove the injector liner from the Split/Splitless injector:



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.



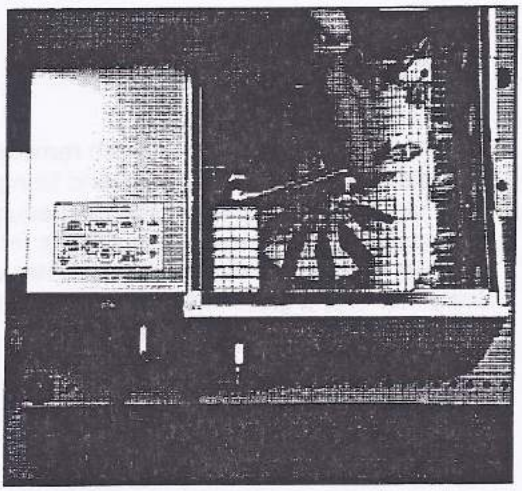
3) Using a 7/16" and 1/2" wrench, loosen the nut and graphite ferrule securing the column to the oven side of the injector.

Chapter: Injectors

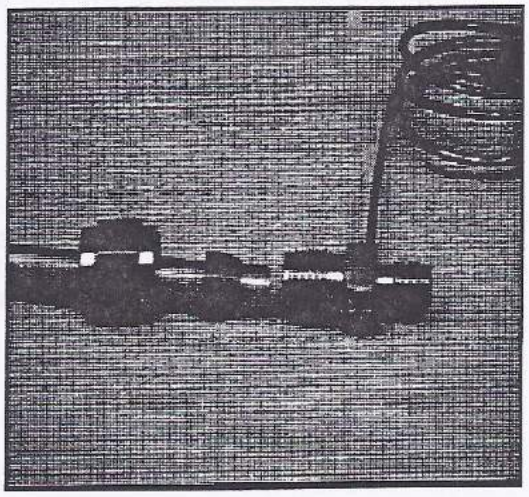
Topic: Heated Split/Splitless Injector

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

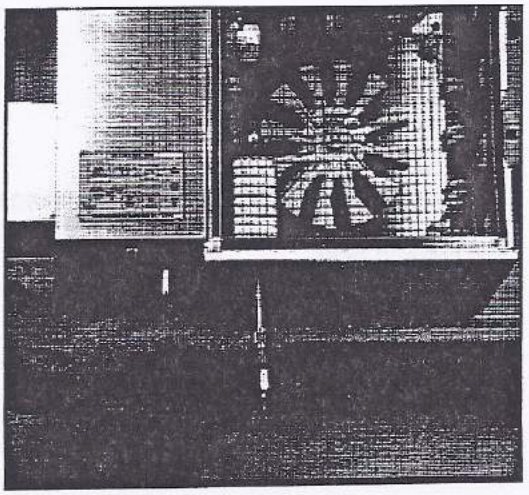
4) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting.



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.



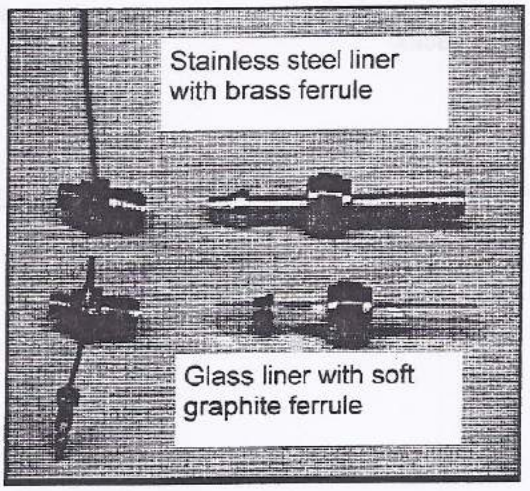
6) The injector liner and carrier inlet fitting can then be removed from the GC by pulling straight out towards the operator.



Chapter: Injectors

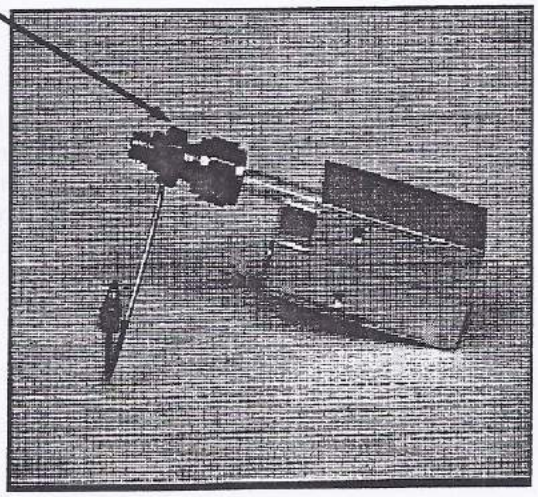
Topic: Heated Split/Splitless Injector

To replace the injection liner:

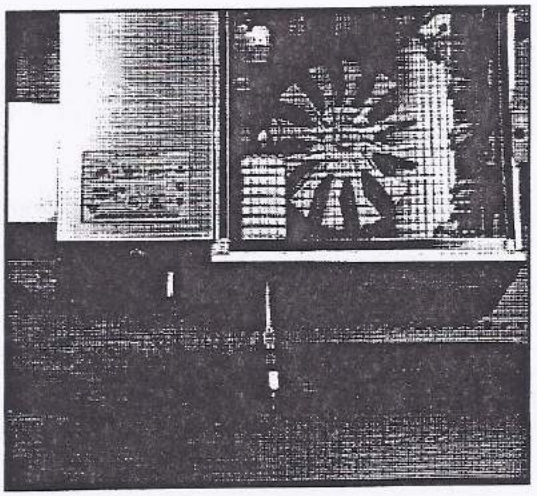


7) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting. The stainless steel liner provided as standard equipment with the split/splitless injector uses a brass ferrule on the septum end of the liner, but if you replace the stainless liner with a glass liner, you will need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

