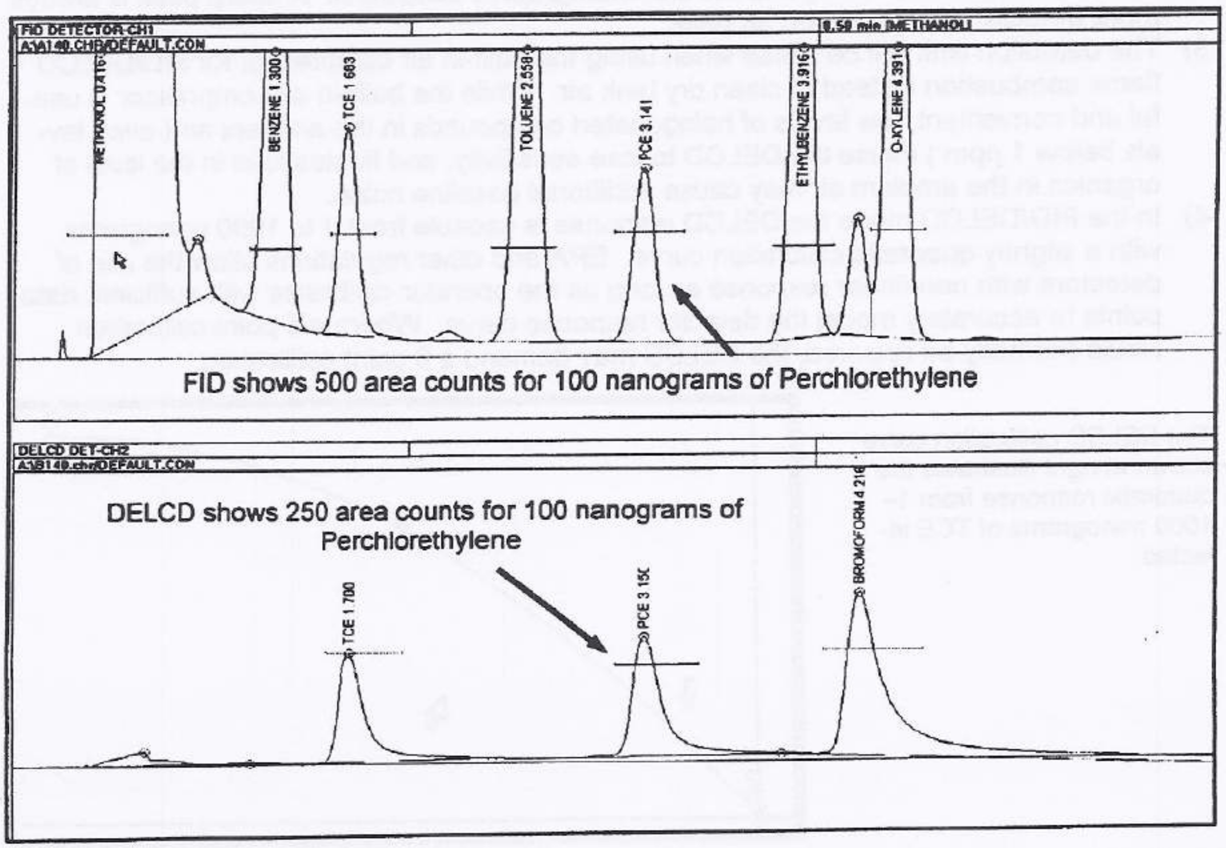
Chapter: Detectors

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).

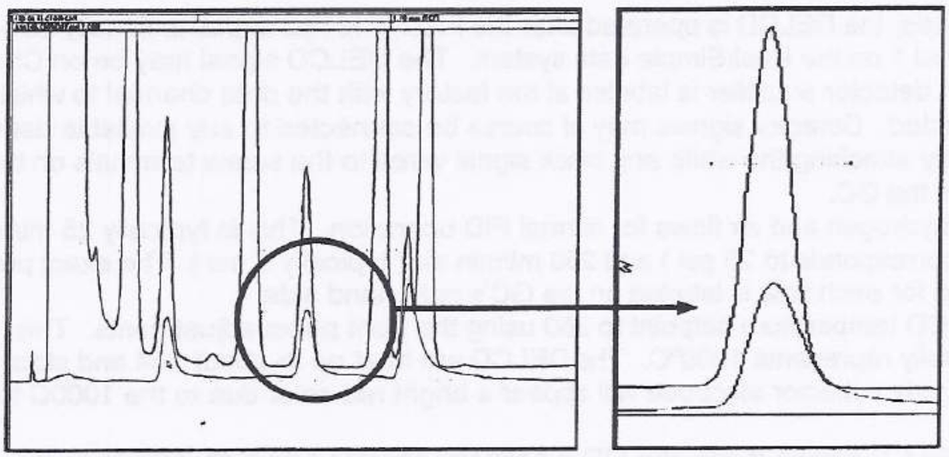


FID shows 500 area counts for 100 nanograms of Perchlorethylene

DELCD shows 250 area counts for 100 nanograms of Perchlorethylene

Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

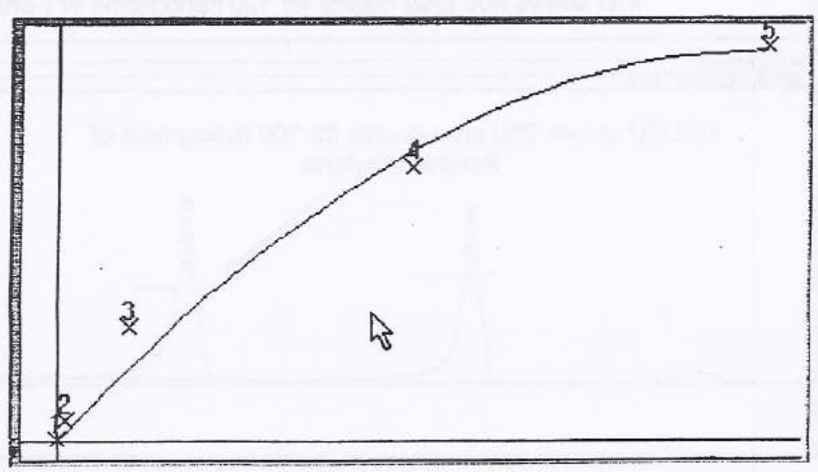


DELCD peak overlaid on FID peak for PCE, then expanded for clarity.

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



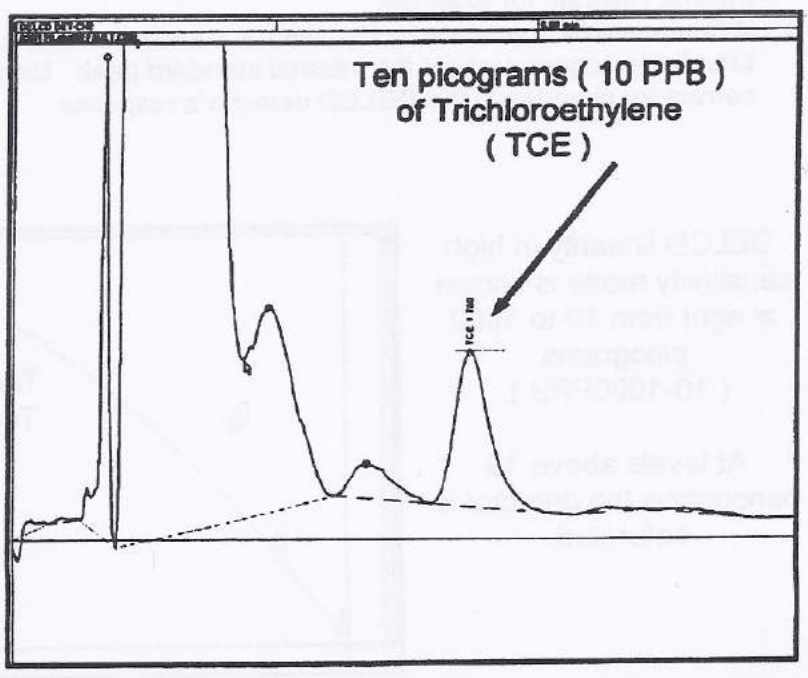
Chapter: Detectors

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 ClO_2-BrO_2. Because the chlorine/bromine atoms prefer to react with hydrogen to form non-detectable HCl-Hbr, than with oxygen to form detectable ClO_2-BrO_2 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 H_2O 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.

