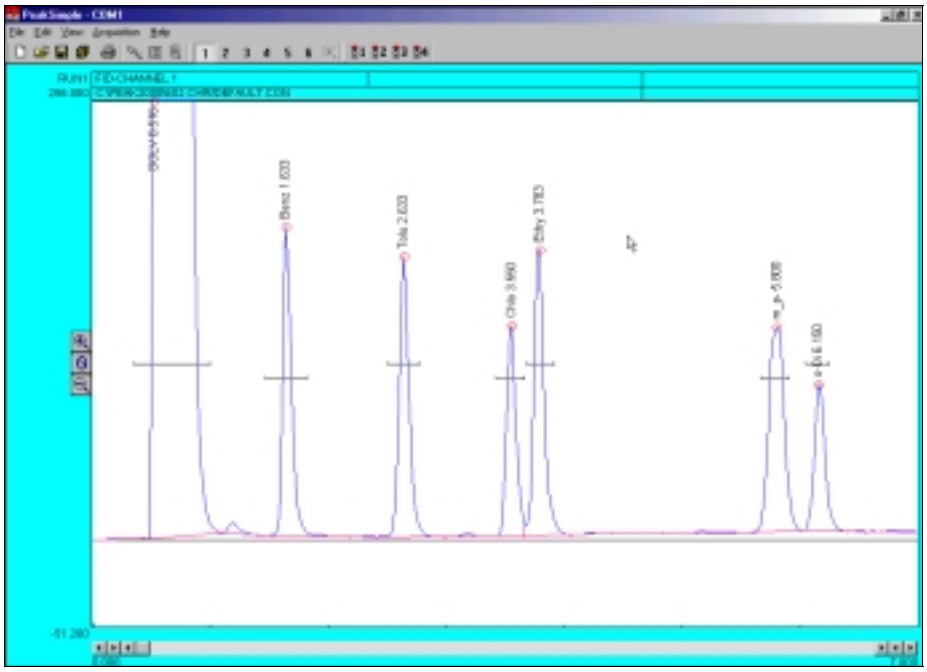
PeakSimple 2000

Chromatography Integration Software

Basic Tutorial



Installing PeakSimple 2000 from floppy disk or CD-Rom

A. Start the Windows operating system in use on your computer. (Windows 95, 98, ME, 2000)

B. Insert the PeakSimple 2000 disk or CD into your floppy disk drive.

C. Go to the **Start** menu in the bottom left hand corner of the windows screen and select **Run** from the set of icons.

D. From the run menu, type **X:\setup** (where **X** is the letter of your computers disk drive).

E. Now click on the **Continue** button with your mouse cursor or press the enter key on your keyboard to begin installation.

F. To complete installation follow the onscreen instructions provided by the installation wizard.

Installing PeakSimple 2000 from software download

A. Start the Windows operating system and use an online browser to access www.srigc.com.

B. From the menu on the left hand side of the screen select **Download our Software** and then download PeakSimple 2000 from the following page.

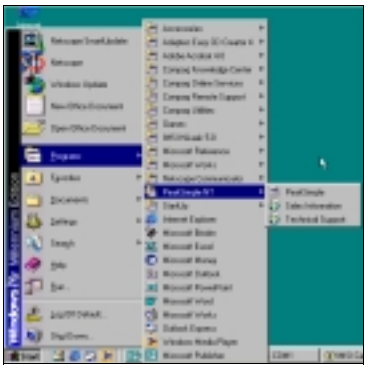
C. Save the file to a temporary folder and then double click on it from My Computer to allow the program to self-extract.

D. Once all the files have been extracted successfully double-click the install file and press the **Continue** button when prompted.

E. Follow the onscreen instructions to complete the installation of PeakSimple.

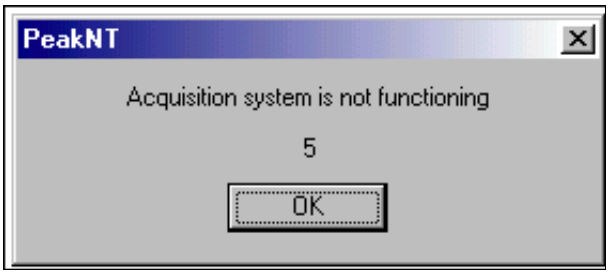
Launching PeakSimple 2000

1. Click on the windows **Start** button in the bottom left-hand corner of the screen. Select **Programs** and then **PeakSimple** from the list of program groups on the screen and then click on **PeakSimple.**



2. This will launch PeakSimple and initialize the data acquisition system.

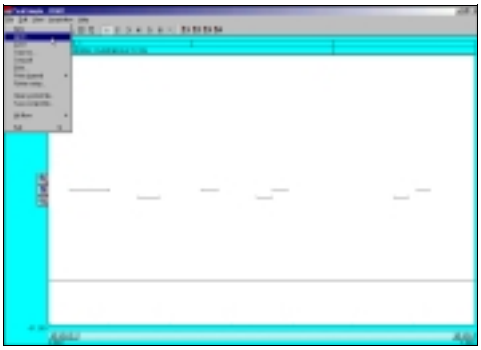
3. If PeakSimple comes up with an error message stating “Acquisition system is not functioning” with a countdown timer, it is indicating that there is a communication problem between the computer and the data system or that the data system and the hardware is not connected. Click **OK** to continue working with PeakSimple.



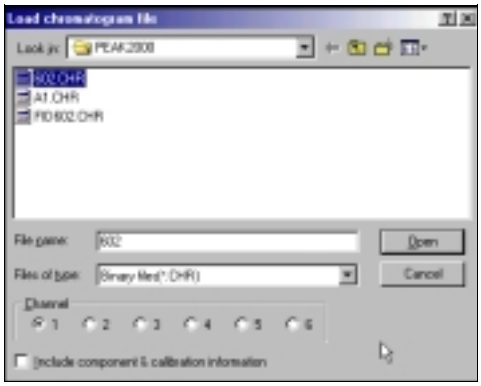
4. Most of the commands and options in PeakSimple are equipped with tool tips that will automatically pop up to display useful information when the mouse cursor is held over a command. To turn off the tool tips deselect the tool tips option in the Help menu.

Opening a PeakSimple Data File

1. To open a PeakSimple data file or chromatogram, begin by selecting **File** in the PeakSimple menu bar and then choose **Open…** from the set of options.



2. The Load Chromatogram File window is now open. The PeakSimple software includes a number of sample chromatogram data files that can be opened, displayed, and manipulated. One file, 602.CHR, will be used throughout the rest of the tutorial. Select file **602.CHR** from the PeakSimple directory, choose **Channel 1** as a destination channel, and then select **Open** to load the file.

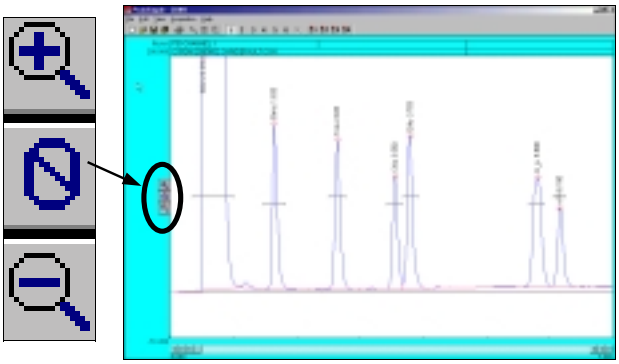


Adjusting Display Limits

1. To adjust the display limits of a chromatogram click on either the **+** magnifying glass icon or the **-** magnifying glass icon to the left of the chromatogram. This will increase or decrease the limits by a factor of two each time you click on the icons.

2. After opening chromatogram 602.CHR, practice making the display limits smaller but the peaks larger by clicking the **+** magnifying glass icon.

3. Practice making the display limits larger but the peaks smaller by clicking on the **-** magnifying glass icon.

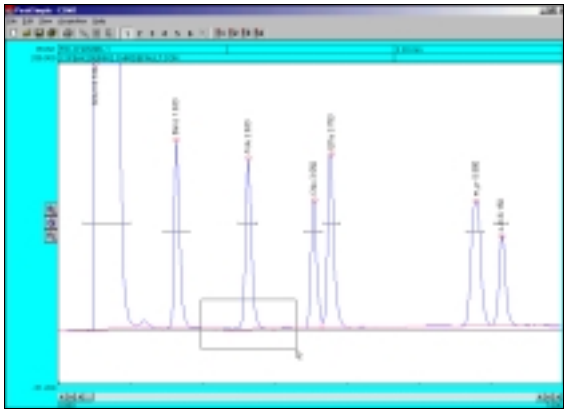


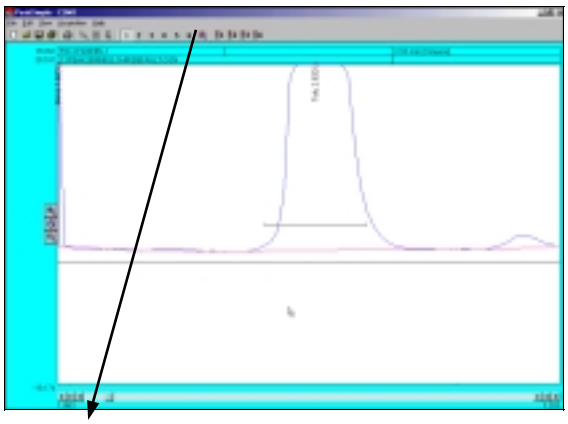
Zooming

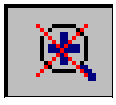
1. To zoom in on a specific part of a PeakSimple chromatogram, click and hold the left mouse button and drag it over the desired area.

2. After opening chromatogram 602.CHR hold the left mouse button and drag it over the base of the toluene peak. Let go of the mouse button and there will be a larger view of the area that was selected.

3. To return to the original display limits of the chromatogram and unzoom the area selected press **F6** or select the unzoom icon located in the PeakSimple toolbar at the top of the screen.







Dragging Retention Windows

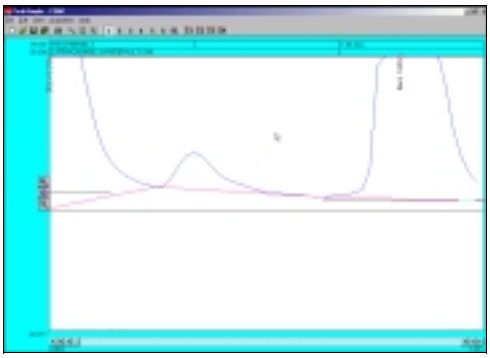
1. To drag a retention window bar place the mouse cursor on the bar until a double sided arrow pops up. Click on the left mouse button and hold and then drag the retention window bar to its desired place.

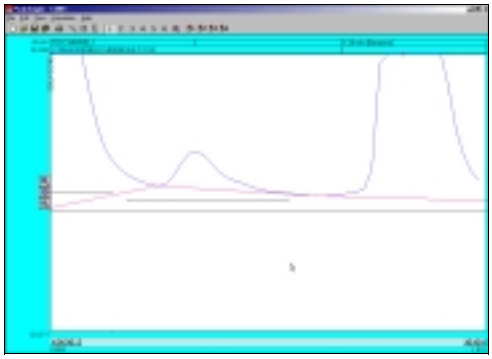
2. After opening the chromatogram 602.CHR zoom in on the benzene peak and the smaller peak to its left. Locate the benzene retention window bar and drag it over to the smaller unnamed peak to the left of the benzene. Because this is a small peak it is not immediately recognized.

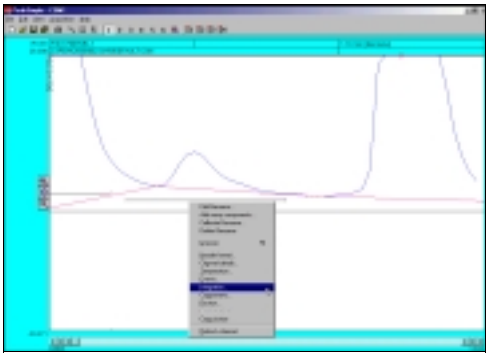
3. Right click on the chromatogram over the unnamed peak and select **Integration** from the resulting menu.

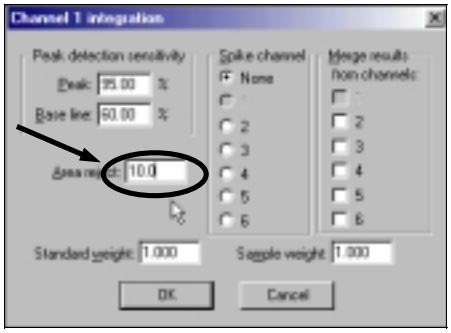
4. From the integration window locate the **Area Reject** dialogue box, erase the 100.0 in the box, and add the number **10.0** to the dialogue box. Click **OK** and the integration window will exit.

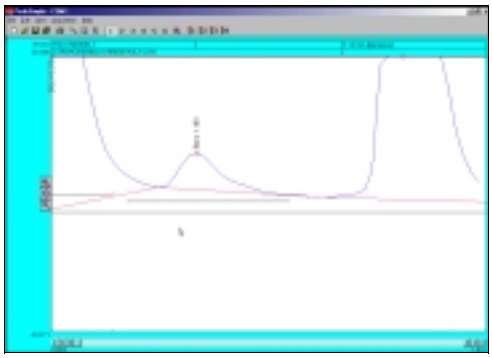
5. Press the **Enter** or **Return** key on your keyboard and the smaller peak will now be recognized as Benzene.





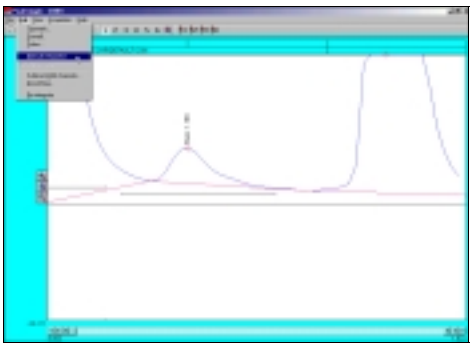




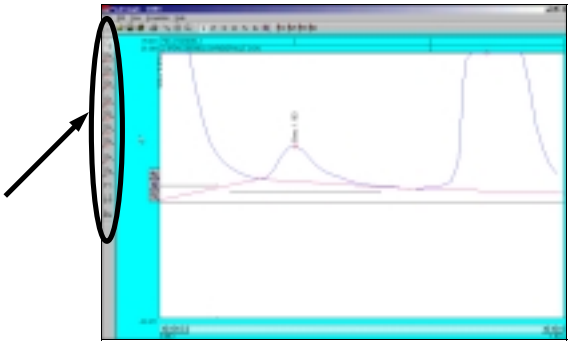


Manual Integration

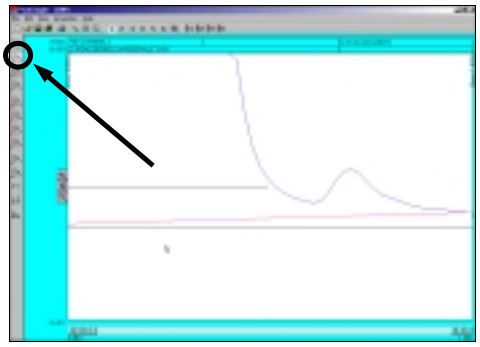
1. To manually adjust the integration baseline and peak separation in a chromatogram use the manual integration toolbar provided by PeakSimple. To open up the manual integration toolbar select **Edit** in the PeakSimple menu bar and then click on the **Manual Integration** option. The manual integration toolbar will now appear to the left of the chromatograph.



2. The manual integration toolbar contains nine types of manual integration options. Four of the most commonly used options are **None** integration, **Drop** integration, **Based** integration, and **Rubber Band** integration.

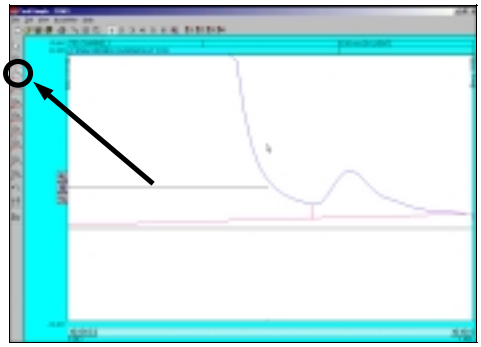


3. To make a baseline ignore a peak use the None integration tool. After opening chromatogram 602.CHR and the manual integration toolbar, zoom in on the baseline of the solvent peak and the smaller unrecognized peak immediately to its right. Click on the **None** integration tool in the manual integration toolbar with the mouse cursor and then click on the valley between the two peaks where they meet the baseline. The area of the small peak is now added to the solvent peak.

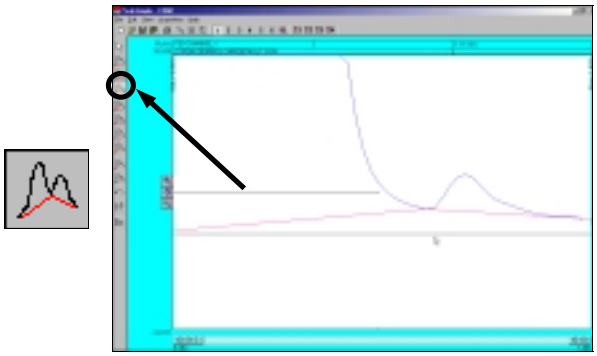


4. To undo the changes made to a chromatogram at any time simply click on the **Undo** integration tool in the manual integration toolbar. After selecting this tool all integration changes made to the chromatogram will be undone.

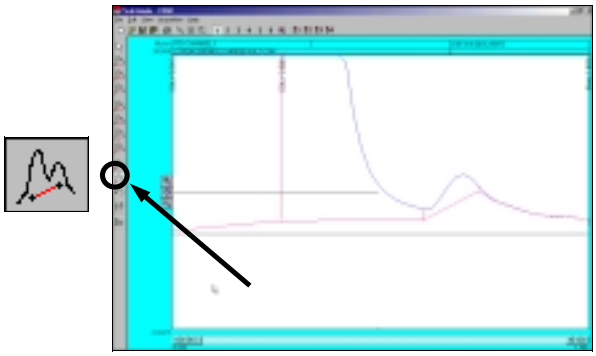
5. Click on the **Undo** tool with your mouse cursor and select the **Drop** integration tool to enable the dropping of the baseline below the between the two peaks. After selecting the Drop tool click where the valley of the peaks meet the baseline with the cursor. The baseline should now be dropped below the base of the peaks and a line should extend from it to the baseline.



6. After the manual integration between the two peaks is dropped use the **Based** integration tool to raise the baseline to the valley between the peaks. Once the Based integration tool is selected, click on the valley between the solvent peak and the smaller peak to its right with the mouse cursor. The baseline will now extend up to meet the valley of the two peaks.

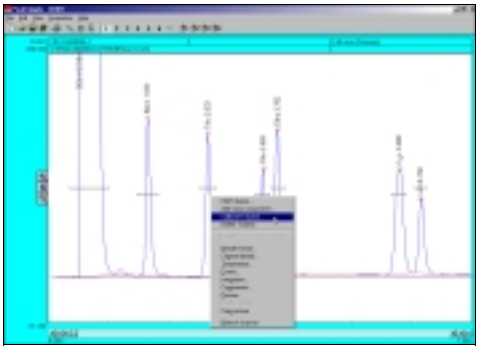


7. Once again click on the **Undo** tool in the manual integration toolbar to remove all changes done to the chromatogram. Select the **Rubber Band** integration tool to manually draw a baseline. Once the Rubber Band tool is selected take the mouse cursor and click on a part of the baseline. While holding down the left mouse button extend the line to another part of the baseline further to the right of the starting point and let go of the mouse button. The base line will now be drawn according to the line that was drawn using the Rubber Band integration tool.

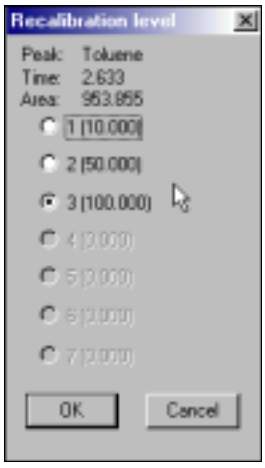


Calibration

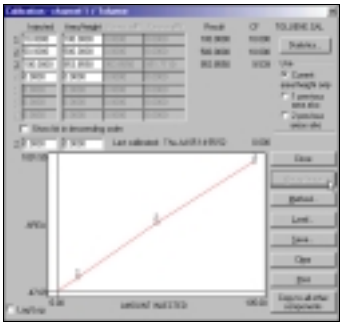
1. To turn the raw area of a peak into a real-world number the peak first needs to be calibrated. To calibrate the Toluene peak in chromatogram 602.CHR, open up the file and then right click using the mouse on the Toluene peak. After right clicking on Toluene select **Calibrate Toluene** from the resulting menu.



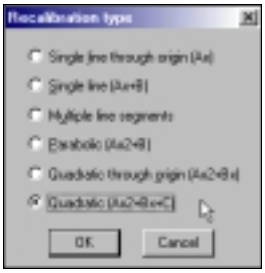
2. From the Recalibration level window click on the third level radio button **3 (100.000)** and then select **OK** with your mouse cursor.



3. After selecting OK from the Recalibration level menu the Calibration menu for Toluene will pop up. Check to make sure the flashing asterisk on the calibration curve is on level 3 and then click on the **Accept New** button to the right of the window.



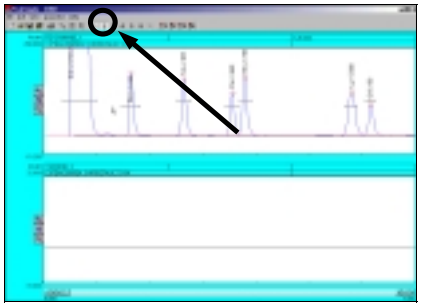
4. Once the new data is accepted, click on the **Method** button immediately below the Accept New button. The Recalibration type window will now open allowing the user to select a method of calibration. By default the calibration type is set at Multiple Line Segments. Select the **Quadratic (Ax2+Bx+C)** radio button and then click on **OK** with the mouse cursor.



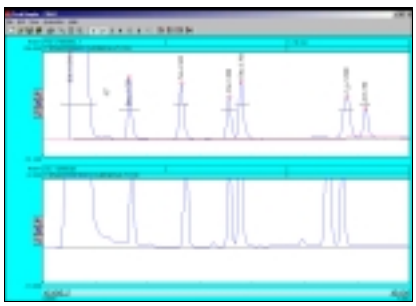
5. After changing the method of calibration click on **Statistics** in the upper right hand corner of the Calibration level window. The Calibration statistics window will pop up revealing the statistics for the calibration of Toluene. Click **OK** with the mouse cursor to close the Calibration statistics window and then select **Close** from the Calibration window to finish calibrating Toluene.

Overlay

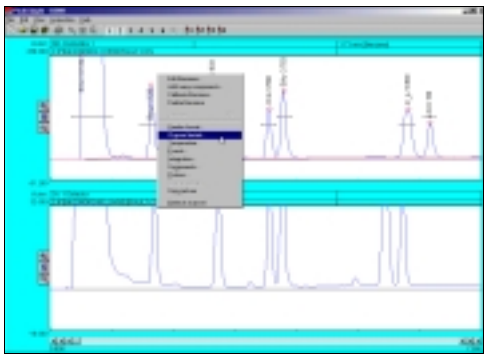
1. To compare two or more chromatograms overlay them using PeakSimple. To overlay two chromatograms first open chromatogram 602.CHR and then click on the **2** button in the PeakSimple toolbar. A second chromatogram channel is now open in the PeakSimple window.



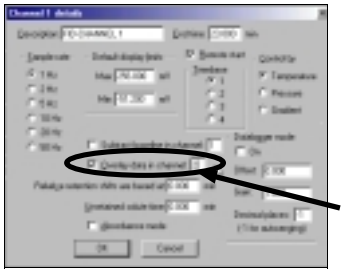
2. Once the second channel is open select **File** from the PeakSimple menu bar and then click on **Open**. The Load chromatogram file window will open up displaying a list of files to load. Select chromatogram **FID602.CHR** to load and then select the **2** channel radio button to load the chromatogram in the second channel.



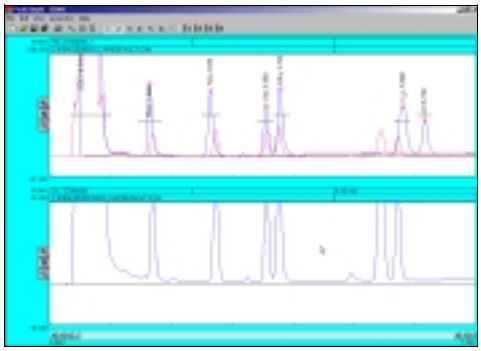
3. Once FID602.CHR is open in the second channel right click using the mouse on the chromatogram in the first channel and select **Channel Details** from the list of options.



4. After the Channel 1 details window appears on the screen locate the **Overlay data in channel** check box and select it. Look to the dialogue box to the right of the Overlay data in channel check box and insert the number **2** in place of the 1. Click on **OK** with the mouse cursor to exit the Channel 1 details window.

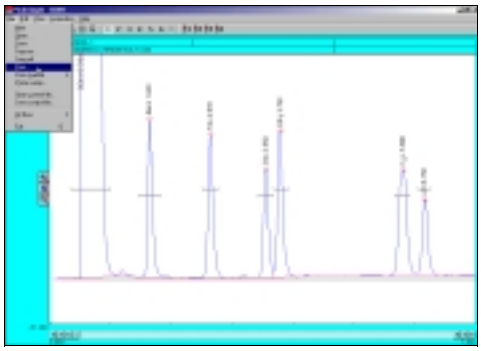


5. The chromatogram FID602.CHR is now in place overlaid on top of chromatogram 602.CHR in channel 1. Chromatogram 602.CHR is in blue while FID602.CHR is in red.

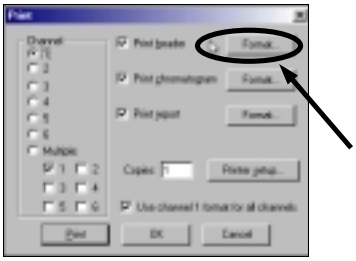


Printing a Chromatogram

1. To print a chromatogram first open chromatogram 602.CHR. Once the chromatogram is open select **File** from the PeakSimple menu bar and then select **Print** from the drop-down menu.



2. The Print window will open and will allow the user to customize the printing of a chromatogram. Click on the **Format** button for the Print header to open up the Header format window. Add or delete any information in the window by clicking on the fields and inserting the desired information. Click on the **OK** button when all the desired information is inputted to close the window.

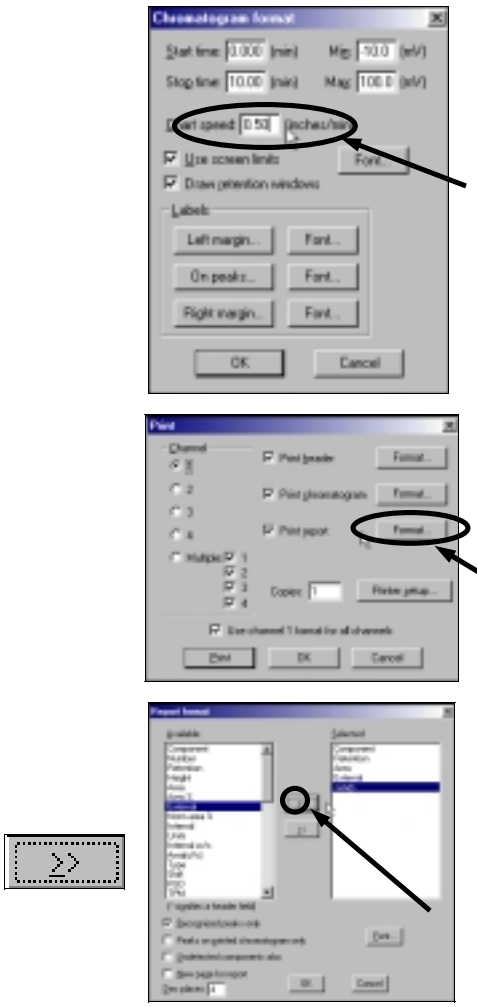


3. In the Print window click on the **Format** button for Print chromatogram to open up the Chromatogram format window. Locate the **Chart speed** dialogue box and insert the number of inches each minute on the chromatogram will take up when printed (for a nine minute run try **0.50** inches per minute). After the Chart speed is entered click on **OK** to exit the window.

4. In the Print window locate the Print report check box and click on the **Format** button to its right.

5. Once the Report format window is open click on **External** in the Available dialogue menu (on the left) and then click with the mouse cursor on the right facing arrow button to add External to the Selected dialogue box (on the right). After External is added to the Selected dialogue box click on **Units** with the mouse cursor and click on the right facing arrow button to add Units to the Selected dialogue box. Click on **OK** with the mouse cursor to exit out of the Report format window.

6. Select **Print** in the Print window to print the chromatogram or click on **OK** in the Print window to exit the window.

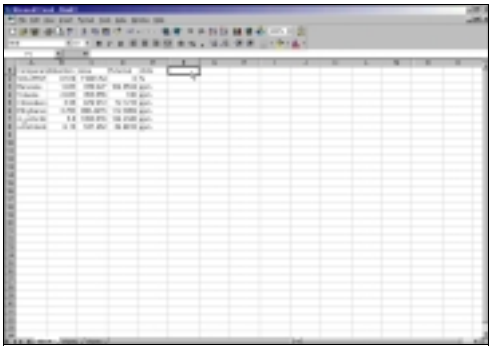


Exporting to Excel

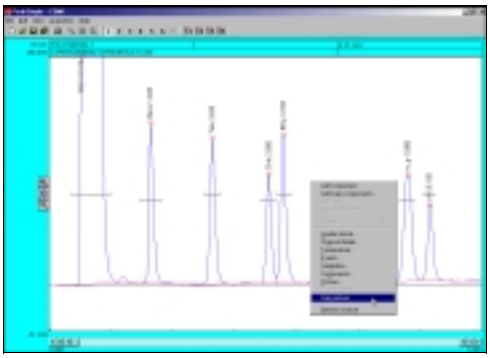
1. In the PeakSimple toolbar click on the **Results** window button to open up the Results window. Once the Results window is open click on the **Copy** button to copy the results data to the Windows clipboard.

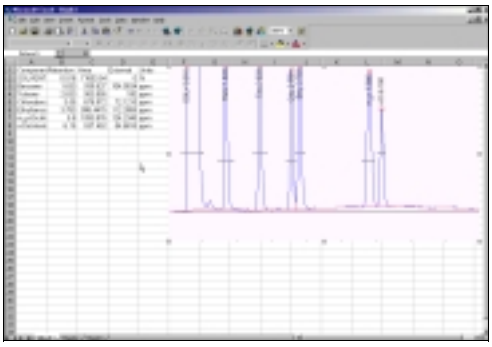
2. Make sure Microsoft Excel is loaded on the computer. If Excel is not loaded you can copy results data and chromatograms to Microsoft Word or PowerPoint. Open up Microsoft Excel by clicking with the mouse cursor on the **Start** button in the bottom left of the Windows screen and then **Programs** and then **Microsoft Excel** in the Windows Program menu.