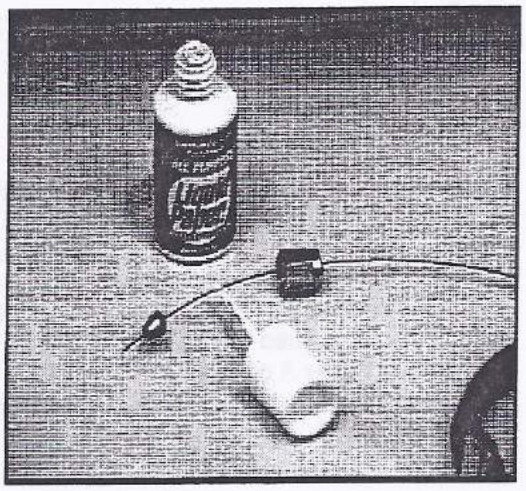


9) The injector liner and carrier inlet fitting can then be installed into the GC by sliding straight in towards the column oven

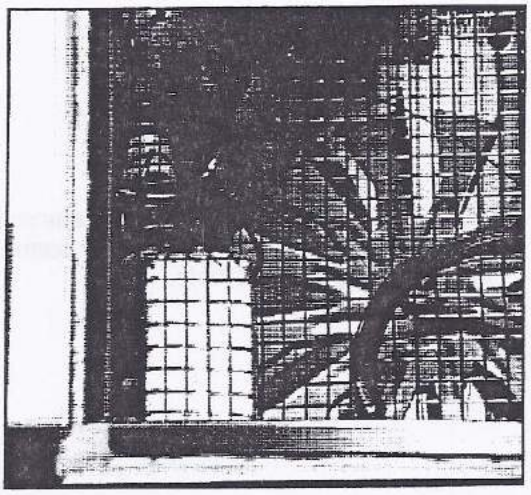
Chapter: Injectors

Topic: Heated Split/Splitless Injector

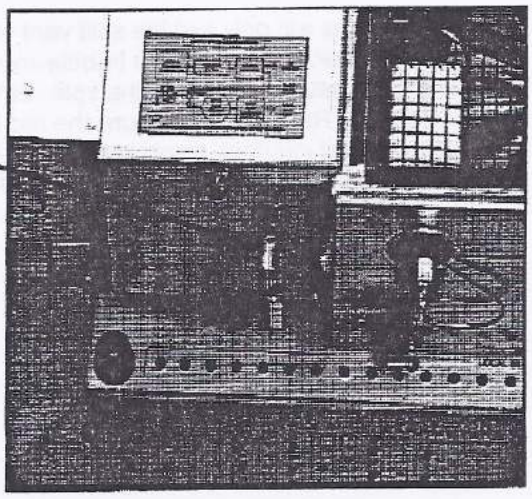
To install a narrow bore (.25mm) capillary column:



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.

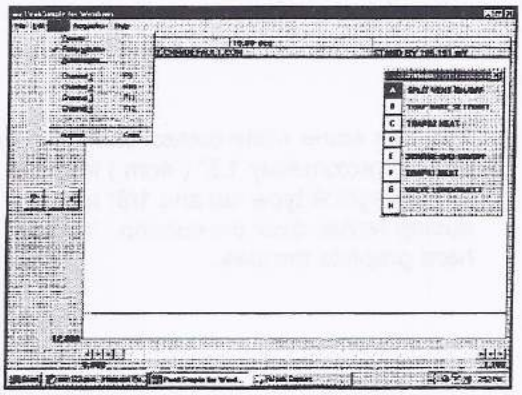


12) Adjust the split flow rate using the needle valve located on the front of the valve oven.

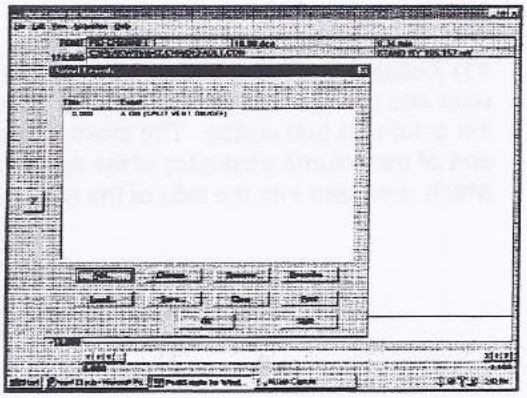
Chapter: Injectors

Topic: Heated Split/Splitless Injector

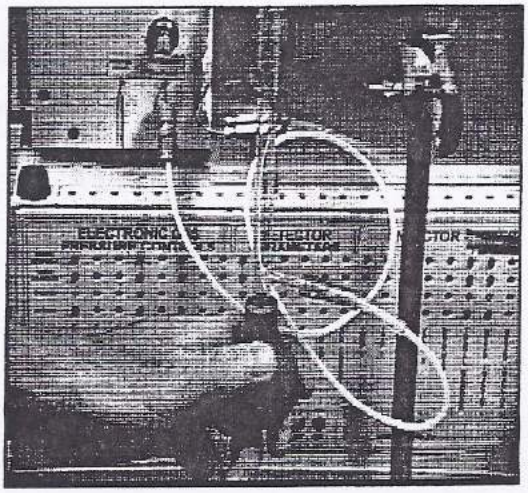
To install a narrow bore (.25mm) capillary column:



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.