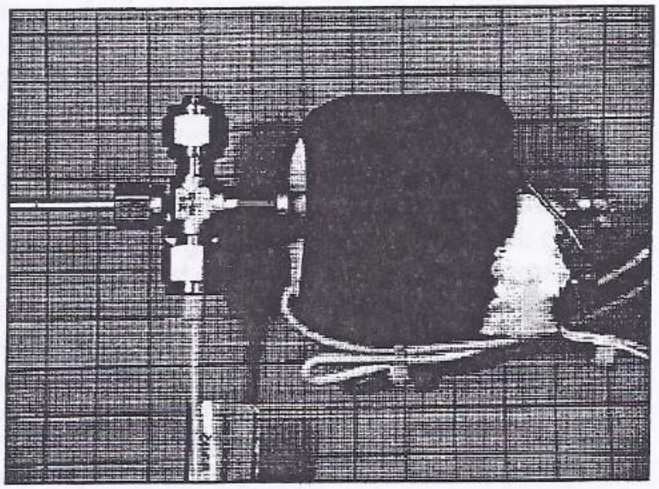


Chapter: Detectors

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.

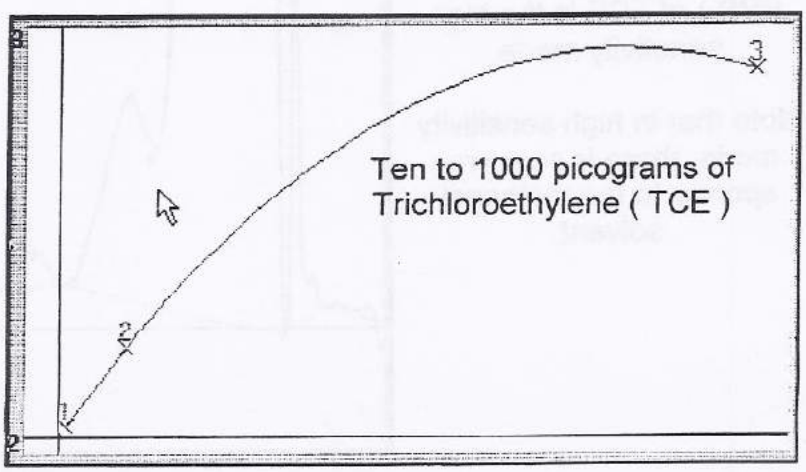
The collector electrode is removed and a 1/4" cap installed instead.



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).

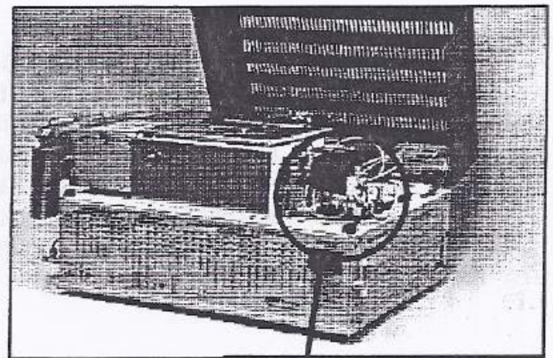
At levels above 10 nanograms the detector is saturated.



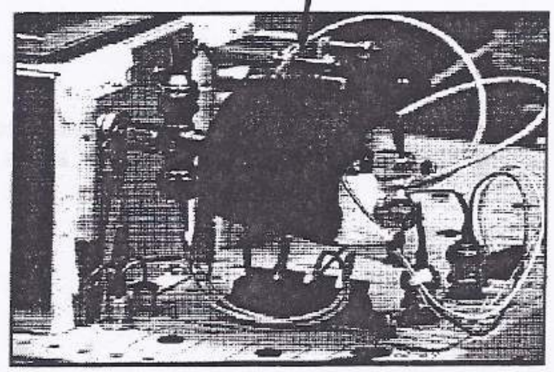
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

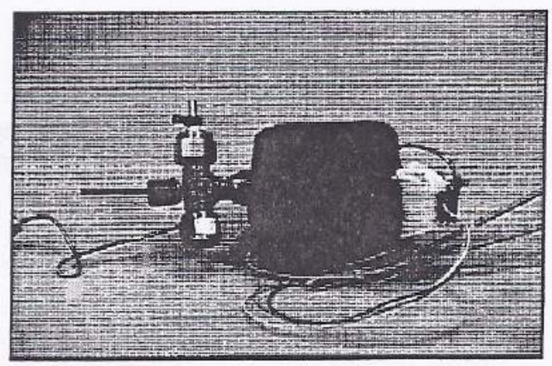
The DELCD detector is only available in combination with the FID detector.



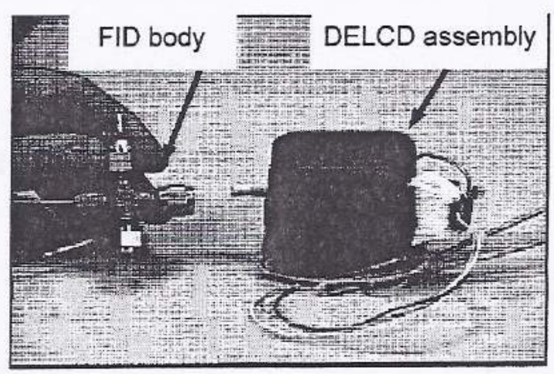
The FID/DELCD combo detector is mounted to a thermostatted aluminum heater block on the right hand side of the column oven.



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



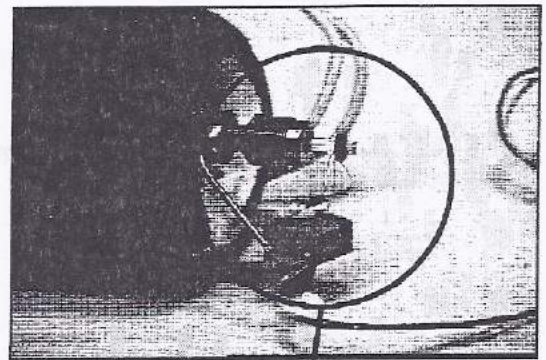
The DELCD part of the detector is the large black cylinder which mounts into the right hand port of the FID detector body. It can be separated from the FID body by loosening the 1/4" Swagelok nut and graphite ferrule which secures it in place.

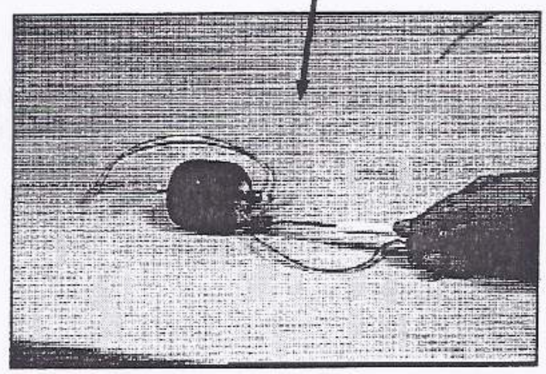


Chapter: Detectors

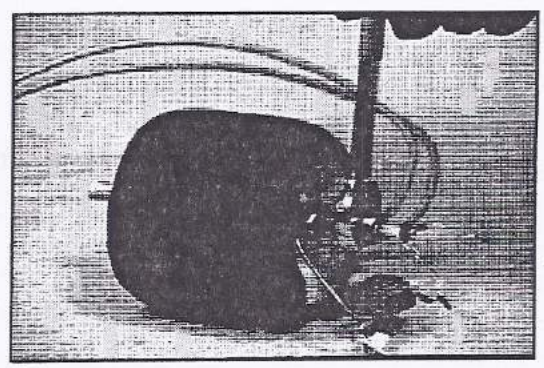
Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

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.

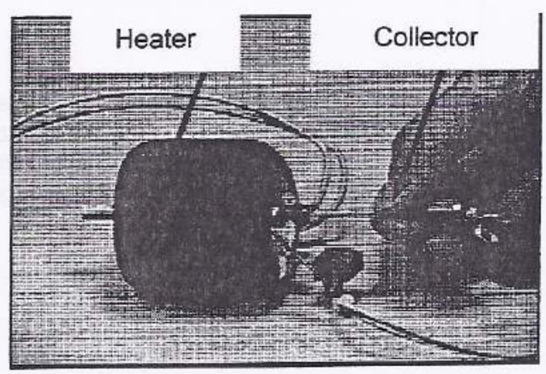




Dis-connect the three small platinum wires from the screw terminals and gently move them aside.



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.

