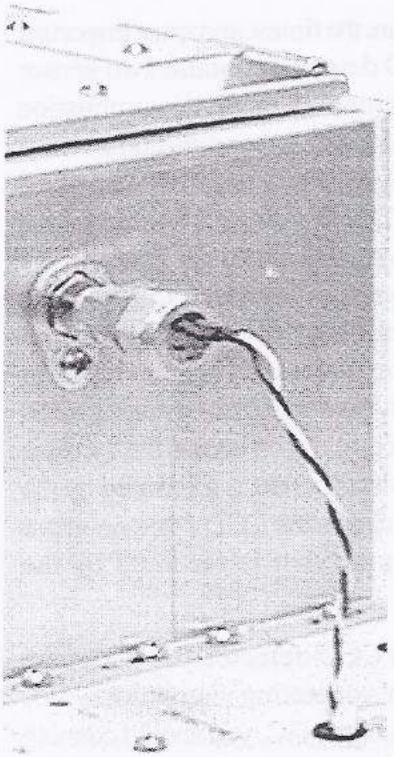
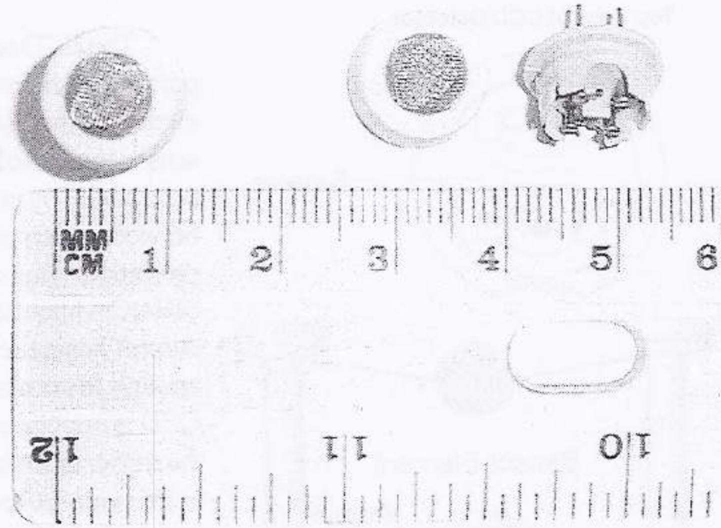
Overview

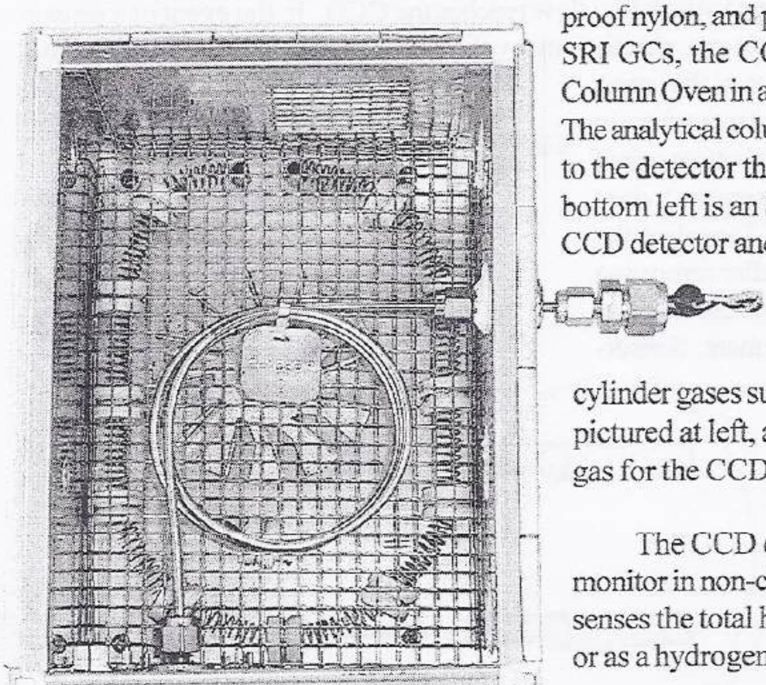


CCD on Column Oven



CCD Detector and protective cap (cap is removed prior to installation)

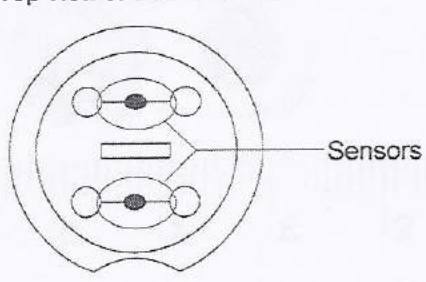
The Catalytic Combustion Detector responds to all hydrocarbons with the selectivity of an FID and the sensitivity of a TCD. The entire detector's diameter is merely one centimeter. Its sensor element consists of a tiny coil of platinum wire embedded in a catalytic ceramic bead. Each CCD detector has a pair of sensor elements. The sensors are housed in high-grade, flame-proof nylon, and protectively capped with a fine steel mesh. In SRI GCs, the CCD detector is mounted on the wall of the Column Oven in a brass housing, as shown in the top left picture. The analytical column residing in the Column Oven is connected to the detector through the oven wall; the example shown at bottom left is an SRI Gas-less™ Educational GC featuring a CCD detector and a 1m (3') Hayesep-D packed column. The CCD detector is especially suited for gas-less operation because it can operate on ambient air, requiring no high pressure cylinder gases such as hydrogen or helium. In the GC system pictured at left, a built-in air compressor supplies the carrier gas for the CCD.

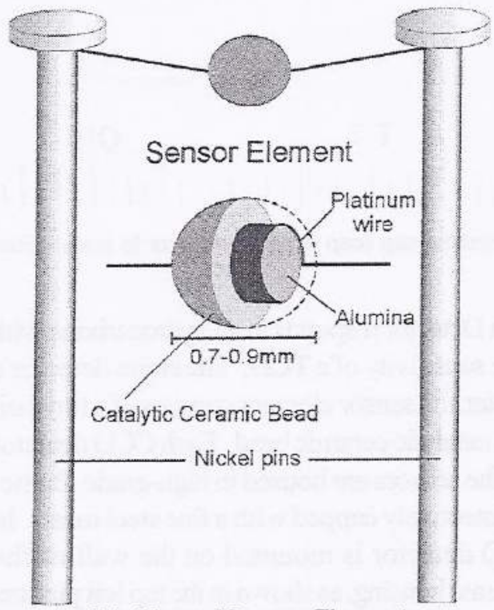


The CCD detector can also be used as a hydrocarbon monitor in non-chromatographic applications where the CCD senses the total hydrocarbon content of a flowing air stream, or as a hydrogen/hydrocarbon leak detector.

Theory of Operation

Top View of CCD Detector

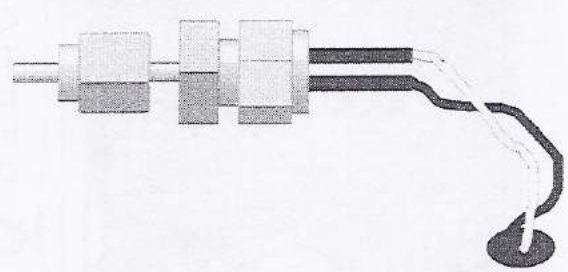


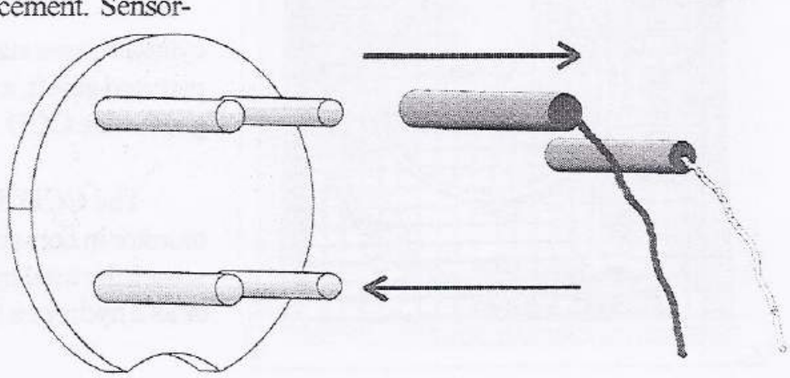


Side View of Sensor Element

The CCD sensor elements are the tiniest and most important part of the detector. Each CCD detector contains two sensor elements, but uses only one at a time. A catalytic combustion sensor consists of a coil of platinum wire around an alumina core surrounded by noble metal catalysts. Each sensor is suspended between a pair of nickel pins. The detector is shipped with a protective nylon cap topped with steel mesh, but is installed on a SRI GC without it. During a chromatogaphic run, a 150 milliamp current heats the catalytic ceramic bead to around 500°C, hot enough to combust hydrocarbon molecules on contact. The CCD is maintained in an oxidative environment by using air as the carrier or make-up gas. This combustion causes the increase in temperature and change in resistance that is measured by the sensor. This change in resistance causes the CCD detector output to change, which produces a peak that is recorded by the PeakSimple data system.

To prolong the life of your CCD detector, use it in strict accordance with your GC system's operating instructions. For instance, if you have an SRI Mud-Logger GC, you should connect your sample streams at 10psi so that no more than 5mL/min of pure hydrcarbon flow reaches the CCD. In the event of a sensor burn-out, simply remove the white and black wires from the top two nickel pins, and move them to the bottom pair of nickel pins to connect them to the second sensor. It does not matter which wire goes on which pin. To replace the CCD detector, unscrew its brass fitting after removing the wires from the nickel pins. Pull out the old one and remove the protective cap from the replacement. Sensor-side first, insert the replacement into the fitting with its half-moon shaped cut-out on the bottom. Replace the fitting and HAND TIGHTEN it. If the detector fitting is screwed on too tightly, the detector will not receive proper gas flow. Next, slip the black and white wire plugs over the pins, and your replacement CCD detector is ready to use.





Expected Performance

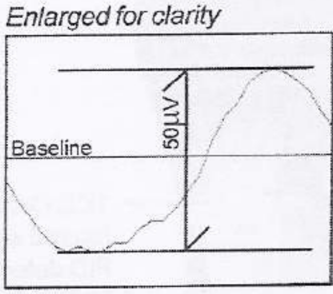
CCD Detector Noise Run

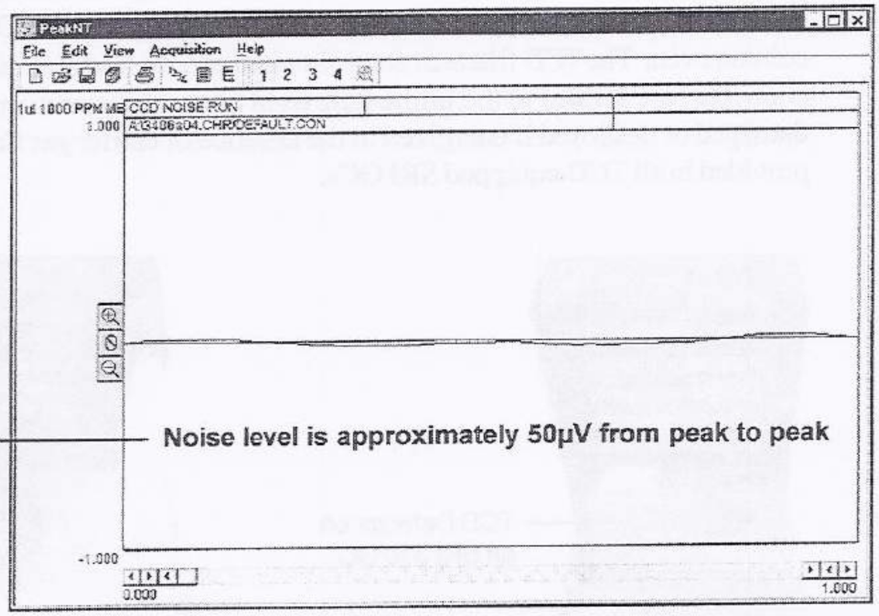
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:

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

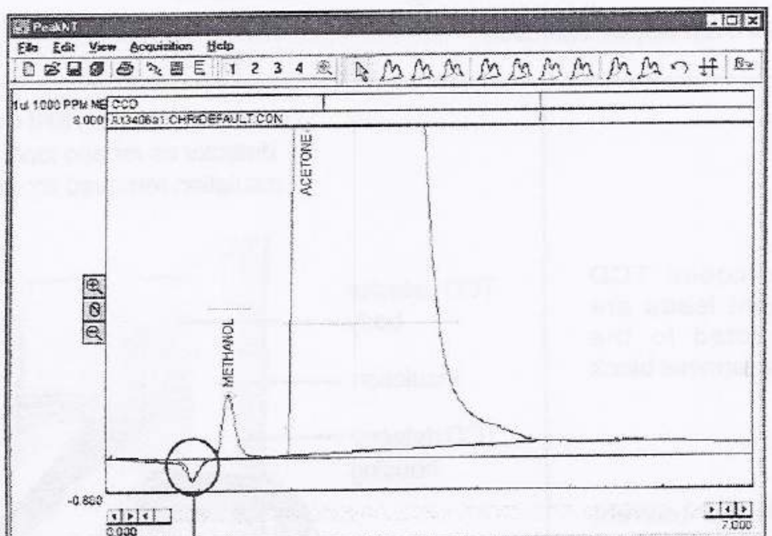
Enlarged for clarity





Noise level is approximately 50µV from peak to peak

Factory Test Run of a Gas-less™ Educational GC System



Negative water peak

Column = 1m Hayesep D
Flow = 37mL/min
Sample = 1µL 1000ppm Methanol/Acetone mix; direct injection

Isothermal Temperature Program:

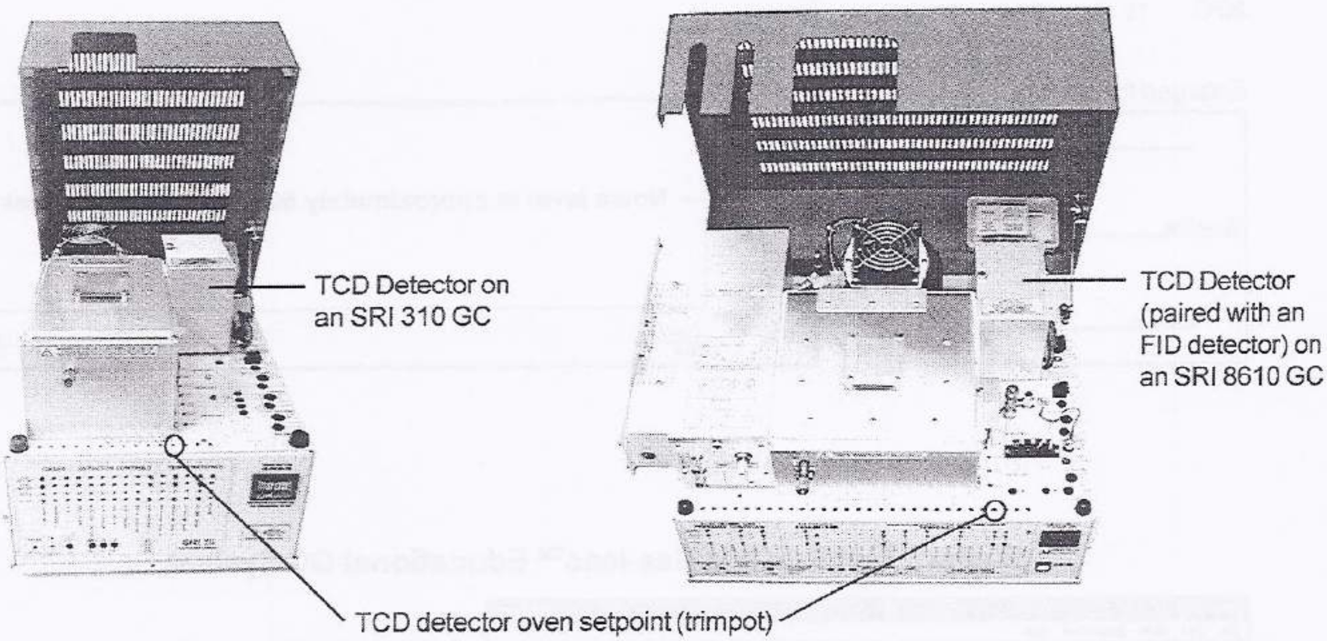
Initial Temp	Hold	Ramp	Final Temp
130°C	10.00	0.00	130°C

RESULTS:

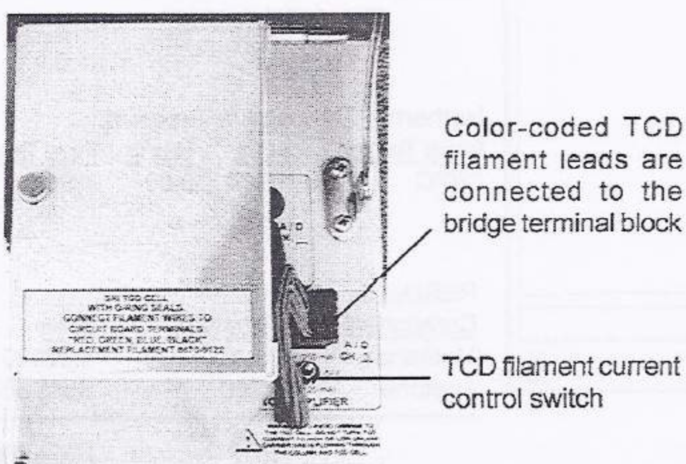
Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
	Total	6958.5600

Overview

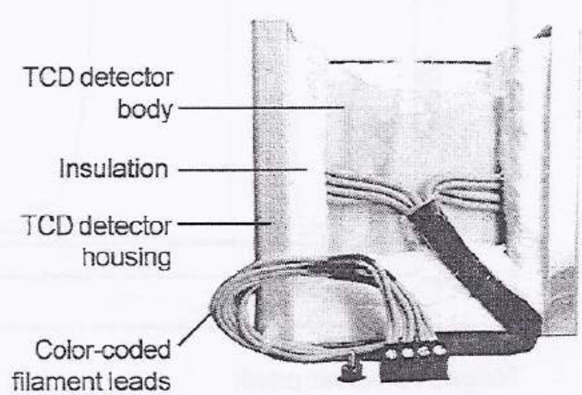
The Thermal Conductivity Detector (TCD) is the most universal detector available. Depending on the compound, the TCD responds with a detection range of 0.01% to 100% (100-1,000,000ppm). The SRI TCD consists of four filaments housed in a stainless steel detector block. The TCD detector block is installed in its own thermostatically-controlled oven for stability. The TCD oven is mounted on the right rear of the column oven. The TCD filament control switch and the bridge terminal block to which the filament leads are connected are located to the immediate right of the detector oven. Since the four TCD filaments can be damaged or destroyed if energized in the absence of carrier gas flow, a TCD filament protection circuit is provided in all TCD-equipped SRI GCs.



TCD Detector on an SRI GC



TCD Detector on an SRI GC with detector cover and top/front insulation removed for clarity

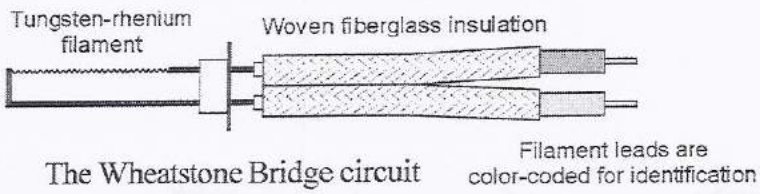


DETECTORS
Thermal Conductivity Detector - TCD

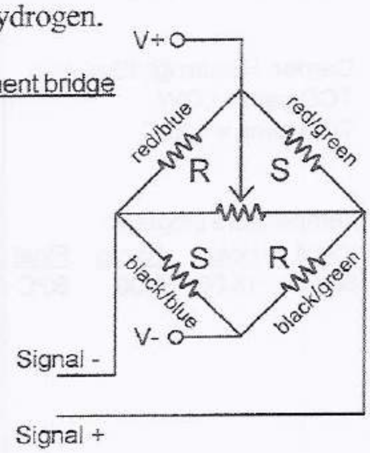
Theory of Operation

The TCD detector measures the difference in thermal conductivity in the carrier gas flow and the analyte peaks. Every compound possesses some degree of thermal conductivity, and may therefore be measured with a TCD detector. Due to its high thermal conductivity and safety, helium carrier is most often used with TCD detectors. However, other gases may be used such as nitrogen, argon, or hydrogen.

One of four TCD filaments

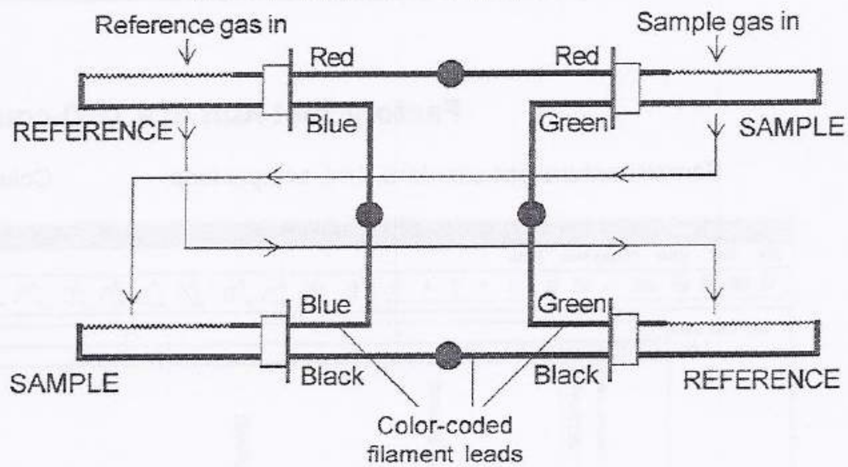


TCD filament bridge



The Wheatstone Bridge circuit design in the SRI TCD uses four general-purpose tungsten-rhenium filaments for sample analysis. Two of the filaments are exposed to the sample-laden carrier gas flow and provide the actual chromatographic signal. The other two filaments are provided with clean carrier flow, enabling them to be used as a baseline reference signal. When the effluent from the column flows over the two sample stream filaments, the bridge current is unbalanced with respect to the reference signal. This deflection is translated into an analog signal which is sent to the data system for analysis.

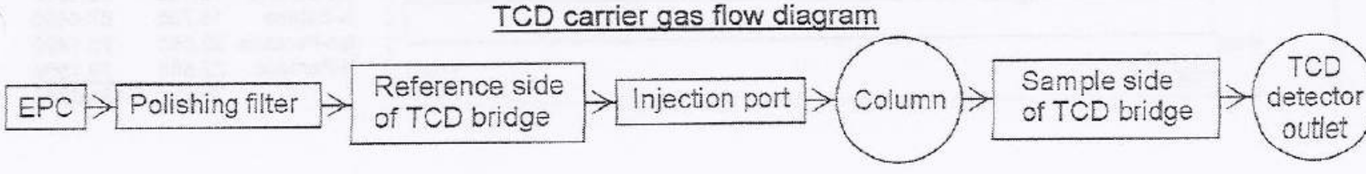
Simplification of filament interconnection



The four pairs of filament leads are color-coded in two-color units; each color is used on two different leads. All eight wires are connected to the bridge current supply via four setscrew-type terminal connectors on the top control panel of the GC. Silkscreened labelling on the chassis indicates which color wire connects to each terminal.

The TCD detector block is divided into two cells containing two filaments each. One cell holds the reference pair while the other cell holds the sample pair. All four TCD filaments are physically identical except for their color-coding. The carrier gas is plumbed so that is exits the Electronic Pressure Controller module, flows through the polishing filter, through the reference side of the TCD bridge, then through the injection port to the column, and from the column to the sample side of the TCD bridge. After the flow passes through the sample cell, it is directed back out of the TCD oven and into the column oven through the TCD detector outlet, where it may be routed to a subsequent detector or to vent. All four TCD detector inlet/outlet tubes are 1/16" stainless steel.