6. Once Excel is opened select **Edit** from the Excel menu bar and then **Paste** from the drop down menu. The results data is now placed into the columns and rows of Excel. Using the mouse cursor, select a box to the right of the results data in the Excel spreadsheet. Go back into the PeakSimple for Windows NT program and hit **Close** to exit the Results window.



7. Right click with the mouse cursor anywhere on chromatogram 602.CHR and select **Copy picture** from the resulting menu. Go back into Excel and select **Edit** from the Excel menu bar and then **Paste** from the drop down menu. The PeakSimple chromatogram will now be displayed next to its results data in the rows and columns of Microsoft Excel.

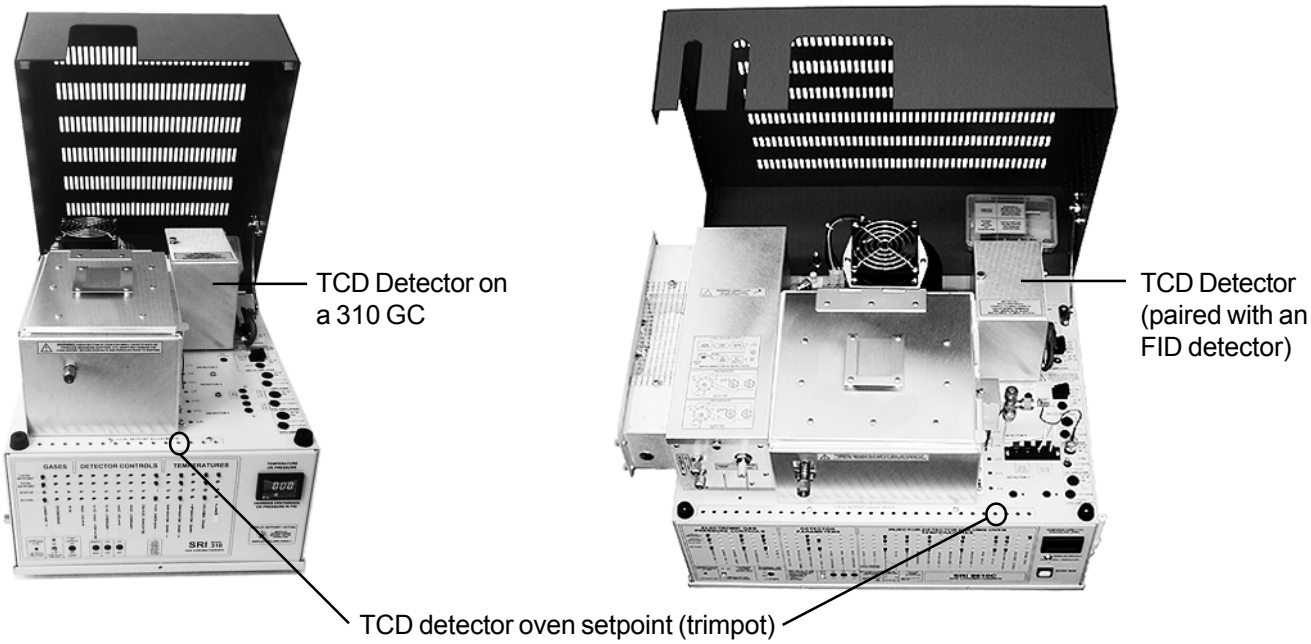




This concludes the PeakSimple 2000 Basic Tutorial

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 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 GCs.

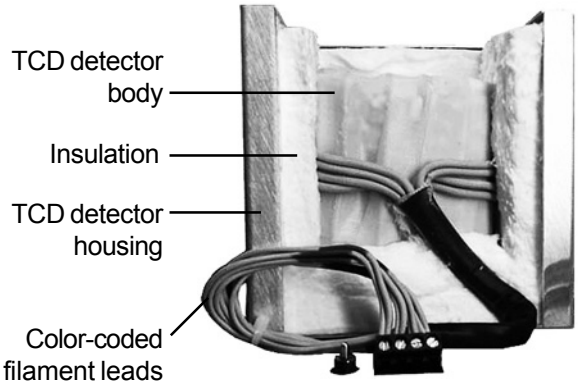


TCD Detector

Color-coded TCD filament leads are connected to the bridge terminal block

TCD filament current control switch

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

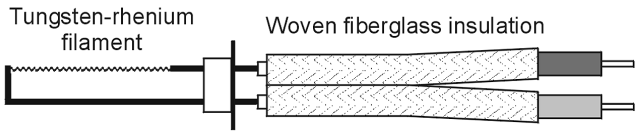


DETECTORS
Thermal Conductivity Detector - TCD

Theory of Operation

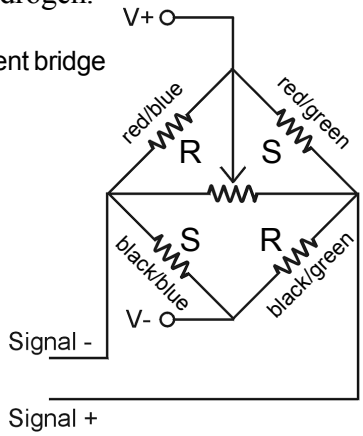
The TCD detector measures the difference in thermal conductivity in the carrier gas flow and the analyte peaks. Every compound possesses some degree of thermal conductivity, and may therefore be measured with a TCD detector. Due to its high thermal conductivity and safety, helium carrier is most often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

One of four TCD filaments



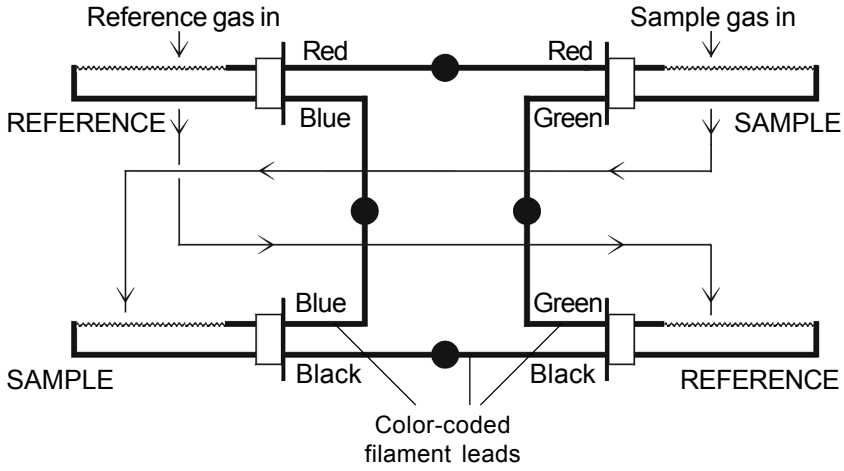
Filament leads are color-coded for identification

TCD filament bridge



The Wheatstone Bridge circuit design in the 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.

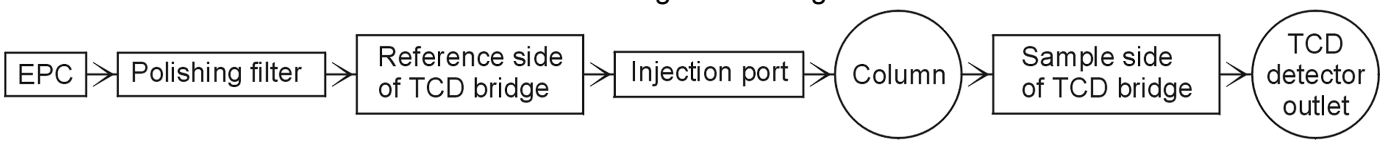
Simplification of filament interconnection



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



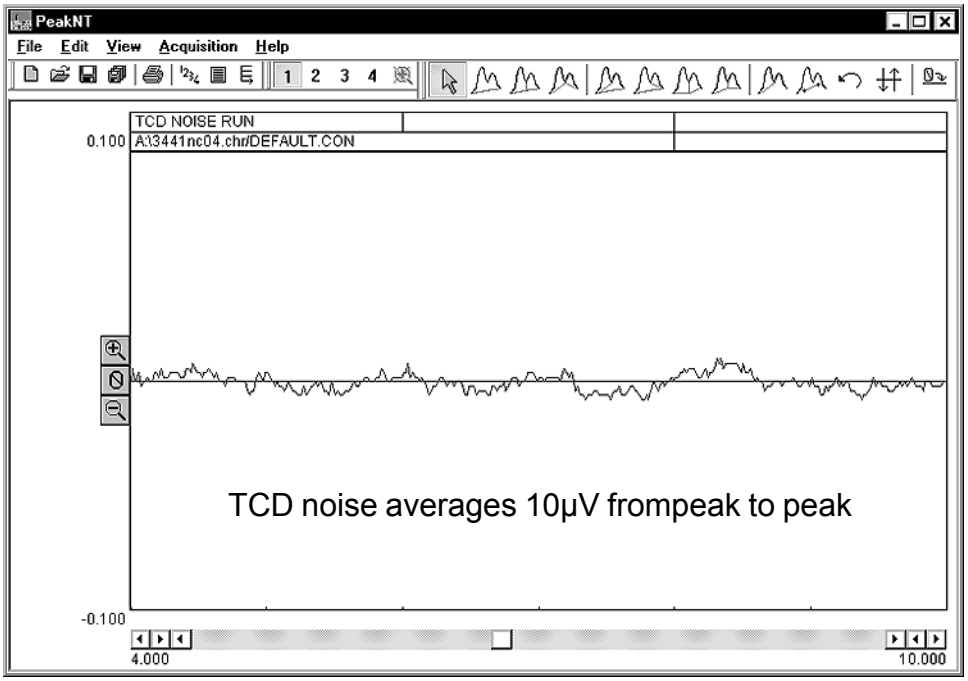
Expected Performance

TCD Noise Run

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

Temperature program:

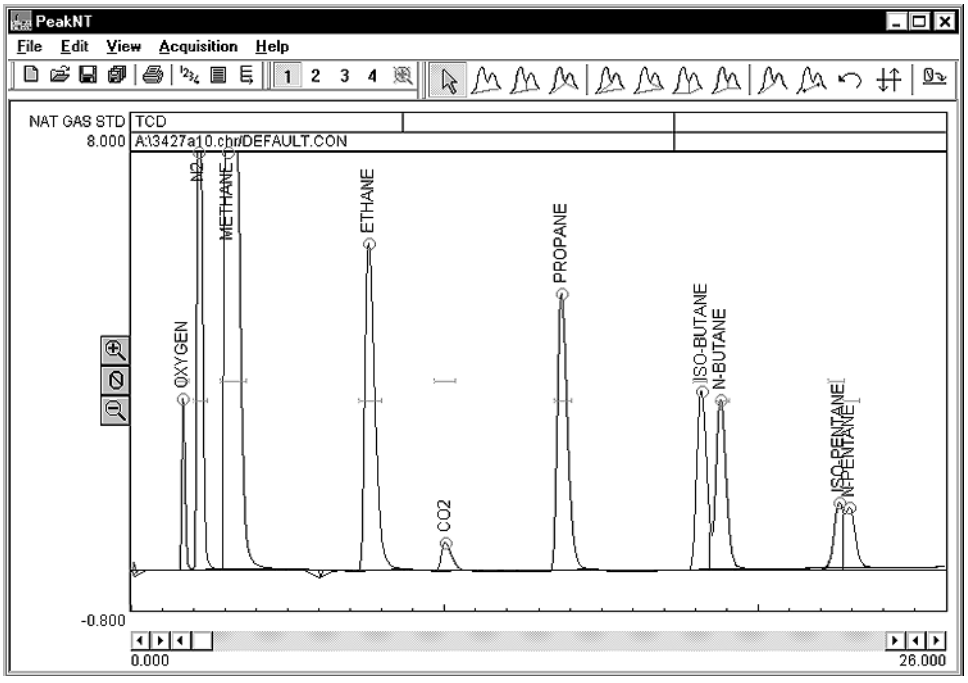
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C



Factory Test Run of a TCD-equipped Buck 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:

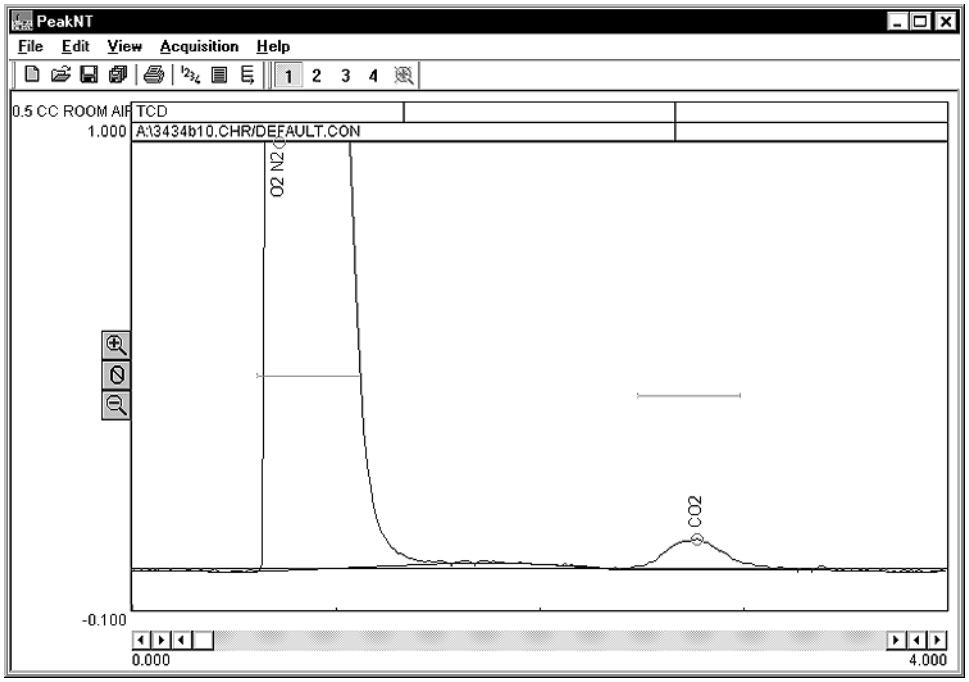
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

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

DETECTORS
Thermal Conductivity Detector - TCD

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

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

Results:

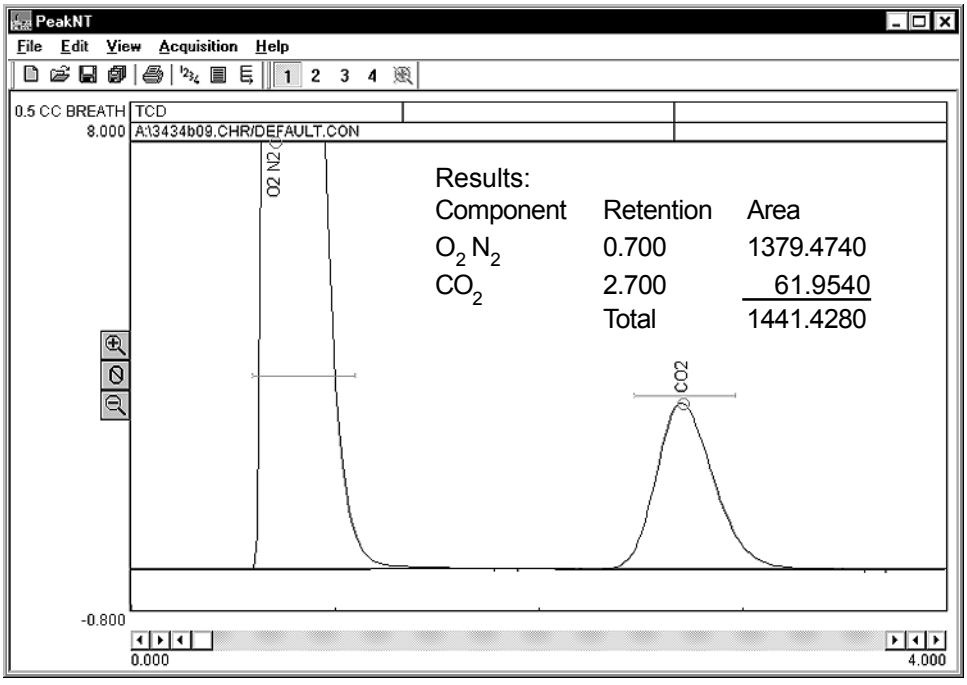
Component	Retention	Area
O_2 N_2	0.716	1021.3830
CO_2	2.766	1.5060
	Total	1022.8890

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



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 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.

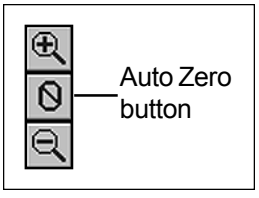
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column 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).



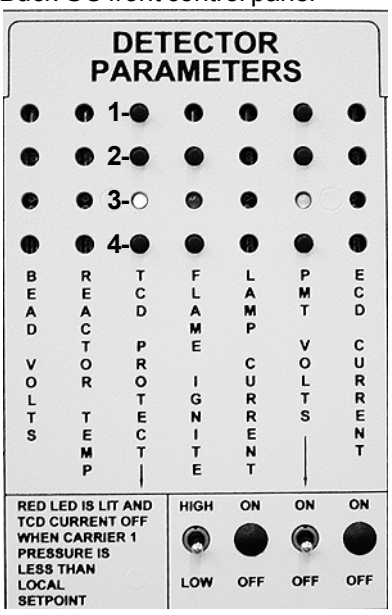
8. Inject sample. Injection volumes of 0.5mL for gas and 1µL for liquid is recommended to prolong TCD filament life.

DETECTORS
Thermal Conductivity Detector - TCD

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 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 the Buck GC front control panel



1- LOCAL SETPOINT button

2- TOTAL SETPOINT button

3- STATUS LED

4- ACTUAL button

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.

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 Buck. In addition to the standard filaments, optional gold-plated filaments for improved corrosion resistance are also available.

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

Buck TCD detector replacement parts

Standard TCD filament with rubber O-ring gasket	670-9120
High temperature TCD filament with copper gasket	690-9123

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

DETECTORS
Thermal Conductivity Detector - TCD

Replacing the TCD Filaments

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!

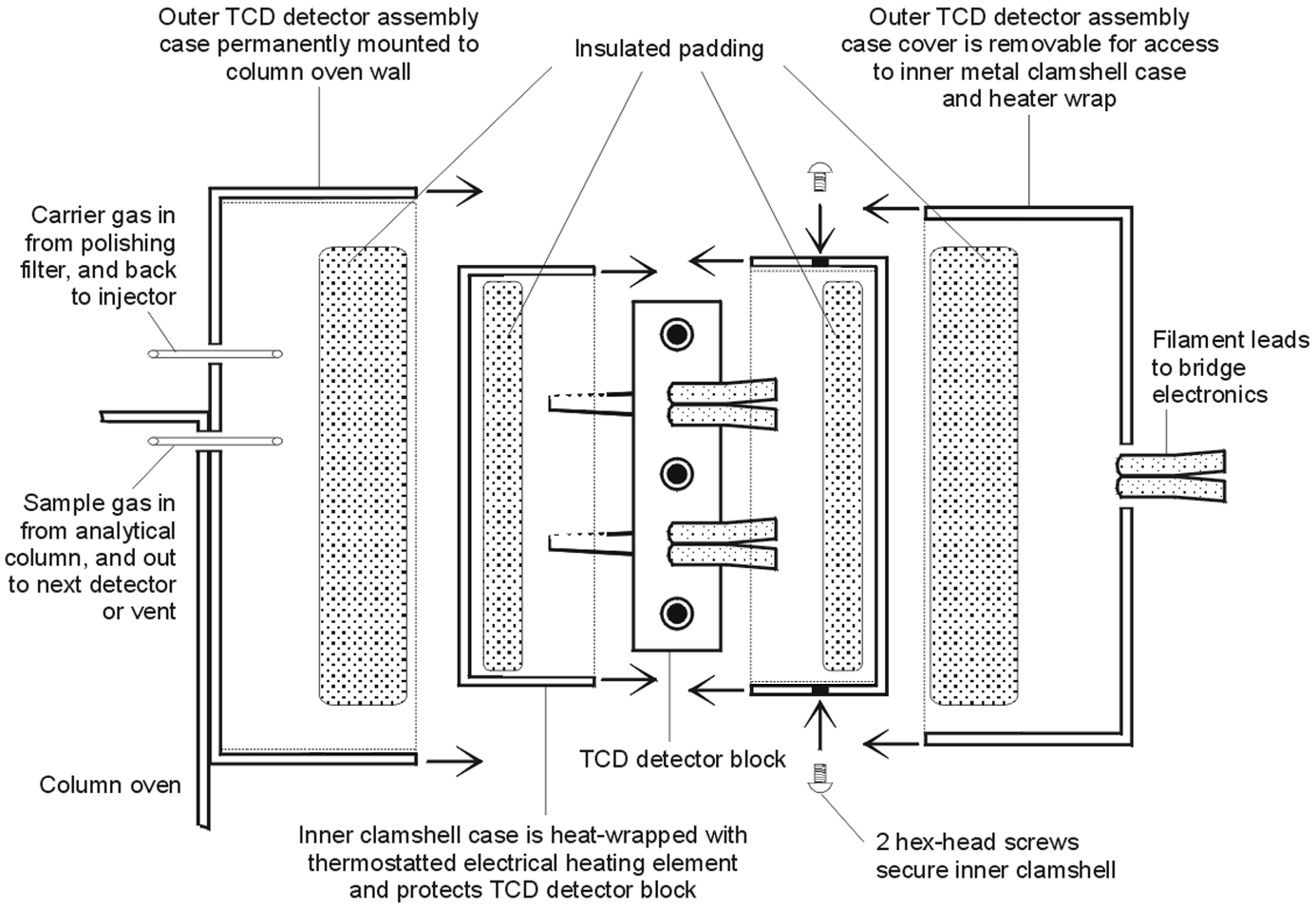
A. Standard TCD detector block access

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.

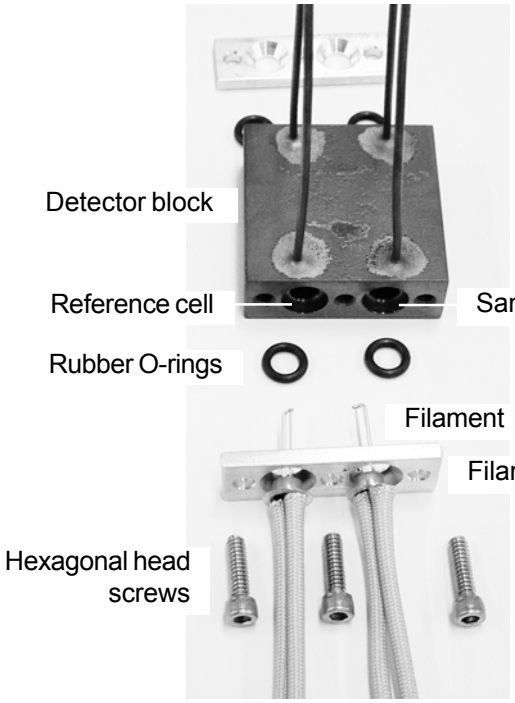
6. The TCD filaments are secured in the detector block by two plates, each of which is held in place with three hexagonal head screws. Holding the detector block with one hand, use an Allen wrench to unscrew and remove the hexagonal head screws from one of the filament securing plates. Then, slide the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

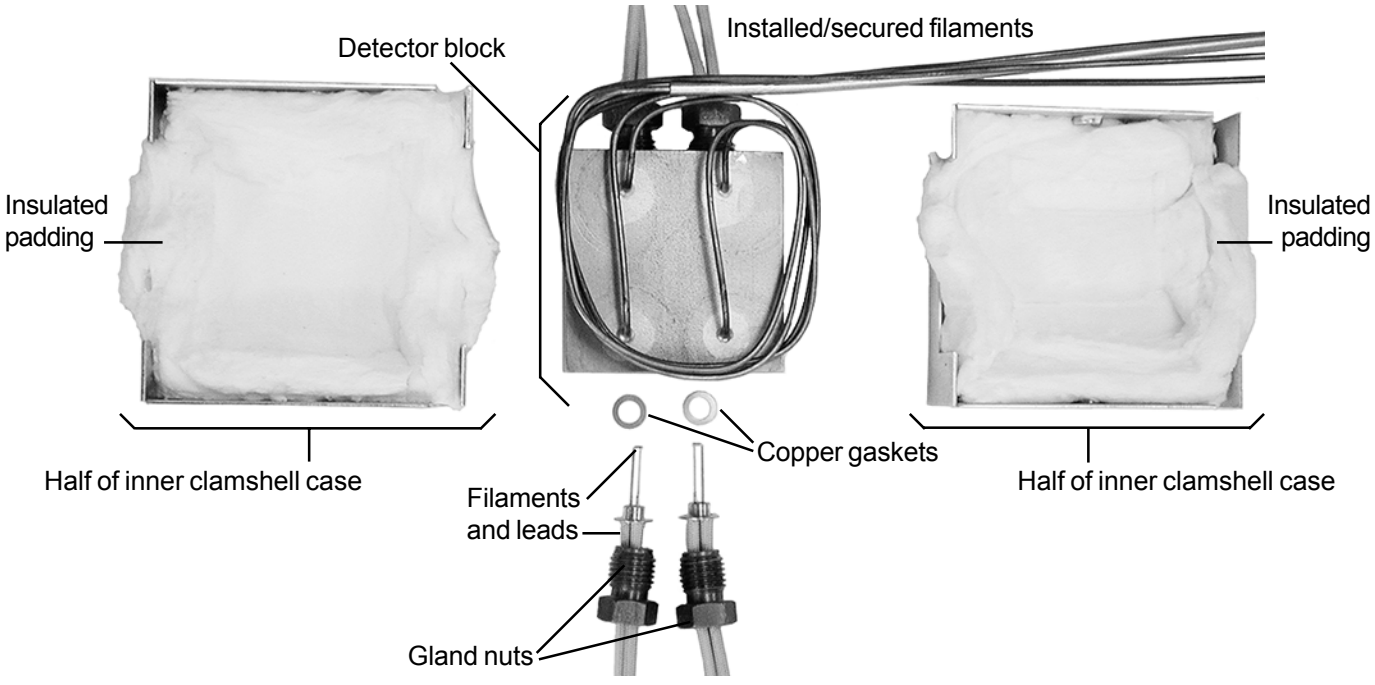
The high temperature TCD assembly is the same as the standard: outer housing around an inner clamshell case. The high temp detector block uses gland nuts and copper gaskets to secure the four filaments in its two cells. Instead of the heater rope, it employs a heating cartridge, which is inside the inner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

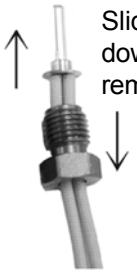


Replacing the TCD Filaments continued

(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.

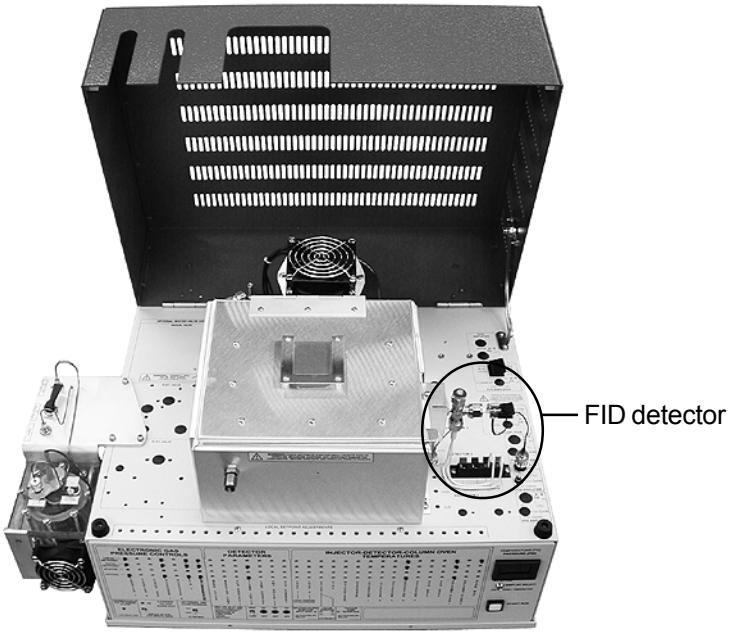
8. Reconnect the TCD detector gas inlets and outlets.

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.

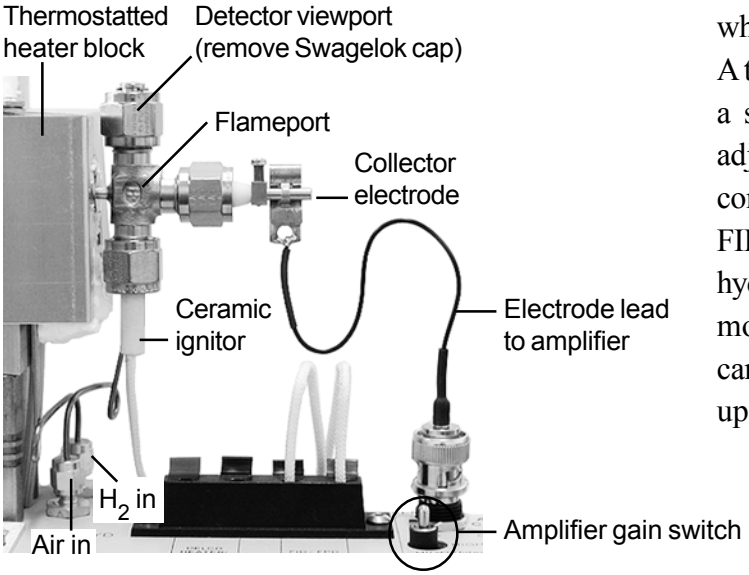
10. Reconnect the filament leads to the bridge current terminal block. Use the color guide labels on the terminal block to insert the color-coded leads into the appropriate terminal.

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.