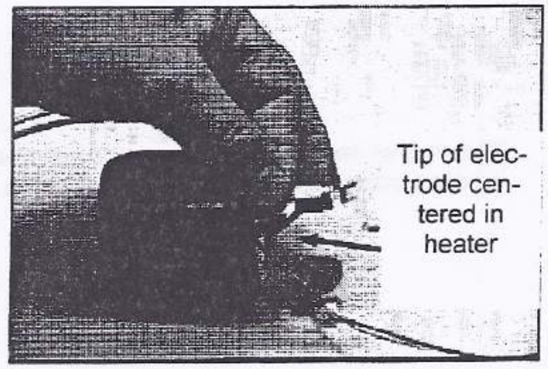


The collector can then be withdrawn from the heater.

Chapter: Detectors

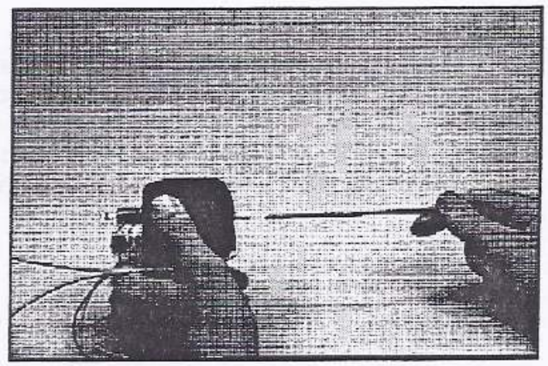
Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

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

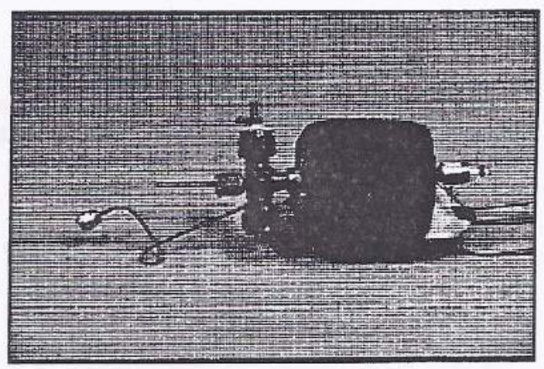


Tip of electrode centered in heater

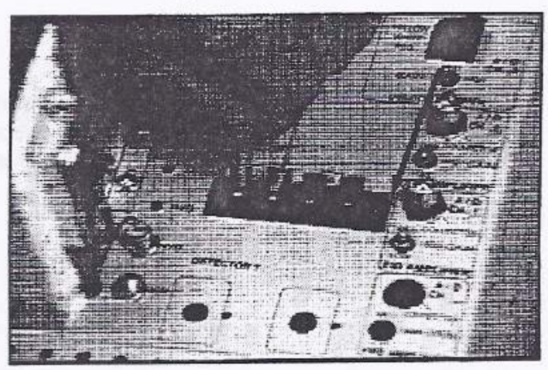
Use a file, rod, screwdriver or other long thin object as a gauge to verify that the electrode tip is centered in the new heater body. Gently re-position the electrode by sliding it through the graphite ferrule to get the proper adjustment. Finally, look down the bore of the heater and check to make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



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



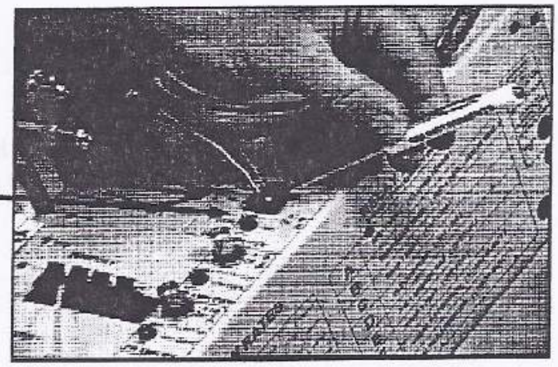
The two DELCD heater wires are connected to the push terminals on the deck of the GC which are labeled DELCD heater.



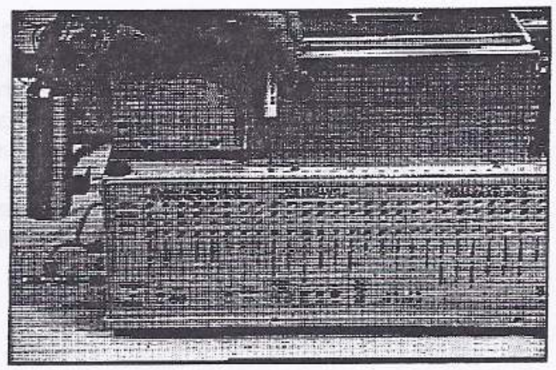
Chapter: Detectors

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.

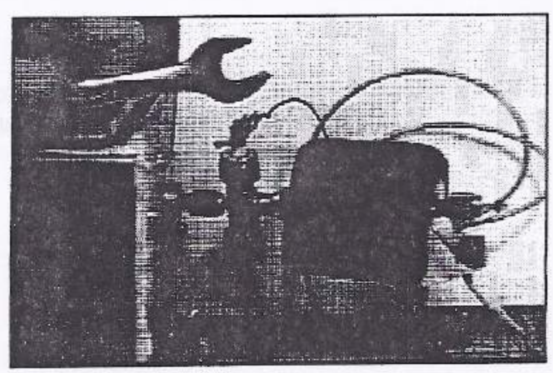


Set the DELCD heater setpoint temperature to 250. This is an arbitrary number which actually corresponds to a temperature of 1000°C. Better sensitivity can be obtained by raising the setpoint to 260 or 270, but at the cost of reduced heater lifetimes.



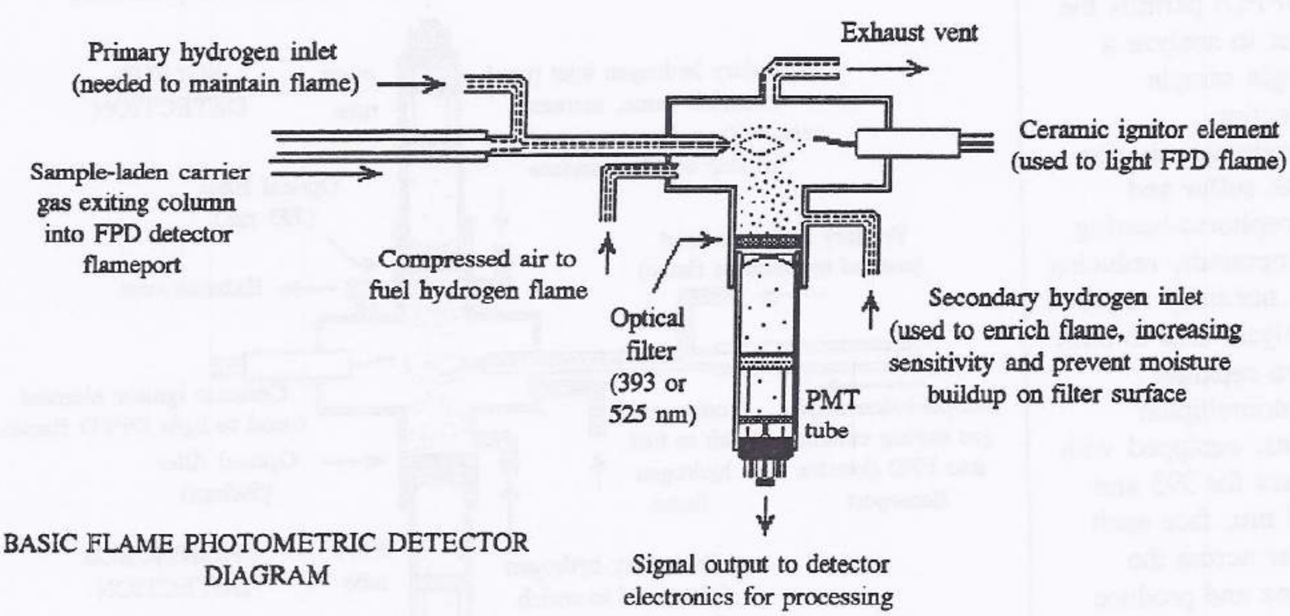
The actual temperature of the DELCD heater will equilibrate to about 7 degrees less than the setpoint within 10 minutes

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.