TCD carrier gas flow diagram



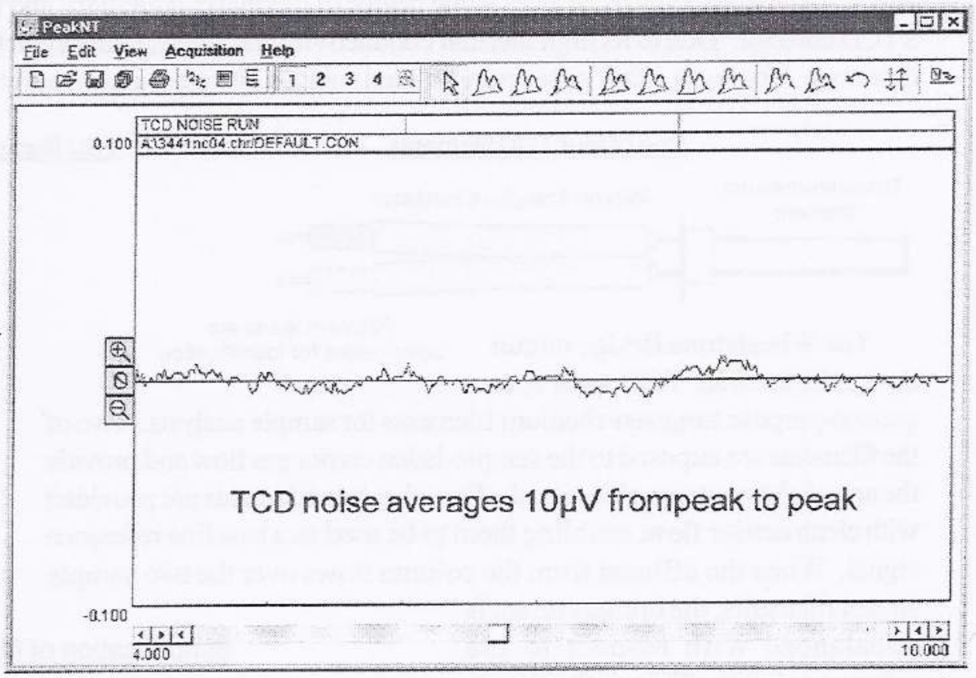
Expected Performance

TCD Noise Run

Carrier: Helium @ 10mL/min
TCD gain = LOW
TCD temp = 100°C

Temperature program:

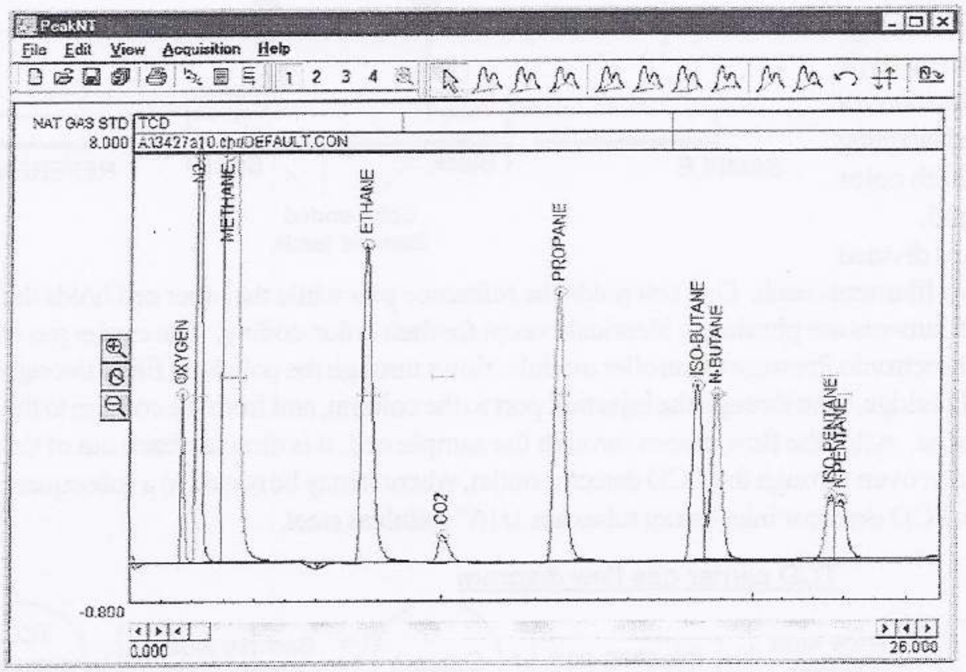
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C



Factory Test Run of a TCD-equipped SRI GC

Sample: natural gas standard, 1mL sample loop

Columns: 1m Molecular Sieve, 2m Silica Gel



Events:

Time	Event
0.00	ZERO
0.050	G ON (valve inject)
6.00	G OFF

Temperature program:

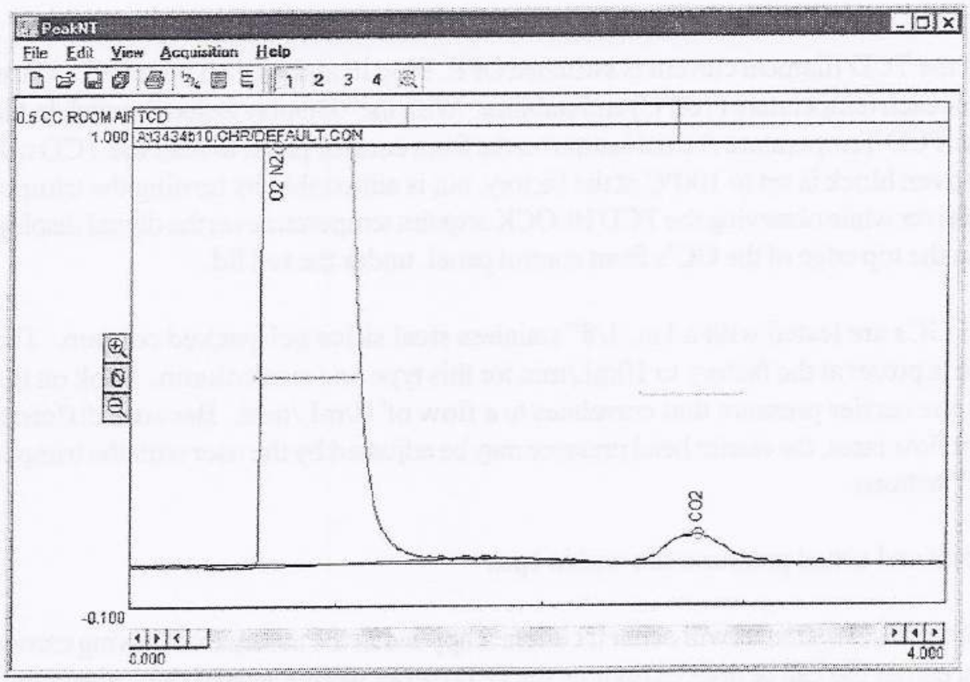
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

Results:

Component	Retention	Area
Oxygen	1.633	19.7500
N2	2.150	121.0880
Methane	3.033	563.6130
Ethane	7.550	128.2185
CO2	9.983	11.9860
Propane	13.683	113.9220
Iso-Butane	18.150	69.4960
N-Butane	18.766	67.4460
Iso-Pentane	22.550	20.1490
N-Pentane	22.866	19.1560
	Total:	1134.8245

Expected Performance

TCD Room Air Analysis



Column: 3' Silica Gel
Carrier: Helium at 10mL/min
Sample: 0.5cc room air, direct injection
TCD current: LOW
TCD temperature: 100°C

Temperature Program:

Initial	Hold	Ramp	Final
80°C	4.00	0.00	80°C

Results:

Component	Retention	Area
O_2N_2	0.716	1021.3830
CO_2	2.766	1.5060
	Total	1022.8890

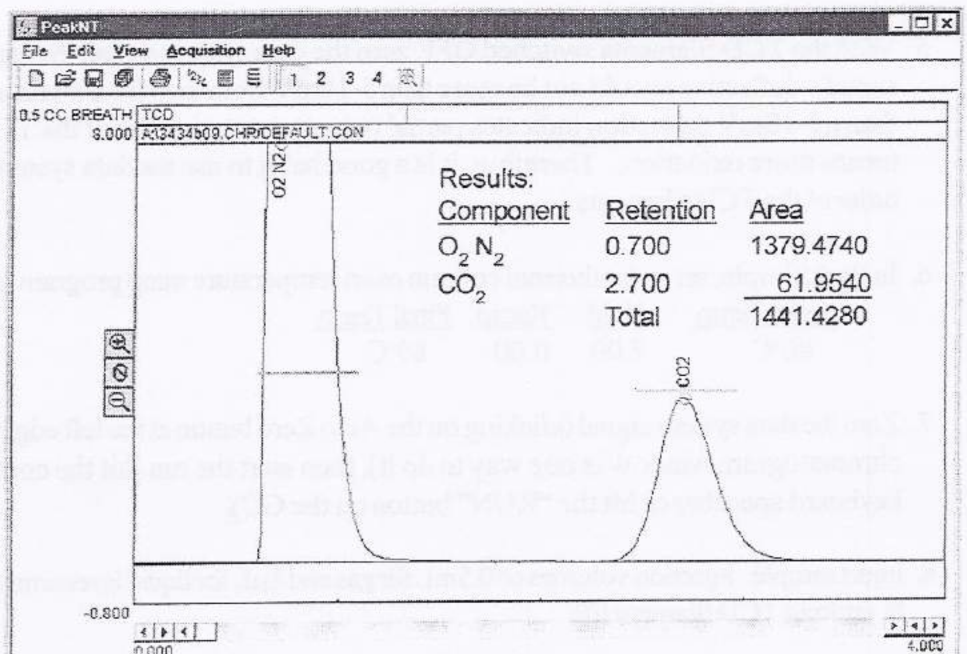
The CO_2 content of the room air analyzed is approximately 350ppm.

TCD Breath Analysis

Column: 3' Silica Gel
Carrier: Helium at 10mL/min
Sample: 0.5cc human breath, direct injection
TCD current: LOW
TCD temperature: 100°C

Temperature Program:

Initial	Hold	Ramp	Final
80°C	24.00	0.00	80°C



Results:

Component	Retention	Area
O_2N_2	0.700	1379.4740
CO_2	2.700	61.9540
	Total	1441.4280

General Operating Procedure

1. Check to make sure that the TCD filament current is switched OFF. Plug in and turn on your GC. Allow the TCD detector oven to reach temperature (100°C) and stabilize. With the "Display Select" switch in the UP position, press on the TCD Temperature Actual button on the front control panel to read the TCD cell temperature. The TCD oven block is set to 100°C at the factory, but is adjustable by turning the trimpot with a small blade screwdriver while observing the TCD BLOCK setpoint temperature on the digital display. The trimpot is located on the top edge of the GC's front control panel, under the red lid.

2. All TCD-equipped SRI GCs are tested with a 1m, 1/8" stainless steel silica gel-packed column. The carrier gas head pressure is preset at the factory to 10mL/min for this type and size column. Look on the right side of the GC for the carrier pressure that correlates to a flow of 10mL/min. Because different columns require different flow rates, the carrier head pressure may be adjusted by the user with the trimpot above the "CARRIER 1" buttons.

3. Make sure that the setpoint and actual pressures are within 1psi.

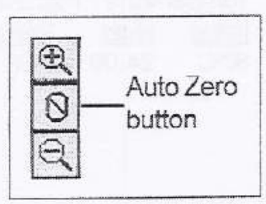
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column Oven on the same side as the detector. Place the end of the tube in liquid and observe (a little spit on a finger can suffice). If there are no bubbles exiting the tube, there is a flow problem. DO NOT turn on the TCD current if carrier gas flow is not detectable. A filament protection circuit prevents filament damage if carrier gas pressure is not detected at the GC, but it cannot prevent filament damage under all circumstances. Any lack of carrier gas flow should be corrected before proceeding.

5. With the TCD filaments switched OFF, zero the data system signal. Switch the filaments to LOW. The signal's deflection should not be more than 5-10mV from zero for a brand-new TCD detector. Any more than a 5-10mV deflection indicates partial or complete oxidation of the TCD filaments; more deflection means more oxidation. Therefore, it is a good habit to use the data system signal to check the working order of the TCD filaments.

6. In PeakSimple, set an isothermal column oven temperature ramp program as follows:

Initial Temp.	Hold	Ramp	Final Temp.
80°C	7.00	0.00	80°C

7. Zero the data system signal (clicking on the Auto Zero button at the left edge of the chromatogram window is one way to do it), then start the run (hit the computer keyboard spacebar or hit the "RUN" button on the GC).

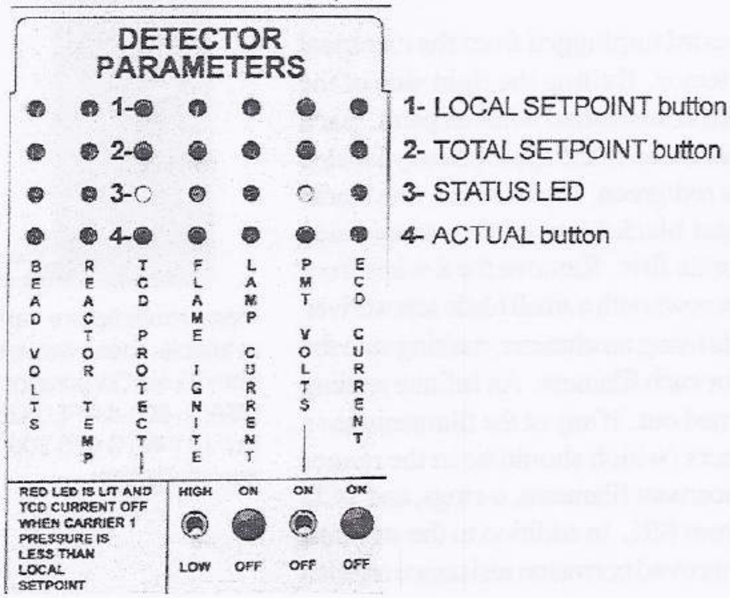


8. Inject sample. Injection volumes of 0.5mL for gas and 1μL for liquid is recommended to prolong TCD filament life.

TCD Filament Protection Circuit

All TCD detectors are susceptible to filament damage or destruction if operated at high current in the absence of carrier and/or reference gas flow. The filaments will incandesce and burn out if the carrier or reference gas flow is interrupted due to a variety of possible factors such as a column break, inadvertent column disconnection during column changes, removal of the septum nut for septum replacement, or when the carrier gas cylinder runs dry during an analysis. The SRI TCD filament protection circuit is a current "cut-out" circuit that monitors the column head pressure during GC operation. Under normal circumstances, there is no reason for the column head pressure to drop below 3psi, with most columns operating at 8psi or above. When the head pressure sensor located in the carrier gas flow path drops below 3psi, the protection circuit is activated, and the current to the TCD filaments is interrupted immediately. A red LED on the GC's front control panel under "DETECTOR PARAMETERS" will light to indicate that the protection circuit has detected a gas pressure loss and shut down the filament current. The cause of the protection circuit activation should be immediately investigated and corrected. As an additional caution, use HIGH current only with helium or hydrogen carrier gases. With nitrogen carrier, use LOW current only, or the filaments may be damaged. The pressure at which the protection circuit activates is user adjustable with the trimpot on the top edge of the front control panel, above the label reading "TCD PROTECT."