Capillary FID GC



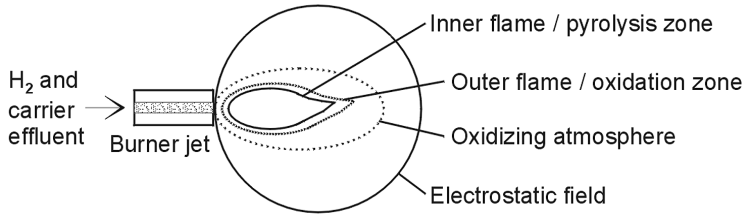
The FID features a unique ceramic ignitor which can run hot continuously, and prevent the flame from extinguishing even with large water injections or pressure surges from column backflush. This ignitor is positioned perpendicular to the stainless steel detector jet and does not penetrate the flame. 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 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

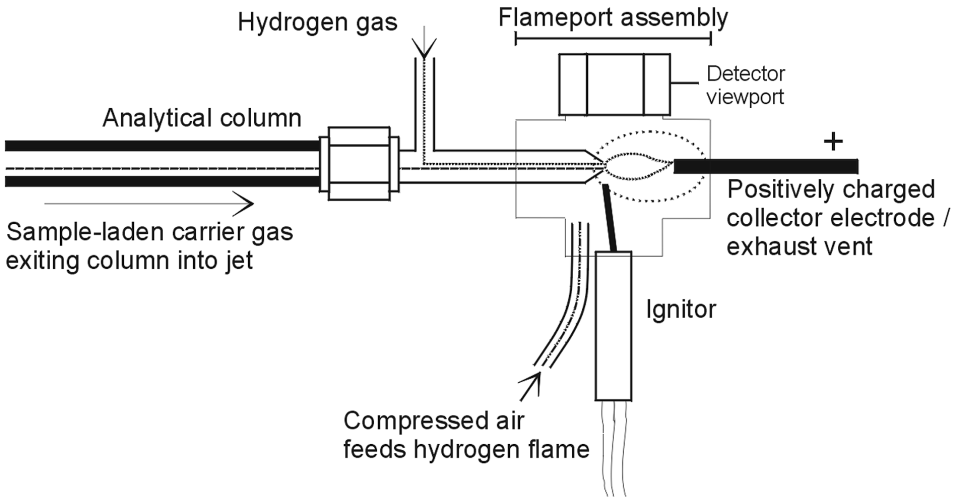
In the FID, the carrier gas effluent from the GC column is mixed with hydrogen, then routed through an unbreakable stainless steel jet. The hydrogen mix supports a diffusion flame at the jet's tip which ionizes the analyte molecules. Positive and negative ions are produced as each sample component is 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



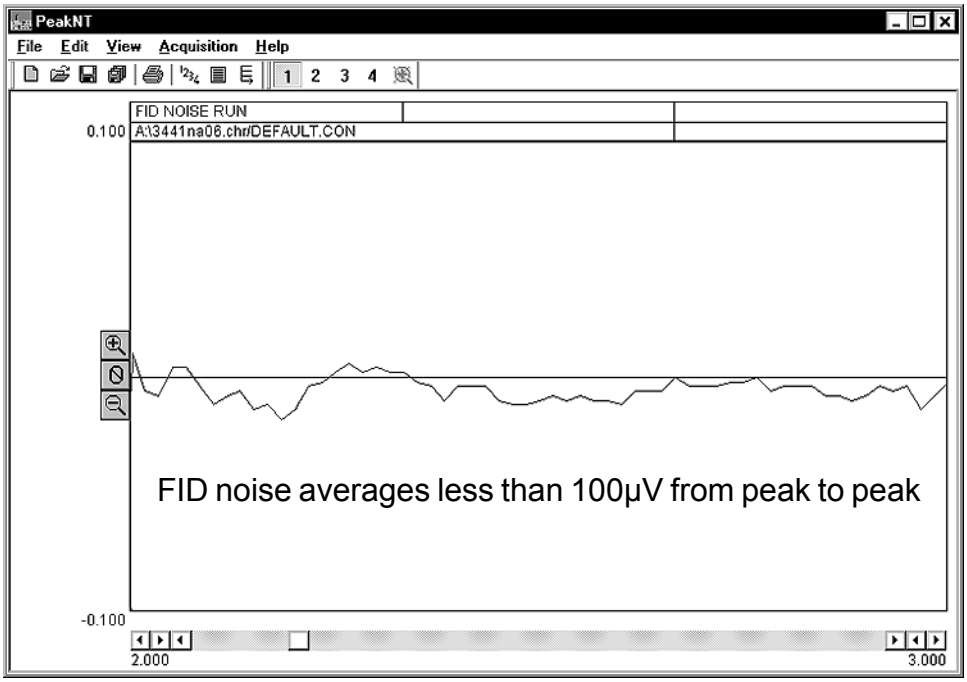
Expected Performance

FID noise run

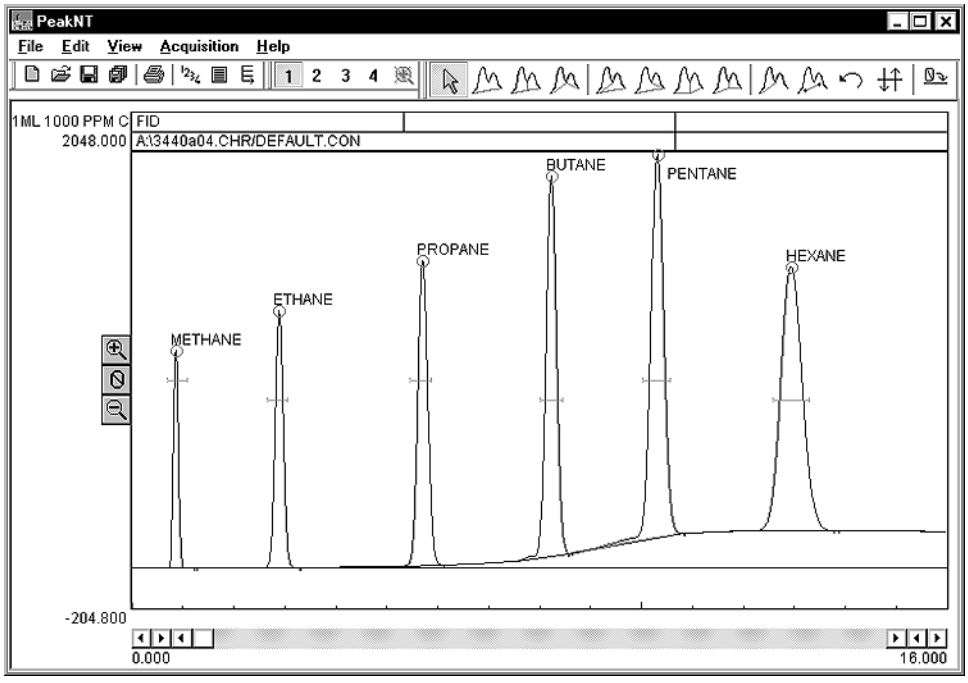
Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

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:

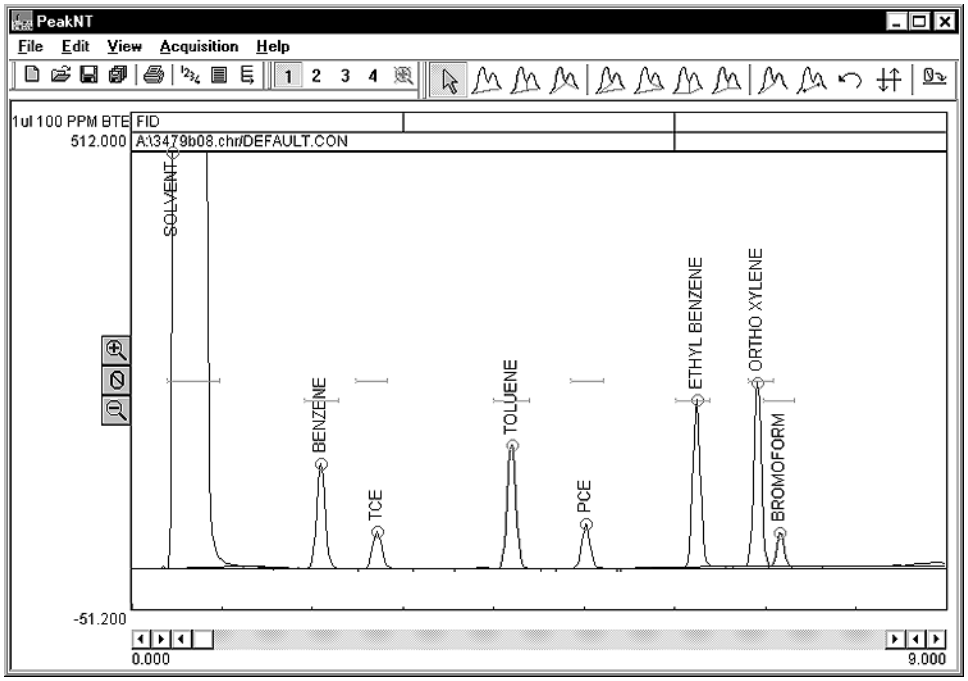
Component	Retention	Area
Methane	0.850	6979.9260
Ethane	2.866	13623.7580
Propane	5.683	19535.8960
Butane	8.200	26456.5980
Pentane	10.283	33053.9680
Hexane	12.916	39419.0870
	Total	139069.2330

DETECTORS
FID - Flame Ionization Detector

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 GC.



1µL 100ppm BTEX sample

15m MXT-VOL capillary column

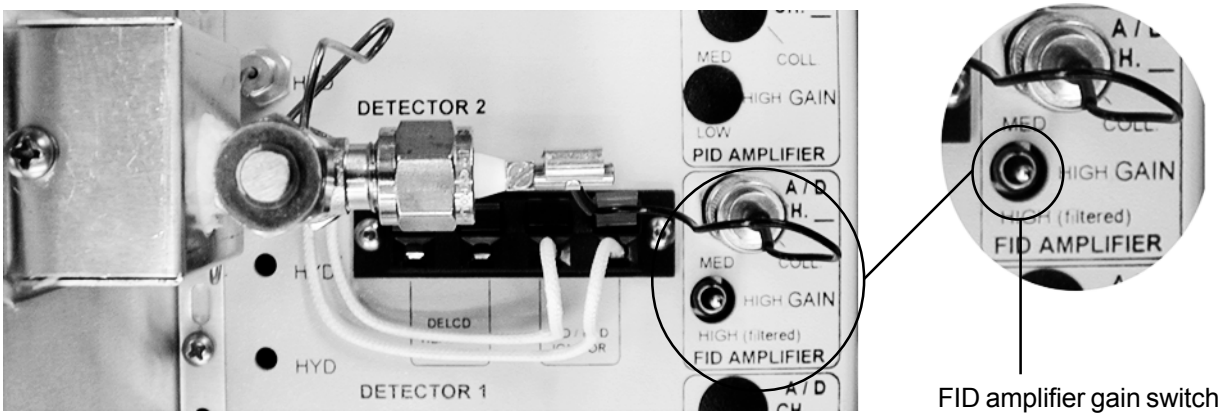
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

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

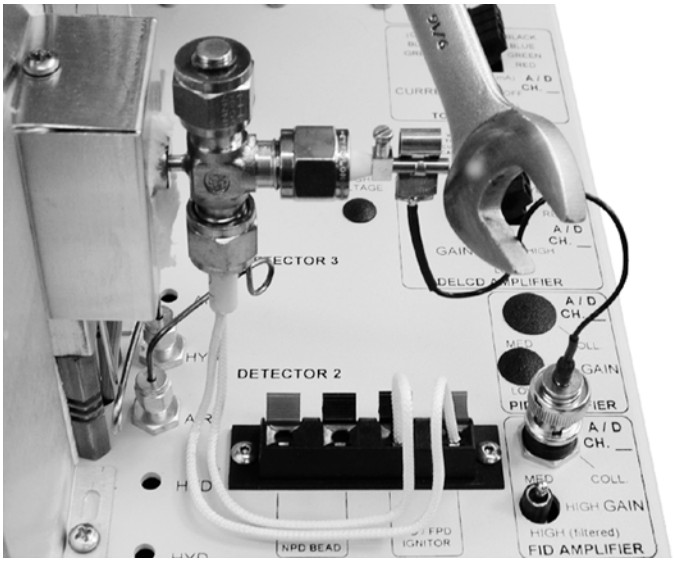
General Operating Procedure

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.



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 GC under the "DETECTOR PARAMETERS" heading (it is labelled vertically: "FLAME IGNITE").



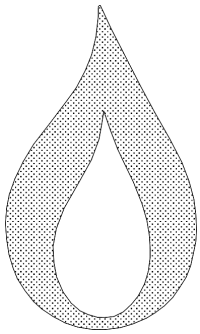
4. Verify that the FID flame is lit by holding the shiny side of a chromed wrench directly in front of the collector outlet/FID exhaust vent. If condensation becomes visible on the wrench surface, the flame is lit.

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



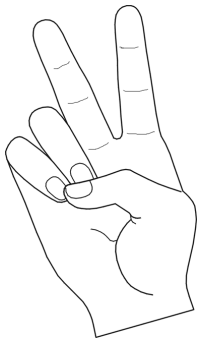
1. Extinguish the flame by turning off the air.

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.

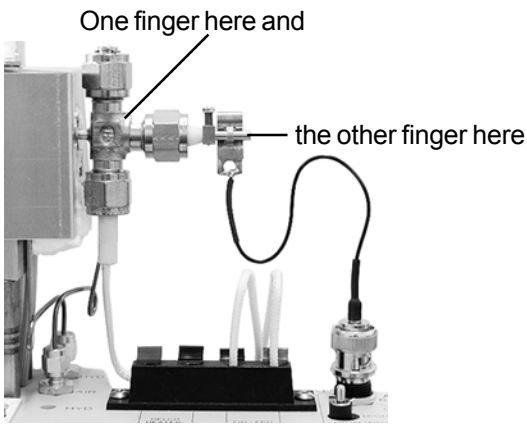
B. Wet Finger Test



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).

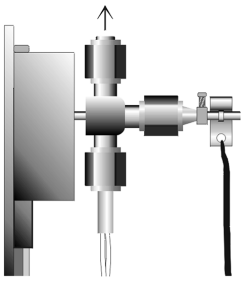
3. Place one finger on the collector electrode, and place the other on bare metal (like the FID detector body or the column oven lid) to ground the collector. Make your contact brief--you need only brush these parts to perform the test. Be careful not to burn yourself; the column oven lid is probably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

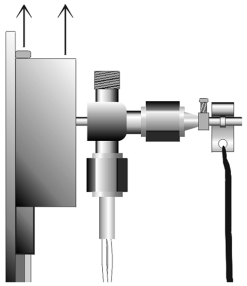
The FID detector rarely requires cleaning or servicing. It may develop a film or coating of combustion desposits in the flameport with extended use. Use the FID detector viewport to check for visible deposits. If you're experiencing problems with your FID detector, try cleaning it, even if you can't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

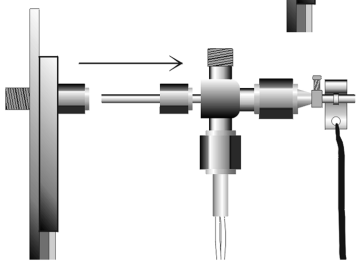
2. Remove flameport assembly from the heater block

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

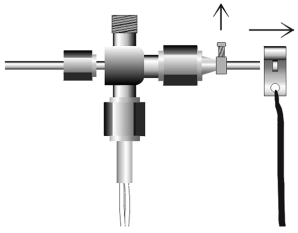


   b. Using a philps head screwdriver, remove the screw on the top of the FID's heater block and pull the aluminum cover up and off.

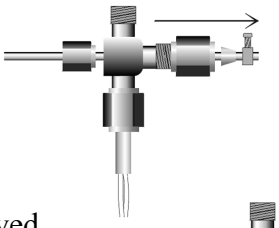
   c. Gently pull off the white insulation to reveal the detector's bulkhead fitting on the column oven wall. Loosen this fitting to disconnect the flameport.



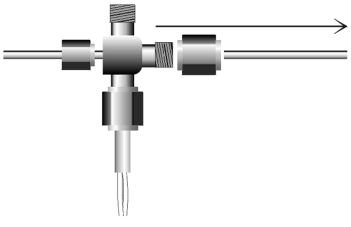
3. Remove the collector electrode



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



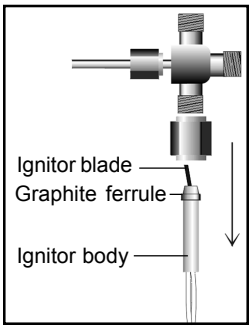
   b. Loosen and remove the nut and ferrule that hold the collector electrode in the flameport body.



   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.

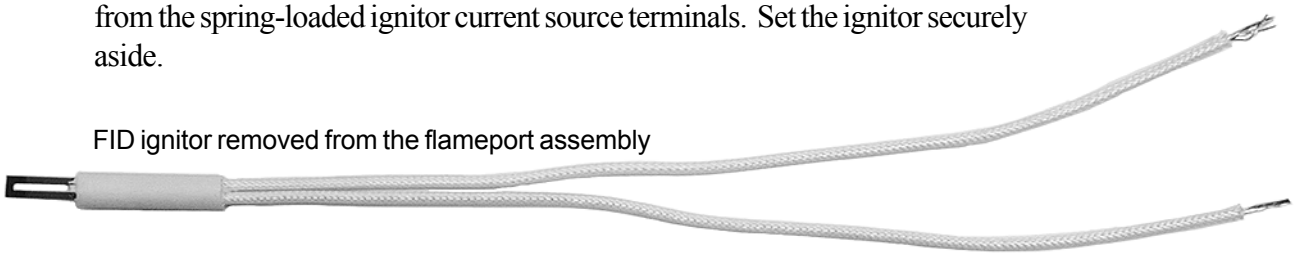
Cleaning the FID continued

4. Remove the FID ignitor element



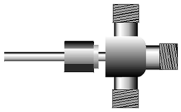
a. The ignitor element is brittle and will break when stressed, so handle the ignitor carefully, mindful of any torque on the blades. While holding the ignitor by the ceramic body with one hand, loosen the 1/4" swagelok-type nut that holds it in place. There is a graphite ferrule inside this nut that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



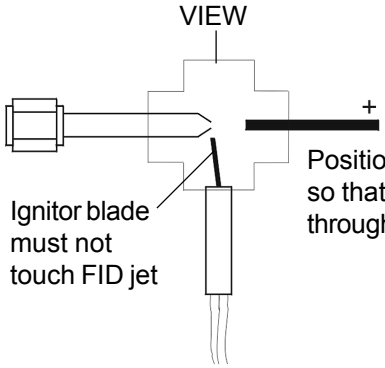
Scrape, rinse, and bake out the FID flameport interior

6. Re-assembly

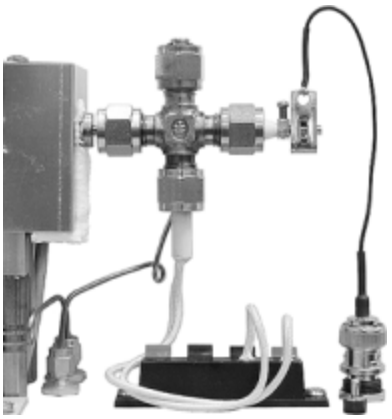
a. Once all the FID parts are cleaned, reverse the disassembly process, starting with the replacement of the ceramic ignitor. Leaving out the cleaning steps, your last step should be reinstalling the flameport assembly onto the heater block. Make sure to position the ignitor so that the blade is slightly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

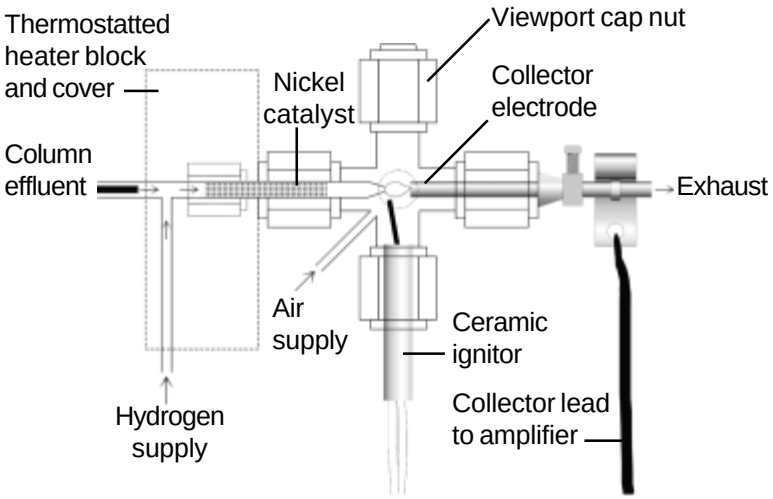
FID ignitor removed from the flameport; note the slight angle of the blade element

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



Methanizer-equipped FID Detector

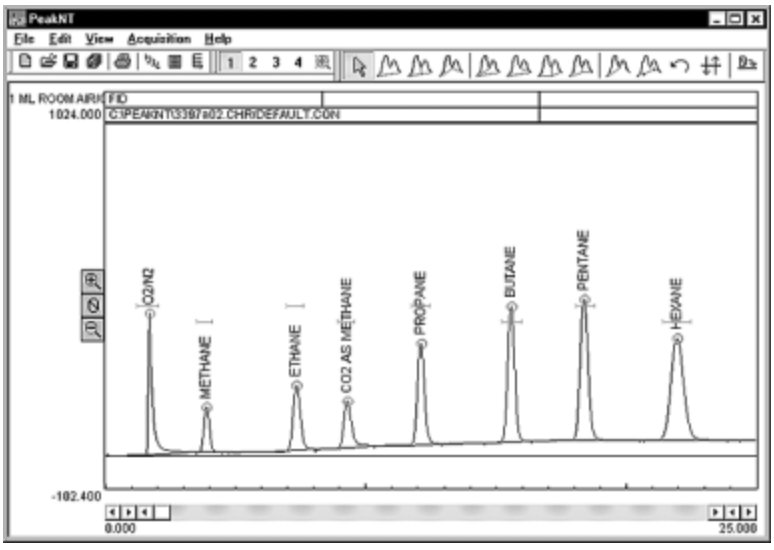




The Methanizer option enables the Flame Ionization Detector to detect low levels of CO and CO_2. It is installed as the removable jet in a special FID detector assembly. The Methanizer / jet delivers the column effluent mixed with hydrogen to the FID detector. The Methanizer is packed with a nickel catalyst powder on glass wool secured with two frits. During analysis, the Methanizer is heated to 380°C with the FID detector body. When the column effluent mixes with the FID hydrogen supply and passes through the Methanizer, CO and CO_2 are converted to methane. Since the conversion of CO and CO_2 to methane occurs after the sample compounds have passed through the column, their retention times are unchanged. Hydrocarbons pass through the Methanizer unaffected. The special Methanizer FID detector assembly operates like the regular FID detector, except that the FID temperature must be set to 380°C. Due to the chemical relationship between nickel and sulfur, the Methanizer can be poisoned by large quantities of sulfur gas.

Expected Performance

The following chromatogram was produced by an Multiple Gas Analyzer #1 equipped with a Methanizer.



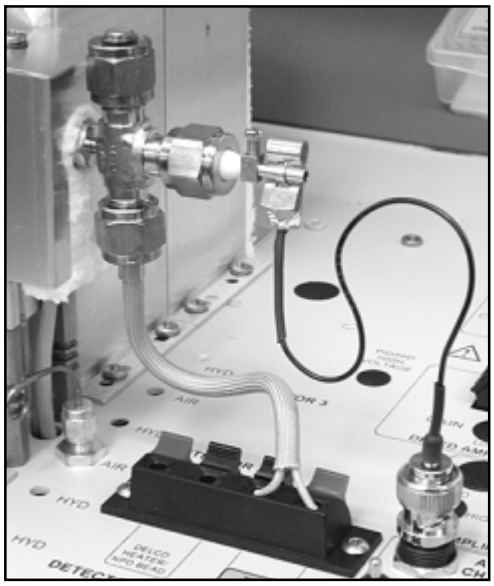
Sample: 41cc room air + 15cc 1000ppm C_1-C_6 injected into the 1mL sample loop = 250ppm CO_2 and C_1-C_6

Results:

Component	Retention	Area
O2/N2	1.650	4731.2140
Methane	3.866	2008.6000
Ethane	7.316	3854.7300
CO2 as Methane	9.250	3142.1040
Propane	12.083	5379.8755
Butane	15.533	7326.4440
Pentane	18.333	9136.3340
Hexane	21.900	10408.3160
	Total	45987.6175

Overview

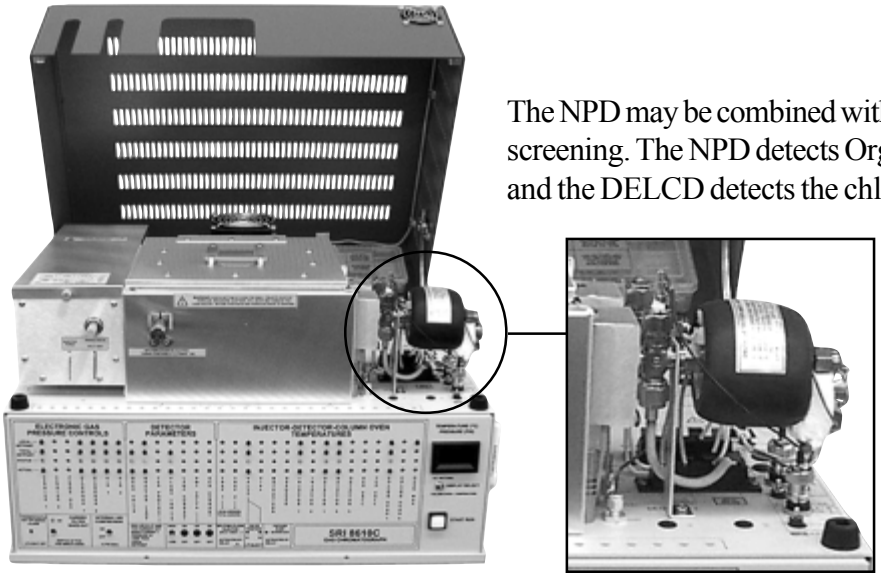
The SRI Nitrogen-Phosphorus Detector (NPD) has a linear response selective to organic compounds containing nitrogen and/or phosphorus. The NPD also responds to normal hydrocarbons, but approximately 100,000 times less than nitrogen or phosphorus containing compounds. Due to its selectivity and sensitivity, the NPD is often used to detect pesticides, herbicides, drugs of abuse, and other trace compounds. Nitrogen is the carrier gas of choice for the NPD detector, but helium is often used, especially when other detectors are installed on the same GC as the NPD.



The NPD is similar to the FID in design.

The NPD is similar in design to the FID, except it uses a thermionic NPD bead to generate ions in a hydrogen and air plasma. Like the FID, the NPD uses a stainless steel jet to deliver sample-laden carrier gas and hydrogen gas to the detector, and a positively charged collector electrode that also serves as the detector exhaust. The NPD bead is positioned between the jet and the collector electrode. The tip of the NPD jet is slightly different from that of the FID jet.

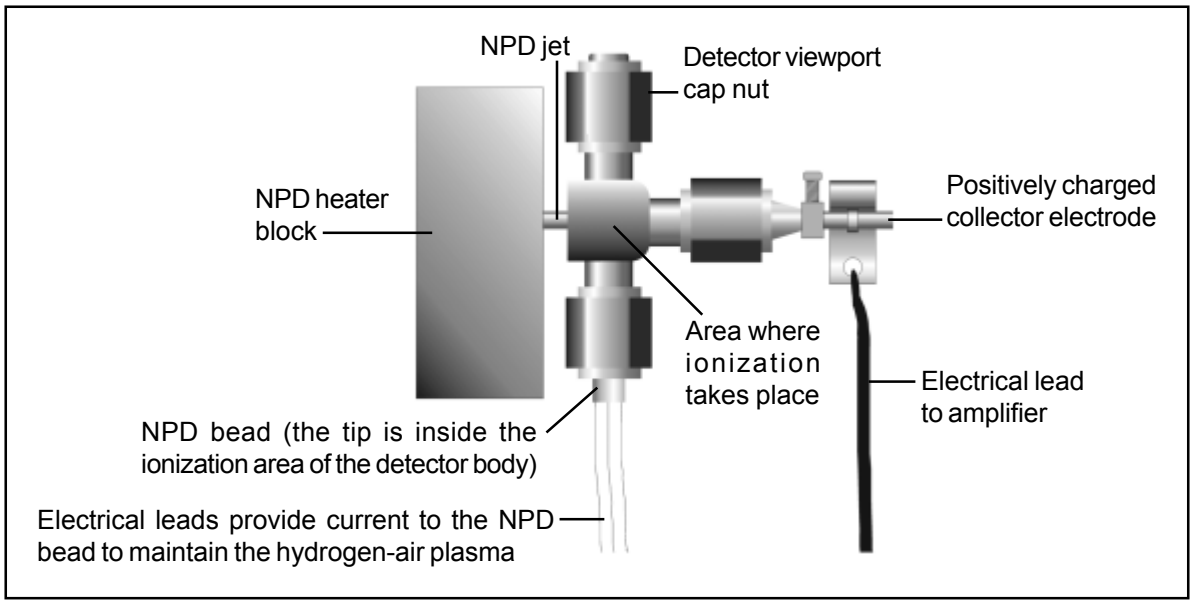
The thermionic NPD bead is coated with an alkali salt.



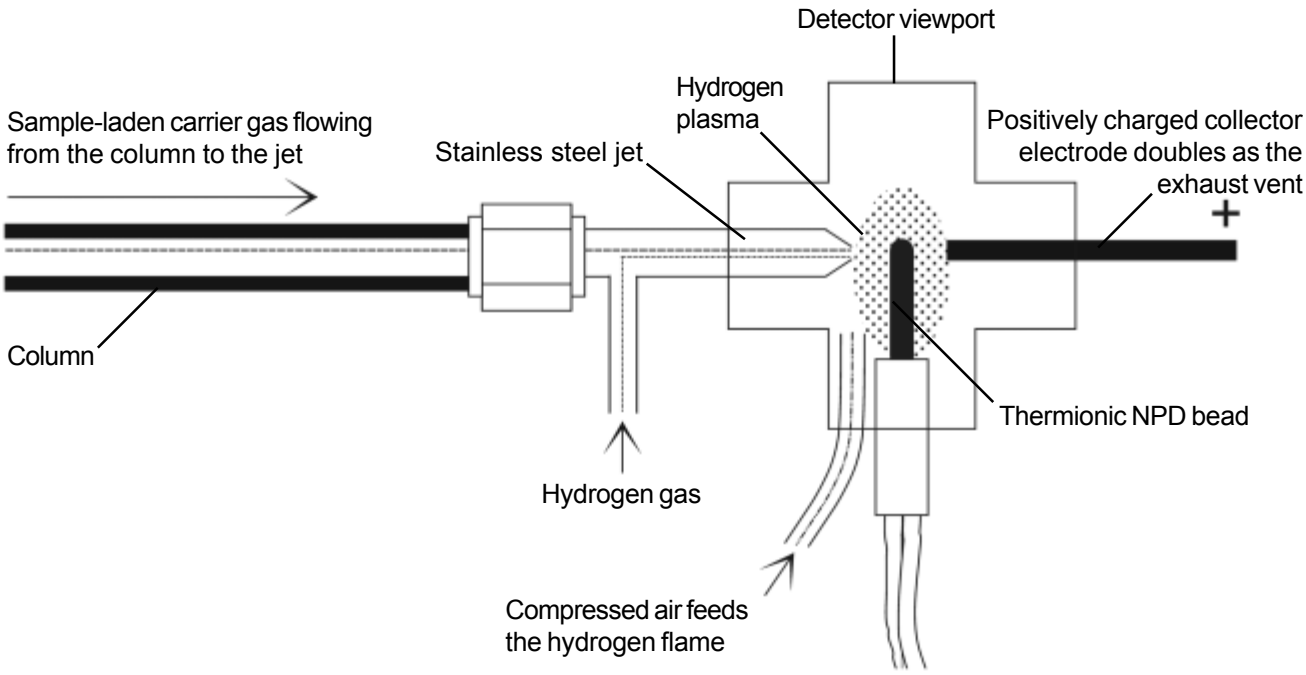
The NPD may be combined with the DELCD for pesticide screening. The NPD detects Organo-phosphate pesticides and the DELCD detects the chlorinated pesticides.