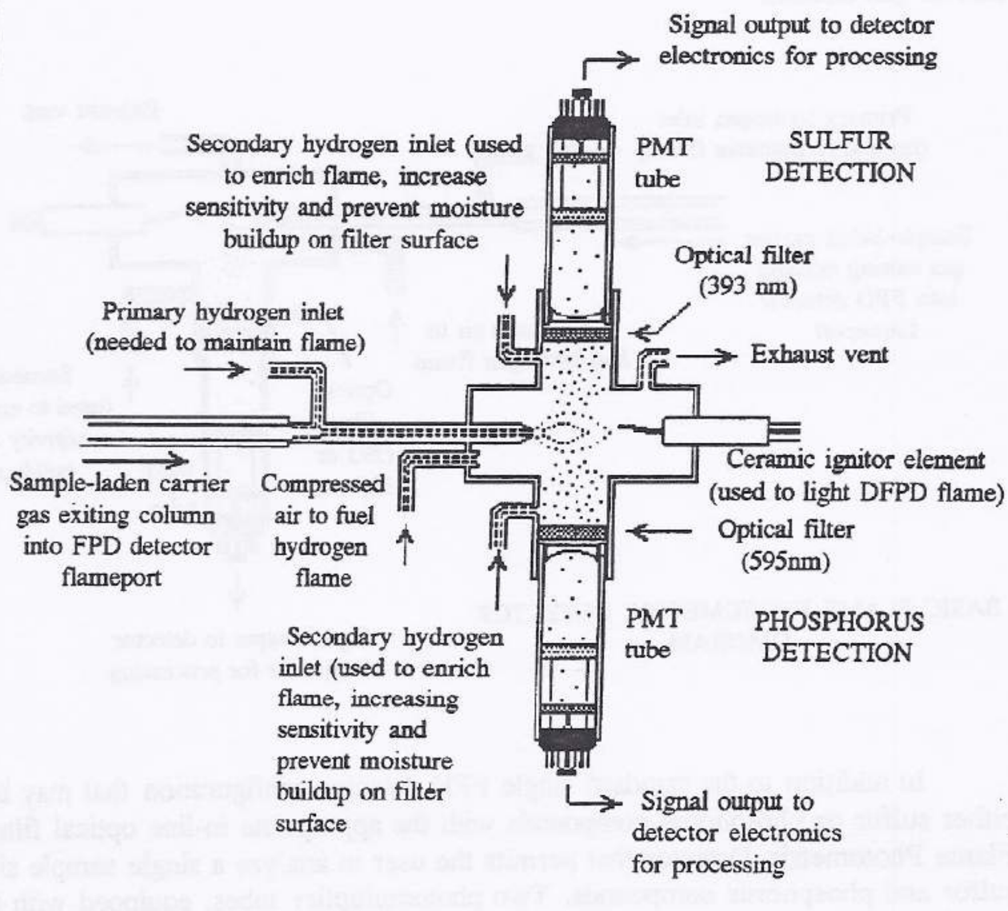
BASIC FLAME PHOTOMETRIC DETECTOR DIAGRAM

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.

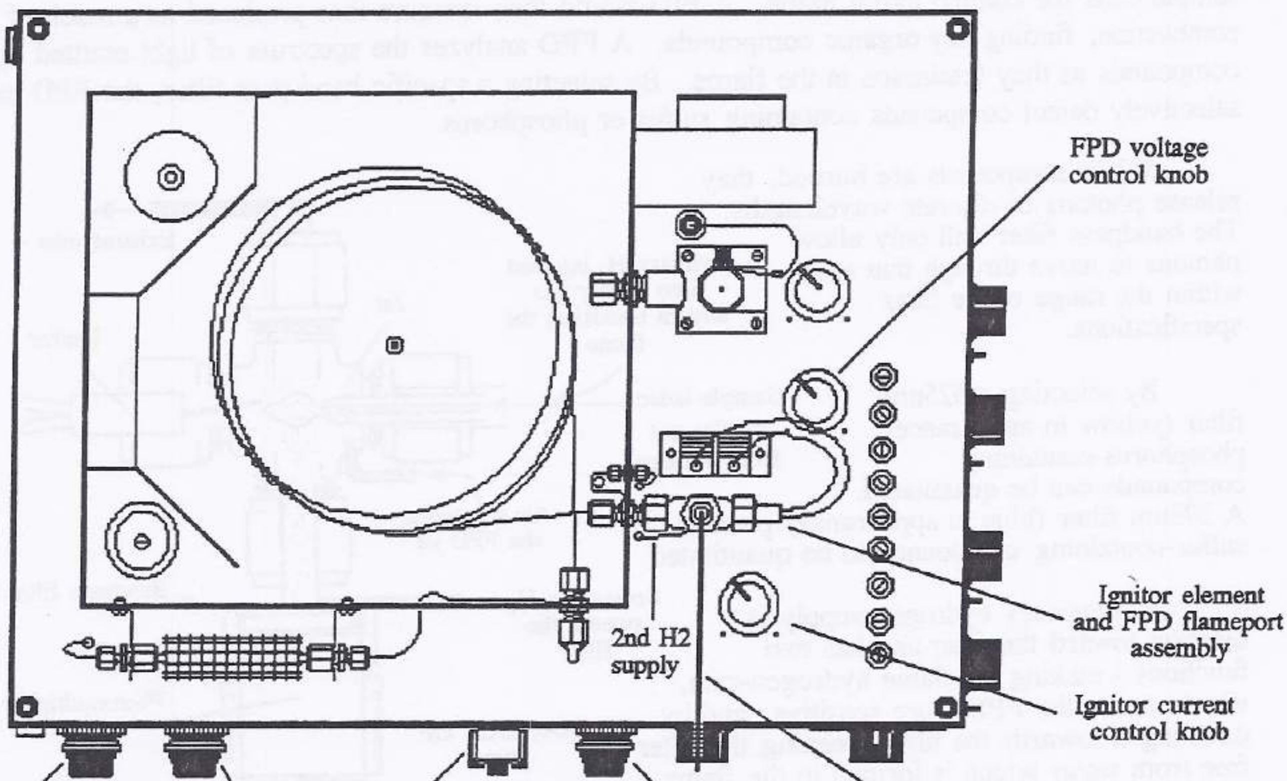
The Dual Flame Photometric Detector, or DFPD, as it is referred to, is typically used to simultaneously analyze for sulfur and phosphorus-containing compounds. The photomultiplier tubes employed are extremely sensitive to a broad portion of the visible light spectrum. Specific filtration inserted into 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



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.

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.

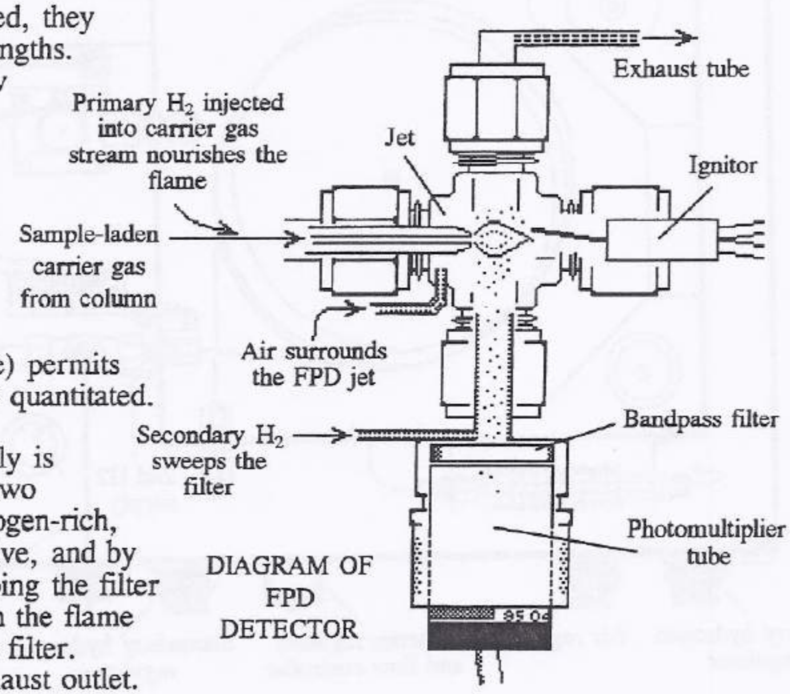


DIAGRAM OF FPD DETECTOR

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.

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.