TCD protection circuit LED lit on an SRI model 8610 GC front control panel



Bright red LED display

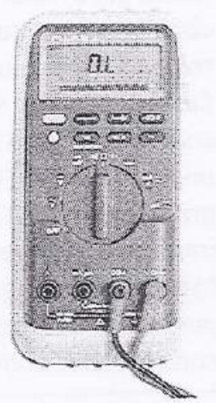
LED panel displays control data corresponding to the button pressed

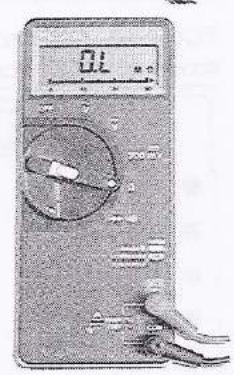
The DISPLAY SELECT switch allows the user to choose between displaying the control zones using the buttons or the column oven temperature

1- Pressing the LOCAL SETPOINT button displays the filament cut-off setpoint value (factory set at 3psi) in the bright red LED display in the upper right corner of the GC's front control panel. If the carrier gas pressure reaches or falls below this value, the filament current will immediately be interrupted.
2- Pressing the TOTAL SETPOINT button displays the carrier gas pressure present in the GC system. Under normal operation, this value will be well above the 3psi cut-off setpoint.
3- The STATUS LED glows bright red only when the TCD protection circuit has been activated.
4- Pressing the ACTUAL button displays the voltage present across one half of the TCD bridge. A value of 3.5 to 4.5 volts is typical when using high current; low current will display 2.5-3.5 volts (note: the LED displays 4 volts as "400," 3.5 as "350," etc.). Any value lower than these indicates a potential problem in the TCD detector bridge.

TCD Troubleshooting

When the TCD fails to perform normally, review operating conditions to ensure that carrier gas flow to the detector is unimpeded, and that the column oven temperature, carrier gas flow rate, and carrier gas EPC pressure are all within the desired operating parameters. If all conditions are properly met and the detector continues to perform poorly or fails to perform at all, check the TCD filaments for damage. The main diagnostic test is to measure the resistance of each filament using the ohmeter function of a multimeter or volt-ohmeter (VOM). At room temperature, the resistance of each filament should be 32-34 ohms. At 100°C, the filaments are around 40 ohms each. If any filament is significantly different from the others, the TCD bridge will be unbalanced, noisy and drifty. All eight filament wires must be disconnected and tested. Since all the leads are bundled together as they exit the TCD detector assembly, you may need to use the multimeter or VOM to determine the actual pairs. It is normal for each filament to have a slightly different reading within the appropriate operating range, so match the readings to determine the lead pairs.





Many multimeters are available; these two are from Fluke Corporation:
USA: 1-800-44-FLUKE
EU: (31 40) 2 678 200
www.fluke.com

With the power turned off and the power cord unplugged from the electrical outlet, raise the red lid to access the TCD detector. Exiting the right side of the TCD detector oven is the bundle of 8 insulated, color-coded wires in pairs. Each pair of wires represents one filament and is connected to the appropriately labeled terminal for its paired colors. One filament has red/green, one red/blue, one black/green, and one black/blue. The red/green and black/blue are the sample side filaments, and the ones which typically deteriorate first. Remove the 8 wires from the bridge terminal by loosening the retaining setscrews with a small blade screwdriver. Measure the resistance across the filament leads using an ohmeter, making sure the correct pair of colored wires is tested together for each filament. An infinite reading is an indication that the filament is open, or burned out. If any of the filaments has a significantly different resistance than the others (which should be in the ranges mentioned above), it should be replaced. Replacement filaments, o-rings, and TCD blocks with four new filaments are available from SRI. In addition to the standard filaments, optional gold-plated filaments for improved corrosion resistance are also available.

SRI TCD detector replacement parts

Standard TCD filament with rubber O-ring gasket	8670-9120
High temperature TCD filament with copper gasket	8690-9123

(filament part #s are also listed on the top of the TCD oven in your SRI GC)

Replacing the TCD Filaments

SRI TCD detectors are made to last a long time without ever replacing the filaments. However, any TCD filaments that fail the diagnostic ohmeter test mentioned previously will have to be replaced. While they share the same outer assembly, there are a few differences between the high temperature TCD detector block and the standard TCD block. Both designs are discussed. All filaments are fragile; handle them with care. Have colored ink pens, electrical tape, whatever you will use for color coding close at hand before you begin. It is best to go slowly, color-coding then replacing each filament one at a time. IF YOU MIX UP THE FILAMENT LEADS, YOUR TCD WILL NOT WORK!

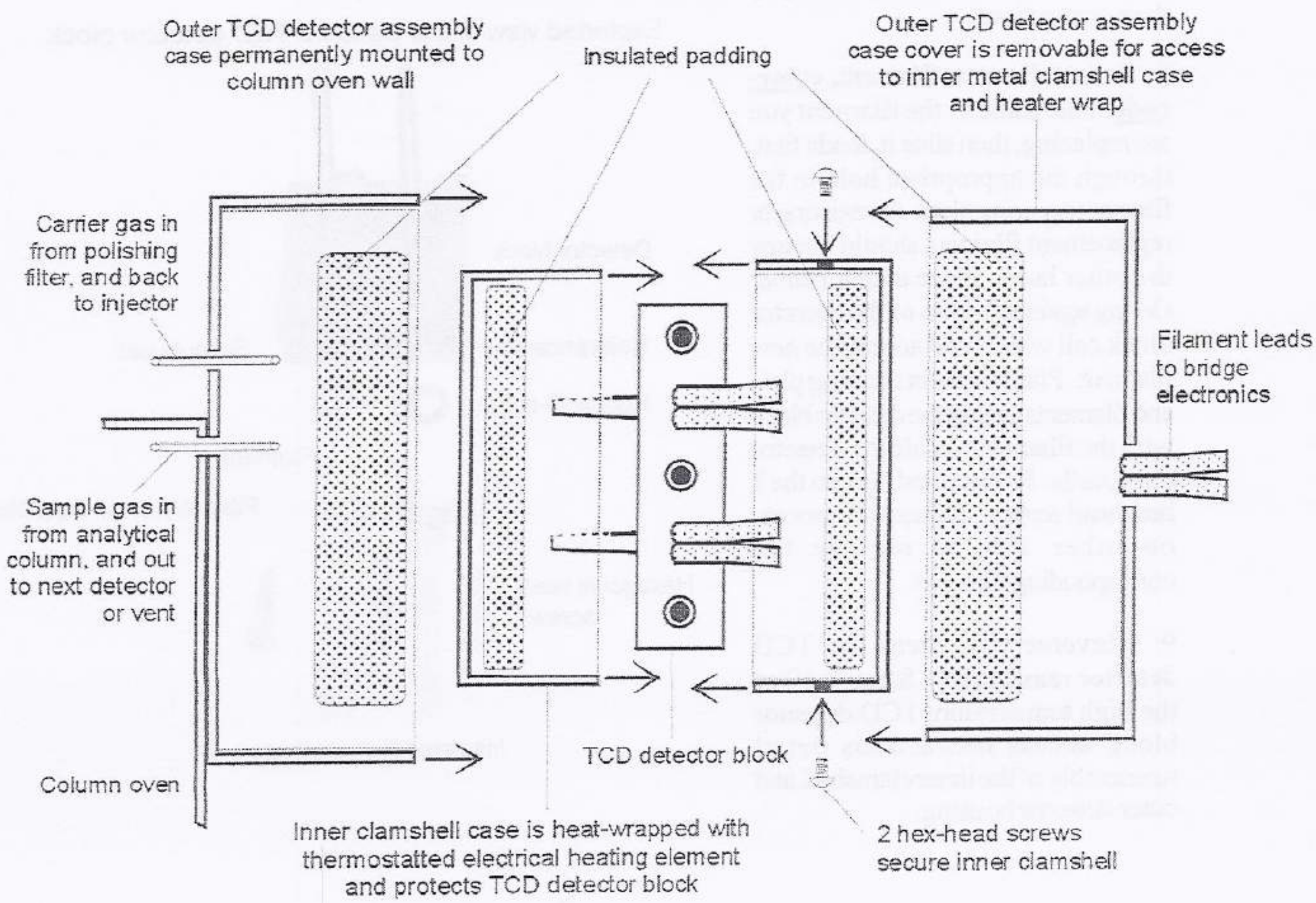
A. Standard TCD detector block access

1. With a small blade screwdriver, free the filament leads from the bridge terminal by loosening the setscrews.

2. Remove the detector assembly cover by unscrewing the thumbscrew then sliding the cover off toward the right-hand edge of the GC; gently remove the white insulation to reveal the detector block.

3. Disconnect the detector block gas inlets and outlets. The reference gas inlet is disconnected at the polishing filter immediately behind the column oven. The reference gas outlet is disconnected inside the column oven. Disconnect the sample gas inlet at the fitting on the column. The detector block sample gas inlet tubing has a copper sheath for identification. The sample gas outlet is usually routed out the right side of the column oven.

Exploded view of the standard TCD detector assembly



Replacing the TCD Filaments continued

(Standard TCD detector block access continued)

4. Cut the fiberglass tape wrapped around the detector block and peel it off. Unwrap and remove the heater rope from the detector block (it is probably affixed to the thermocouple wires with more fiberglass tape).

5. Disconnect the thermocouple by loosening the small philips head screw which holds it on the detector block clamshell. Next, remove the clamshell by unscrewing the two small philips head screws that hold its halves together. Gently remove the white insulation to reveal the detector block.

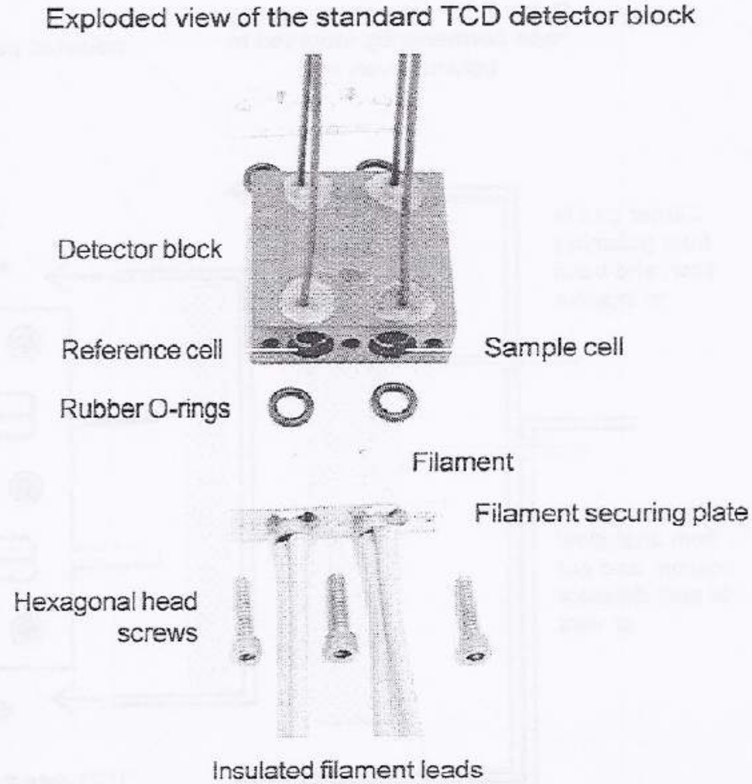
6. The TCD filaments are secured in the detector block by two plates, each of which is held in place with three hexagonal head screws. Holding the detector block with one hand, use an Allen wrench to unscrew and remove the hexagonal head screws from one of the filament securing plates. Then, slide the filament securing plate off the filaments and leads. Set it securely aside.