DETECTORS
Nitrogen/Phophorus Detector - NPD

Theory of Operation



Inside the NPD detector body, an electrically heated thermionic bead (NPD bead) is positioned between the jet orifice and the collector electrode. The bead is coated with an alkali metal which promotes the ionization of compounds that contain nitrogen or phosphorus. Hydrogen and air flows create a hydrogen plasma around the hot NPD bead. When molecules containing nitrogen or phosphorus enter the plasma from the column and jet orifice, they undergo a catalytic surface chemistry reaction, producing thermionic electrons. The resulting ions are attracted to a positively charged collector electrode, then amplified and output to the data system. The hydrogen to air ratio is too lean to sustain a flame, therefore minimizing hydrocarbon ionization and contributing to the NPD detector's selectivity.



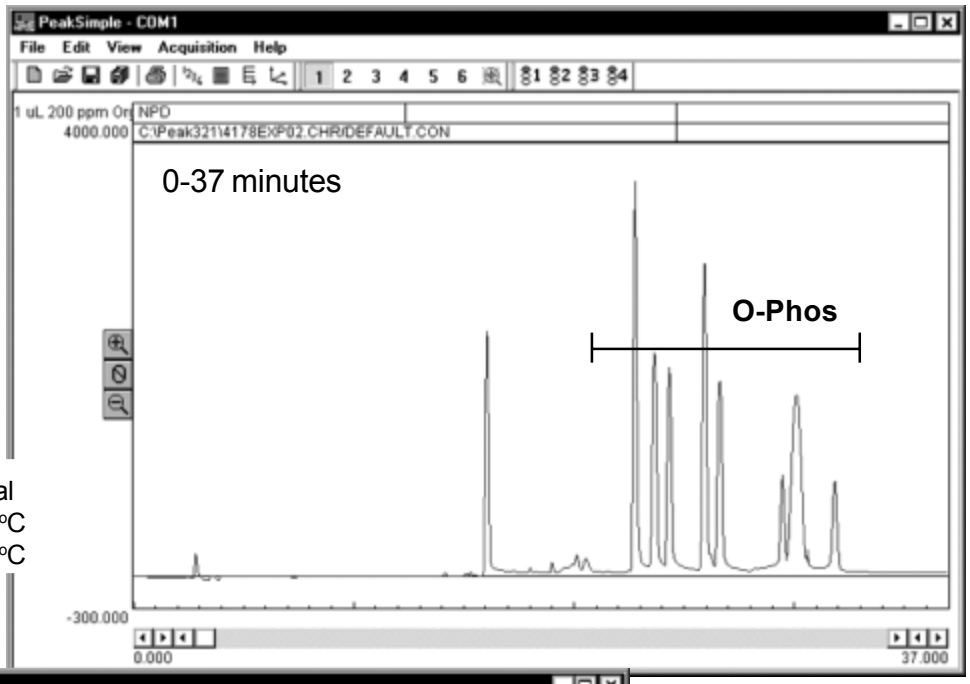
Expected Performance

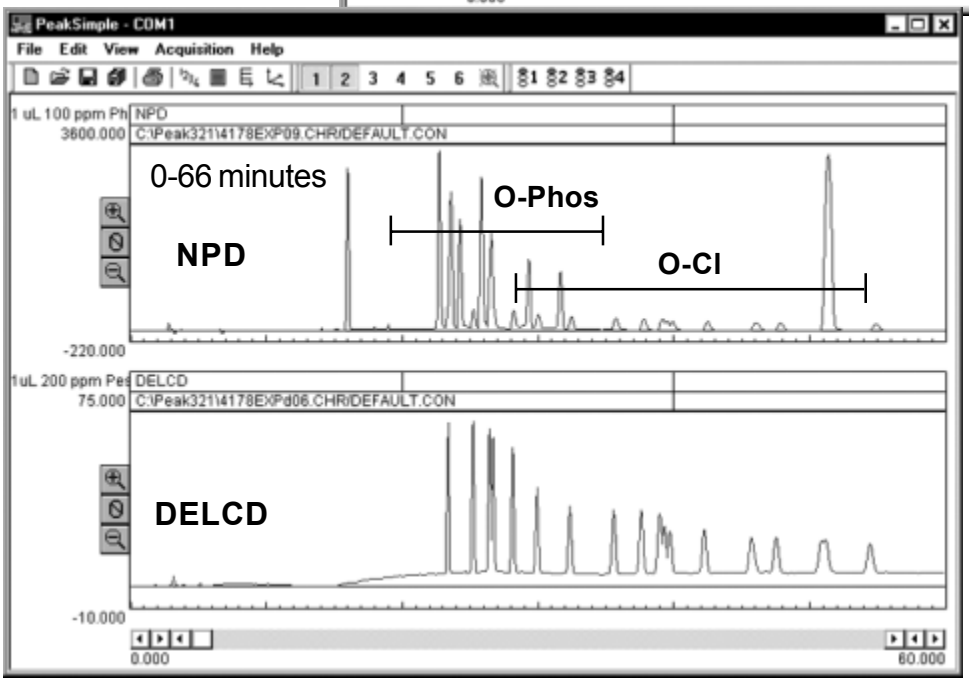
The following chromatograms are from an SRI GC equipped with NPD, DELCD and FID detectors. Since the NPD is not the only detector, helium carrier gas was used instead of nitrogen. The first chromatogram shows a separation of 200ppm Organophosphorus pesticide standard, Mix 8270. The second chromatogram shows both the NPD and DELCD responses to a mixture of 100ppm Mix 8270 and 100ppm Organochlorine pesticide standard, Mix 8081. Other than the sample and length of time, the analytical parameters were the same for both runs. The NPD has a much smaller response to the Organochlorine standard (Mix 8081). Since the DELCD is selective to chlorinated molecules, its response to Mix 8081 supplements the NPD response for better identification. and quantification.

Sample: 1µL 200ppm Organophophorus standard mix 8270
Method: direct injection
Column: 60-meter MXT-VOL
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -370
Injector temperature: 150°C

Temperature program:

Initial	Hold	Ramp	Final
100°C	6.00	15.00	280°C
280°C	20.00	0.00	280°C

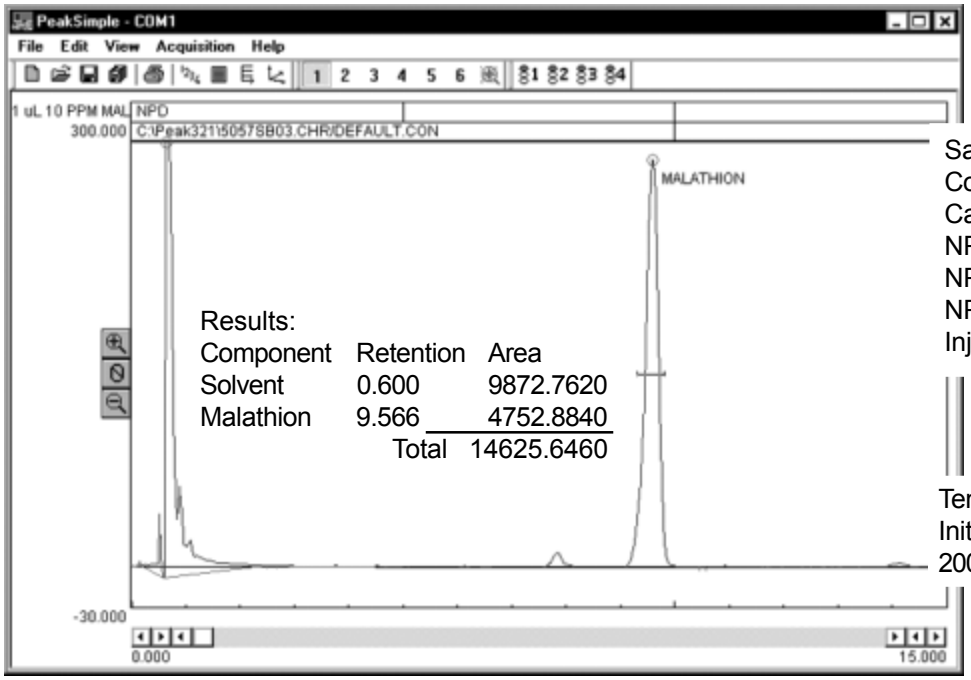




Sample: 1µL 100ppm Organophophorus pesticide standard mix 8270 + 100ppm Organochlorine pesticide standard mix 8081
Method: direct injection
Injector temperature: 150°C
Column: 60-meter MXT-VOL
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -370
DELCD gain: LOW
DELCD reactor: 260
DELCD temp: 300°C

Expected Performance

This chromatogram shows the NPD response to an isothermal analysis of a 10ppm malathion sample. Compare the NPD response to the 100% hydrocarbon solvent with the response to the 10ppm malathion sample.

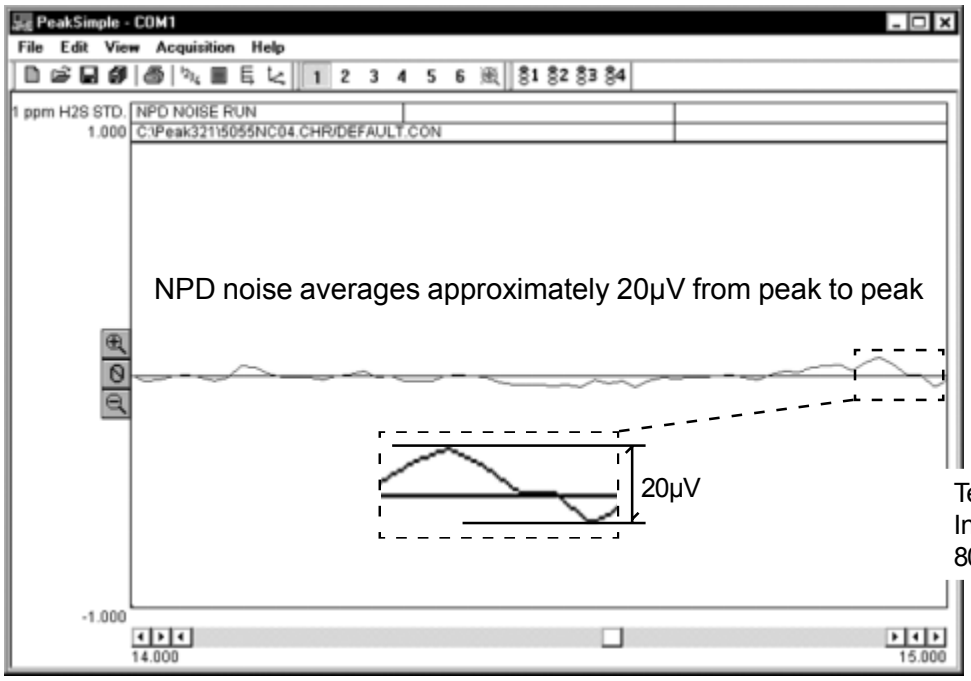


Sample: 1 µL 10ppm malathion
Column: 15-meter MXT-5
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -380
Injector temperature: 200°C

Temperature program:

Initial	Hold	Ramp	Final
200°C	100.00	0.00	200°C

The following chromatogram shows an NPD noise run using helium carrier gas and an 80 degree isothermal temperature program.



Column: 15-meter MXT-5
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -380

Temperature program:

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

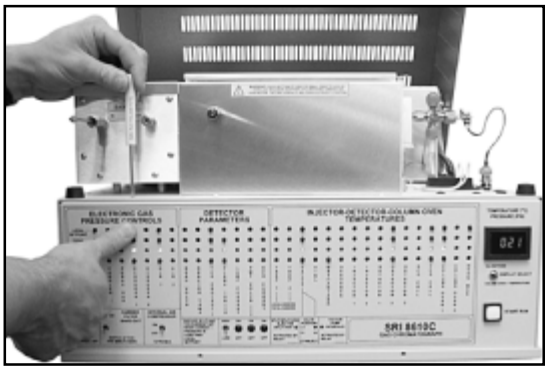
Note: Most SRI NPDs are installed on a GC with one or more other detectors. Therefore, SRI tests its NPD detectors with helium carrier gas.

General Operating Procedures

NPD Detector

1. Set the NPD amplifier gain switch to HIGH for most applications.

2. The approximate pressures required for the correct hydrogen and air flows are labeled on the right-hand side of the GC chassis under "GAS FLOW RATES." Set the hydrogen flow to 3mL/minute and the air flow to 100mL/minute using the trimpots on the top edge of the GC's front control panel. To adjust a pressure setting, hold down the SETPOINT button while turning the corresponding trimpot until you can read the desired pressure setting in the LED display (make sure the LED "DISPLAY SELECT" switch is on "ALL BUTTONS").



3. Set the NPD detector temperature to 250°C: hold down the SETPOINT button while turning the detector heat trimpot until the desired setpoint is visible in the LED display.

4. Set the NPD bead current to -360. Higher current settings may be used, but the life and subsequent sensitivity of the NPD bead will be reduced.

5. Press the ACTUAL button to observe the temperature of the NPD in the LED display. When the detector has reached the set temperature and the signal appears stable, the NPD is ready for use.

NPD/DELCD Combination Detector

1. Set the DELCD amplifier gain switch to LOW, and the NPD gain to HIGH.

2. Set the NPD/DELCD hydrogen to 3mL/minute and the air to 100mL/min using the correlating pressure labeled on the right-hand side of the GC.

3. Set the NPD/DELCD detector heat to 150°C.

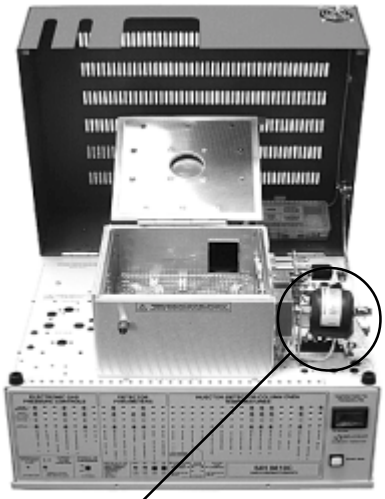
4. Set the DELCD reactor temeprature to 260. The number 260 represents 1000°C; the DELCD will heat to about 254 and stabilize. The visible end of the reactor tube will glow bright red with the high temperature.

GAS FLOW RATES

CARRIER 1:		:	4	PSI =	10	ml/min
CARRIER 2:		:		PSI =		ml/min
P&T PURGE:		:		PSI =		ml/min
HYDROGEN 1:	NPD/DELCD	:	10	PSI =	3	ml/min
HYDROGEN 2:	FID	:	19	PSI =	25	ml/min
AIR 1:	NPD/DELCD	:	3	PSI =	100	ml/min
AIR 2:	FID	:	8	PSI =	250	ml/min

Example Gas Flow Rates table: this particular GC is equipped with NPD/DELCD and FID detectors.

5. Inject sample when the combination detector has reached the set temperatures and their signals appear stable.



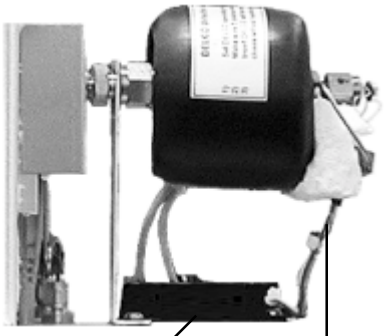
DELCD on an 8610C GC

Overview

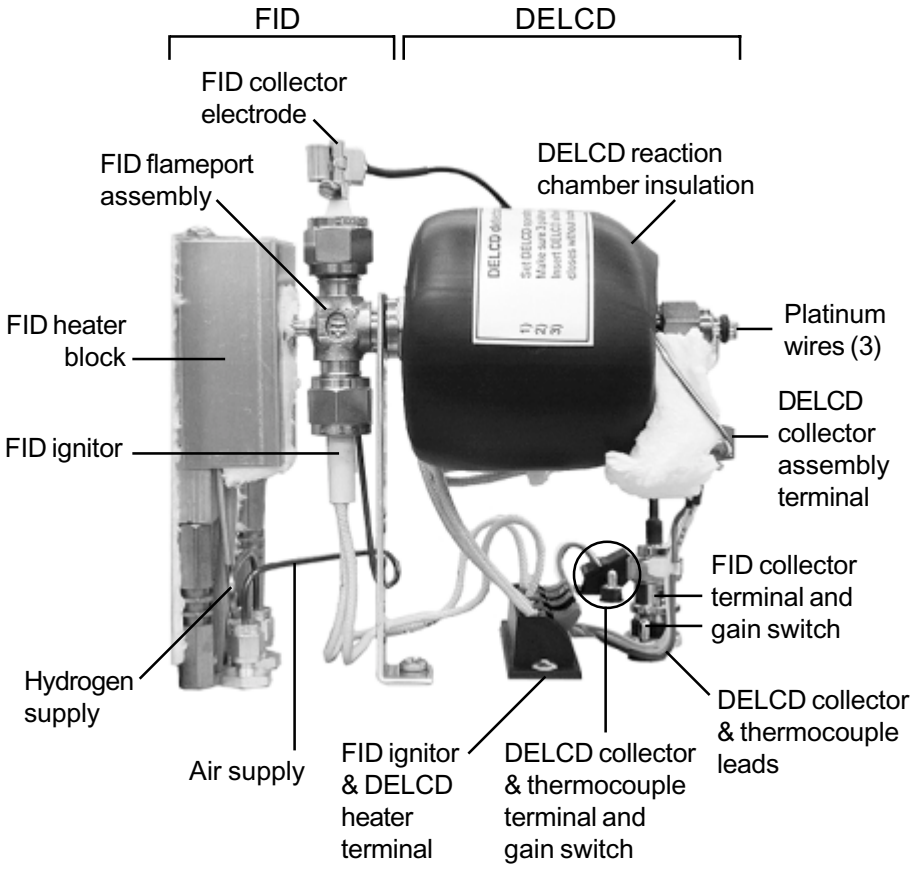
The Dry Electrolytic Conductivity detector, or DELCD, is selective to chlorinated and brominated molecules. It differs from the traditional wet ELCD in that it does not use a solvent electrolyte, and the reaction products are detected in the gaseous phase. The SRI DELCD is available alone or in combination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

DELCD - À la carte

Close-up of the same DELCD detector



FID / DELCD Combo Detector



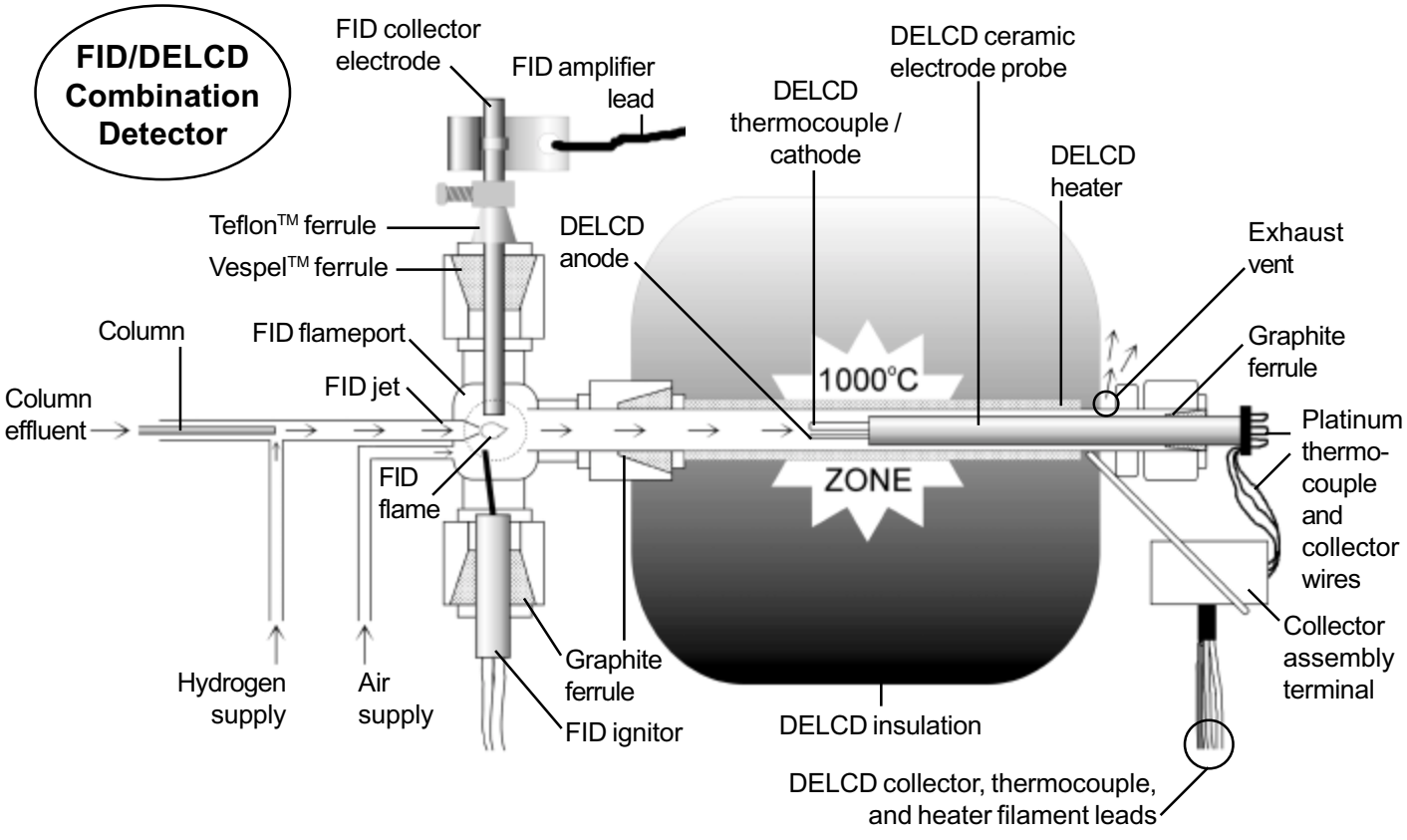
DETECTORS
Dry Electrolytic Conductivity Detector - DELCD

Theory of Operation

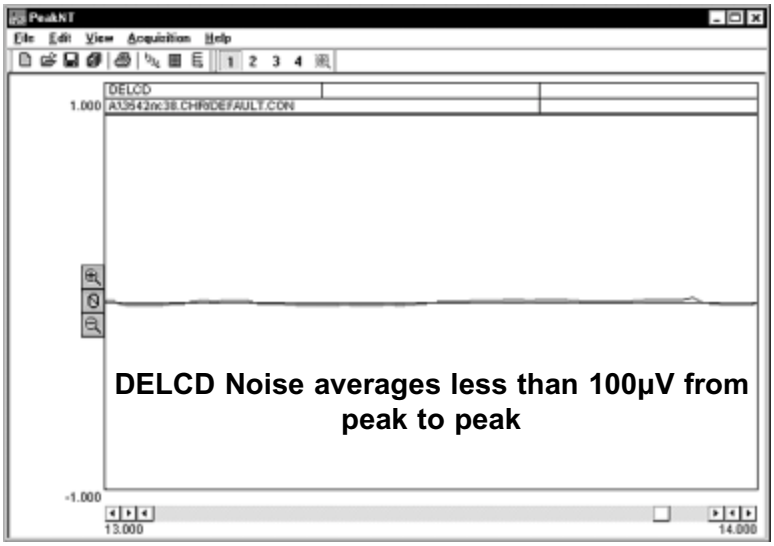
The DELCD consists of a small ceramic tube—the DELCD reactor—heated to 1000°C. Inside the reactor, a platinum thermocouple measures the detector temperature, and a nichrome collector electrode measures the conductivity of the gases flowing through the DELCD. The detector response is dependent upon its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

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

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



Expected Performance



DELCD Noise Run

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

Temperature program:

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

FID / DELCD Combo Test Run

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

Temperature program:

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

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

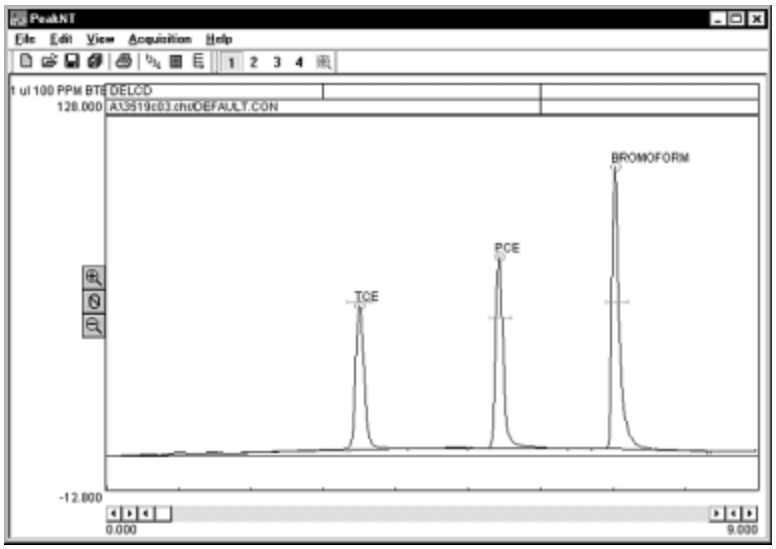
DELCD Results:

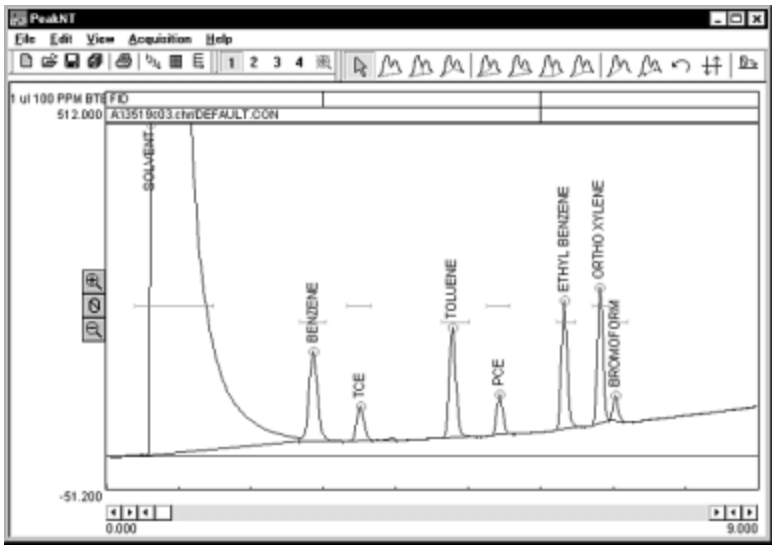
Component	Retention	Area
TCE	3.483	463.5080
PCE	5.416	532.2900
Bromoform	7.016	759.6650
	Total	1755.4630

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

FID Results:

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





DETECTORS
Dry Electrolytic Conductivity Detector - DELCD

General Operating Procedure

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

Combo Mode

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

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

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

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

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

High Sensitivity Mode

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

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

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

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

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

General Operating Procedure continued

FID/DELCD - FID Only

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

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

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

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

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

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

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

DELCD Only

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

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

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

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

Troubleshooting and Maintenance

Installing the Spare DELCD Cell

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

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

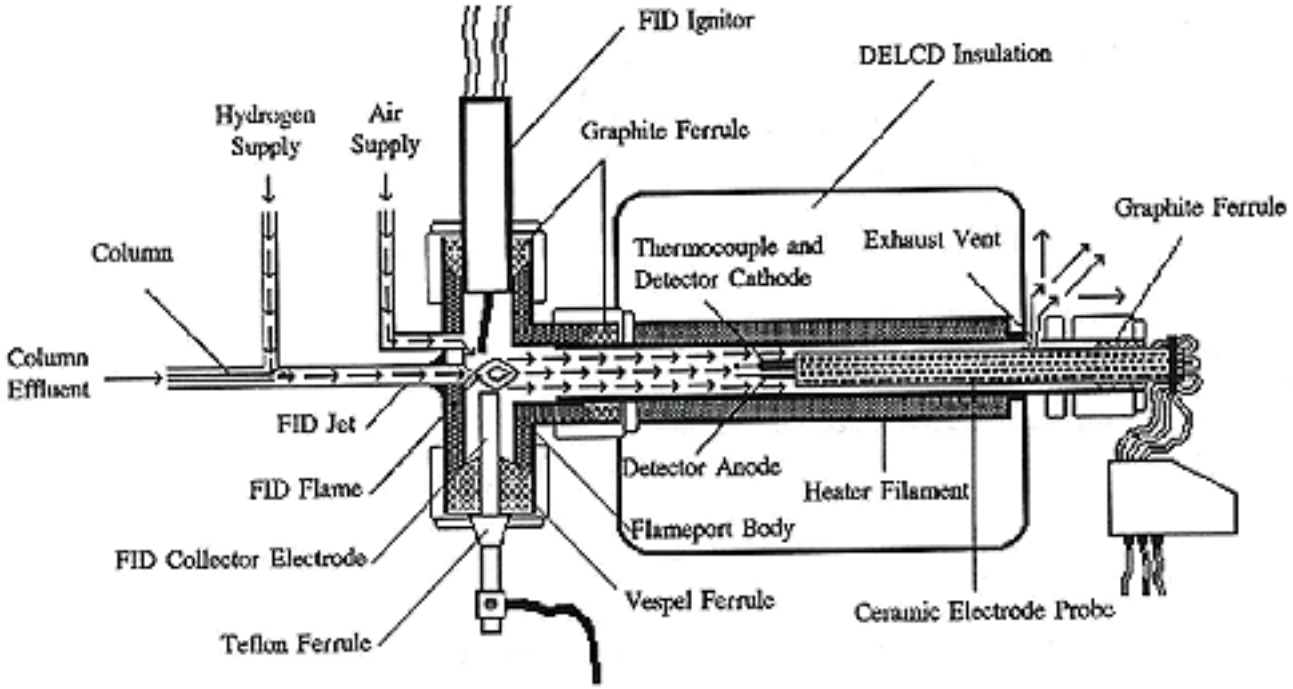
2. Remove the DELCD cell by using a wrench to loosen the 1/4" fitting that secures it on the FID exhaust port or on the heater block. You may have to hold the insulation aside to freely access the fitting; it is soft and may be compressed by hand.