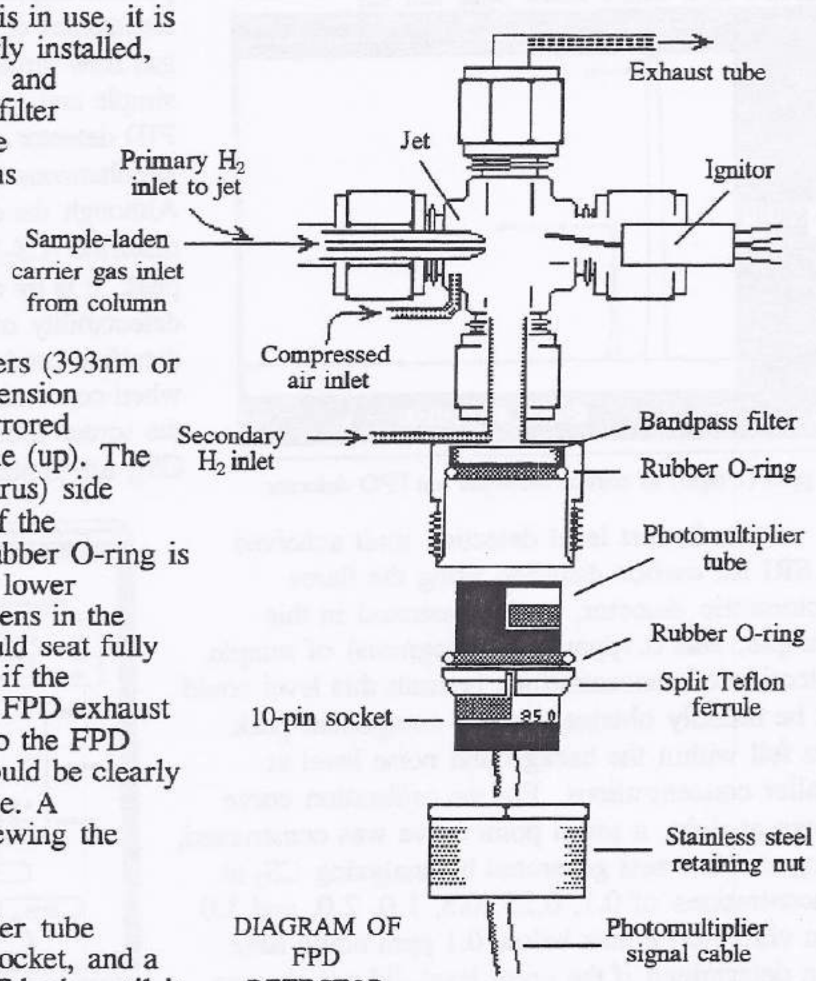


DIAGRAM OF FPD DETECTOR

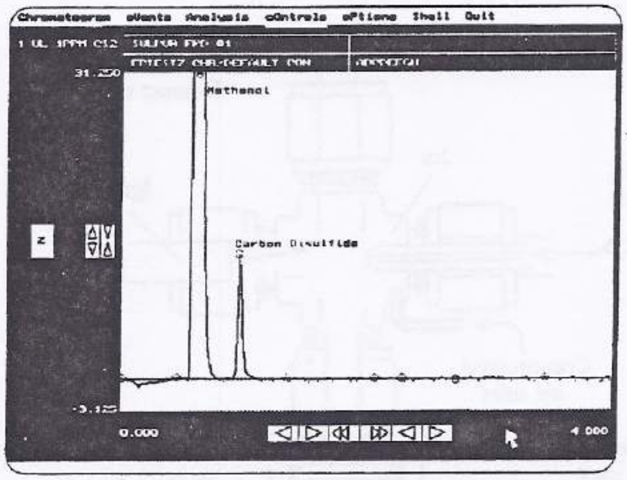
As illustrated in the diagram at right, either of the filters (393nm or 525nm) installs into the lower extension of the FPD assembly with the mirrored surface of the lens facing the flame (up). The blue (sulphur) or yellow (phosphorus) side of the filter should face the lens of the photomultiplier tube (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.

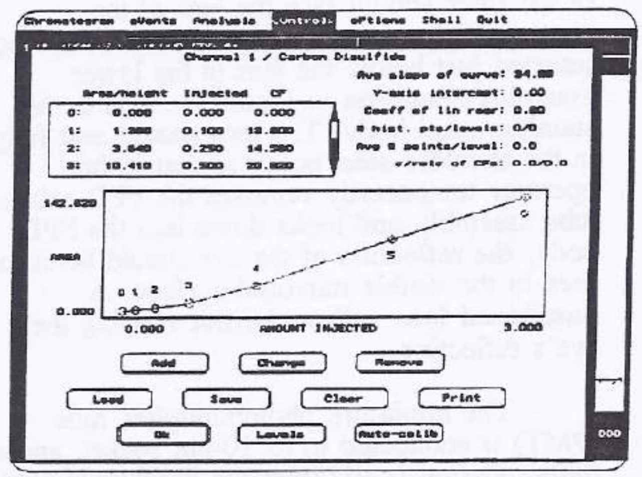
This discussion of the servicing of the FPD detector will be especially useful for users who must change filters on a regular basis to switch from sulphur mode over to phosphorus mode and back again. The FPD detector is provided with one user-specified filter. Replacement and secondary filters are also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

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.

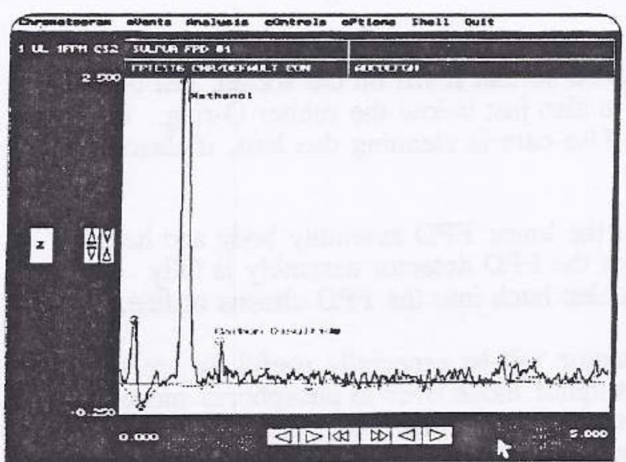


1 ppm (1 ngm) of carbon disulfide via FPD detector

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.

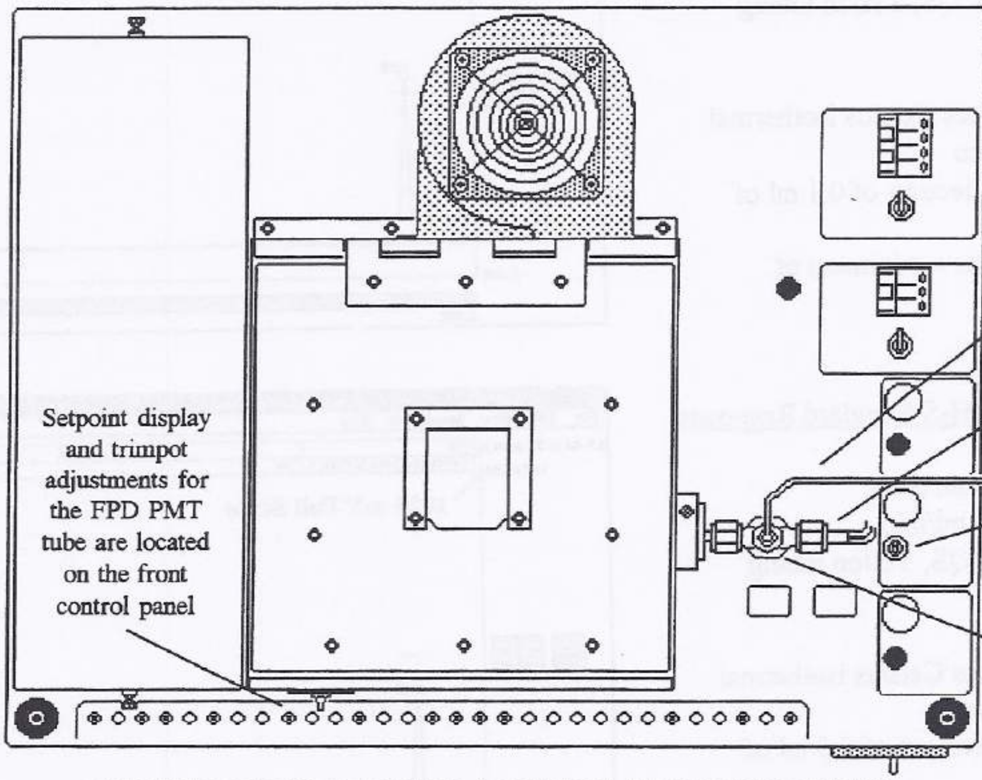


PeakSimple calibration curve for CS_2 via SRI FPD



0.1 ppm of CS_2 detectable well above background

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.