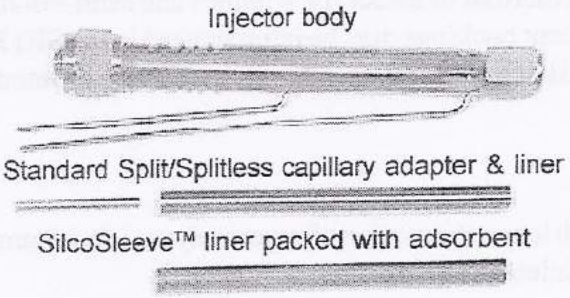


14) The relay can be turned on/off automatically during an analysis by entering the relay commands in the PeakSimple event table.



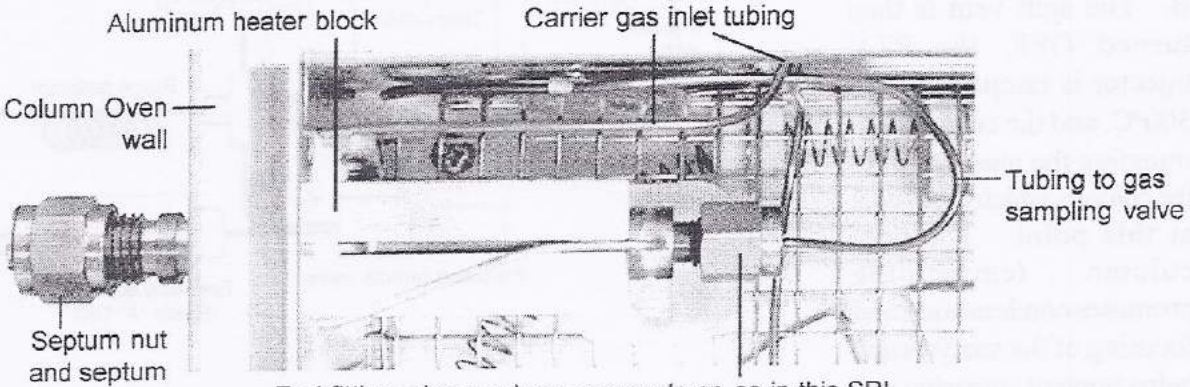
15) Carrier gas will only exit the split vent when Relay A is activated. Connect your bubble-meter or other flow measuring device to the split vent exit tube. Activate Relay A. Make sure the red lid of the GC is down (lid interlock disables solenoid function). Adjust the needle valve to obtain desired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

Overview

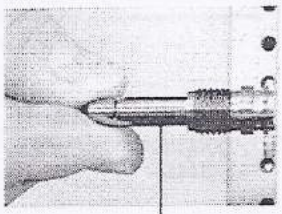


PTV and Split/Splitless injector components

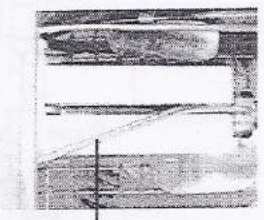
The Programmed Temperature Vaporization (PTV) injector is composed of the same parts as the Heated Split/Splitless injector: the injector body, a SilcoSleeve™ liner, an injector purge restrictor, a precision needle valve for adjustment of split flow rate, a split flow solenoid that turns on & off from 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

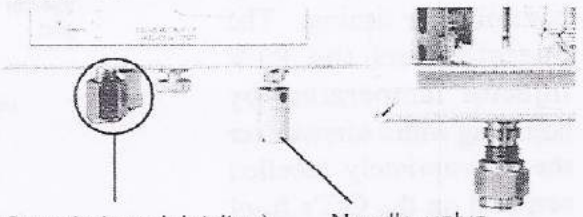


Tenax-GR™ packed SilcoSleeve™ liner, partially slid out for visibility



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

INJECTORS
PTV - Programmed Temperature Vaporization Injector

Theory of Operation

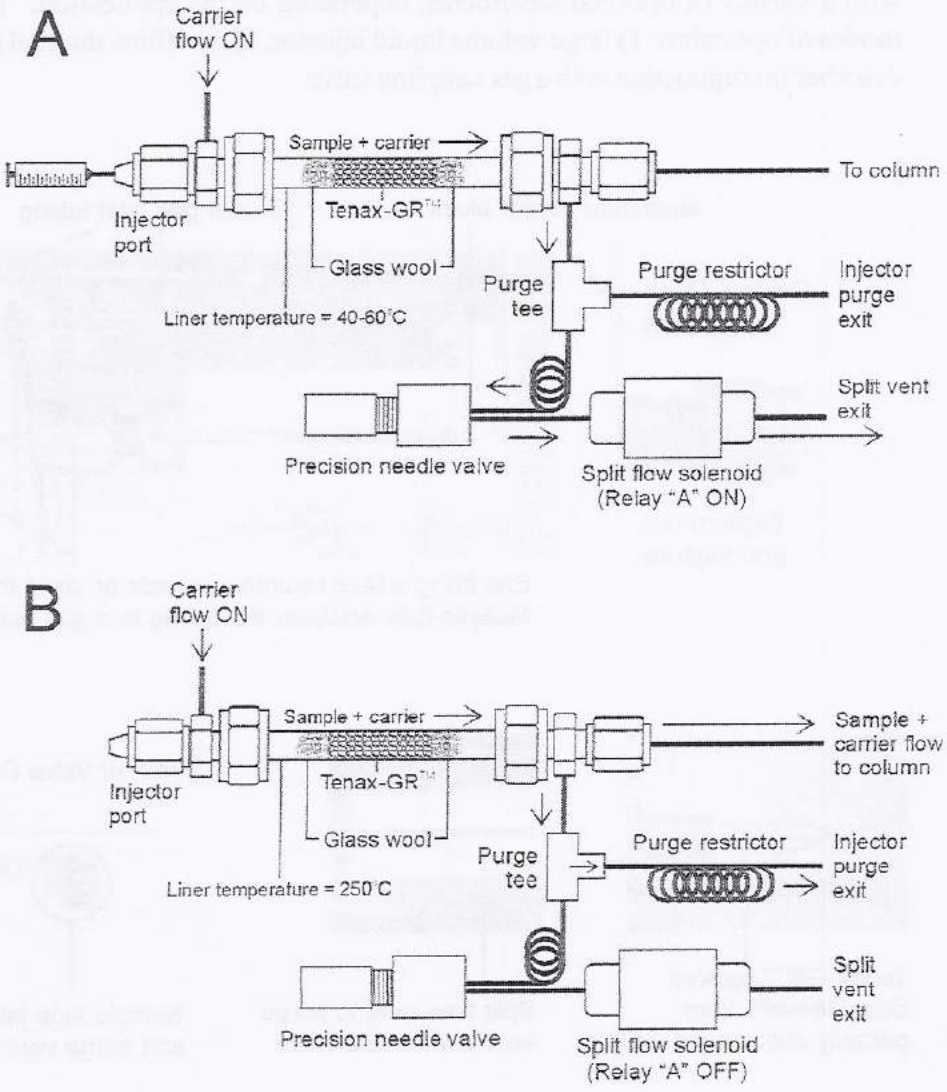
The Programmed Temperature Vaporization injector is basically a Heated Split/Splitless injector with the ability to rapidly heat to 300°C. This ballistic heating capability enables large volume liquid sample injections. The PTV injector can be used as a thermal desorber for volatiles and semi-volatiles, 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.