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

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

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

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

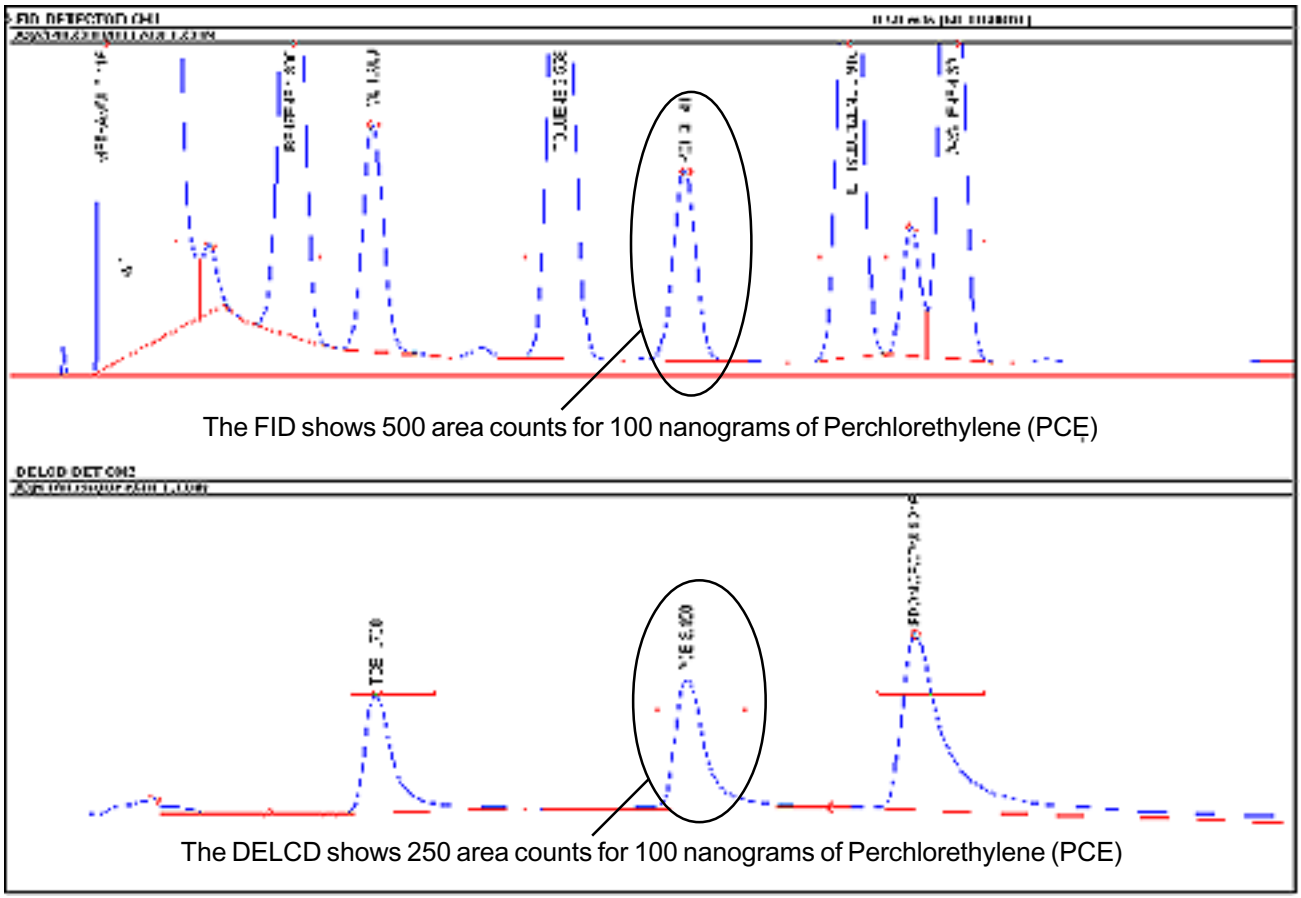


As diagrammed above, the sample enters the FID flame from the column where hydrocarbons are ionized and combusted. Electrons liberated in the ionization are collected by the FID collector electrode. About half the gas effluent (carrier gas + hydrogen + air + combustion products) flows out through the FID collector electrode which has an internal diameter of .040 (1 mm.). The restriction caused by the small collector i.d. splits the flow of exhaust gases so that the other half of the gases pass through the DELCD. The DELCD consists of a small ceramic tube which is heated to 1000ºC. In the center of the heated tube is a platinum thermocouple which measures the temperature and a DELCD collector electrode which measures the conductivity of the gases flowing through the DELCD. Since the response is very dependent on the temperature , the control circuit must maintain the temperature within a fraction of a degree at 1000ºC. ClO_2-BrO_2 exhibits extremely high conductivity at 1000ºC. So the DELCD actually responds to the ClO_2-BrO_2 concentration of the gases in the FID exhaust. Because other molecules are not detected, the DELCD is almost completely selective for chlorine and bromine. Fluorine and iodine are not well detected.

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 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.

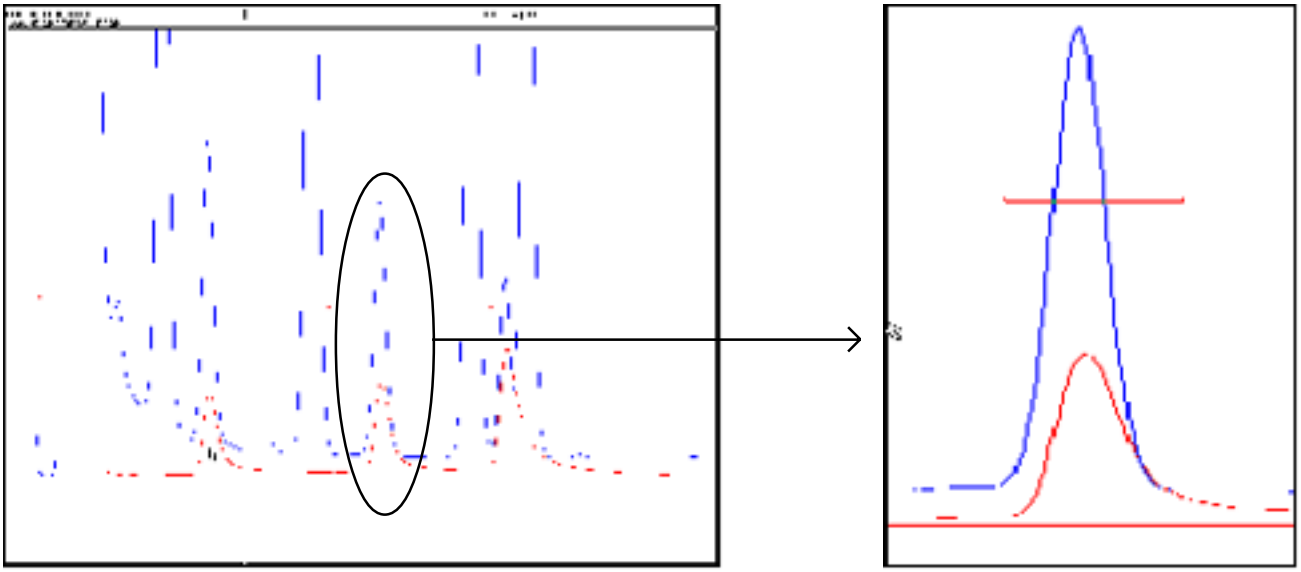
1) Set the FID hydrogen and air flows for normal FID operation. This is typically 25 ml/min hydrogen (corresponds to 25 psi) and 250 ml/min air (typically 6 psi). The exact pressure required for each flow is labeled on the GC's right hand side.
2) Set the DELCD temperature setpoint to 260 using the front panel adjustments. This number actually represents 1000ºC. 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).



The FID shows 500 area counts for 100 nanograms of Perchlorethylene (PCE)

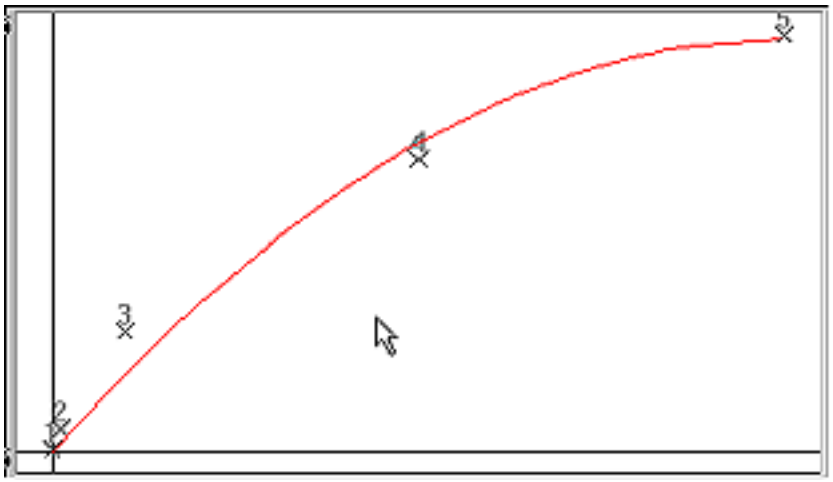
The DELCD shows 250 area counts for 100 nanograms of Perchlorethylene (PCE)

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.



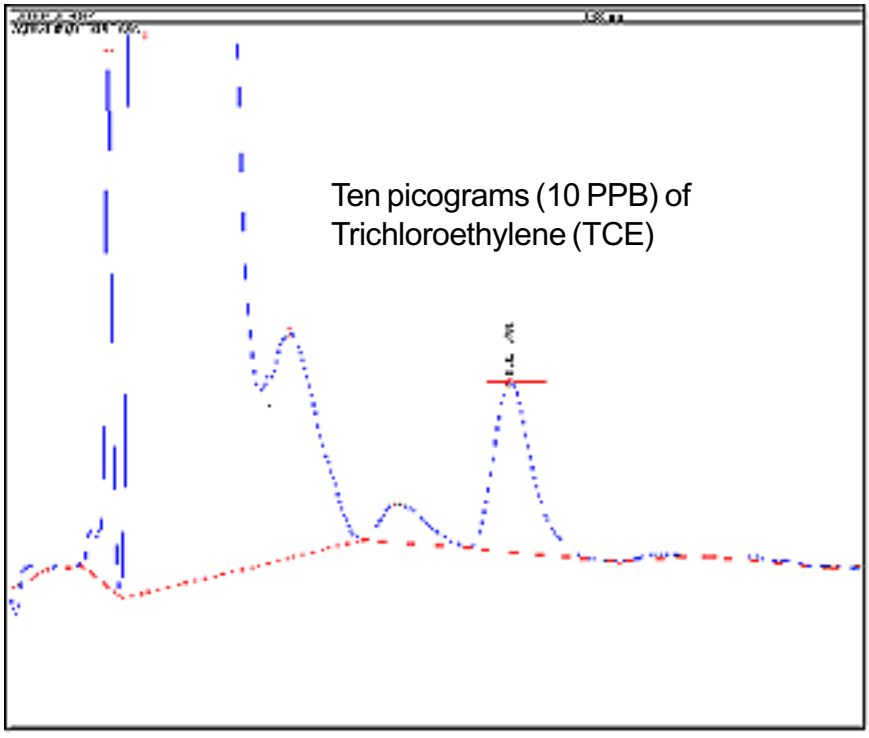
The DELCD calibration curve shown at right illustrates the quadratic response from 1–1000 nanograms of TCE injected

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 (0.25mm) 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.

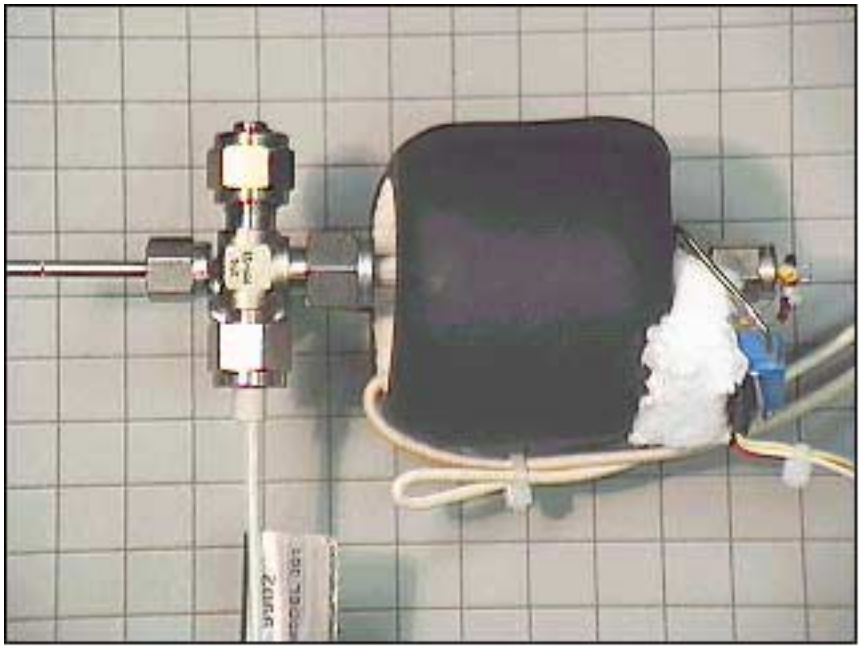
Note that in high sensitivity mode, there is some response to the methanol solvent.



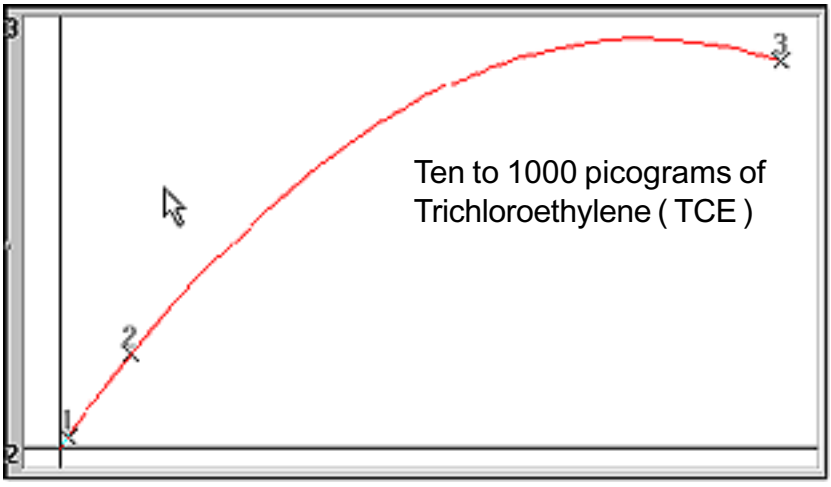
Operating the FID/DELCD in the high sensitivity DELCD only mode

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 µl 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.

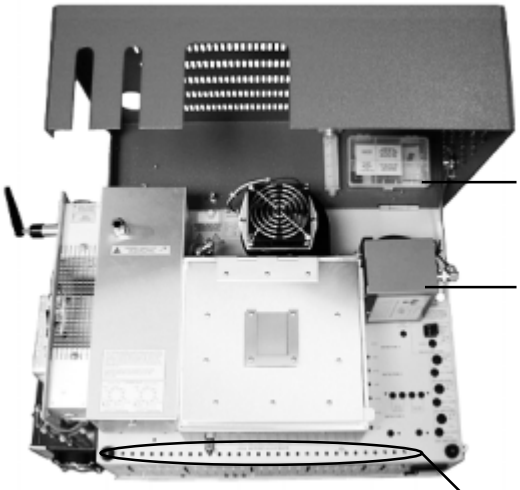


DELCD linearity in high sensitivity mode is shown at right from 10 to 1000 picograms (10-1000PPB).

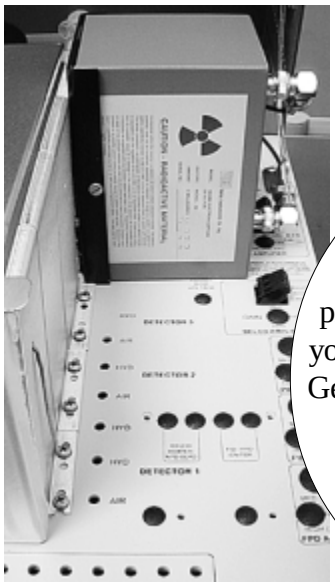
At levels above 10 nanograms, the detector is saturated.

OVERVIEW

The Electron Capture Detector (ECD) is selective to electronegative compounds, especially chlorinated, fluorinated, or brominated molecules. It is sensitive to some of these compounds in the parts per trillion (ppt) range. The ECD detector requires nitrogen or argon / 5% methane (P5) to operate. The ECD detector is mounted immediately adjacent to the right rear column oven wall on your GC chassis. Two BNC cables connect the anode and cathode, respectively, to the ECD amplifier. The ECD detector consists of a stainless steel cylinder containing 5 millicuries of radioactive Nickel 63 in an oven enclosure that is thermostatically controllable from ambient temperature to 375°C. Since the detector contains only 5 millicuries of Nickel-63, the ECD is covered by a "General License" requiring a periodic wipe test and the filing of a form with your state's Department of Health. The documentation necessary to authorize your possession of a radioactvie source is included in the ECD manual from Valco, the manufacturers. This documentation transfers possession of the ECD directly to you from SRI; provides the ECD installation service and the GC. There are four important documents to look for: 1) Certification of Sealed Source, 2) Conditions for Acceptance of a Generally Licensed Device, 3) Test Specifications, and 4) Record of Source Transfer. Valco may print your address on the multiple copies of the Record of Source Transfer, which are to be completed by you and filed with the appropriate state and local authorities. The other documents remain with the ECD detector, and are necessary to prove authorized possession of the ECD.

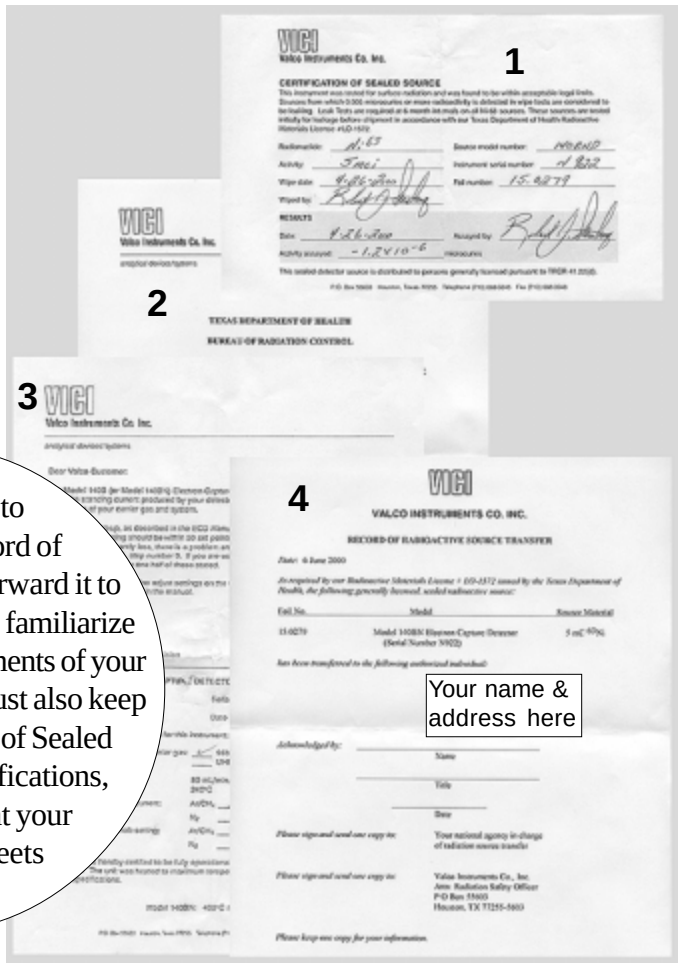


ECD-equipped SRI GC



Close-up view of the ECD detector on an SRI GC

It is imperative to complete the Record of Source Transfer and forward it to proper authorities, and to familiarize yourself with the requirements of your General License. You must also keep on file the Cerification of Sealed Source and Test Specifications, which are proof that your ECD detector meets regulations.

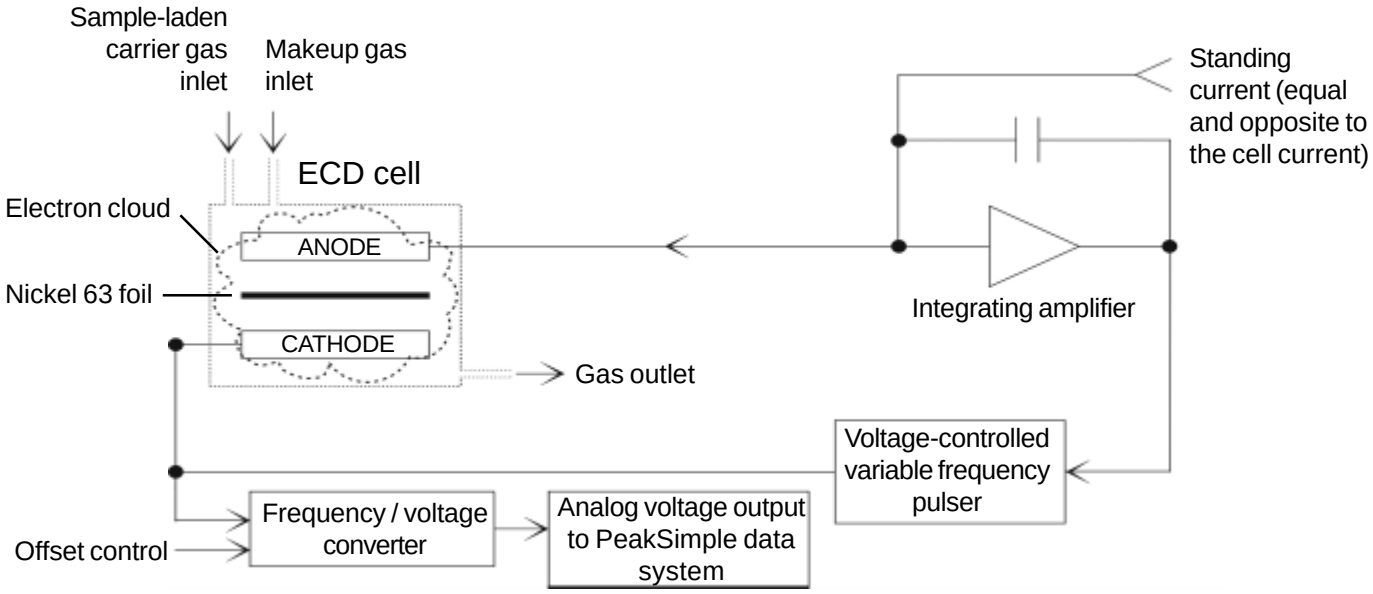


DETECTORS
Electron Capture Detector - ECD

Theory of Operation

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

ECD Detector Operational Diagram



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

Example Pulse Trains

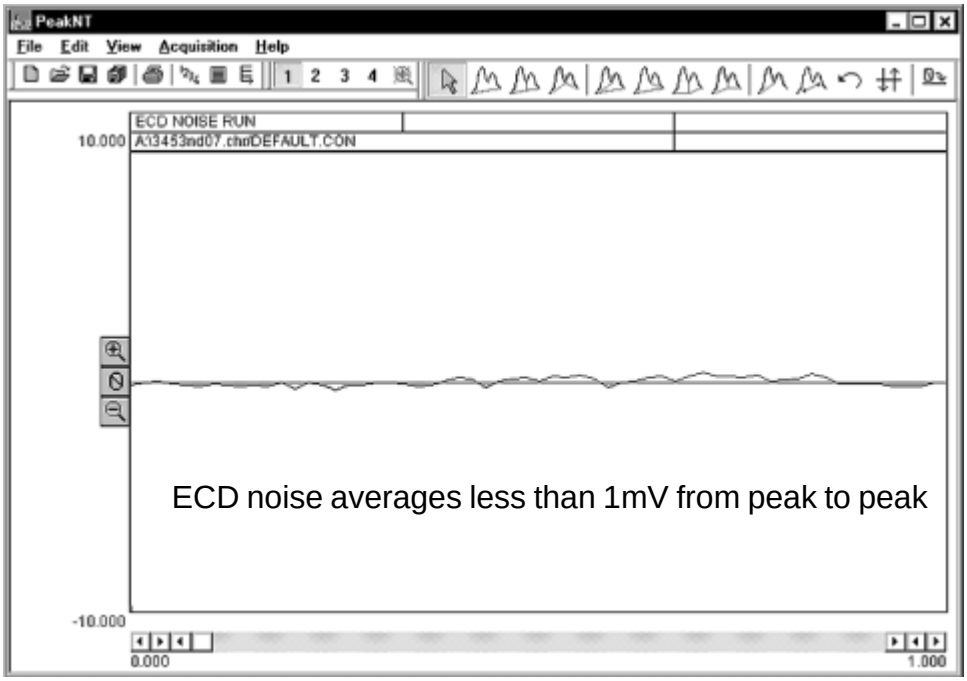
Detector electronics pulse to maintain the standing current

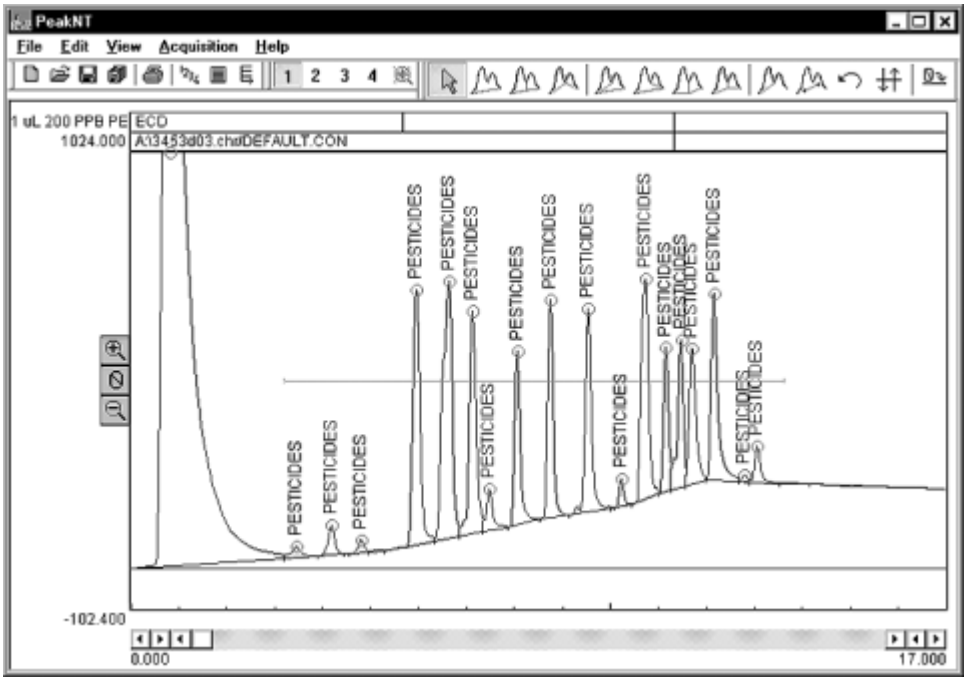
The pulse rate is increased in the presence of electronegative compounds

Expected Performance

ECD Noise Run

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





ECD Pesticide Analysis

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

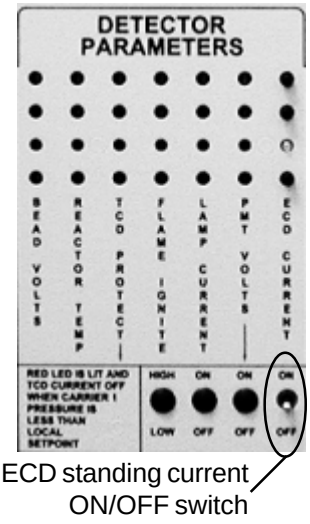
Results:

Component	Retention	Area
Pesticides	2.850	45792.4350

DETECTORS
Electron Capture Detector - ECD

General Operating Procedure

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



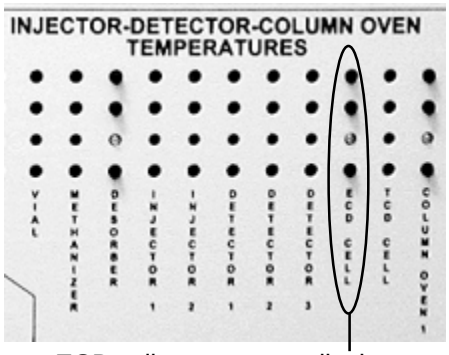
ECD standing current ON/OFF switch

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

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

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

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



ECD cell temperature display

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

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

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

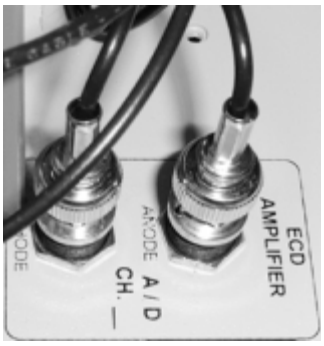
ECD Troubleshooting

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

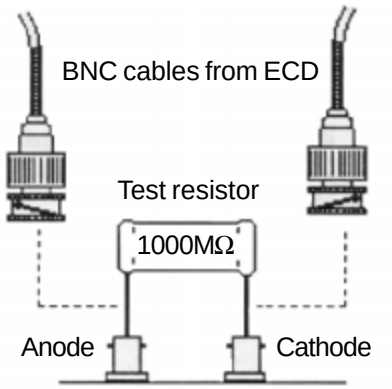
1. Verify that the ECD amplifier electronics are working properly by removing the detector from the circuit and inserting a 1000MOhm test resistor in its place. The parts kit in the tackle box included with your GC under the red lid contains a 1000MOhm resistor for this test. Turn the ECD current off.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

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

Anode and cathode BNC connectors



ECD electronics test

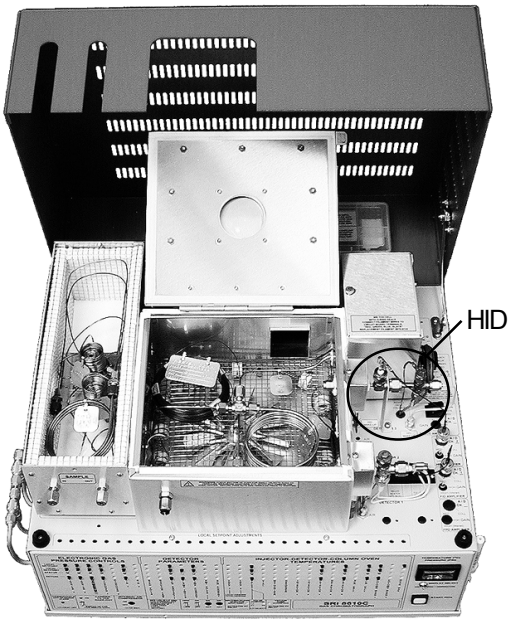


2. Operate the ECD on make-up gas only by disconnecting the column from the ECD. With the standing current still set at 300, observe the signal offset and noise. If it drops, then the problem is being introduced into the GC and ECD by the carrier gas through the column.