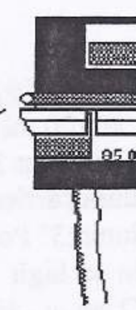


TOP VIEW OF FPD DETECTOR LOCATION ON 8610C GC CHASSIS

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)

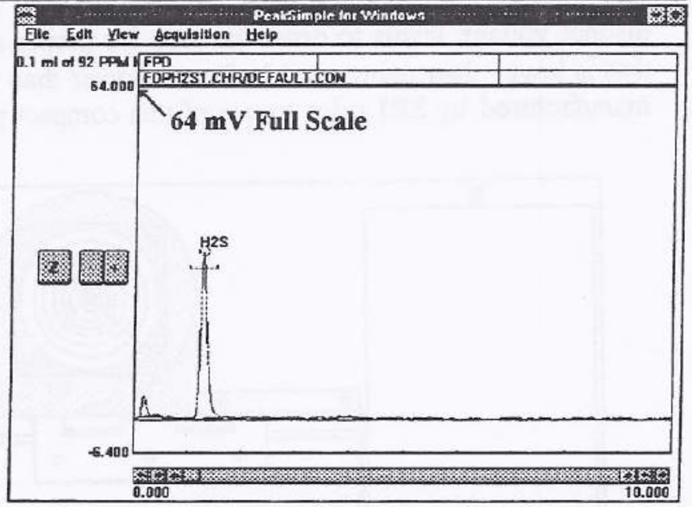


Top view of PMT tube, with lens visible

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.

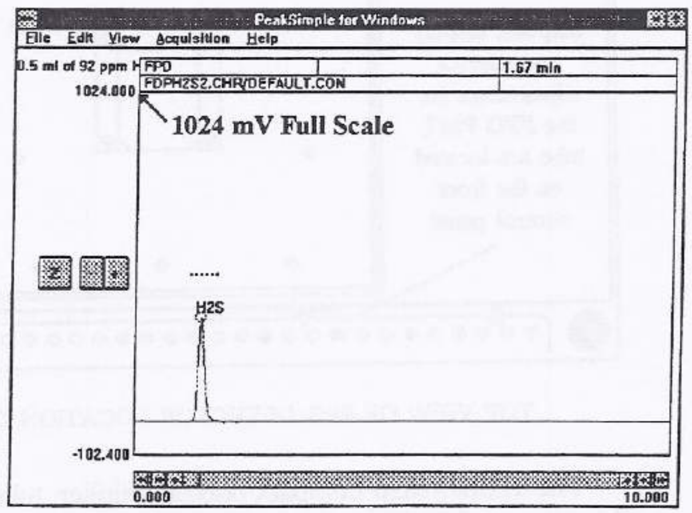
FPD 0.1 ml of 92 ppm H_2S Standard Response

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



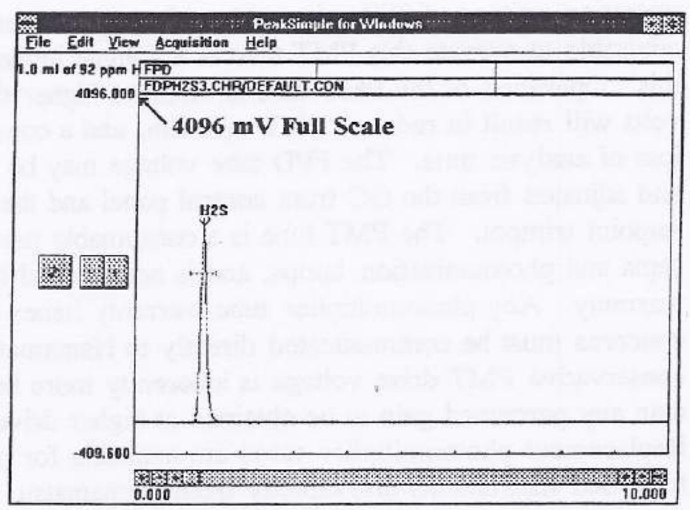
FPD 0.5 ml of 92 ppm H_2S Standard Response

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

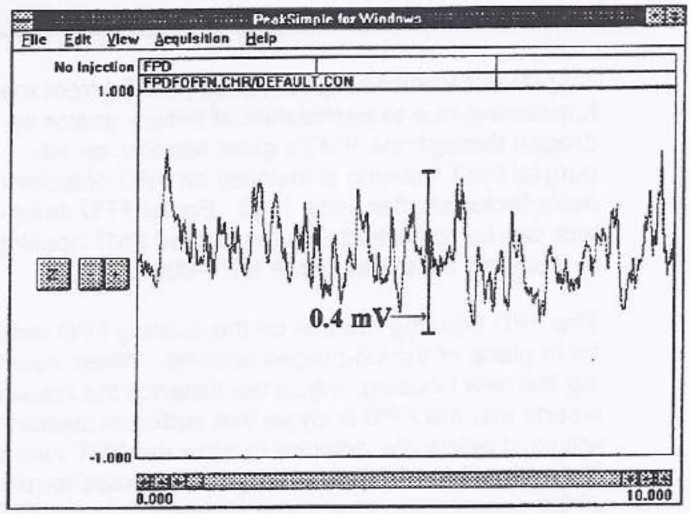


FPD 1.0 ml of 92 ppm H_2S Standard Response

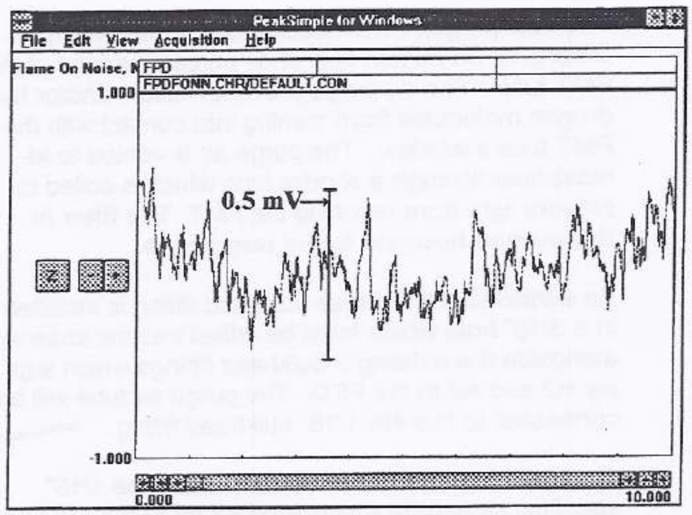
1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 1.0 ml of 92 ppm H_2S
9) Expected area counts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



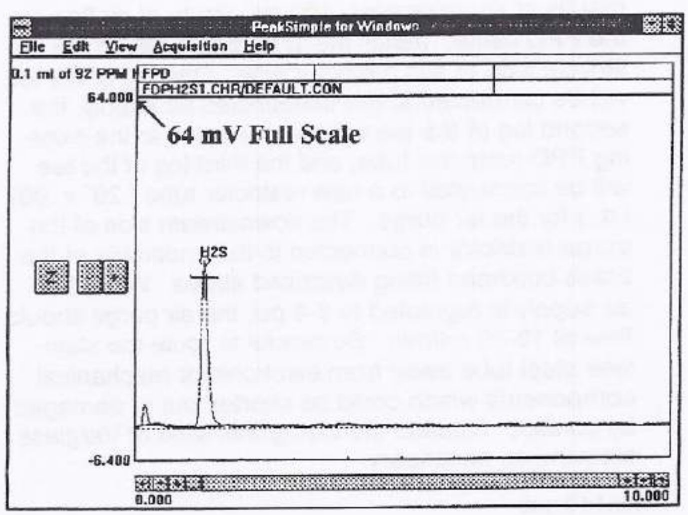
FPD Expected Performance



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

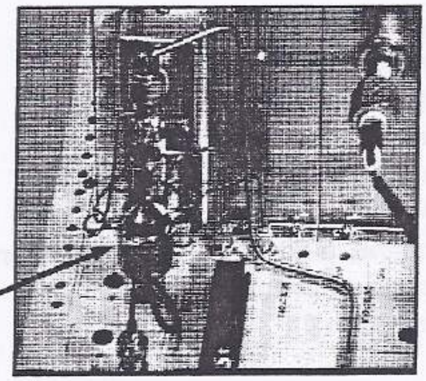


FPD 0.1 ml of 92 ppm H_2S Standard Response
1) Air at 100 ml/min
2) Hydrogen at 30 ml/min each
3) Helium carrier at 10 ml/min
4) Column:3' PoraPak QS, Teflon tubing
5) Gain on high
6) FPD set at -400 V
7) 10 minute, 60 degrees Celsius isothermal temperature program
8) Direct on column injection of 0.1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