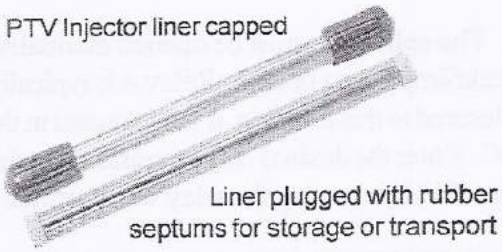
B. The split vent is then turned OFF, the PTV injector is ramped to 200-300°C, and the carrier flow transfers the analytes onto the column, which is still cool at this point. The cool column temperature promotes condensation and focusing of the analytes and helps prevent smearing and excessive tailing. 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.

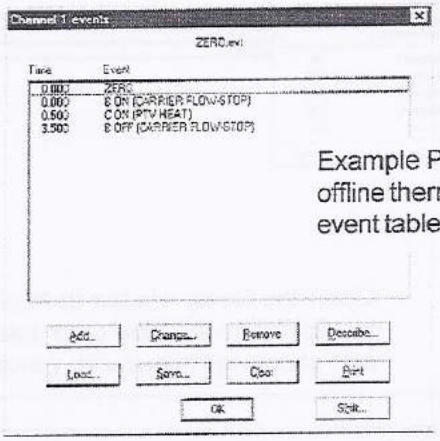


Theory of Operation continued

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.

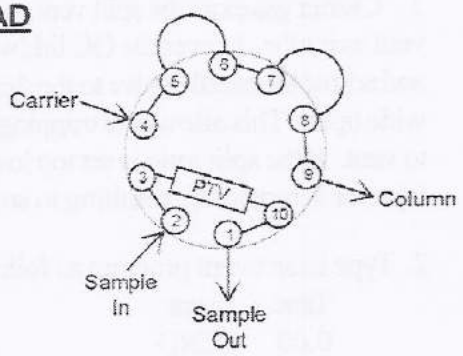
LOAD Position:
(Relay "G" OFF)

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.

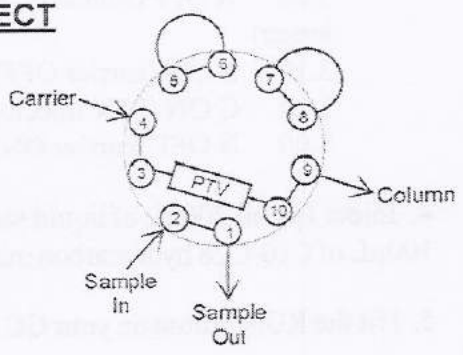
INJECT Position:
(Relay "G" ON)

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

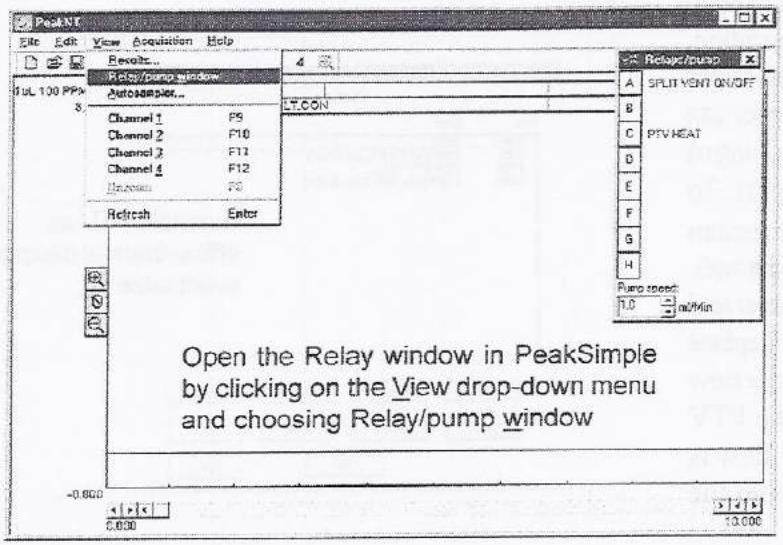


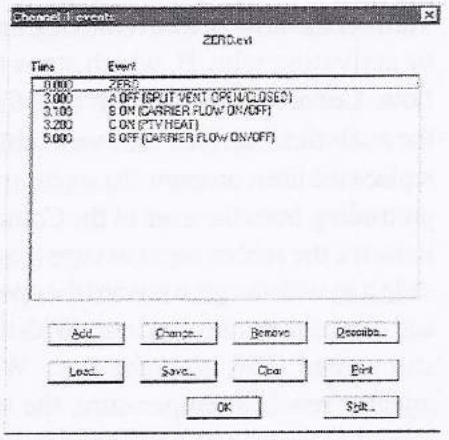
INJECT



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:

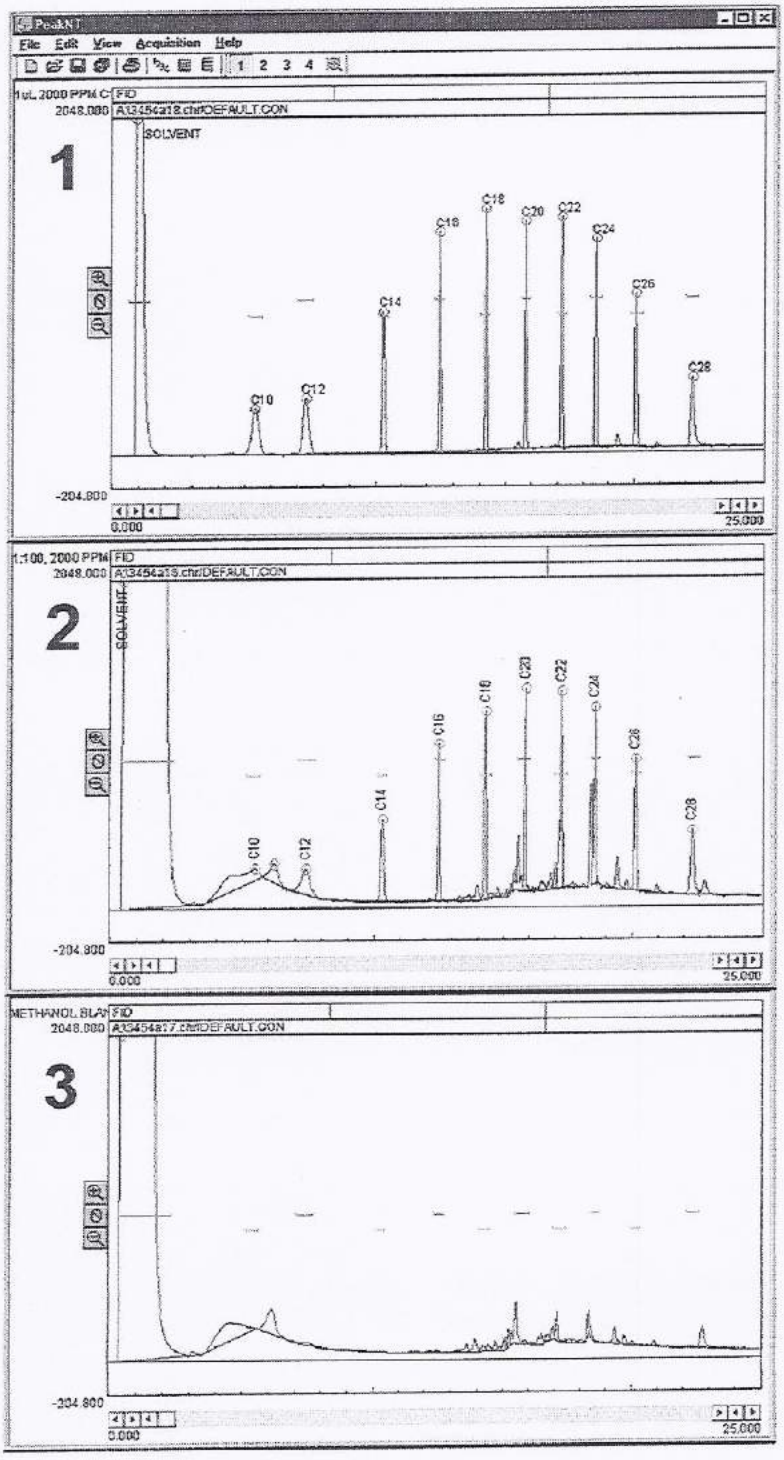
Time	Event
0.00	ZERO
3.00	A OFF (split vent closed; if you get too large a solvent peak, keep the split vent open longer)
3.10	B ON (carrier OFF)
3.20	C ON (PTV injector heat)
5.00	B OFF (carrier ON)

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.

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	4600.0300
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

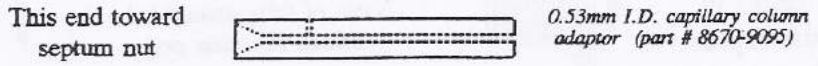
Temperature program:

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

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.

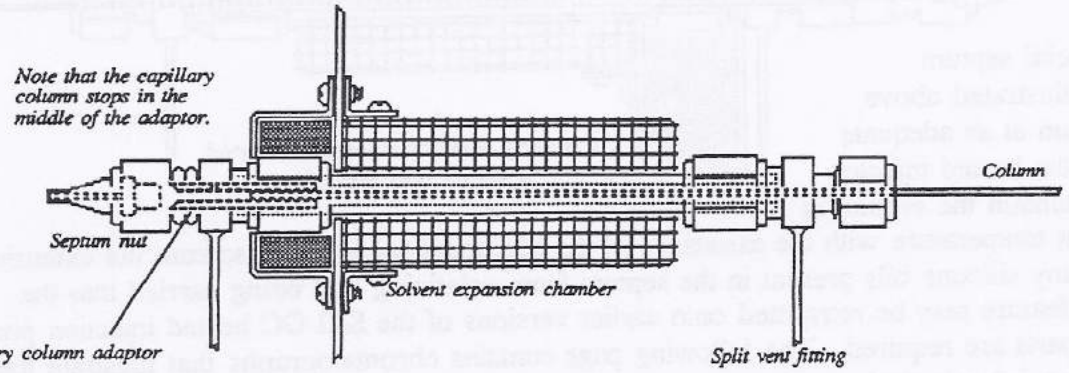
Use the toggle switch to select the flow controller to regulate carrier gas.