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

Overview

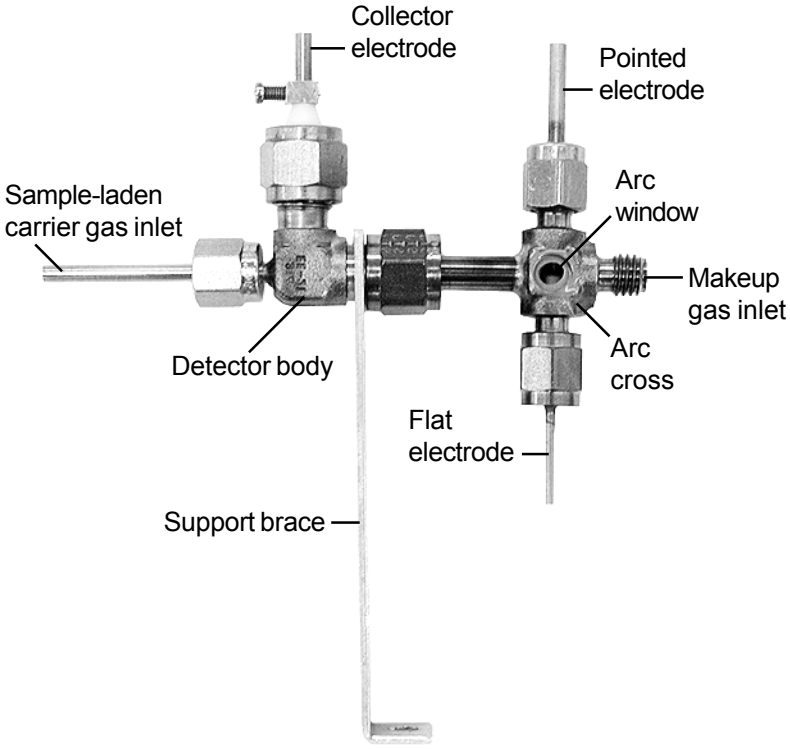
The Helium Ionization Detector is a universal detector, responding to all molecules except neon. It requires only helium carrier and make-up gas, and is sensitive to the low ppm range. The HID is particularly useful for volatile inorganics to which the FID and other selective detectors will not respond, 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 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 Buck GCs, the HID is mounted on the right-hand side of the Column Oven.



HID detector between TCD and FID detectors

Close-up of the same HID detector

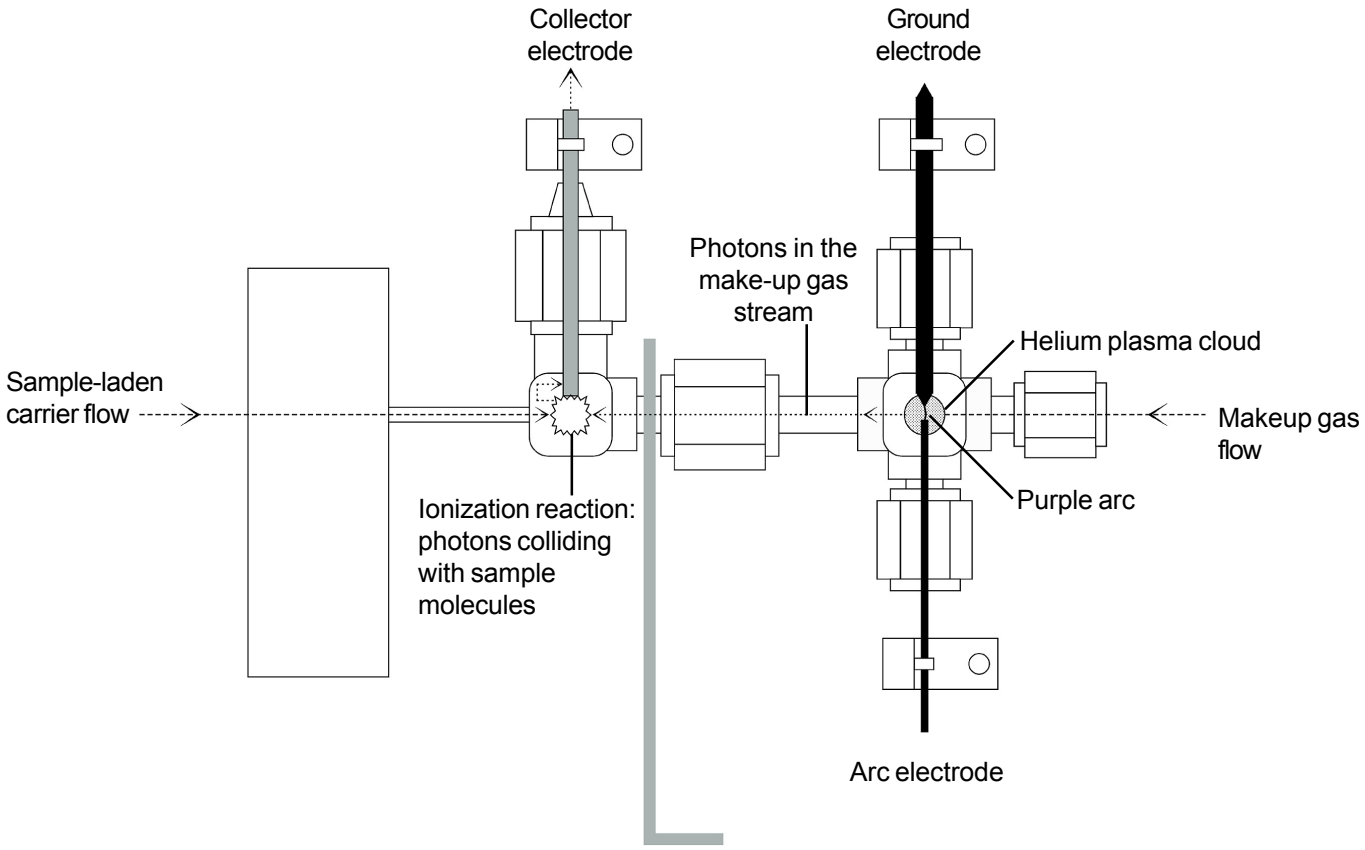
HID detector removed from GC and heater block



DETECTORS
HID - Helium Ionization Detector

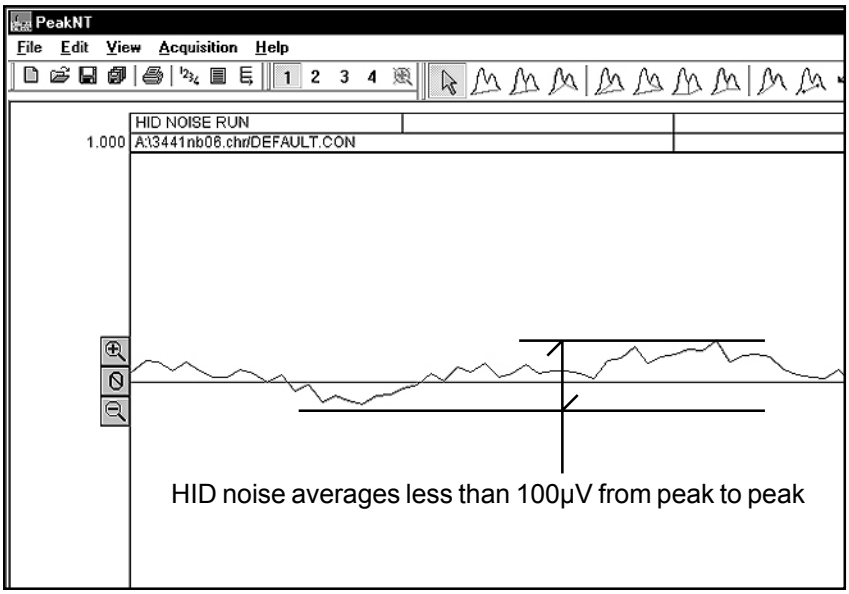
Theory of Operation

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



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

Expected Performance



HID noise run

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

Temperature Program:

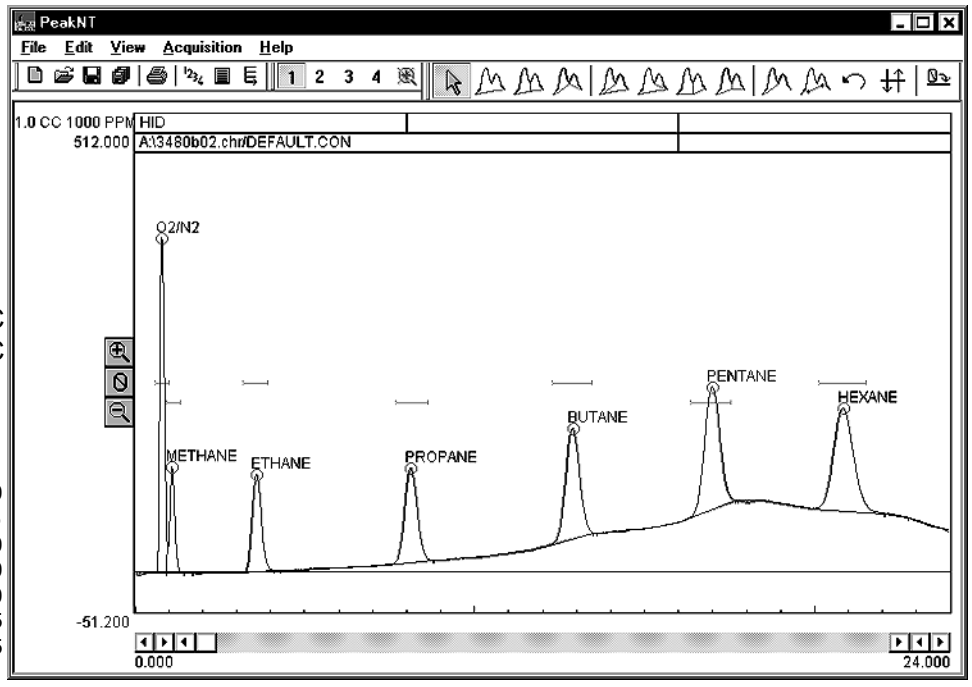
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

Test Analysis of 1cc 1000ppm C_1-C_6

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

Temperature program:

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



Results:

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

DETECTORS
HID - Helium Ionization Detector

General Operating Procedure

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

2. Set the helium make-up gas flow to 40mL/min, and the helium carrier gas flow to 10mL/min. The make-up gas flow is critical to the HID's performance. With insufficient make-up flow, the chlorinated peaks will be inverted on the chromatogram; see the chromatograms compared on the ***HID Make-up Gas Flow*** page. 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.

HID Make-up Gas Flow

The following chromatograms were produced by a HID equipped GC. Excepting the make-up gas flows, all run conditions are identical. The first chromatogram resulted from a make-up gas flow of 20mL/min. Drastically different in appearance from the first, the second chromatogram was produced with a make-up gas flow of 10mL/min. In the absence of sufficient make-up gas flow, the chlorinated peaks are negative. Not every HID has the same optimum make-up flow; experiment with different flow rates until you find the best range for your detector.

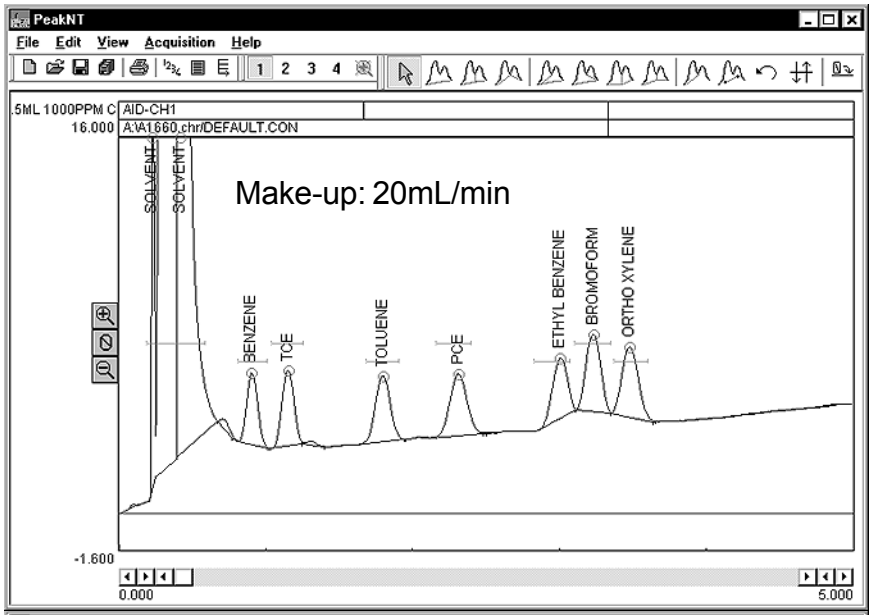
Sample: 0.5mL 1000ppm C_1-C_6
Column: 30m MXT1-5
Carrier: Helium @ 10mL/min

Temperature program:

Initial	Hold	Ramp	Final
50°C	1.00	10.00	140°C

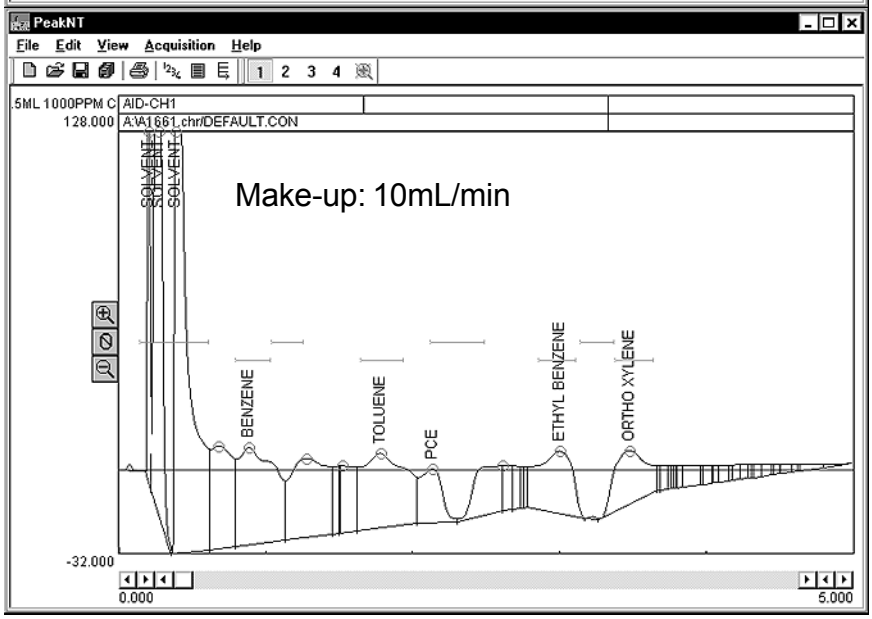
Results:

Component	Retention	Area
Solvent	0.305	4791.9566
Benzene	0.896	14.5888
TCE	1.145	17.9614
Toluene	1.790	19.6294
PCE	2.305	21.3786
Ethyl Benzene	2.998	23.5176
Bromoform	3.221	22.0414
Ortho Xylene	3.470	26.3280
	Total:	4937.4018



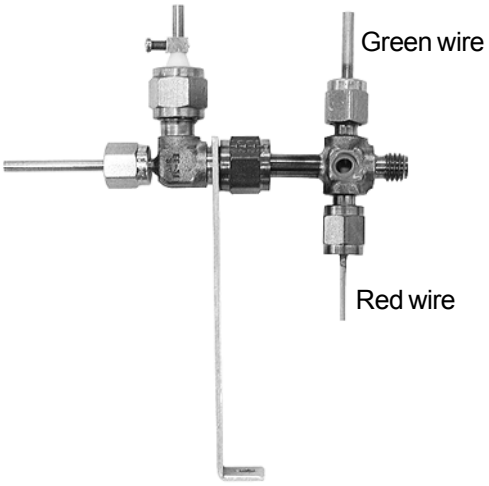
Results:

Component	Retention	Area
Solvent	0.381	1771.5762
Benzene	0.876	622.0096
TCE	1.266	527.2432
Toluene	1.771	571.1129
Ethyl Benzene	2.993	379.2581
Ortho Xylene	3.468	312.9010
	Total:	4184.1010

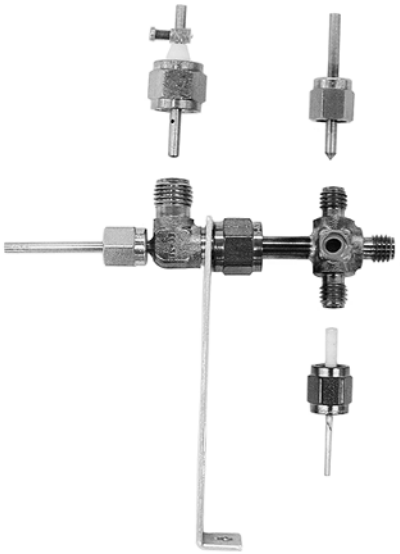


Cleaning the HID

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).

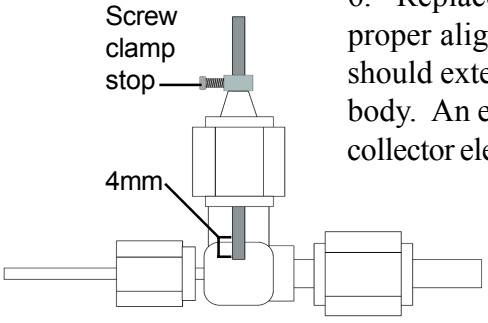


2. Remove the the arc and ground electrodes by loosening the 1/8" fittings that hold the electrodes in the arc cross.

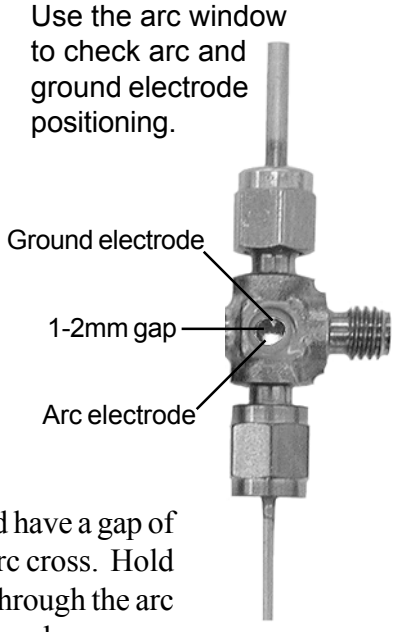
3. Remove the collector electrode by loosening the 1/4" fitting that secures it in the detector body.

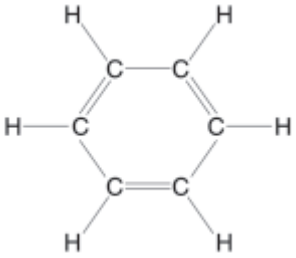
4. Use a piece of 100-400 grain sandpaper to clean the surface of the collector electrode and the point of the ground electrode. Sand the tip of the arc electrode so that it is flush against the ceramic insulation, and to remove any residue. While handling the electrodes, try to minimize hand contact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



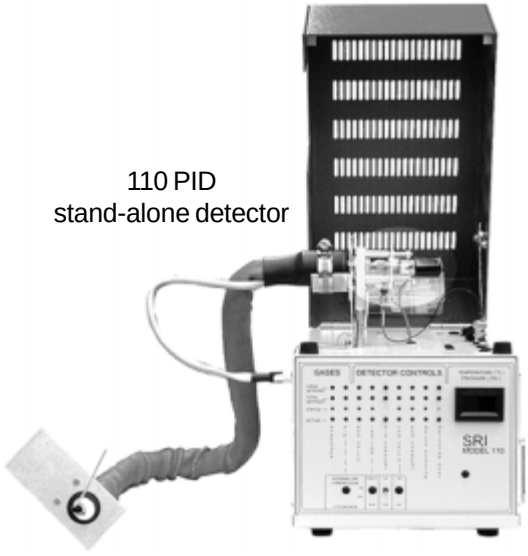


Benzene

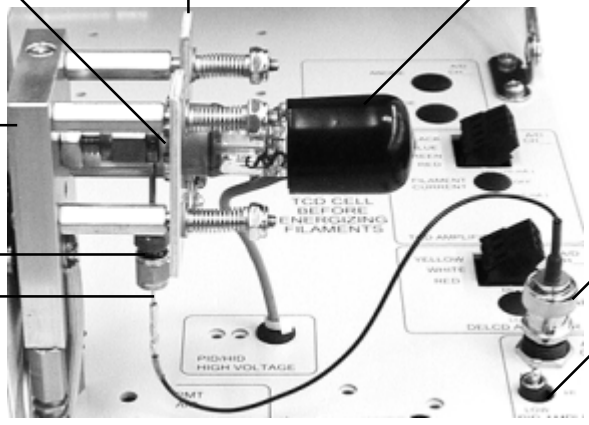
Overview

The Photo Ionization Detector (PID) responds to all molecules whose ionization potential is below 10.6eV, including aromatics and molecules with carbon double bonds. The PID is nondestructive, so the sample can be routed through the PID and on to other detectors. It is often used in series with the FID and / or DELCD. PID detection limits for aromatics are in the ppb range; purge and trap concentration of the sample can lower detection limits to the ppt range. Because of its selective sensitivity, use of the PID is mandated in several EPA methods. The PID detector consists of a 10.6 electron volt (eV) UV lamp mounted on a thermostatted, low-volume (100µL), flow-through cell. The temperature is adjustable from ambient to 250°C. Three detector gain levels (LOW, MEDIUM and HIGH) are provided for a wide range of sample concentrations. The PID lamp is held in place by a spring-loaded plate, so that the lamp may be quickly removed for cleaning and replaced without any special tools. The PID can run on air carrier for gasless operation, or for stream monitoring applications where the entire stream of sample is directed through the detector (no column is used).


PID in series with a DELCD and an FID on a 910 chassis



110 PID
stand-alone detector



100µL detector cell (the area in the center of the Teflon™ seal ring)

Spring-loaded retaining plate

High voltage band in black plastic hood over lamp anode ring

Heater block

Collector electrode support union

Collector electrode

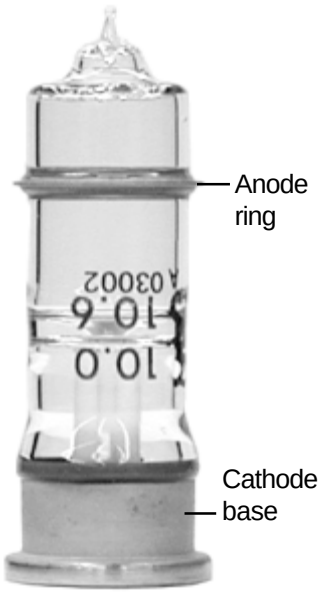
BNC connector to amplifier

Amplifier gain switch

DETECTORS
Photo Ionization Detector - PID

Theory of Operation

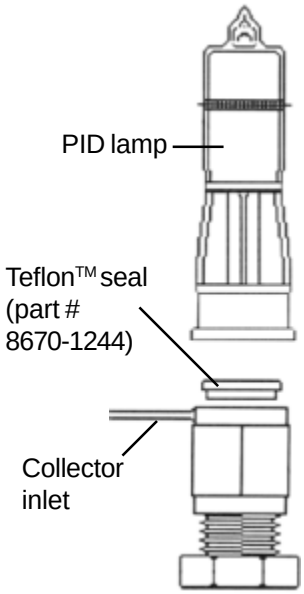
10.6eV PID Lamp (Part # 670-1242)



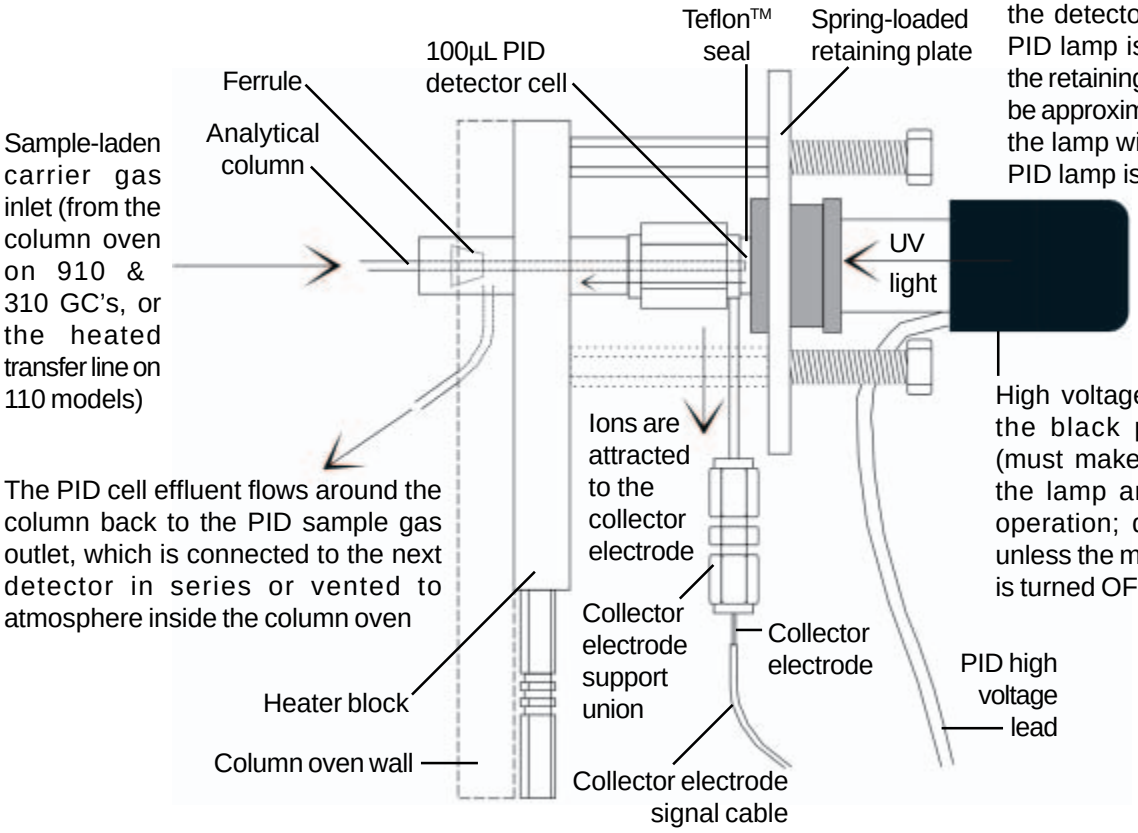
The PID design uses a 10.6eV lamp with a high voltage power supply. Sample laden carrier gas flows from the analytical column into the PID sample inlet, where it is streamed through a 100µL flow-through cell. When sample molecules flow into the cell, they are bombarded by the UV light beam. Molecules with an ionization potential lower than 10.6eV release an ion when struck by the ultraviolet photons. These ions are attracted to a collector electrode, then sent to the amplifier to produce an analog signal, which is acquired by the PeakSimple data system.

Unlike other PID designs that heat the entire lamp, only the lamp window of the SRI PID is heated. This results in a longer lamp life for PID detectors.

Partial PID Assembly - Exploded View



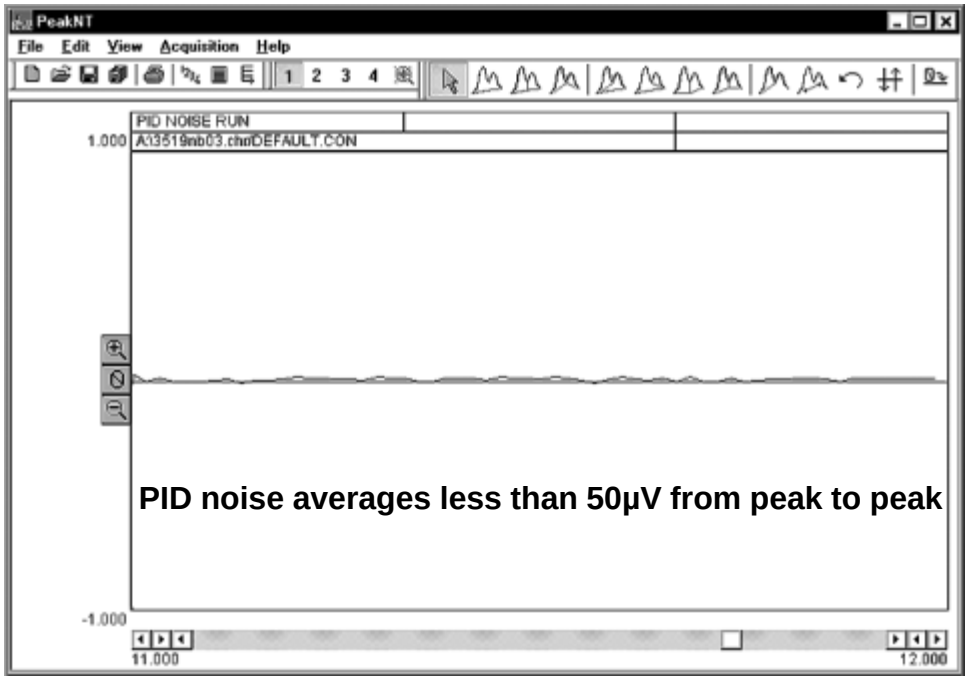
Simplified PID Operational Diagram



NOTE: The end of the column must be visible in the detector cell when the PID lamp is removed from the retaining plate. It should be approximately 1mm from the lamp window when the PID lamp is in place.