7. Once the securing plate is removed, the filament and rubber O-ring that seals it can be gently pulled out of the detector block cell. When replacing a filament, its rubber O-ring should also be replaced. Check the lip of the detector block cell for fragments of the old O-ring and if any are present, remove them as they will interfere with proper sealing of the cell. If you're replacing one reference or sample filament, replace the other at the same time. If you didn't have fun disassembling the TCD detector block, replace all the filaments while you have it open. It's a good idea to remove then replace one plate and corresponding pair of filaments at a time to avoid mixing up their connections.

8. To install a new filament, **color-code** it the same as the filament you are replacing, then slide it, leads first, through the appropriate hole in the filament securing plate. An existing or replacement filament should occupy the other hole. Place a new rubber O-ring against the rim of the detector block cell which will accept the new filament. Place filament securing plate and filaments against the detector block with the filaments inside the detector block cells. Replace and tighten the 3 hex-head screws. Repeat this process on other side to replace the corresponding filament.

9. Reverse your steps for TCD detector reassembly. Steps 7-10 of the high temperature TCD detector block access instructions detail reassembly of the inner clamshell and outer detector housing.

Exploded view of the standard TCD detector block



Replacing the TCD Filaments continued

B. High temperature TCD detector block access

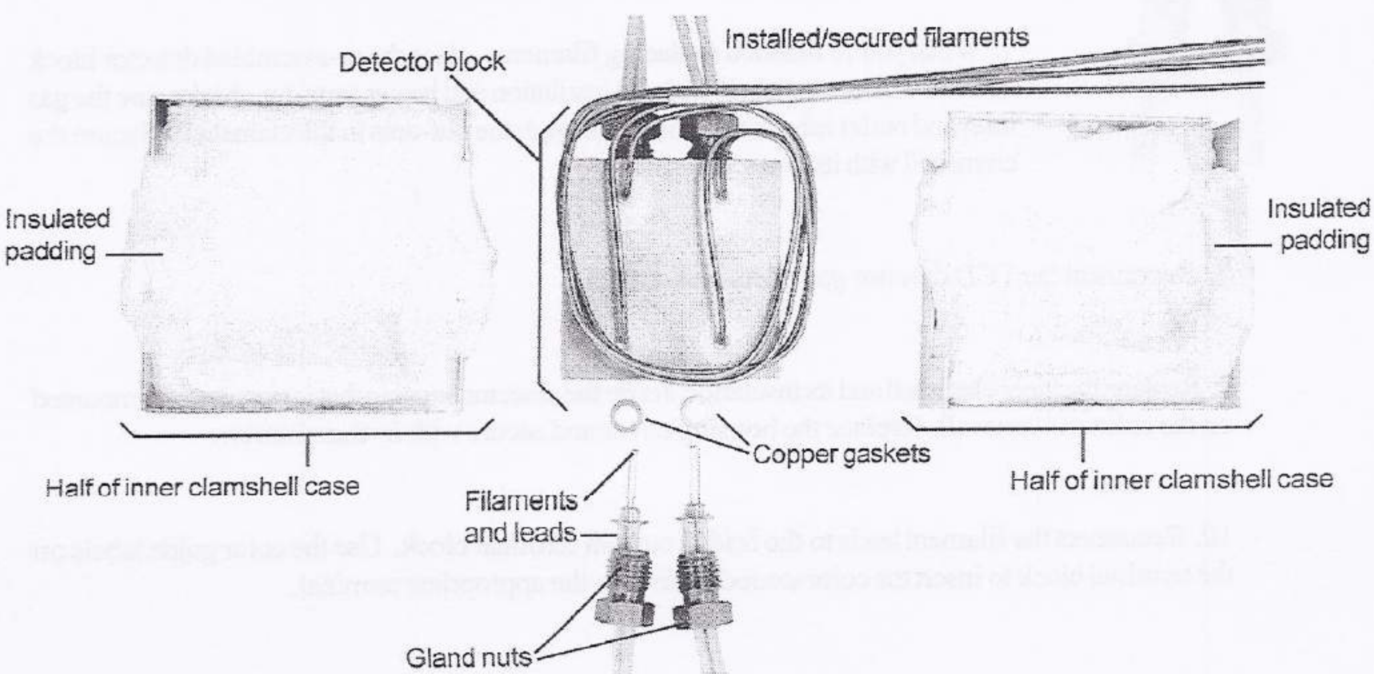
The high temperature TCD assembly is the same as the standard: outer housing around an inner clamshell case. The high temp detector block uses gland nuts and copper gaskets to secure the four filaments in its two cells. Instead of the heater rope, it employs a heating cartridge, which is inside the inner clamshell case with the detector block.

1. With a small blade screwdriver, disconnect the filament leads from the bridge terminal by loosening the setscrews.

2. Remove the detector housing by unscrewing the thumbscrew then sliding the housing cover off toward the right-hand edge of the GC. Gently remove the white insulation to reveal the detector block.

3. Disconnect the detector block gas inlets and outlets. The reference gas inlet is disconnected at the polishing filter immediately behind the column oven. The reference gas outlet is disconnected inside the column oven. Disconnect the sample gas inlet at the fitting on the column. The detector block sample gas inlet tubing has a copper sheath for identification. The sample gas outlet is usually routed out the right side of the column oven. Once these three fittings are loosened and the detector block tubing freed, gently pull the detector block away from the housing.

Exploded view of high temperature TCD detector block and inner clamshell

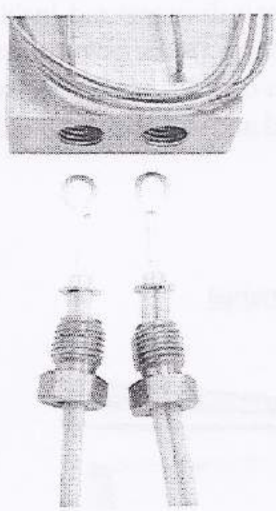


Replacing the TCD Filaments continued

(High temperature TCD detector block access continued)

4. Open the inner clamshell case by unscrewing the two small philips head screws that hold the two halves together. Gently remove the white insulation to access the detector block.

5. The filaments are held in place by gland nuts; loosen these nuts to remove the filaments and copper gaskets. **Color-code** the new filament the same as the one you are replacing (you can use colored marker pens, electrical tape, etc.) before completely removing the old one. Slide the gland nut off the existing filament, toward the ends of the filament leads.



6. Put the new filament's leads through the gland nut. Slide the gland nut up the filament's leads until it rests against the base of the filament. Place the copper gasket against the rim of the detector block cell opening. Carefully insert the filament and gland nut together into the cell opening. Tighten the gland nut to secure the filament in the cell.

7. When you're finished replacing filaments, place the re-assembled detector block inside the inner clamshell with the insulation and heater cartridge. Make sure the gas inlet and outlet tubes are running through the cut-outs in the clamshell. Secure the clamshell with its two screws.

8. Reconnect the TCD detector gas inlets and outlets.

9. Replace the inner clamshell and its insulation inside the detector housing that is permanently mounted on the column oven wall. Replace the housing cover and secure with its thumbscrew.

10. Reconnect the filament leads to the bridge current terminal block. Use the color guide labels on the terminal block to insert the color-coded leads into the appropriate terminal.

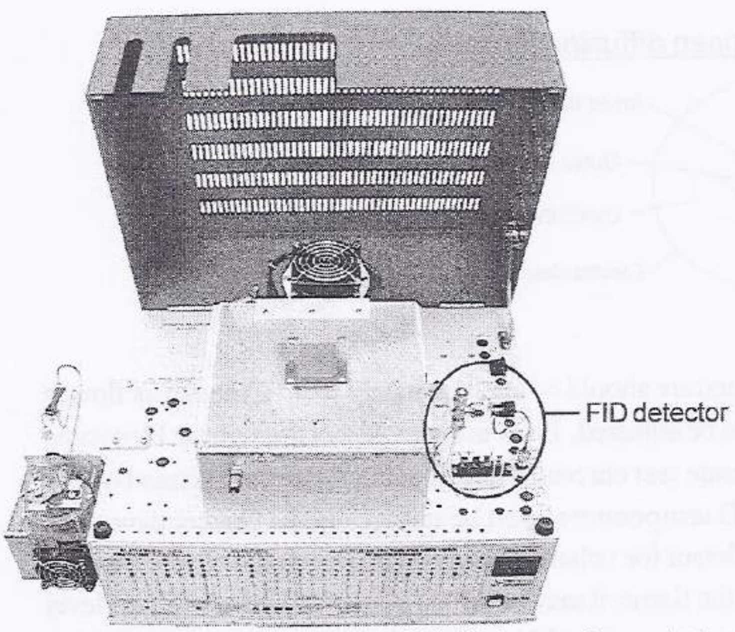
As illustrated by the table below, Helium and Hydrogen have the highest thermal conductivities of any gases. The TCD detector responds to the difference between the thermal conductivity of the carrier gas and the analyte peak. The greater the difference, the better the sensitivity. For this reason, Nitrogen is only used as a carrier gas when hydrogen or helium is the target analyte. Argon is sometimes used as a carrier gas, but would have little sensitivity towards ethane or propane, for example, because the thermal conductivity of the argon (39) is very close to that of ethane (43) or propane (36).

THERMAL CONDUCTIVITIES OF SOME COMMON GASES

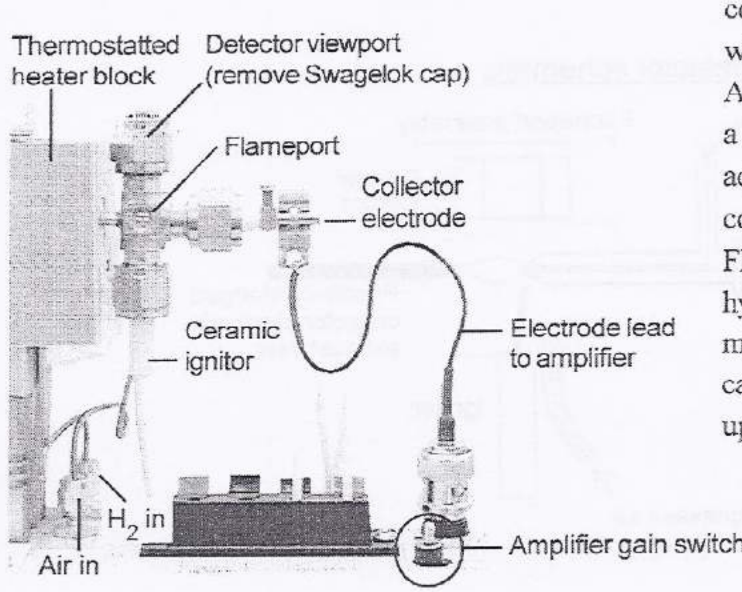
Air	58
Argon	39
CO	53
CO2	34
H2	419
HE	343
N2	57
O2	58
Neon	109
Methane	73
Ethane	43
Propane	36
Butane	32

Overview

The Flame Ionization Detector responds to any molecule with a carbon-hydrogen bond, but its response is either poor or nonexistent to compounds such as H_2S, CCl_4, or NH_3. Since the FID is mass sensitive, not concentration sensitive, changes in carrier gas flow rate have little effect on the detector response. It is preferred for general hydrocarbon analysis, with a detection range from 0.1ppm to almost 100%. The FID's response is stable from day to day, and is not susceptible to contamination from dirty samples or column bleed. It is generally robust and easy to operate, but because it uses a hydrogen diffusion flame to ionize compounds for analysis, it destroys the sample in the process.



(SRI Capillary FID GC with built-in Hydrogen Generator)



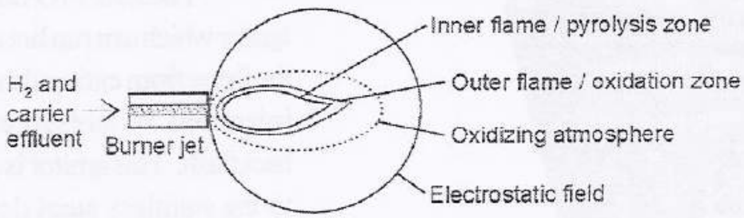
The SRI FID features a unique ceramic ignitor which can run hot continuously, and prevent the flame from extinguishing even with large water injections or pressure surges from column backflush. This ignitor is positioned perpendicular to the stainless steel detector jet and does not penetrate the flame. Opposite this flame is the collector electrode. This positively charged metal tube serves as a collector for the ions released as each sample component elutes from the column(s) and is pyrolyzed in the flame; it doubles as a vent for the FID exhaust gas. The FID is equipped with an electrometer amplifier which has HIGH, HIGH (filtered), and MEDIUM gain settings. On an SRI GC, the hydrogen and air gas flows are controlled using electronic pressure controllers, which are user adjustable via the GC's front panel. A thermostatted aluminum heater block maintains a stable detector temperature which is user adjustable up to 375°C. The optional built-in air compressor may be used to supply the air for the FID, eliminating bulky air cylinders. The built-in hydrogen generator is another option: the standard model can produce 20mL/min for use as both carrier gas and FID combustion gas at pressures up to 25 psi.