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.

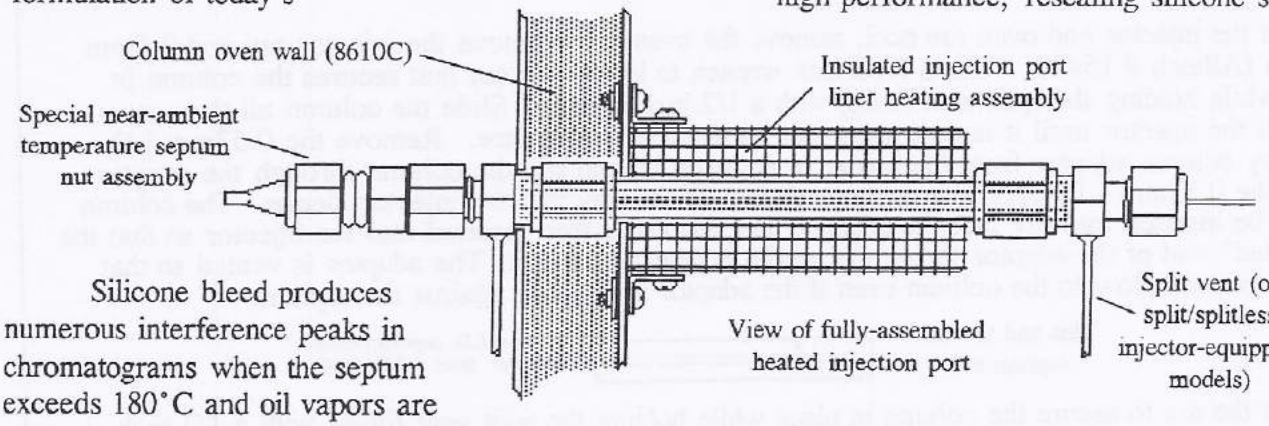
CORRECT PLACEMENT OF COLUMN AND CAPILLARY ADAPTOR FOR COLD ON-COLUMN MODE:



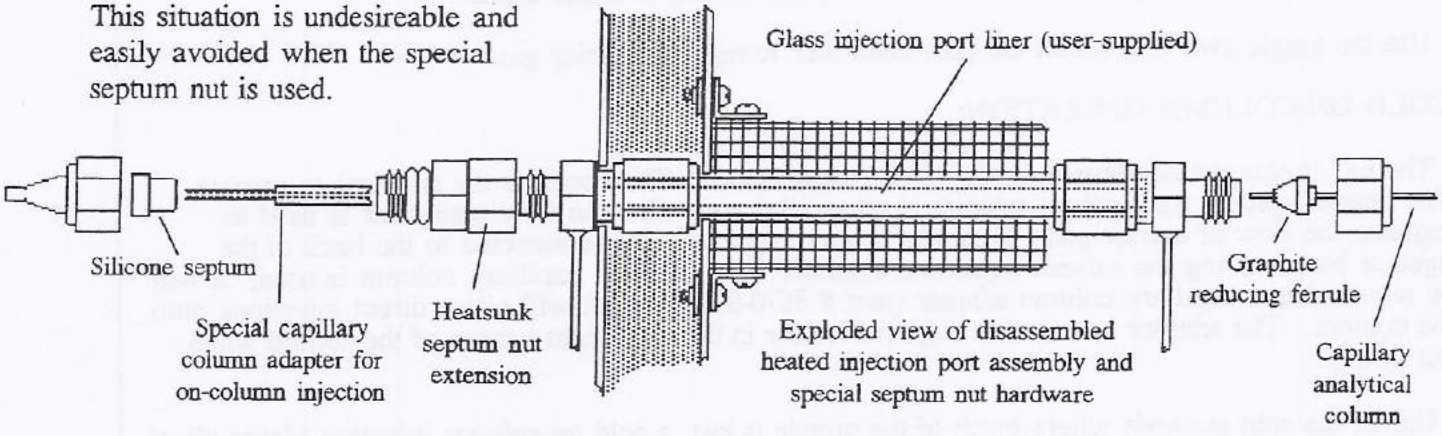
Chapter: INJECTORS AND GAS VALVES

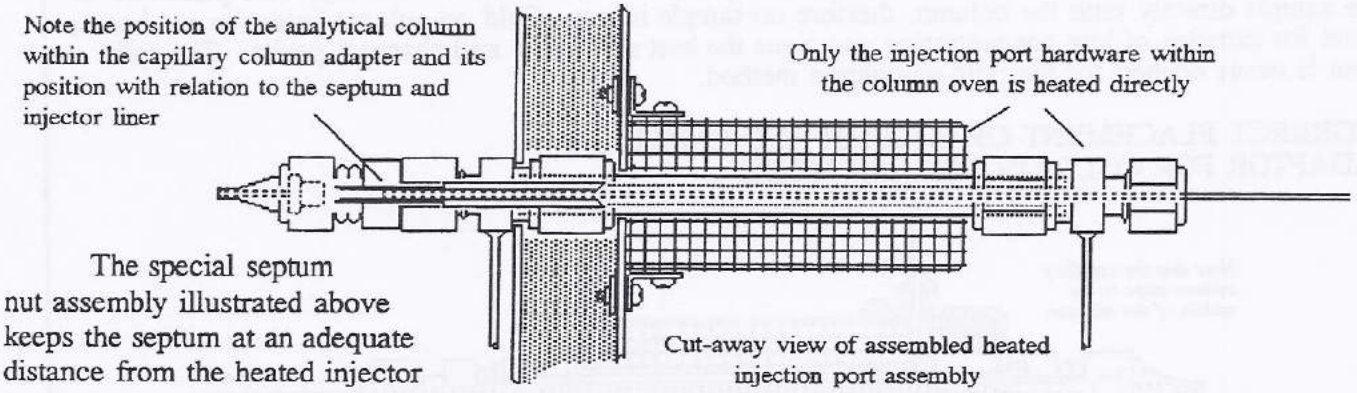
Topic: Near-Ambient Temperature Septum Nut Assembly For Heated Injectors

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.



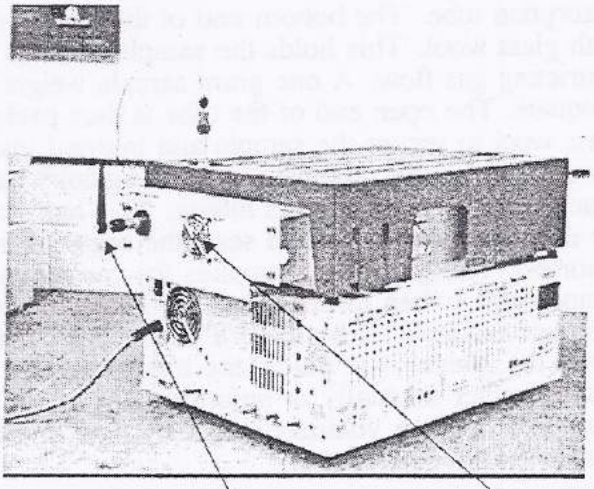


The special septum nut assembly illustrated above keeps the septum at an adequate distance from the heated injector assembly to maintain the septum at or near ambient temperature with the assistance of the additional mass of the septum nut extension. This prevents any silicone oils present in the septum 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

Chapter: INJECTORS

Topic: THERMAL DESORBER OPERATION

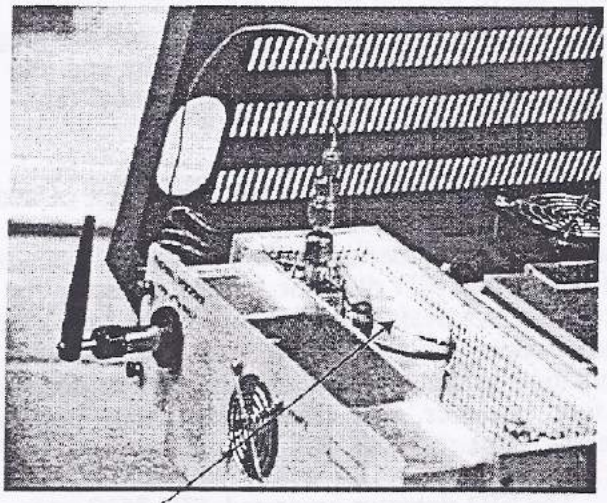
The SRI Thermal Soil Desorber accessory is useful for the analysis of volatile and especially semi-volatile compounds in soil or other granular solids. Because the analyte can be extracted from the soil by heat alone, with little or no sample preparation, field analyses can be performed without liquid 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.

Chapter: INJECTORS

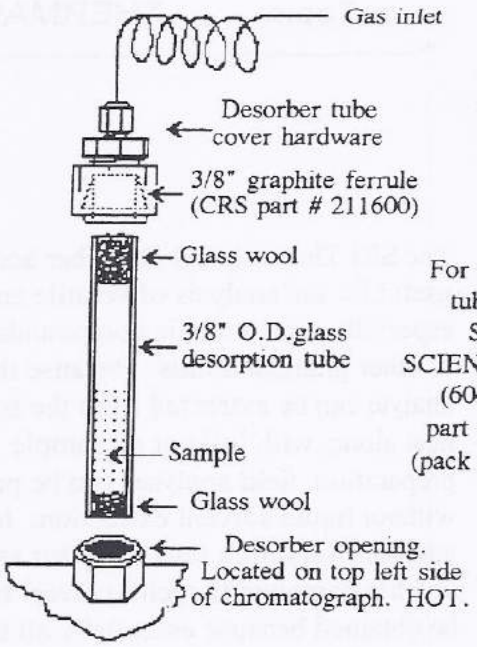
Topic: THERMAL DESORPTION

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.

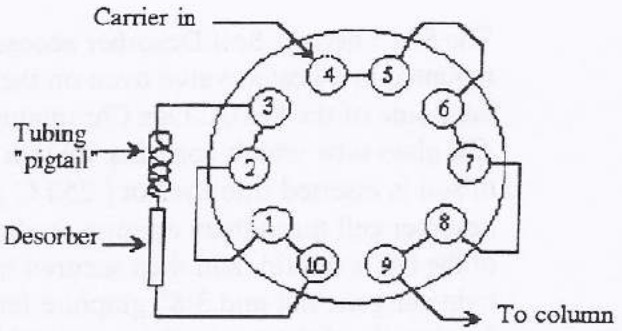
In the past, direct thermal desorption of average soil samples had been difficult due to the massive amounts of water liberated. This tended to extinguish the flame of the FID detector (typical detector for hydrocarbon analysis). Water elutes along with the early gasoline components and may interfere 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.