High voltage band inside the black plastic hood (must make contact with the lamp anode for PID operation; do not adjust unless the main GC power is turned OFF)

Expected Performance



PID Noise Run

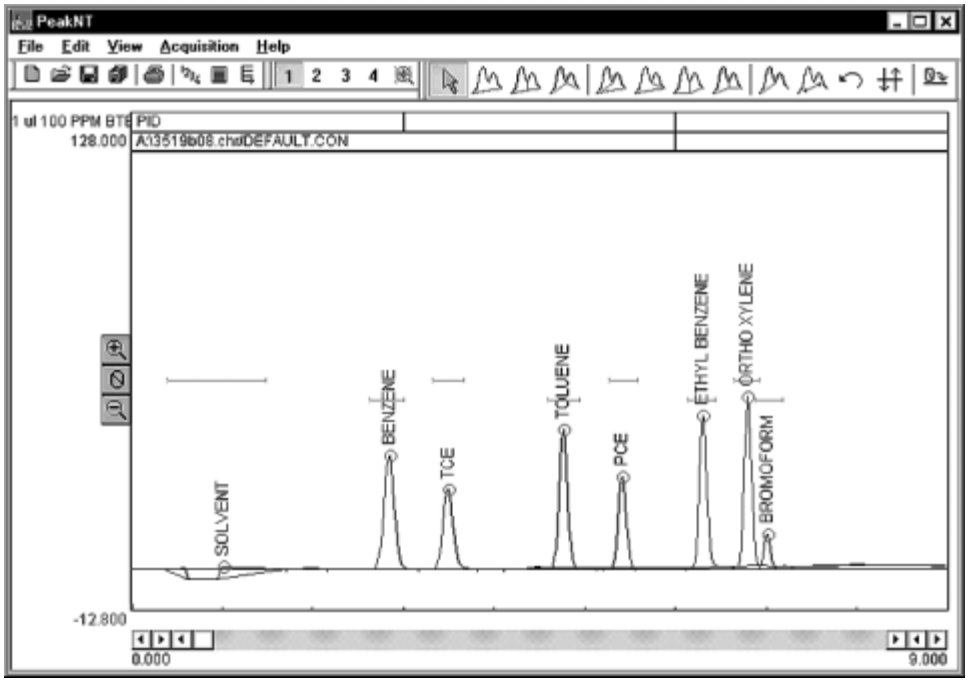
Column: 15m MXT-VOL
Carrier: Helium @ 10mL/min
PID gain: LOW
PID temp: 150°C
PID current: 70

PID BTEX Analysis (in series with FID and DELCD)

Sample: 1µL 100ppm BTEX plus
Column: 15m MXT-VOL
Carrier: Helium @ 10 mLs/min
PID gain: LOW
PID temp: 150°C
PID current: 70

Temperature program:

Initial	Hold	Ramp	Final
40°C	2.00	15.00	240°C
240°C	10.00	0.00	240°C



Results:

Component	Retention	Area
Benzene	2.416	313.0540
TCE	3.066	231.5120
Toluene	4.600	309.2120
PCE	5.433	216.6230
Ethyl Benzene	6.700	286.0900
Ortho Xylene	7.383	298.9190
Bromoform	7.650	55.9460
	Total	1711.3560

General Operating Procedure

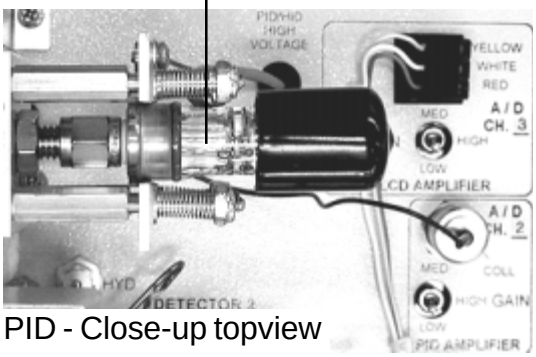
The capillary column enters the PID cell from inside the column oven through the bulkhead fitting in the insulated oven wall. The column may be installed with the lamp in place. Insert the capillary column into the PID detector inlet until the column stops at the lamp window inside the PID cell, then pull it back about 1mm from the lamp window. Tighten the 1/8" nut with the graphite ferrule at the PID inlet to secure the column in place. The collector electrode is positioned at the factory and should not touch the column under normal circumstances.

1. Always ensure that the black plastic hood is in place on the lamp prior to operating the PID detector. The hood contains the high voltage band which is maintained at a high potential; never attempt to adjust the PID high voltage band unless the main GC power is turned off.

2. Turn ON the GC. Turn ON the PID lamp current with the flip switch on the GC's front control panel.

3. Set the PID current to 70 (= 0.70ma) with the trimpot setpoint on the top edge of the GC's front control panel. Use the flat blade screwdriver provided with your GC to adjust the trimpot. The lamp should emit a violet-colored light visible down the center of the tube.

The violet light is visible here when the lamp is on



PID - Close-up topview

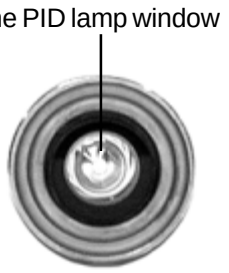
4. Confirm that the lamp is operating at or near 0.70ma by pressing the PID detector ACTUAL display button on the front control panel. The sensitivity of the lamp increases proportionally to the current applied, but operation at higher currents reduces lamp life. The PID operating current range is 70-125. A setting of 70 should provide the user with sufficient sensitivity and lamp durability. Most PID applications can be performed using LOW gain.

5. Set the PID temperature to 150°C.

6. Once the detector has reached temperature and the signal appears stable, sample may be introduced.

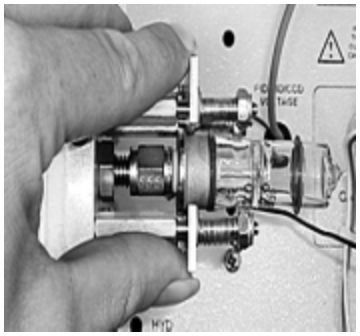
NOTE: Lamps are a consumable part of the PID detector. It is recommended to have a spare lamp available if critical analyses are being performed at remote field sites. Spare and replacement 10.6eV PID lamps are available under part number 670-1242. Teflon seals are available under part number 670-1244.

Troubleshooting and Maintenance
Cleaning the PID Lamp



Over time, during normal operation, a film of contaminants will condense on the PID lamp window. Typically, this film is a result of stationary phase column bleed. To minimize contaminant condensation and thus lamp window cleaning, avoid heating the column any higher than absolutely necessary. Contaminant condensation can block the photons, reducing lamp emissions and sensitivity. Therefore, the PID lamp window must be cleaned when an appreciable change in sensitivity has been observed by the operator. Because the response change resulting from cleaning the lamp window usually requires detector recalibration, frequent cleaning is not recommended.

1. Turn the PID current OFF with the switch on the GC's front control panel. Turn the GC OFF and let the PID detector assembly cool enough to touch it without getting burned.

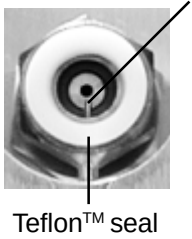


2. Disconnect the high-voltage band from the lamp anode by removing the black plastic hood.

3. Grasp the spring-loaded retainer plate with the fingers of one hand and push or pull it toward the PID lamp; it doesn't take much force to move the plate enough for lamp removal. Slide the PID lamp up and out of the PID detector assembly.

4. Clean the lamp window using a mild abrasive cleanser like Bon Ami or Comet. Wet your finger, and make a paste with a small amount of cleanser. Scrub the lamp window clean in a circular motion with your finger.

5. Rinse the lamp window clean with water. Dry the lamp with a paper towel.



6. Inspect the Teflon™ seal for cuts or nicks. A damaged seal will not affect the PID response, but it may provide a leak site that will reduce the amount of sample delivered to any subsequent detector.

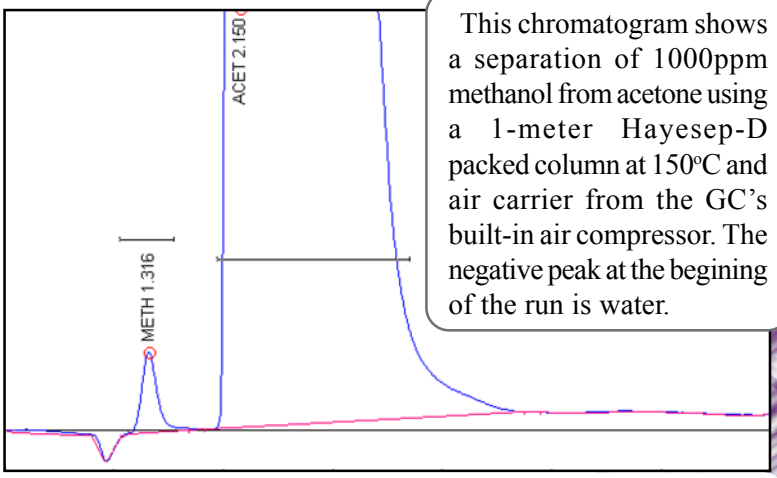
7. With the lamp removed, the collector electrode is visible where it protrudes into the cell. Check the collector electrode for any visible residues, films, discolorations, etc. If present, they may impede the flow of ions from the sample molecules to the collector electrode. To clean the collector electrode, gently use a small file to remove any residues from its tip. Blow the residue off the collector electrode and surrounding areas.

8. Open the spring-loaded retainer plate and replace the PID lamp snug against the seal. The lamp window has a slightly larger diameter than the seal; try to center it against the seal. Replace the high voltage band / black plastic lamp hood.

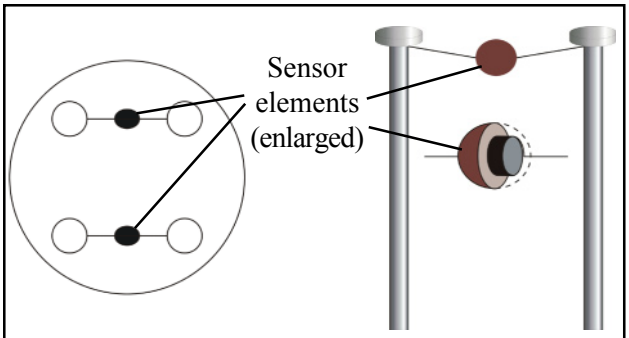
9. Recalibrate the PID detector before returning it to service.

CCD - Catalytic Combustion Detector

- ***Hydrocarbon and H_2 Selective***
- ***Detects Down to 500ppm***
- ***Gas-less Operating Capability***
- ***Inexpensive and Rugged***
- ***Built-in Spare!***



This chromatogram shows a separation of 1000ppm methanol from acetone using a 1-meter Hayesep-D packed column at 150°C and air carrier from the GC's built-in air compressor. The negative peak at the begining of the run is water.



The CCD housing is about 1cm in diameter. Inside, there are 2 sensor elements consisting of a platinum wire embedded in a catalytic ceramic bead.

The CCD is about as sensitive as a TCD, but it has the hydrocarbon selectivity of an FID while capable of operating on air alone. Because the CCD needs no compressed gases like hydrogen or helium, it can be used in the Gas-less™ GCs where a built-in, "whisper quiet" air compressor supplies the ambient air carrier gas.

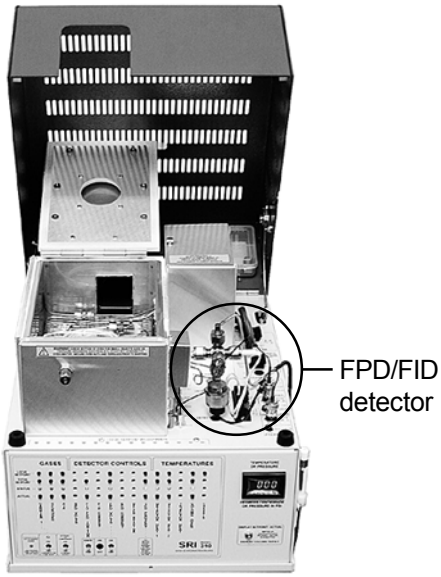
The CCD can also be used as a hydrocarbon monitor in non-chromatographic applications where the CCD senses the total hydrocarbon content of a flowing air stream, or as a hydrogen/hydrocarbon leak detector.

The CCD detector sensor is rugged and can be expected to last a long time. A second sensor is included in the detector housing at no extra cost, providing a built-in replacement should the first sensor become inoperable. Replacement sensor sets install in minutes without tools and are very economical, making this detector a good choice for academic settings where the detector may be damaged by inexperienced operators.

The Catalytic Combustion Detector consists of a tiny coil of platinum wire embedded in a catalytic ceramic bead. A small electric current flows through the platinum coil, heating the ceramic bead to around 500°C. The CCD is maintained in an oxidative environment typically by using air carrier gas. When a hydrogen or hydrocarbon molecule impacts the hot bead, it combusts on the surface and raises the temperature and resistance of the platinum wire. This resistance change causes the detector output signal to change, thus producing a peak. The brass detector housing is mounted on a stainless steel bulkhead fitting, which is secured directly to the wall of the GC column oven.

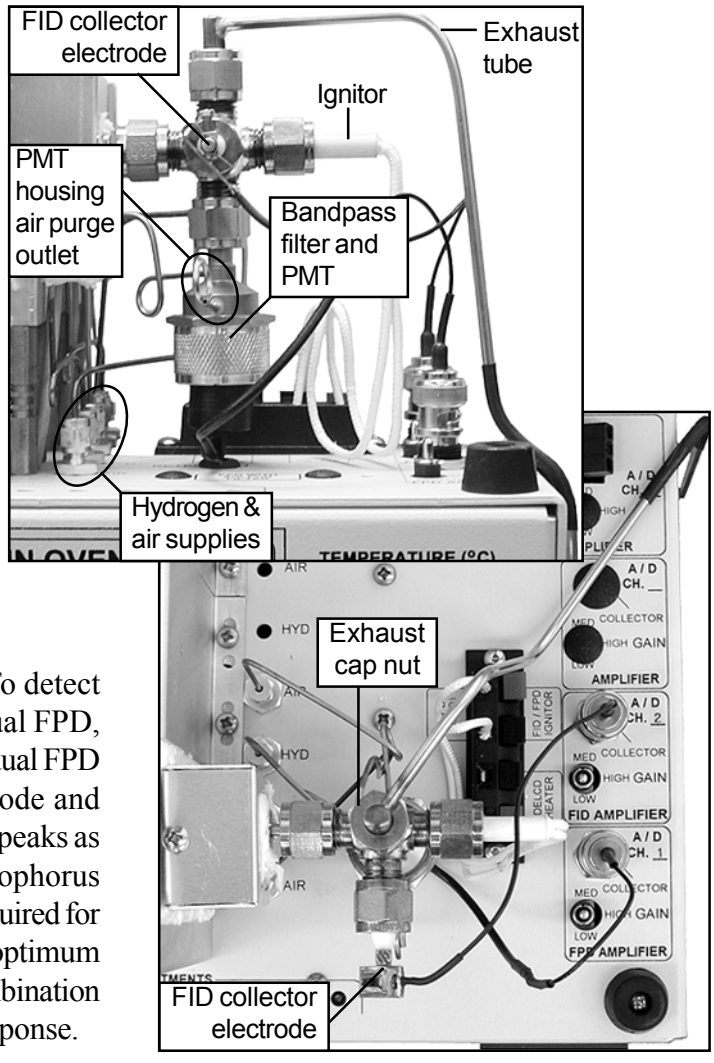
8690-2007	CCD detector
8670-2007	Replacement CCD detector housing (2 sensors in 1 housing)

Overview



The Flame Photometric Detector is similar to the FID in that the sample exits the analytical column into a hydrogen diffusion flame. Where the FID measures ions produced by organic compounds during combustion, the FPD analyzes the spectrum of light emitted by the compounds as they luminesce in the flame. The detector chamber must be light tight so that only light from the flame will be "seen" by the photomultiplier tube (PMT) and analyzed. The FPD uses a second hydrogen flow to purge the optical path between the PMT and the hydrogen diffusion flame. This second hydrogen flow helps to augment the FPD sensitivity by making the flame hydrogen rich. The FPD also uses a second air flow directed across of the face of the PMT to prevent helium and/or hydrogen molecules from permeating the PMT's glass window and causing malfunction. This purge air is vented to atmosphere through a short tube, coiled to prevent light from reaching the PMT. The FPD uses one of two available band pass filters to selectively detect compounds containing sulfur or phosphorus. Compounds containing phosphorus are detectable with the 526nm filter, which is yellow on one side. The 394nm filter (blue on one side) allows detection of sulfur-containing compounds. While not completely selective, the FPD is 100,000 times more sensitive to sulfurous and phosphorous compounds than it is to hydrocarbons. Sulfur compounds like H_2S or SO_2 can be detected down to about 200ppb; phosphorus compounds can be detected down to 10ppb. To detect phosphorus and sulfur at the same time, the Dual FPD, featuring two PMTs, may be used. The single or dual FPD may be equipped with an FID collector electrode and electrometer which will detect the hydrocarbon peaks as the PMTs are responding to the sulfur and phophorus compounds. Because the hydrogen-rich flame required for optimum sulfur and phosphorus detection is not optimum for the best hydrocarbon response, the FID in combination with the FPD is less sensitive than a pure FID response.

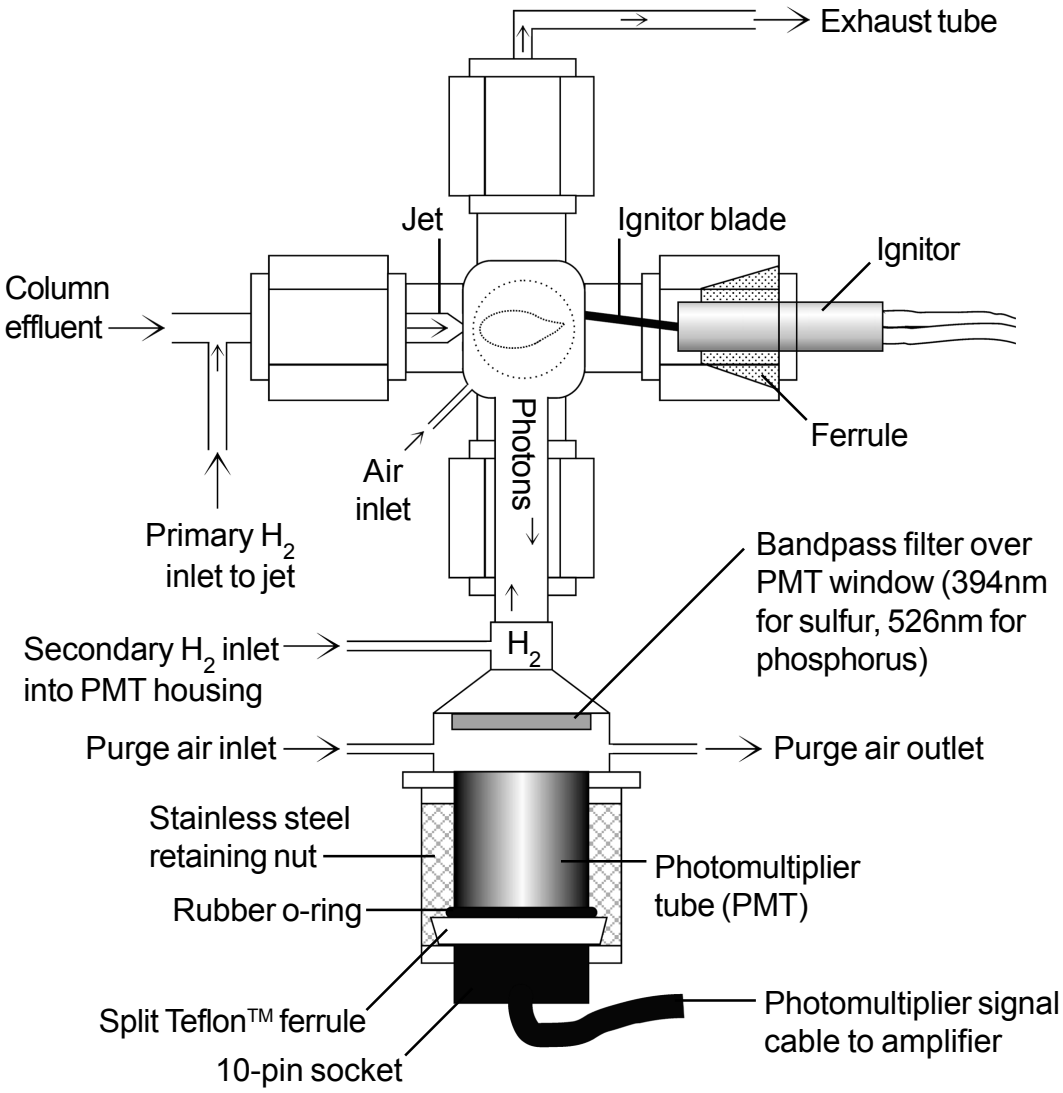
FPD detector equipped with an FID collector

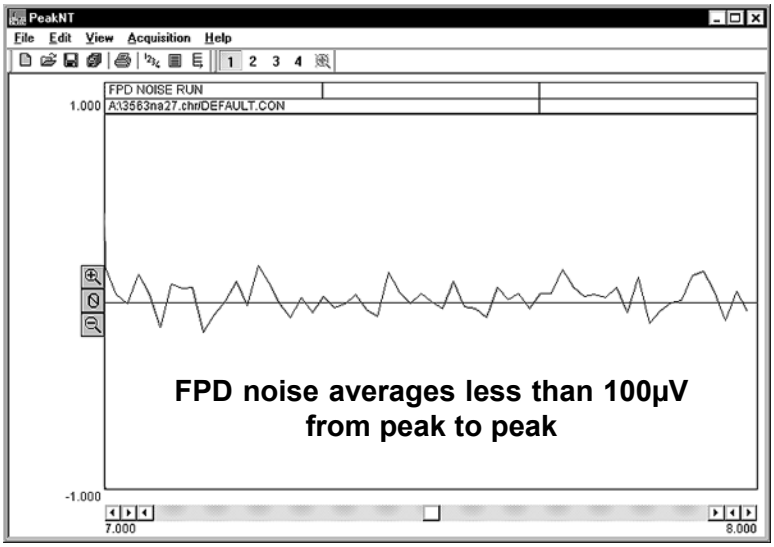


Theory of Operation

The FPD uses one of two available band pass filters over a photomultiplier tube (PMT) to selectively detect compounds containing sulfur or phosphorus as they combust in the hydrogen flame. When compounds are burned in the FPD flame, they emit photons of distinct wavelengths. Only those photons that are within the frequency range of the filter specifications can pass through the filter to the PMT. The PMT converts the photons it "sees" through the bandpass filter to an analog signal, which is acquired by the Peak Simple data system.

Simplified FPD Schematic





Expected Performance

FPD Noise Run (FID/FPD Combo)

Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FPD gain: HIGH
FPD temp: 150°C
FPD PMT volts: -400
FPD H_2: 60mL/min (30mL/min for each of the two hydrogen flows)
FPD air: 100mL/min

FPD Sulfur (FID/FPD Combo)

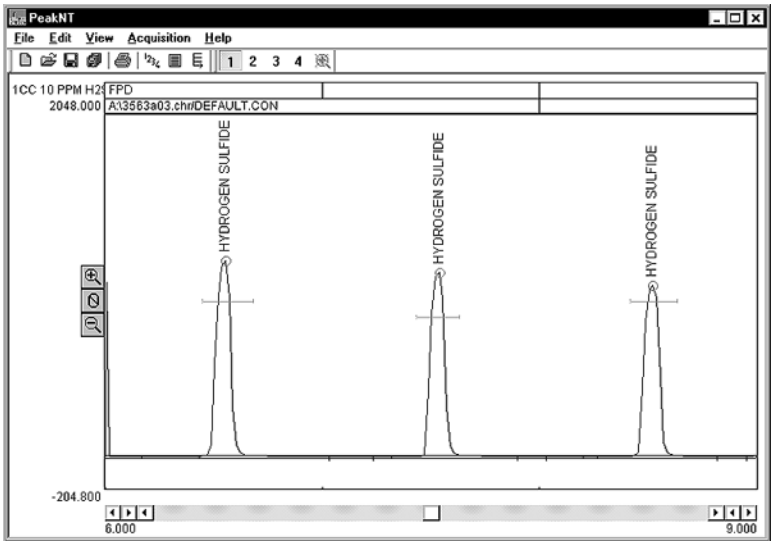
Sample: 1cc 10ppm H_2S in 3 replicate injections to demonstrate the consistency of the FPD response
Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FPD gain: HIGH; FPD temp: 150°C
FPD PMT volts: -400
FPD H_2: 60mL/min (30mL/min for each of the two hydrogen flows)
FPD air: 100mL/min

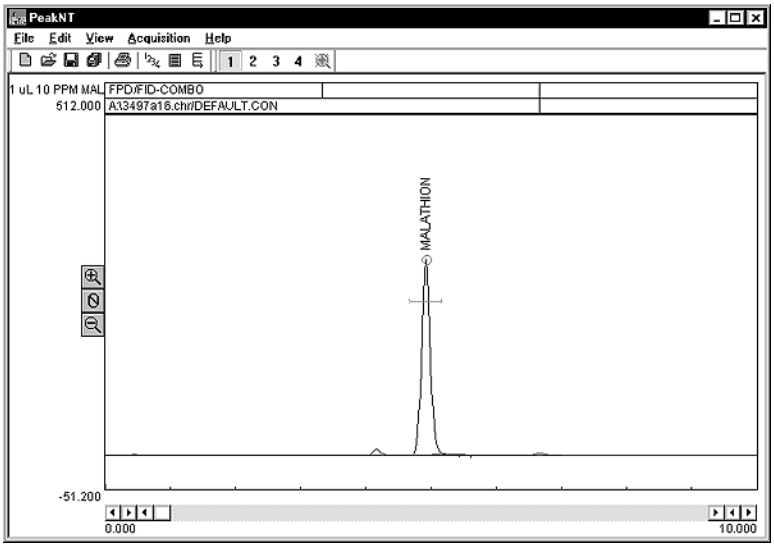
Temperature Program:

Initial	Hold	Ramp	Final
60°C	60.00	0.00	60°C

Results:

Component	Retention	Area
Hydrogen Sulfide	6.550	5216.6680
Hydrogen Sulfide	7.533	4828.8710
Hydrogen Sulfide	8.516	4810.5420
	Total	14856.0810





FPD Phosphorus

Sample: 1µL 10ppm malathion
Column: 15m MXT-1
Carrier: helium @ 10mL/min
FPD PMT volts: -400
FPD H_2: 30mL/min
FPD air: 100mL/min
FPD temp: 250°C

Temperature Program:

Initial	Hold	Ramp	Final
225°C	10.00	0.00	225°C

Results:

Component	Retention	Area
Malathion	4.916	2819.0520

DETECTORS
Flame Photometric Detector - FPD

General Operating Procedure

1. Set the hydrogen flow to 60mL/min. This correlates to a flow of 30mL/min each for the primary and secondary hydrogen. Set the air supply to 100mL/min. The air supply tubing is T'd inside the GC so that 10-30mL/min of air flows across the face of the PMT. Set the carrier gas flow between 5 and 20mL/min.

2. Use the switch on the GC's front control panel to light the FPD flame. Sometimes the flame is difficult to light because of the hydrogen-rich atmosphere inside the FPD detector body. If you are having difficulty lighting it, make sure the PMT voltage is OFF, then remove the cap on the FPD exhaust port and try the ignitor switch again. When the flame lights, there will be a loud noise like the backfiring of a car; this is normal and does not indicate a problem. KEEP YOUR FACE AWAY FROM THE DETECTOR WHILE LIGHTING THE FLAME; the loud noise is accompanied by a flash of flame. Replace the exhaust cap nut after lighting the flame.

3. Switch on the PMT voltage and set it to 400 by using the provided flat blade screwdriver to adjust the trimpot setpoint on the top edge of the GC's front control panel (vertically labeled "PMT VOLTS" under "DETECTOR PARAMETERS").

4. Set the FPD temperature to 150°C by adjusting the appropriate trimpot setpoint. Set the FPD gain to HIGH. Allow the FPD signal to stabilize, then inject the sample.

Optimizing Sensitivity

To optimize your FPD detector's sensitivity, inject the same sample at varying air and hydrogen pressures and observe the fluctuations in sensitivity.

1. Inject sample and observe the FPD response.

2. Turn the air up a tiny bit, less than 1psi, inject sample and observe the FPD response again. If you see an improvement in sensitivity, adjust the air up a little more, inject again and observe the response. Keep adjusting the air pressure up until sensitivity drops again to find the window of optimum sensitivity.

3. If there is no improvement in sensitivity after turning the air up, turn the air down less than 1psi, inject sample and observe the FPD response. If you see an improvement in sensitivity, adjust the air down a little more, inject again and observe the response. Keep adjusting the air pressure down until sensitivity drops again to find the window of optimum sensitivity.

4. Now, turn the hydrogen pressure up 1-2psi and re-optimize the air. Repeat this until you've found the optimum air and hydrogen pressure settings.

Note:

When a large hydrocarbon peak elutes simultaneously with a target sulfur compound, the hydrocarbon peak will quench the sulfur response. For this reason, we recommend the addition of an FID collector electrode to the FPD detector body. The FID collector electrode will allow the operator to "see" the hydrocarbon peaks and their retention times, so that chromatographic separation can be optimized for the elution of sulfur compounds.

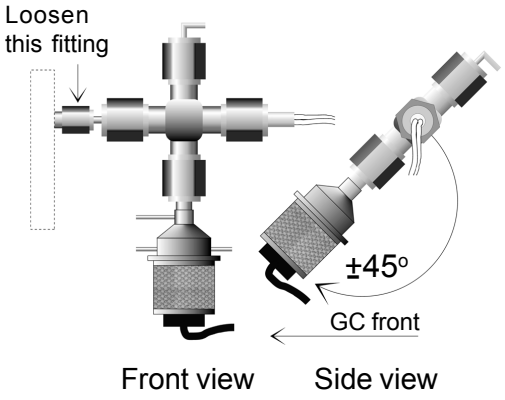
Switching Between Sulfur and Phophorus Modes

The bandpass filter specified when the instrument was ordered comes installed in the FPD detector assembly. There are two options for switching between sulfur and phophorus modes. You can purchase either an additional filter or an additional PMT housing and switch them as necessary. Phosphorus wavelength filters are available under part number 670-0083; sulfur filters are available under part number 670-0082. A PMT housing with a bandpass filter of the other optional wavelength specifications is available under part number 670-0084 (specify sulfur or phosphorus).

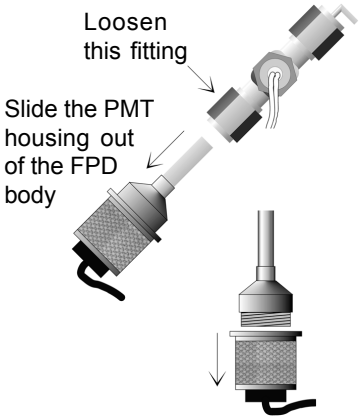
1. Turn OFF the GC power.

2. Unplug the BNC cable connecting the PMT to the amplifier. Disconnect the secondary hydrogen and both air supply lines at their bulkhead fittings on the GC deck.