DETECTORS
FID - Flame Ionization Detector

Theory of Operation

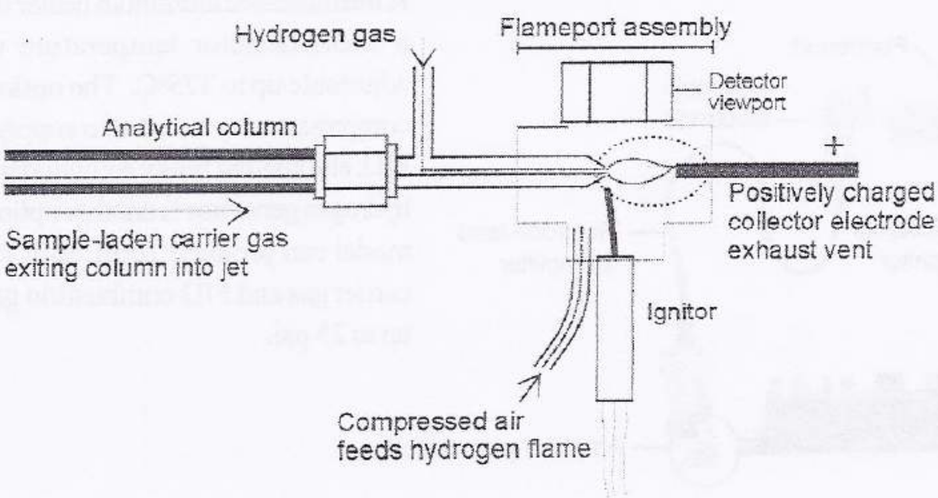
In the SRI FID, the carrier gas effluent from the GC column is mixed with hydrogen, then routed through an unbreakable stainless steel jet. The hydrogen mix supports a diffusion flame at the jet's tip which ionizes the analyte molecules. Positive and negative ions are produced as each sample component is eluted into the flame. A collector electrode attracts the negative ions to the electrometer amplifier, producing an analog signal for the data system input. An electrostatic field is generated by the difference in potential between the positively charged collector electrode and the grounded FID jet. Because of the electrostatic field, the negative ions have to flow in the direction of the collector electrode.

The FID hydrogen diffusion flame



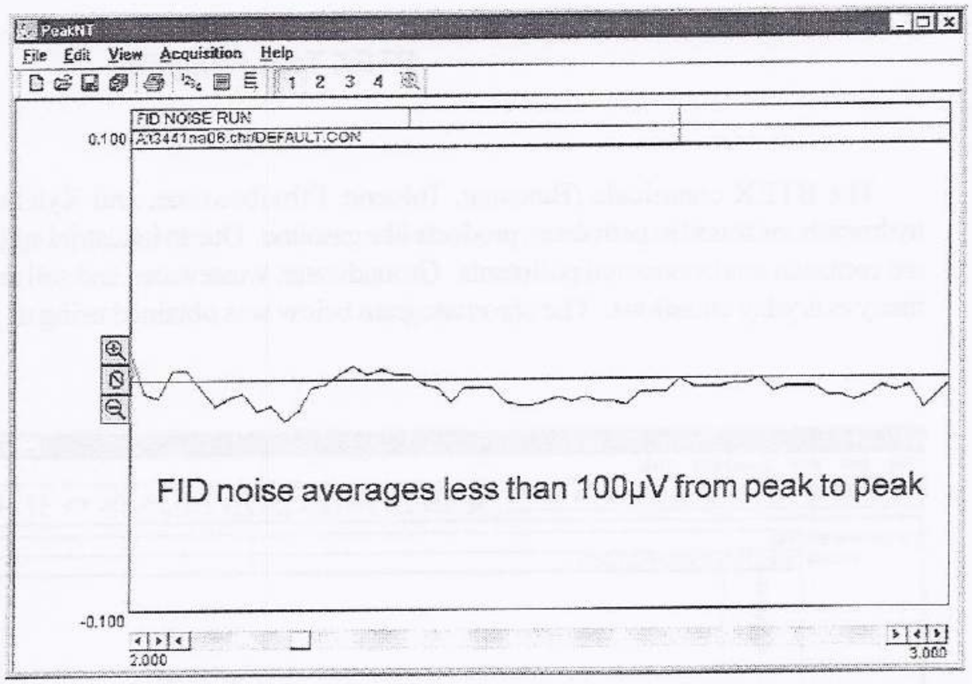
The ratio of air to hydrogen in the combustion mixture should be approximately 10:1. If the carrier flow is higher than normal, the combustion ratio may need to be adjusted. Flow is user adjusted through the Electronic Pressure Controllers (EPC); the rates used to generate test chromatograms at the factory are printed on the right side of the GC in the flow rate chart. The FID temperature must be hot enough so that condensation doesn't occur anywhere in the system; 150°C is sufficient for volatile analytes; for semi-volatiles, use a higher temperature. In addition to using the ignitor to light the flame, it may be left on at an intermediate voltage level to prevent flameout (-750 or 7.5 volts). The ignitor is very durable and will last a long time, even at high temperatures.

FID detector schematic



Expected Performance

FID noise run

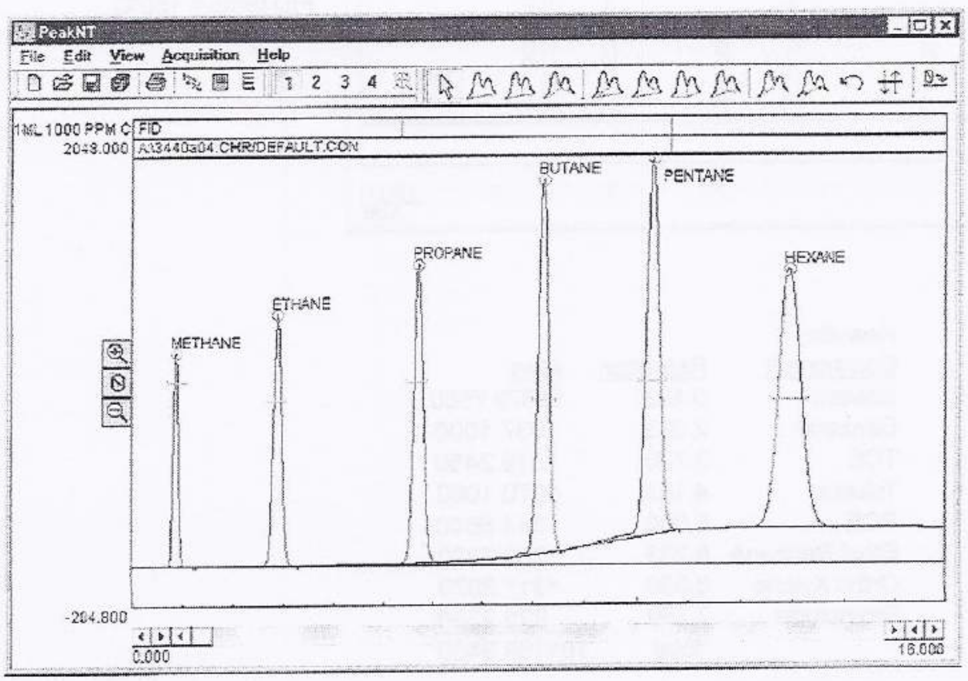


Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Temperature program:

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

C_1-C_6 Hydrocarbon Test Analysis



Sample: 1mL of 1000ppm C_1-C_6
Carrier: Helium @ 10mL/min
FID H_2 at 25psi = 25mL/min
FID air at 6psi = 250mL/min
FID temp = 150°C
FID ignitor = -750
FID gain = HIGH
Valve temp = 90°C

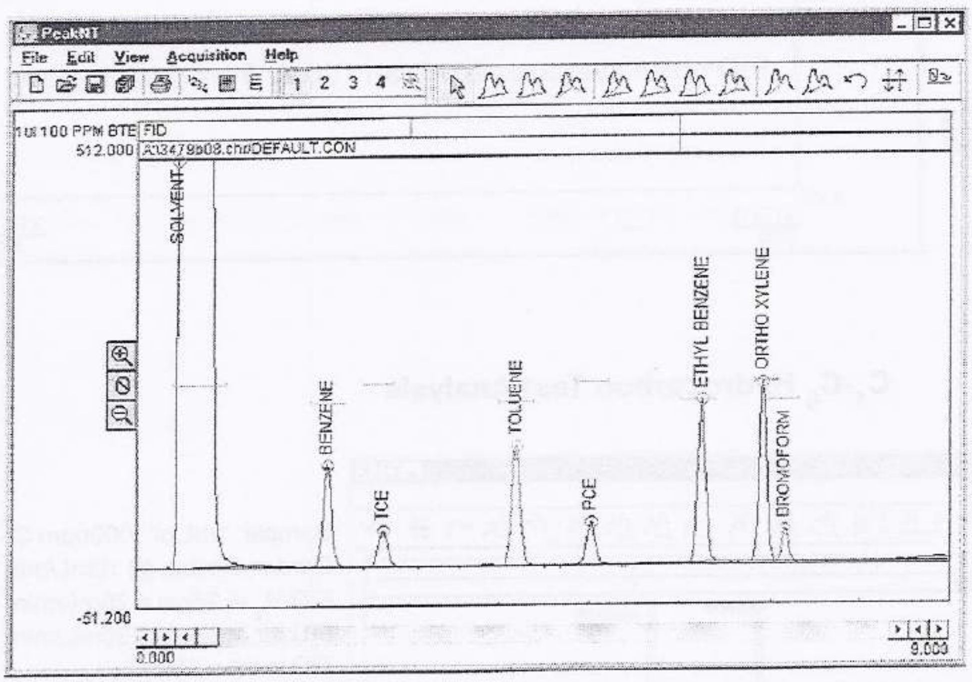
Results:

Component	Retention	Area
Methane	0.850	6979.9260
Ethane	2.866	13623.7580
Propane	5.683	19535.8960
Butane	8.200	26456.5980
Pentane	10.283	33053.9680
Hexane	12.916	39419.0870
	Total	139069.2330

Expected Performance

BTEX Test Analysis

The BTEX chemicals (Benzene, Toluene, Ethylbenzene, and Xylenes) are volatile monoaromatic hydrocarbons found in petroleum products like gasoline. Due to industrial spills and storage tank leakage, they are common environmental pollutants. Groundwater, wastewater, and soil are tested for BTEX chemicals in many everyday situations. The chromatogram below was obtained using an FID-equipped SRI GC.