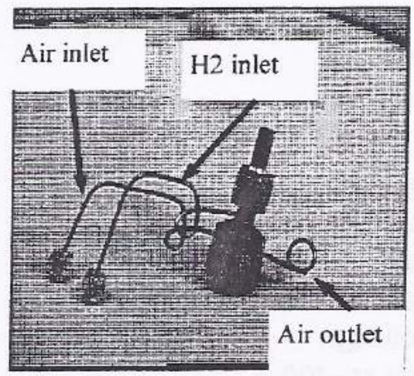
Chapter: FPD DETECTOR

Topic: Retrofit of air purged PMT tube housing

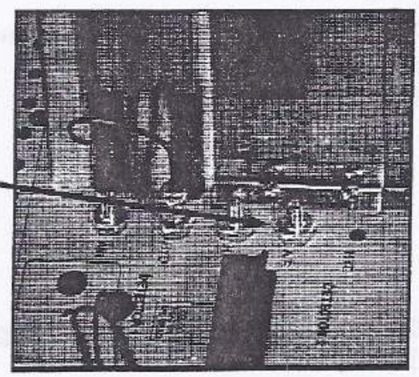
To prevent PhotoMultiplier Tubes (PMT) from malfunctioning due to permeation of helium and/or hydrogen through the PMT's glass window, an air-purged PMT housing is installed on FPD detectors manufactured after June 1998. Earlier FPD detectors can be retrofitted with the purged PMT housing by ordering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



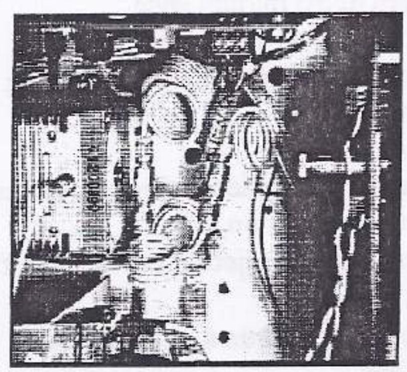
The FPD housing mounts on the existing FPD detector in place of the un-purged housing. When installing the new housing, adjust the distance the housing inserts into the FPD body so that sufficient clearance will exist below the detector to allow the PMT tube to fit comfortably. Only then snug up the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



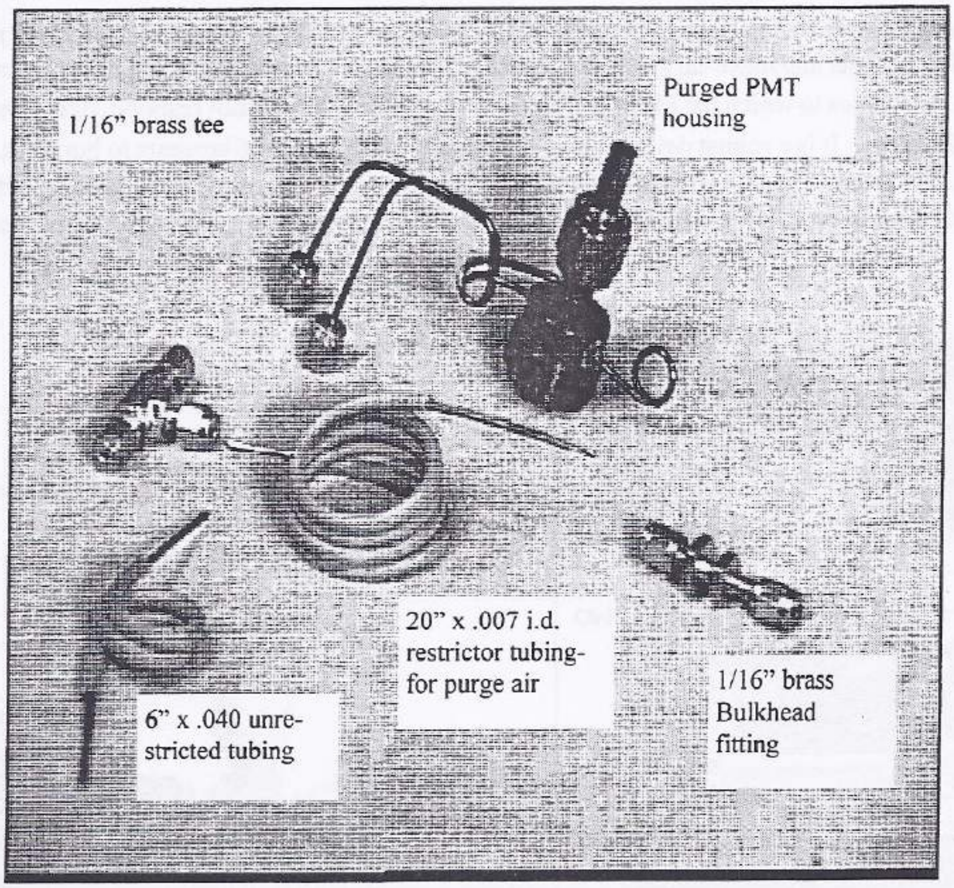
An additional 1/16" brass bulkhead fitting is installed in a 3/16" hole which must be drilled into the chassis alongside the existing 3 bulkhead fittings which supply H2 and Air to the FPD. The purge air tube will be connected to this 4th 1/16" bulkhead fitting.



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

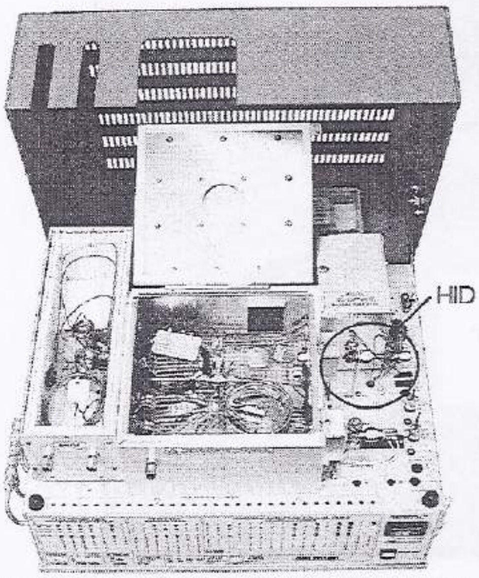


Contents of air purged PMT housing retrofit kit for FPD detector. SRI Part# 8670-0084.

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

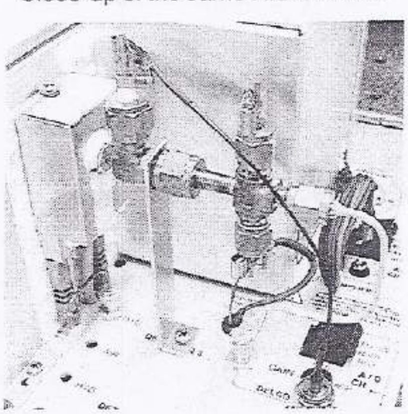
Overview

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.