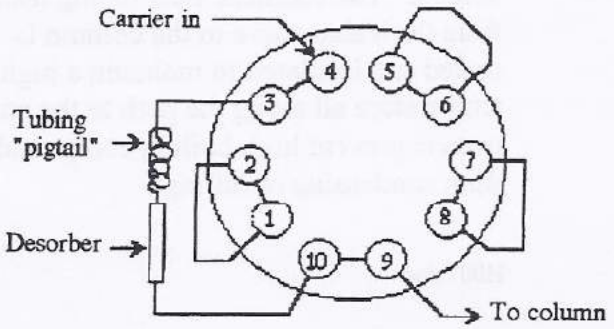


THERMAL DESORBER DETAIL

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

Chapter: INJECTORS

Topic: OPERATION OF THE THERMAL DESORBER

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

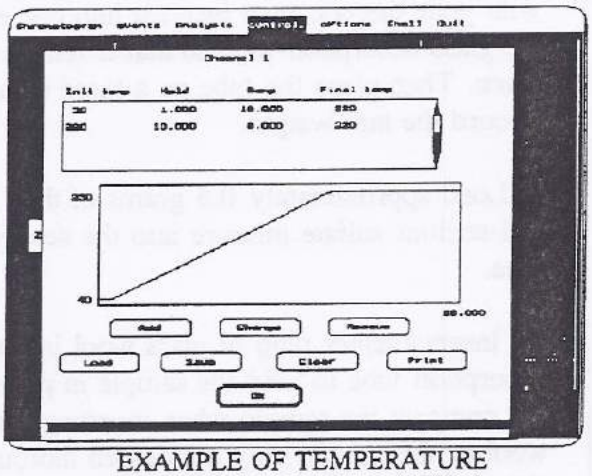
1. Place a clean desorption tube with a glass wool plug at one end on a scale of known accuracy. The tare weight is obtained. This is done by either weighing the clean, empty tube and recording the weight, or by placing the tube on the weighing platform and zeroing the balance.
2. Load the sample into 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.

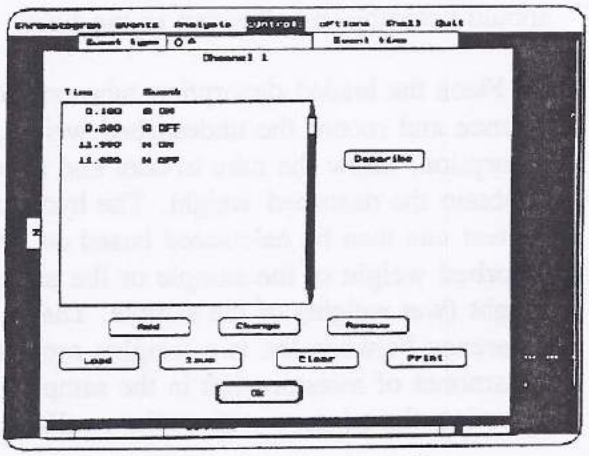
The column is connected to the injection valve inside the valve oven using a 1/16" to 1/8" adapter and 0.040" I.D. stainless steel tubing (1/16" O.D.). This ensures a uniform sample temperature while en route to the column and eliminates any possible cold spots.

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.

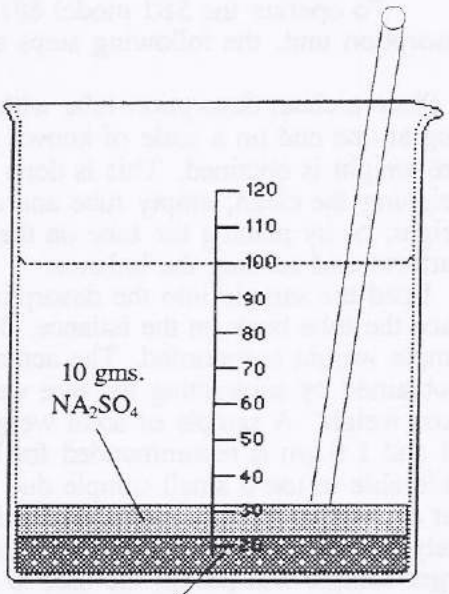
2) Add 10 grams of granular sodium sulfate (Na_2SO_4) to the beaker and mix with a stirring rod or spatula. The granular sodium sulfate, when mixed with the soil, absorbs most of the moisture from the soil, allowing clay soils to be ground into smaller particles. This is important because dense clay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

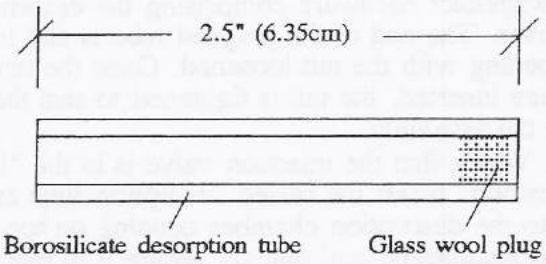
3) Roll a small amount of glass wool into a ball with your fingers, then insert it into one end of the glass desorption tube so that it remains in place. Then place the tube on a tared balance. Record the tare weight.

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

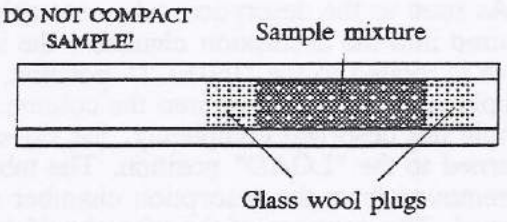


Diagram of assembled sample desorption tube containing 0.5 gms of soil - Na_2SO_4 sample mixture