1µL 100ppm BTEX sample

15m MXT-VOL capillary column

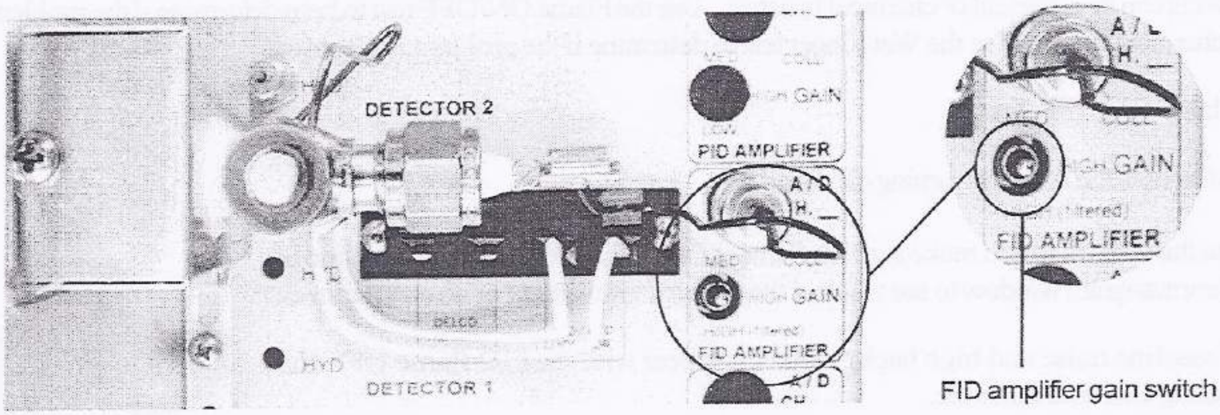
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Results:

Component	Retention	Area
Solvent	0.433	95879.7560
Benzene	2.083	837.1000
TCE	2.700	319.2450
Toluene	4.183	1070.1060
PCE	5.000	344.8640
Ethyl Benzene	6.233	1200.3320
Ortho Xylene	6.900	1312.3070
Bromoform	7.150	225.2360
	total	101188.9460

General Operating Procedure

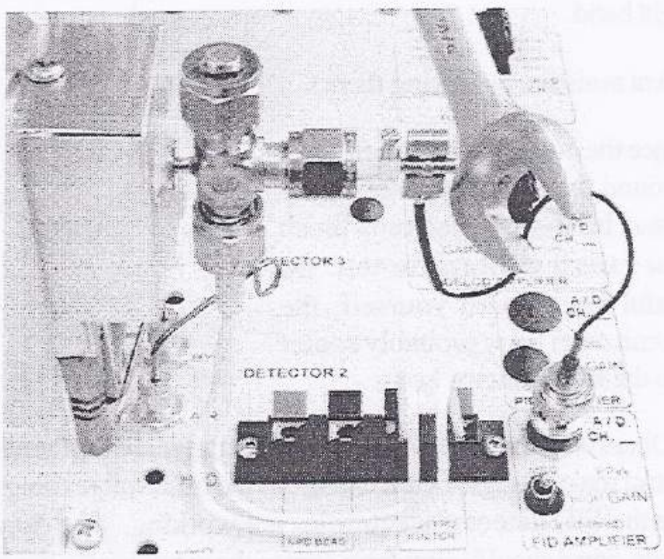
1. Set the FID amplifier gain switch to HIGH for most hydrocarbon applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM. When peaks of interest are 20 seconds wide or more at the base and extra noise immunity is desired, set the gain switch to HIGH (filtered).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



FID amplifier gain switch

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

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



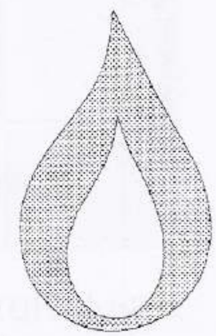
4. Verify that the FID flame is lit by holding the shiny side of a chromed wrench directly in front of the collector outlet/FID exhaust vent. If condensation becomes visible on the wrench surface, the flame is lit.

5. If you wish to keep the ignitor ON to prevent flameout, set the ignitor voltage to -750 by adjusting the trimpot on the "FLAME IGNITE" zone with the supplied screwdriver.

FID Troubleshooting

Whenever you experience problems with your FID, review your operating procedures: check the detector parameters, check to make sure you are on the correct channel of the data system display, check the mixture of hydrogen (25mL/min) and air (250mL/min), check gas pressures and connections, check the oven and detector temperatures, and all the other variables that compose your analysis. Having ruled out operating procedure as the source of the problem, there are two simple diagnostic tests you can perform. Detector problems can be electrical or chemical in nature. Use the Flame ON/OFF test to help determine if the problem is of chemical origin. Use the Wet Finger test to determine if the problem is electrical.

A. Flame ON/OFF Test



1. Extinguish the flame by turning off the air.

2. Use the wrench test to make sure the flame is OFF. If it is, observe the baseline in the chromatogram window to see whether there is an improvement or no change at all.

3. If baseline noise and high background disappear with the FID flame OFF, the problem is chemical in nature.

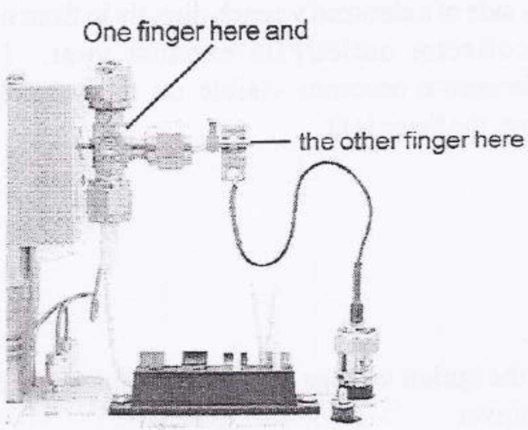
4. Isolate the column by capping off the column entrance to the detector with a swagelok-type cap or a nut and septum. Turn the air back on and light the FID flame. If the detector noise is similar to the background that was observed with the flame OFF, the column is suspect.

B. Wet Finger Test

1. Make a V sign with the first two fingers of your right hand.

2. Moisten those two fingers (you can achieve sufficient moisture by licking them).

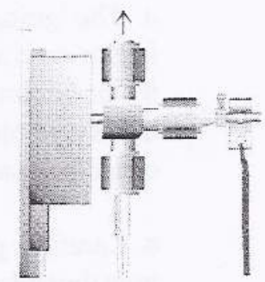
3. Place one finger on the collector electrode, and place the other on bare metal (like the FID detector body or the column oven lid) to ground the collector. Make your contact brief--you need only brush these parts to perform the test. Be careful not to burn yourself; the column oven lid is probably cooler than the FID detector body.



5. Observing the milliVolt reading on the screen. If your contact makes a significant change in the milliVolt reading, then the FID detector electronics are working. The data system signal should jump from zero to the maximum voltage (5,000mV), then come back down when you remove your fingers.

Cleaning the FID

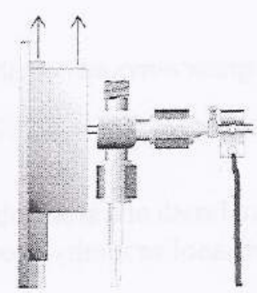
The FID detector rarely requires cleaning or servicing. It may develop a film or coating of combustion desposits in the flameport with extended use. Use the FID detector viewport to check for visible deposits. If you're experiencing problems with your FID detector, try cleaning it, even if you can't see deposits through the viewport.



1. Unscrew the viewport cap nut and examine the flameport interior for coatings or films. If residue is found, the collector electrode and the flameport will need cleaning.

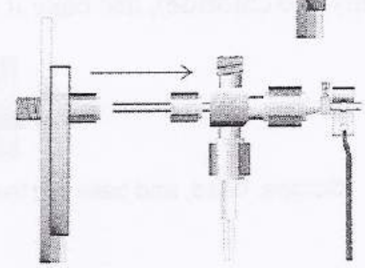
2. Remove flameport assembly from the heater block

   a. Disconnect the FID air supply line at the 1/16" bulkhead fitting.

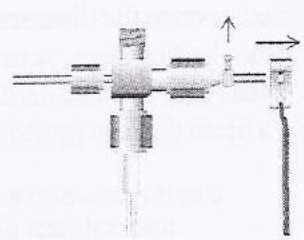
   

   b. Using a philps head screwdriver, remove the screw on the top of the FID's heater block and pull the aluminum cover up and off.

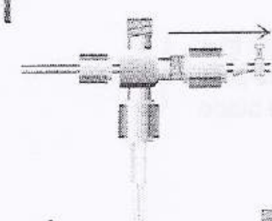
   c. Gently pull off the white insulation to reveal the detector's bulkhead fitting on the column oven wall. Loosen this fitting to disconnect the flameport.

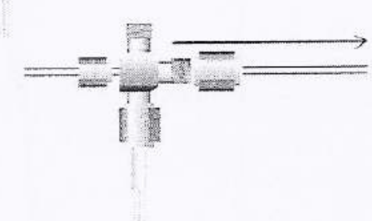
3. Remove the collector electrode

   a. Unclip the electrode lead terminal and slide it off the electrode.

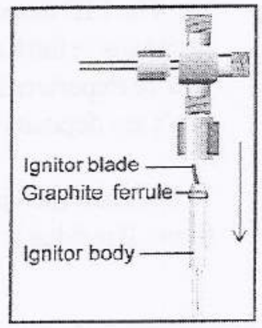
   b. Loosen and remove the nut and ferrule that hold the collector electrode in the flameport body.

   c. Slide the collector electrode out of the nut. Once removed, spin it between your fingers in a piece of sandpaper to clean the stainless steel surface. A wire brush may also be used to scrub the electrode. Once cleaned, set it aside with the ignitor.

Cleaning the FID continued

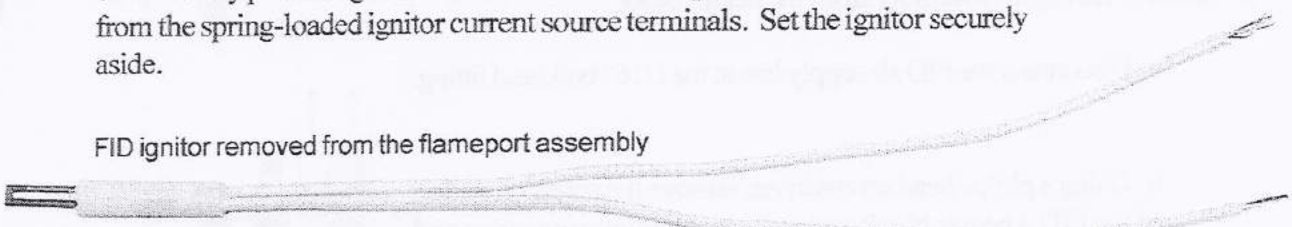
4. Remove the FID ignitor element



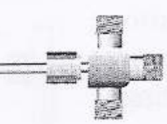
a. The ignitor element is brittle and will break when stressed, so handle the ignitor carefully, mindful of any torque on the blades. While holding the ignitor by the ceramic body with one hand, loosen the 1/4" swagelok-type nut that holds it in place. There is a graphite ferrule inside this nut that secures the ceramic ignitor body when the nut is tightened.

b. Carefully pull the ignitor down out of the flameport. Disconnect the ignitor from the spring-loaded ignitor current source terminals. Set the ignitor securely aside.

FID ignitor removed from the flameport assembly



5. Use a wire brush or a sharp object to remove any residue from the flameport interior, then rinse it with solvent (methanol or methylene chloride), and bake it out in the GC's column oven at 250°C for 10-15 minutes.



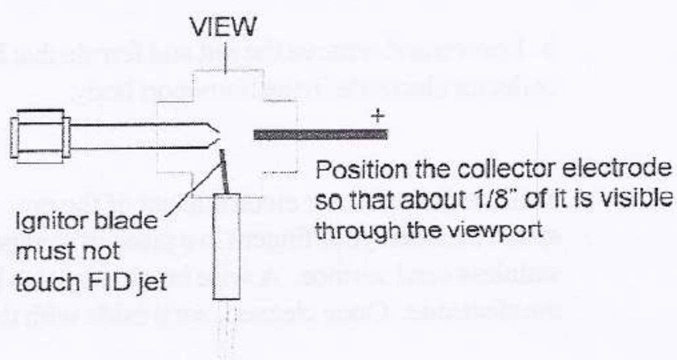
Scrape, rinse, and bake out the FID flameport interior

6. Re-assembly

a. Once all the FID parts are cleaned, reverse the disassembly process, starting with the replacement of the ceramic ignitor. Leaving out the cleaning steps, your last step should be reinstalling the flameport assembly onto the heater block. Make sure to position the ignitor so that the blade is slightly below and angled 10-15° toward the jet's tip so that the ignitor will not interfere with the flame or create turbulence.

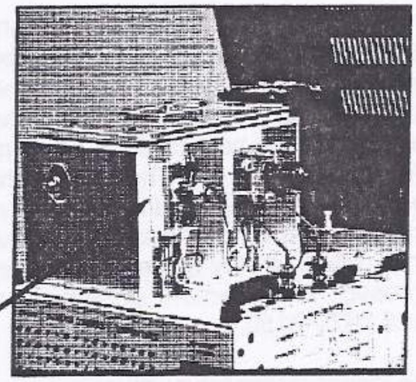
FID ignitor removed from the flameport; note the slight angle of the blade element

Use the viewport to correctly position the FID ignitor and collector electrode inside the flameport



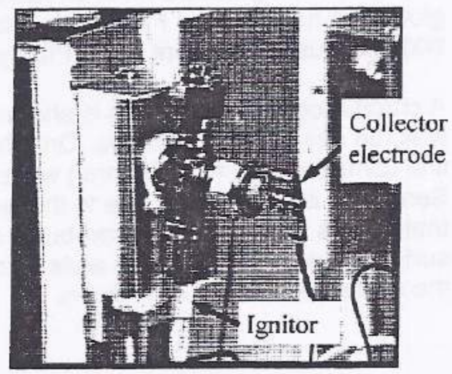
Chapter: FID DETECTOR

Topic: Operation of FID detector without hydrogen (FLID mode)

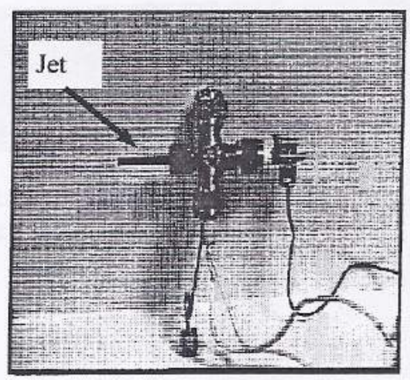


There are situations where it would be helpful to operate the FID detector using just the built-in air compressor for carrier gas and no other gases. SRI distributors demonstrating the GC and software may find it useful to run live chromatograms without the inconvenience of providing hydrogen and helium. Service personnel troubleshooting other GC functions may be able to test the GC without gases, and under some circumstances, the response of the flameless ionization detector (FLID) may actually be useful for non-quantitative applications.