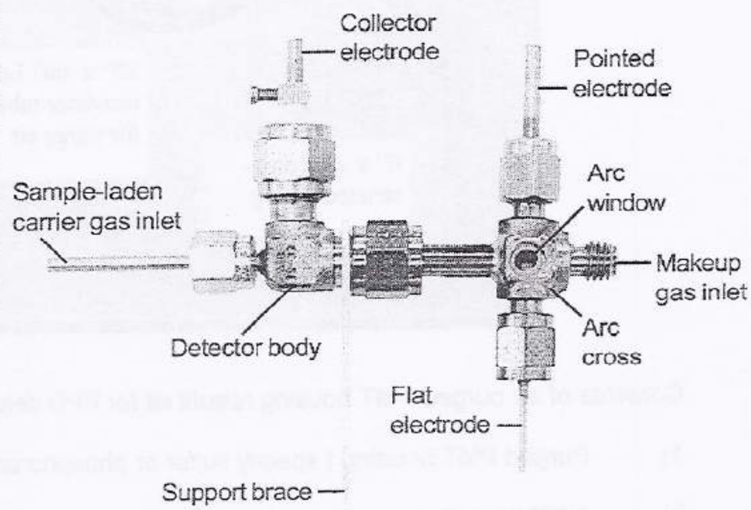


HID detector between TCD and FID detectors on an SRI GC

Close-up of the same HID detector

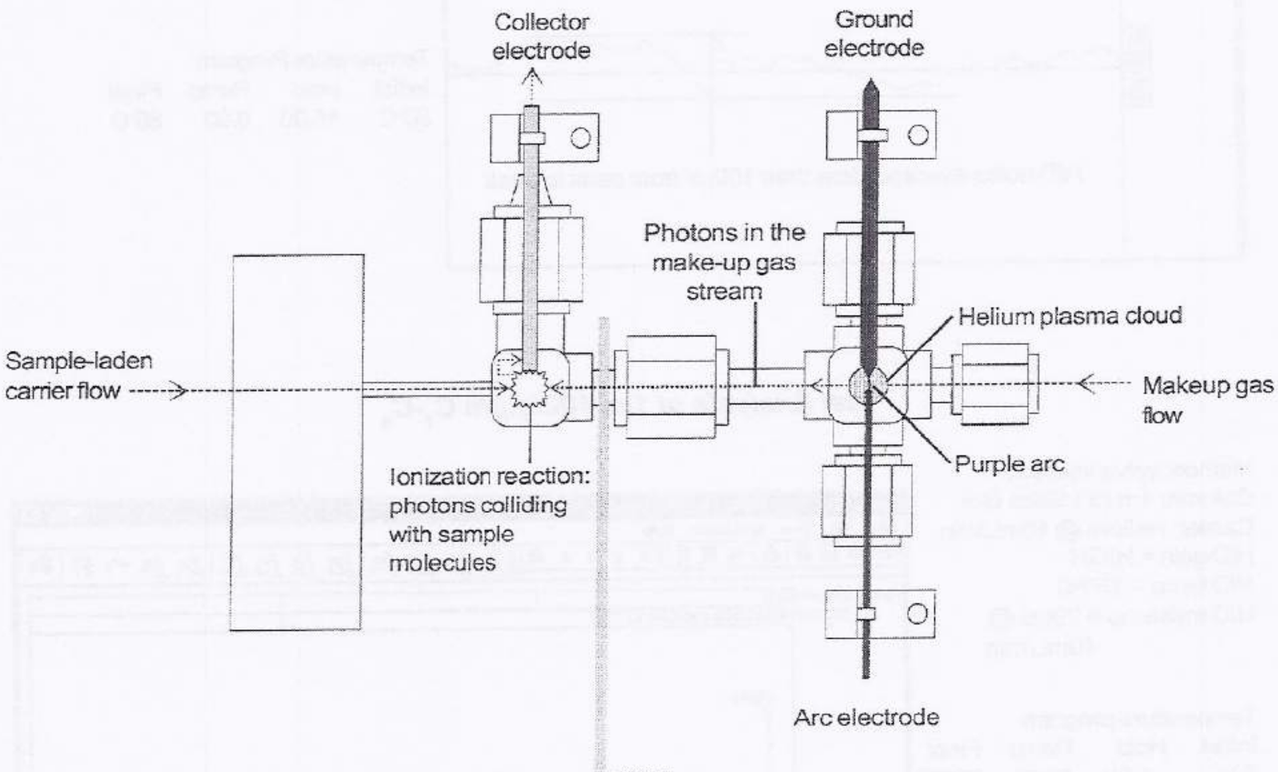


HID detector removed from GC and heater block



Theory of Operation

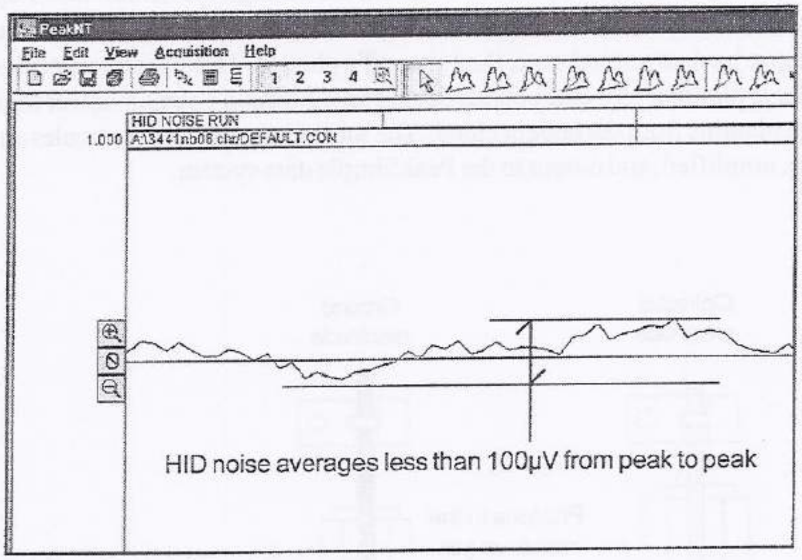
The SRI HID detector uses two electrodes which support a low current arc through the helium make-up gas flow. The helium molecules between the electrodes are elevated from ground state to form a helium plasma cloud. As the helium molecules collapse back to ground state, they give off a photon. The sample 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

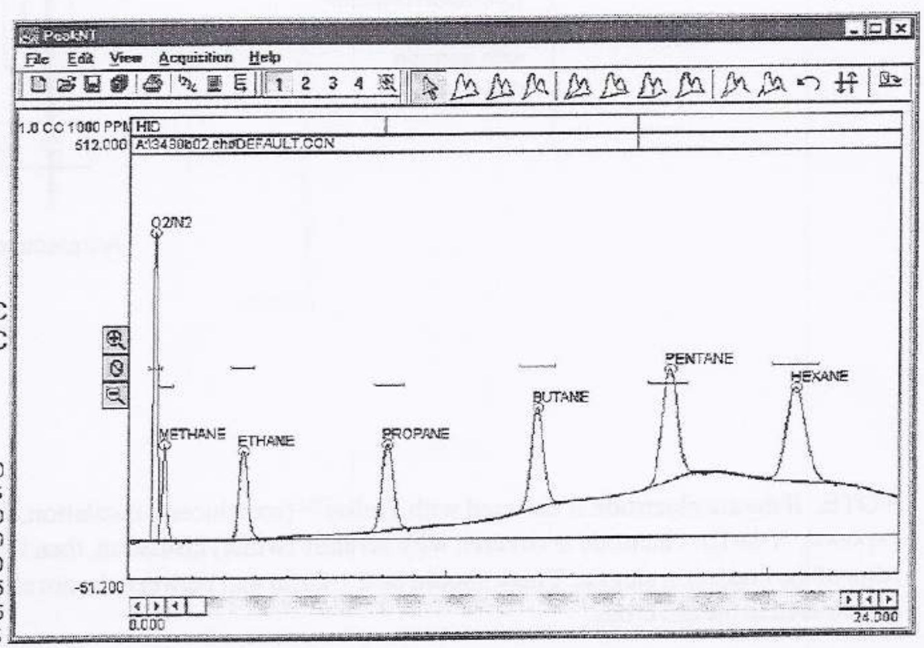
Method: valve injection
Column: 1m (3') Silica Gel
Carrier: Helium @ 10mL/min
HID gain = HIGH
HID temp = 150°C
HID make-up = 29psi @ 40mL/min

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



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).

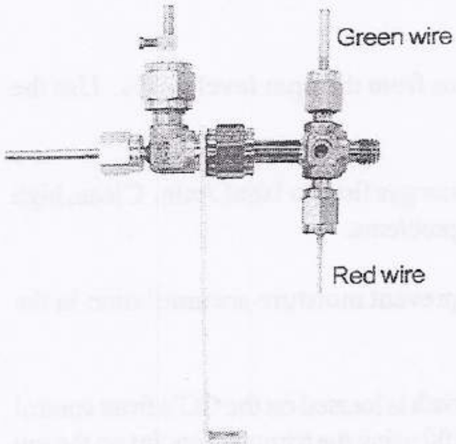
B. Look through the arc window at the arc and ground electrodes. If they appear to be touching, disconnect the red electrode lead wire then check the continuity between the electrodes using a multimeter; the reading should be open or infinite.

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

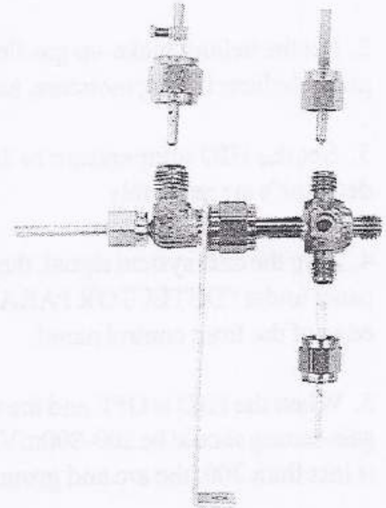
7. Let the milliVolt reading stabilize, then begin the analytical run.

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).

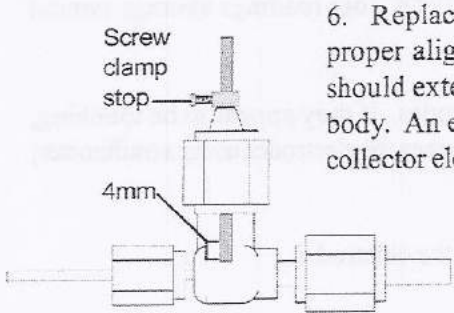


2. Remove the the arc and ground electrodes by loosening the 1/8" fittings that hold the electrodes in the arc cross.

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.

Use the arc window to check arc and ground electrode positioning.