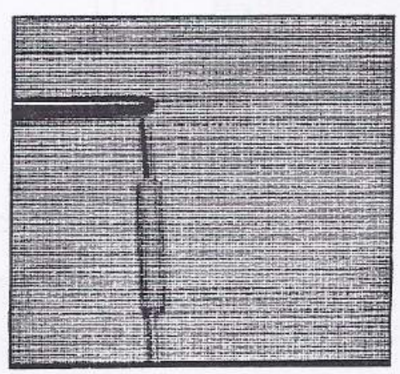
The FID detector is normally located on the right hand side of the column oven.



The FID normally requires a flow of 20-30 ml/min of hydrogen and 200-300 ml/min of air to support a hydrogen flame at the tip if the jet. The heat of the flame ionizes the analyte molecules, and the negative ions allow a small electric current to flow between the collector electrode and the grounded flame jet. The ignitor normally serves only to ignite the flame.



The FID detector body is shown at right in the normal configuration, but removed from the detector heating block on the GC for clarity.

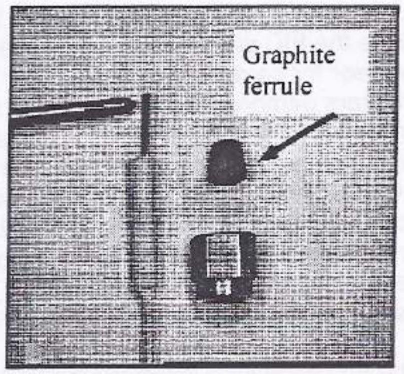


Inside the FID detector body, the ignitor is normally positioned just below and behind the tip of the jet. Notice that the ignitor blade is tilted at a 15 degree angle from the ceramic tube in which it is fabricated. In normal FID operation, the ignitor is positioned below and behind the jet so it will not disrupt or distort the flame, yet close enough to easily ignite the hydrogen/air mixture.

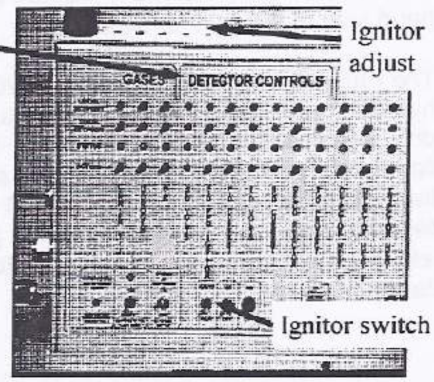
Chapter: FID DETECTOR

Topic: Operation of FID detector without hydrogen (FLID mode)

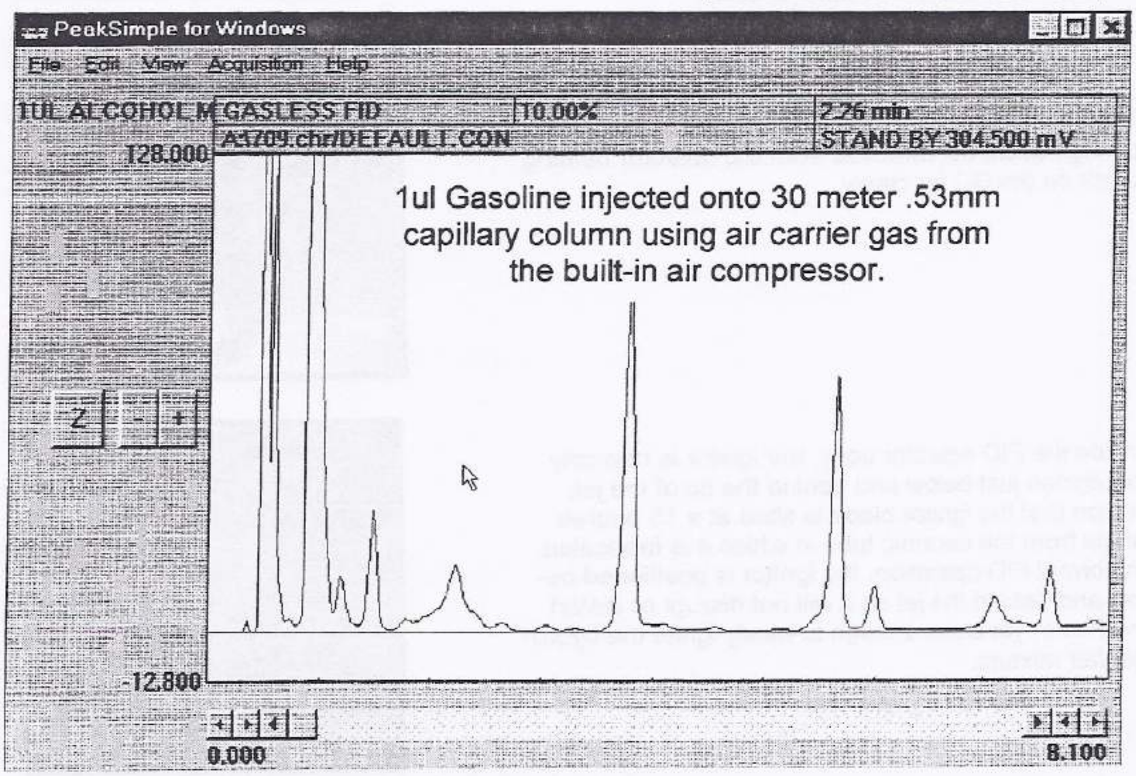
In the FLID mode, the ignitor itself provides the heat necessary to ionize the sample molecules. Accordingly, the ignitor needs to be positioned directly in front of the jet. The slight angle of the ignitor allows the ignitor tip to be located 1-2 mm in front and slightly above the jet. The ignitor is held in place by a soft graphite ferrule and a swagelok nut. Be careful when manipulating or twisting the ignitor because the ignitor blade is very brittle ceramic, and will snap if stressed. Replacement ignitors are available using part# 8670-0150.



The ignitor temperature must be raised so that it glows red hot. Set the FID ignitor volts to at least 900-1000 using the front panel FID Ignitor control.



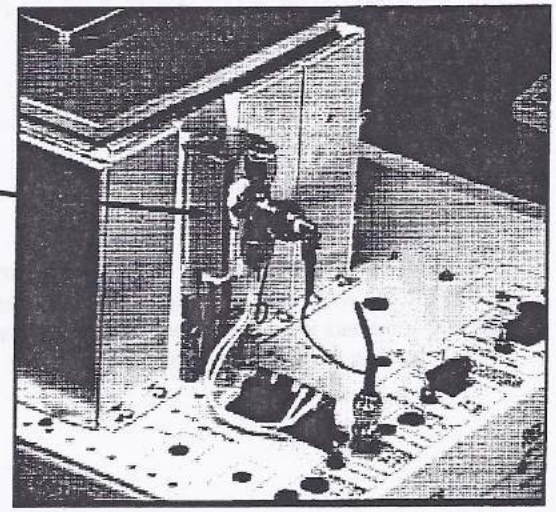
A chromatogram of gasoline is shown below which was run using the FLID mode. Only the larger gasoline components (> 1000 ppm) were detected. Sensitivity is exponential due to the temperature rise that occurs when the peak combusts on the ignitor surface. Large peaks which elute quickly may cool the ignitor resulting in split peaks.



Chapter: Detectors

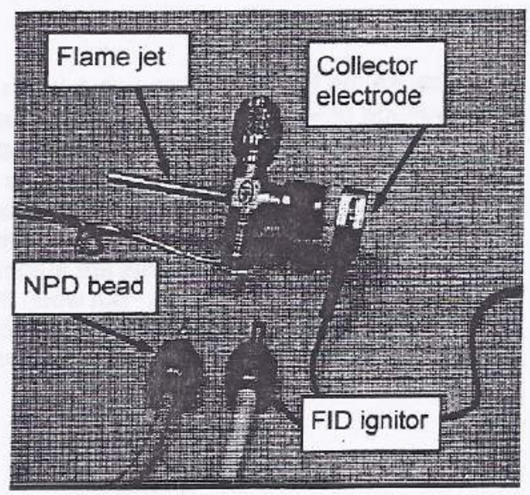
Topic: Converting from FID to NPD mode

To convert the FID detector to NPD detector:

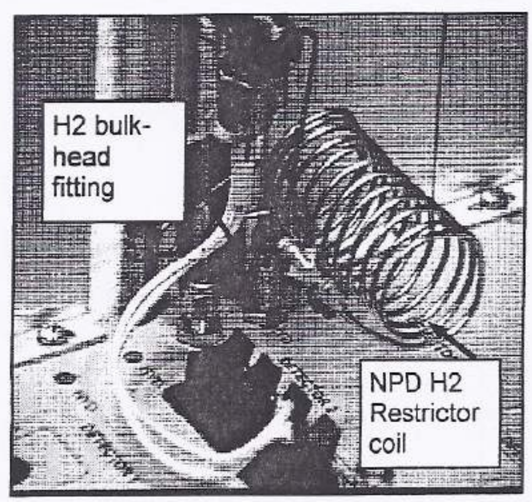


1) The FID and NPD detectors are almost identical. The detector body is mounted on a heated aluminum block on the right hand side of the GC oven.

The NPD body is slightly different from the FID in that the NPD flame jet does not protrude as far into the detector body as it does on the FID. This allows the NPD thermionic bead to be positioned directly in front of the jet. Remove the FID body from the heated aluminum block and replace it with the NPD body.



2) The photo at right shows the FID/NPD detector body and both the FID ignitor and NPD thermionic bead side by side for comparison. Both the FID ignitor and NPD thermionic bead are inserted into the detector body from the bottom. The ignitor is inserted until the tip of the ignitor is just below the tip of the flame jet, while the NPD bead is inserted until the heated part of the bead is directly in front of the flame jet. For NPD operation, the sample molecules must collide with the bead in order to be ionized and detected.



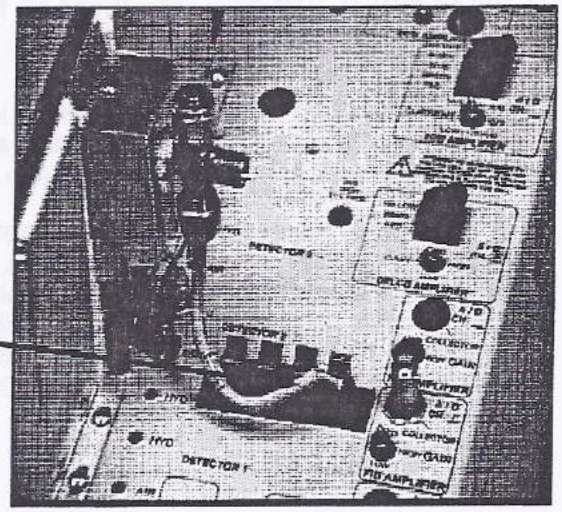
3) The gas flows to the NPD detector are different than the FID gas flows. The NPD hydrogen flow is normally about 3 ml/min while the FID hydrogen flow is about 25ml/min. To obtain this lower H2 flow rate, an additional restrictor coil is attached to the hydrogen bulkhead fitting immediately below the detector body. With this additional restrictor coil in place, 10 psi hydrgen pressure will result in a flow rate of about 3ml/min. The NPD air flow rate is typically about 100 ml/min, but this flow rate can be achieved by simply reducing the air pressure from 8 psi to about 3 psi.

Chapter: Detectors

Topic: Converting from FID to NPD mode

To convert the FID detector to NPD detector:

4) The NPD bead plugs into the push terminal block on the GC directly beneath the detector. The terminals are labelled FID ignitor because this is where the FID ignitor is normally connected.



5) Because the NPD bead can only tolerate a maximum voltage of –4.50 volts, be careful not to set the FID volts setpoint higher than –4.50. Be especially careful not to flip the FID ignite switch to the up position, as this will apply 10 volts to the NPD bead and burn it out. When an NPD detector is ordered separately from the FID, the NPD volts are automatically limited to –4.50 volts maximum. But when the FID and NPD share the bead/ignitor circuit, the operator must be careful not to apply more voltage than the bead can tolerate.