3. Remove the heater block cover by unscrewing the philips head screw on top of it. Slide the cover up and off the block, and carefully remove the white insulation.

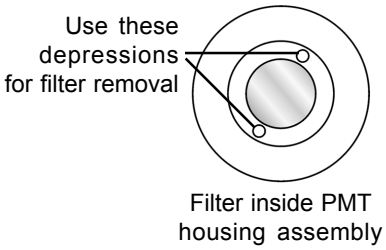


4. Loosen the 1/8" Swagelok fitting that secures the FPD detector assembly on the heater block enough to gently rotate the FPD assembly about 45° toward the front of the GC (see the Front View and Side View illustrations to the right).



5. Stabilize the FPD assembly while you loosen the 1/4" Swagelok fitting that secures the PMT housing to the FPD detector body. Slide the PMT housing out of the detector body. If you're switching PMT housing assemblies, set aside the PMT housing you just removed and skip to step number 7. If you're switching filters, proceed to the next step.

6. Unscrew the PMT housing stainless steel retaining nut, then set it (with the PMT and its socket) aside. The bandpass filter is screwed into the top part of the PMT housing against a black o-ring, and has 2 depressions in its frame. It can be unscrewed using open needle-nosed pliers but you must be very careful not to damage or scratch the filter. FPD sensitivity will be reduced if the filter is improperly installed or dirty.



6. Inspect the black o-ring for any nicks or cuts and replace it if necessary. Place the alternate filter, with its colored side facing down toward the PMT, into the top part of the PMT housing against the black o-ring and tighten it. Remember, it must be light-tight and gas-tight. Replace and tighten the stainless steel retaining nut with the PMT in its socket.

7. Slide the top of the PMT housing containing the alternate filter into the FPD detector body, and tighten the 1/4" fitting that secures it in place.

8. Gently rotate the FPD assembly back to its original angle, and tighten (with a wrench) the 1/8" fitting that secures it to the heater block. Reconnect the gas supply lines and the BNC cable. See the note on the following page regarding proper jet positioning.

Troubleshooting and Maintenance

Changing the Photomultiplier Tube (PMT)

The Photomultiplier Tube (PMT) is a consumable part, and will eventually need replacement. Additional PMTs are available under part number 670-0080.

1. Follow steps 1-4 on the ***"Switching Between Sulfur and Phophorus Modes"*** page.

2. Unscrew the stainless steel retaining nut and remove it, with the PMT and its socket, from the FPD assembly. Slide the retaining nut down the PMT amplifier lead to access the PMT.

3. Unplug the PMT from its socket. Remove the split Teflon™ ferrule and black o-ring from the PMT, inspect them for any damage, and replace them if necessary.

4. Slide the Teflon™ ferrule and black o-ring onto the new PMT. Plug the new PMT into the socket.

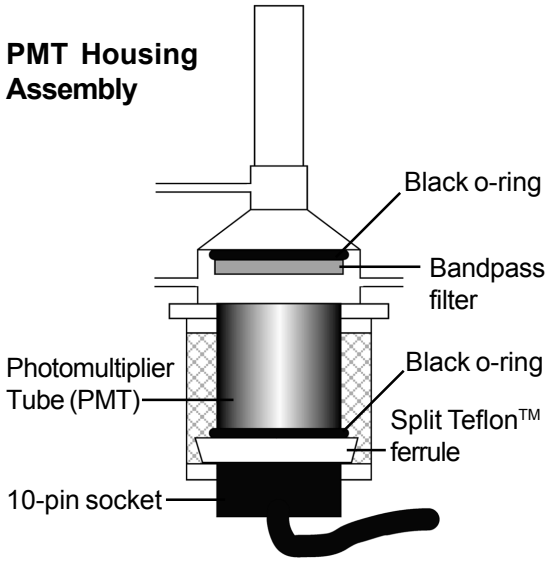
5. Slide the stainless steel retaining nut up and around the PMT, and screw it into place.

6. Gently rotate the FPD assembly back to its original angle, and hand tighten the 1/4" fitting that secures it.

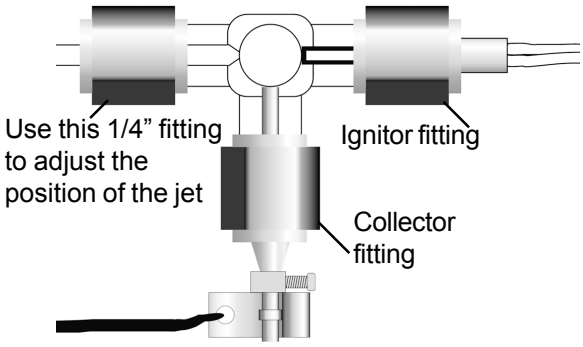
7. Reconnect the BNC cable and the gas supply lines.

Note: When your FPD detector was assembled at the factory, the ignitor and jet (and collector electrode, if present) were all properly positioned within the FPD body. It is advisable to familiarize yourself with their proper positioning in case they require adjustment. Remove the 1/4" Swagelok cap nut on the FPD exhaust port to see inside the detector body. Use a small flashlight to see the position of the jet inside the FPD detector body. The jet's tip should be flush with the cylindrical wall of the opening that you're looking through, and just barely visible from a slight angle. When looking straight down into the opening, you should not be able to see anything protruding into it. To adjust the jet, loosen or tighten the 1/4" Swagelok fitting to move the jet forward or backward. Keep in mind that if the jet actually protrudes into the PMT's line of sight, it could interfere with the FPD's performance. The ignitor should be similarly positioned across from the jet, with the tip of its blade just visible but not protruding into the FPD detector body chamber. To adjust the position of the ignitor, loosen the 1/4" fitting enough to move the ignitor forward or backward as necessary. Tighten the fitting when the ignitor is properly positioned. If there is an FID collector electrode installed on the FPD detector assembly, it must be positioned in the same manner.

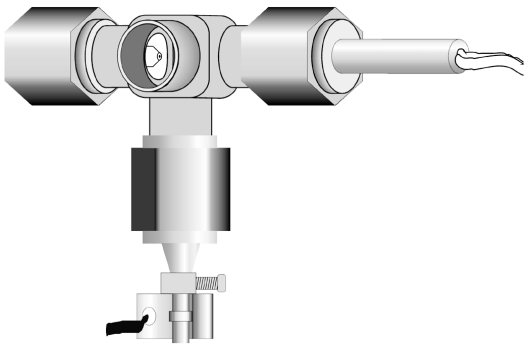
PMT Housing Assembly



Top view of FPD with exhaust cap nut removed



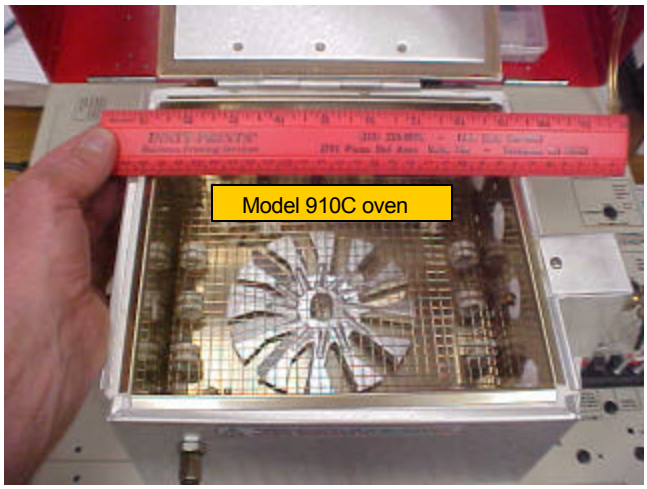
An angled view reveals the tip of the jet



Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

Buck Gas Chromatographs are designed to use both packed and capillary columns. The Model 910 has an oven which allows for columns coiled on a 7" (17.5 cm) diameter or smaller, while the Model 310 GC (which has a smaller oven) can fit columns coiled on a 5" diameter or smaller. The column installation procedure is identical on either GC since both GCs use the same injector and detector hardware. Only the oven size is different.

The most common type of column used with Buck GCs is the MXT type. This is a fused silica lined stainless steel capillary column which is very durable and easy to use. It is available in many different coil diameters, lengths and tubing diameters. Buck typically suggests using the .53mm internal diameter tubing size (sometimes called a "megabore" or "wide bore" capillary column. We normally have the columns coiled to a 3.5" (9cm) diameter so they fit easily in either the 910C or 310 column oven.


Model 910C oven


Model 310 oven

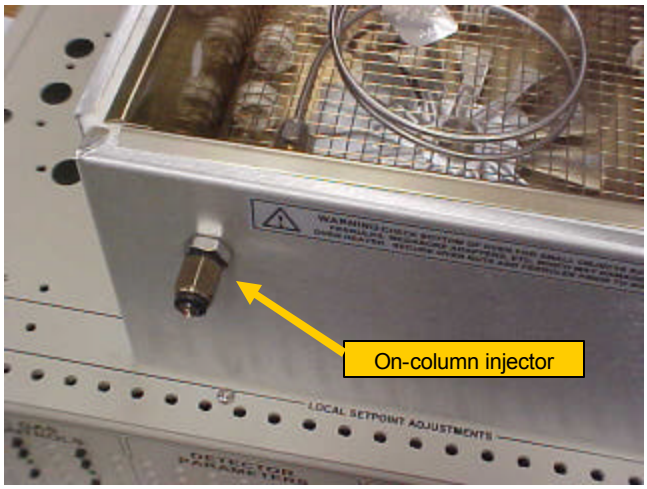
.53mm wide bore MXT metal capillary on 3.5" bundle

1/8" Packed Column

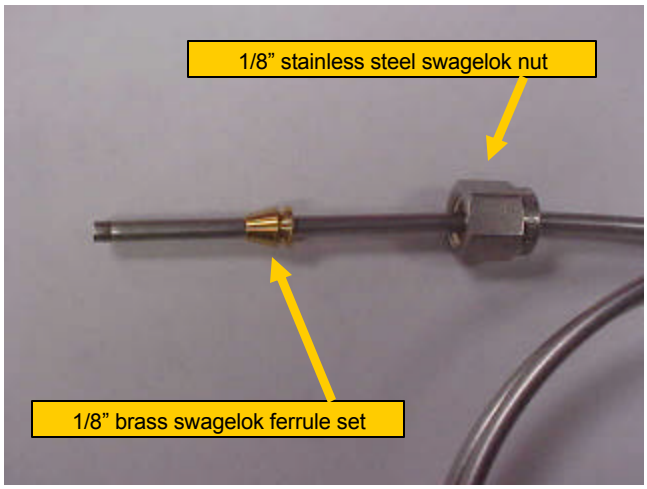
.25mm capillary on 7" cage

Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

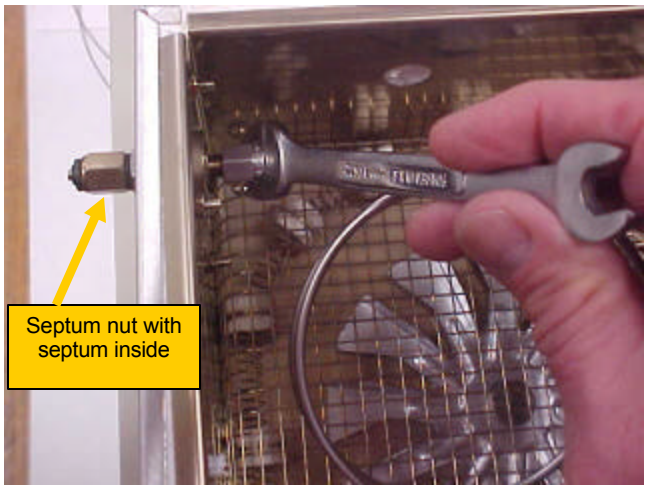
Buck GCs can be factory equipped with several types of injectors. The most common is the "on-column" injector which is suitable for .53mm capillary, 1/8" packed and (in some cases) narrow-bore capillary columns (where the sample does not require splitting).



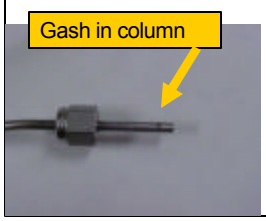
To install a 1/8" packed column in the on-column injector, slide a 1/8" swagelok nut and brass ferrule set (front and back ferrule) on one end of the column. We like to use a stainless steel nut becase at temperatures over 200C a brass nut discolors and starts to become very soft. We like brass ferrules because they seal better than stainless ferrules especially the second of third time they are removed and re-tightened.



Slide the column into the on-column injector from the oven side until it bumps up against the septum. Pull the column back about 1 centimeter and tighten the nuts and ferrules using a 7/32" wrench. If you tighten the column with the end of the column touching the septum, this could stop the carrier gas flow since the septum would seal the end of the column.



Some packed columns purchased from Buck have a gash near one end of the column specifically to provide a path for the carrier gas in the event it is installed with the end of the column jammed up against the septum



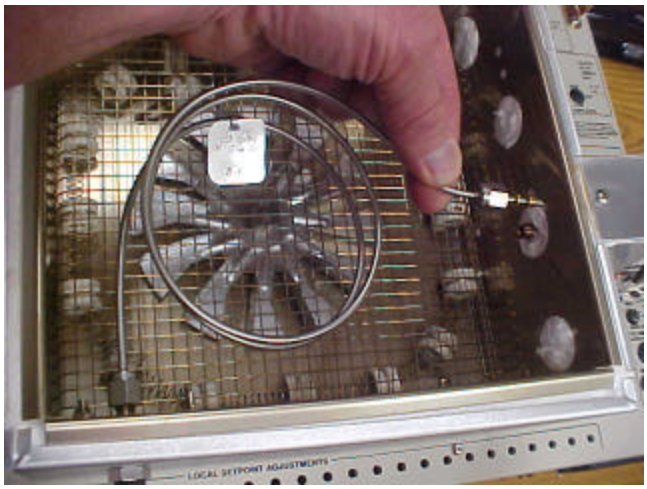
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

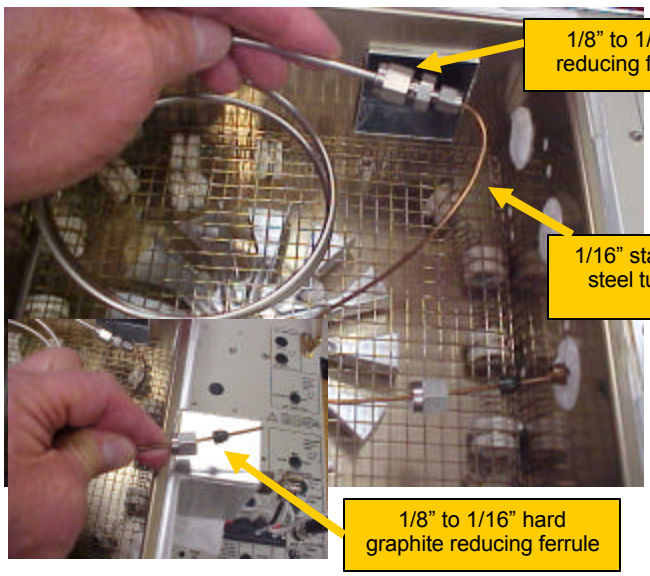
Bend the column gently so it lines up with the detector bulkhead fitting. Attach a stainless swagelok nut and brass ferrule set to the end of the column. It's a

good idea to first tighten the nut and ferrule into a standard swagelok fitting so the ferrule is positioned at a standard depth from the end of the column tubing.

Then tighten the end of the column into the detector bulkhead fitting using a 7/32" wrench. The detector shown in the photo is an FID detector mounted in detector location #2 along the right hand side of the column oven. Other available detectors may appear slightly different or be mounted in positions #1, #3 or #4 along the right hand side of the column oven but all detectors will have 1/8" swagelok connectors to attach the column to.

Some packed columns may be made of glass or may be packed with fragile packing material and it is not possible to bend the column. In this case, attach some empty tubing to the end of the column and bend the empty tubing to connect to the detector. 1/16" stainless tubing is ideal for this. Use a hard graphite reducing ferrule to connect the 1/16" tubing to the 1/8" detector bulkhead.



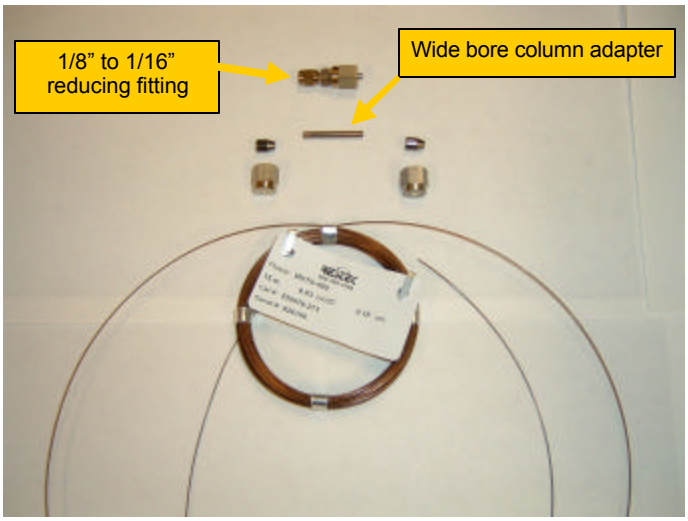


Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

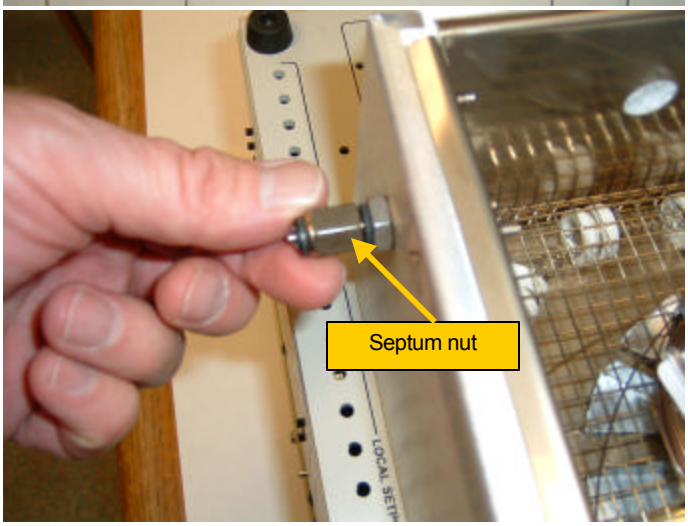
To install a .53mm capillary column in the on-column injector you will need two 1/8” swagelok nuts, two 1/8” to .8mm soft graphite reducing ferrules, the Buck wide bore column adapter, a triangle file to cut the column and a 7/32” wrench.

You have the option of using a swagelok 1/16” to 1/8” reducing fitting and 1/16” ferrule instead.

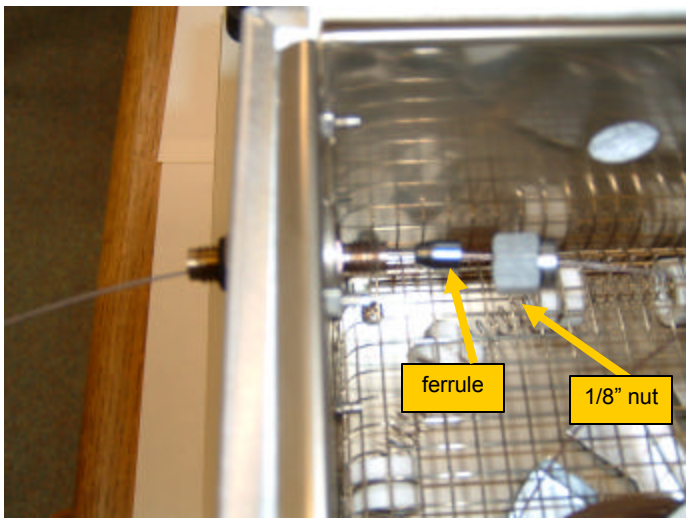
You can use MXT type metal (strongly preferred) capillary columns as shown, or regular fused silica columns. The regular fused silica columns are more likely to break especially in the injector due to the syringe fracturing the fragile fused silica.



Remove the septum nut from the outside of the oven. Now is a good time to inspect and change the septum if it is worn.

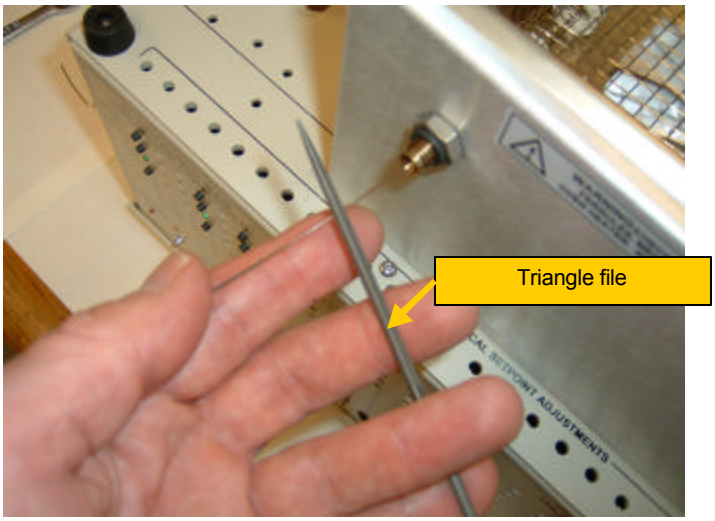


Slide a 1/8” stainless steel swagelok nut and 1/8” to .8mm soft graphite reducing ferrule onto the column. Then slide the column through the injector so it pokes out the front of the oven.



Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

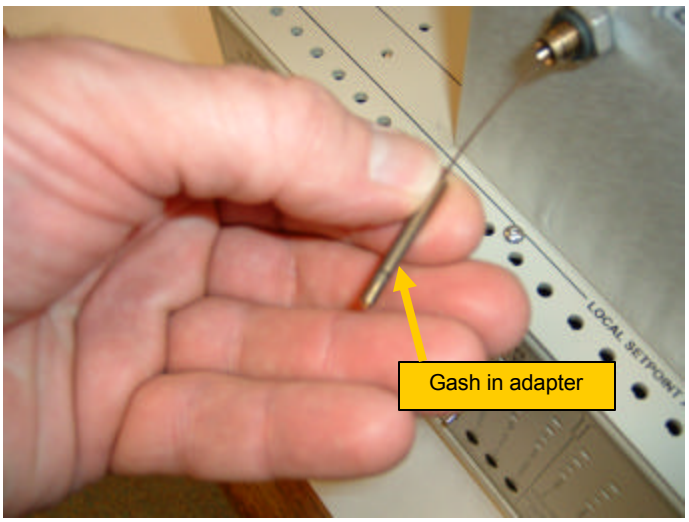
Use a sharp triangle file to cut a few centimeters from the end of the MXT column. It is a good idea to ALWAYS trim the end of the column anytime you run it through a graphite ferrule as the sharp edge of the column can shave graphite off into the bore of the column where it can cause the peaks to tail.
Support the column with your fingertips and drag the file firmly across the tubing in one swift motion.



Place your fingernail under the file cut and bend the column. It will break cleanly at the file cut.

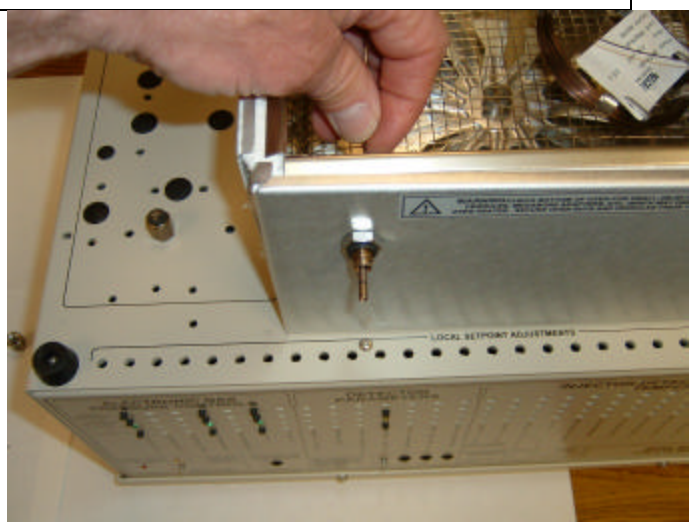
Slide the wide bore adapter onto the column and position the end of the column about halfway in the adapter.

Notice the adapter has a gash at one end. The end with the gash should face out towards the operator.



Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

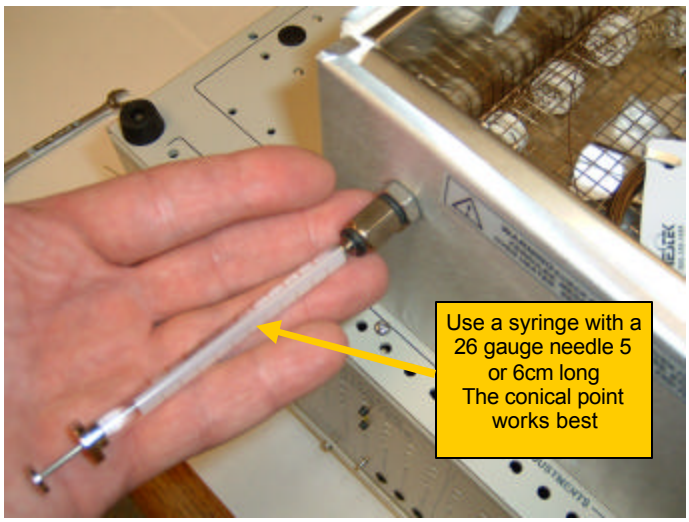
Pull the adapter back into the on-column injector.



Tighten the nut and ferrule securely so that the column will not move even if you pull on it with your fingers. Do not over-tighten the ferrule un-necessarily. You want to feel the ferrule compress a little bit but not tighten so much that the ferrule becomes distorted.

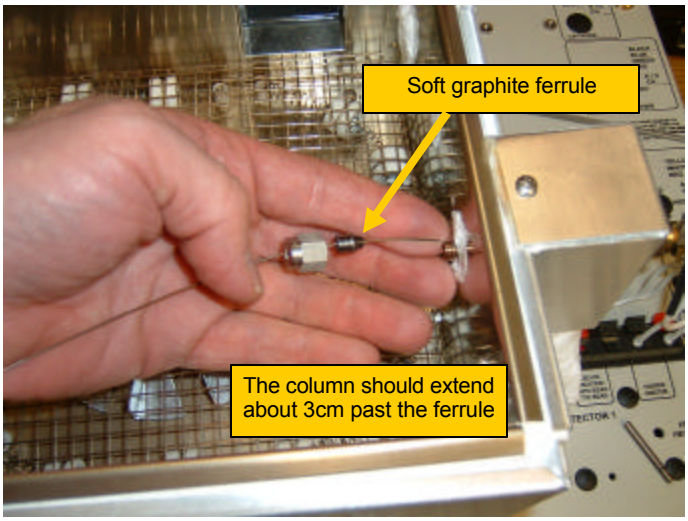
Replace the septum nut. Be careful not to over-tighten the septum nut. If the septum is too tightly compressed, the syringe needle will make a hole in the septum. If the septum is lightly compressed, the silicone septum cleaves and re-seals permitting hundreds of injections before failure and will not clog the syringe or column with septum particles.

Insert an empty syringe through the septum and onto the column to verify that the syringe slides smoothly into the column without catching or snagging. If it feels like the syringe needle catches on the end of the column, then reposition the column 1 cm closer to the center of the oven.

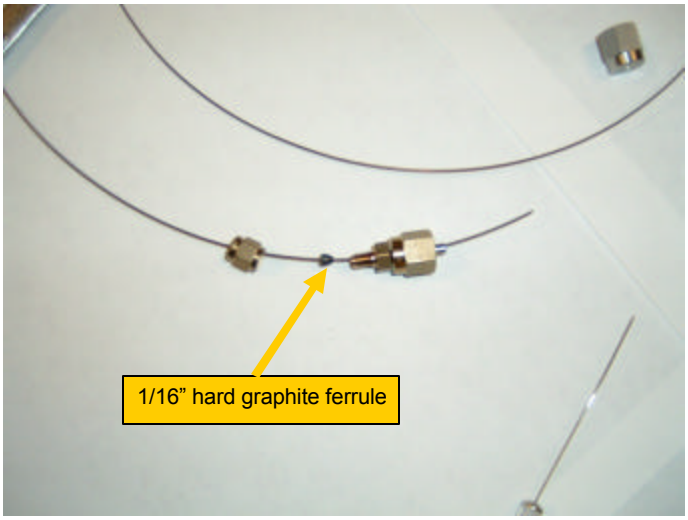


Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

Connect the other end of the column to the detector. The simplest way is to use a 1/8" swagelok nut and a second 1/8" to .8mm soft graphite reducing ferrule. The detector shown in the photo is the FID detector. The end of the column should be positioned about 3cm past the ferrule or about halfway from the inlet fitting to the tip of the FID jet.



Because the soft graphite ferrule can sometimes outgas contaminants which contribute to baseline bleed at high temperatures, we sometimes prefer to connect the column to the detector using a swagelok reducing fitting and a 1/16" hard graphite ferrule. The hard graphite ferrule does not release as much contaminant and it is physically much smaller as well so the baseline shift due to temperature is sometimes much less using this type of connector.



Position the end of the column about halfway between the inlet fitting and the tip of the jet and then tighten the fitting.

Note:
Each type of detector (PID, ECD, NPD ,TCD etc) may require a different position for the end of the column. Consult the detector instructions for positioning details.

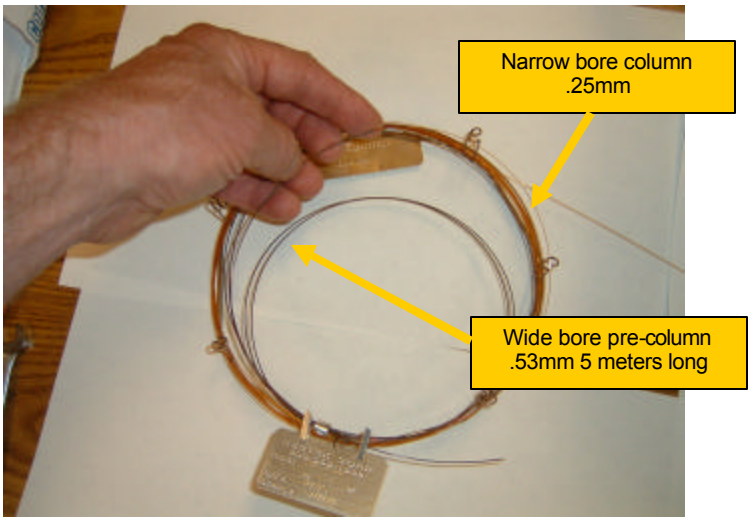
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the on-column injector

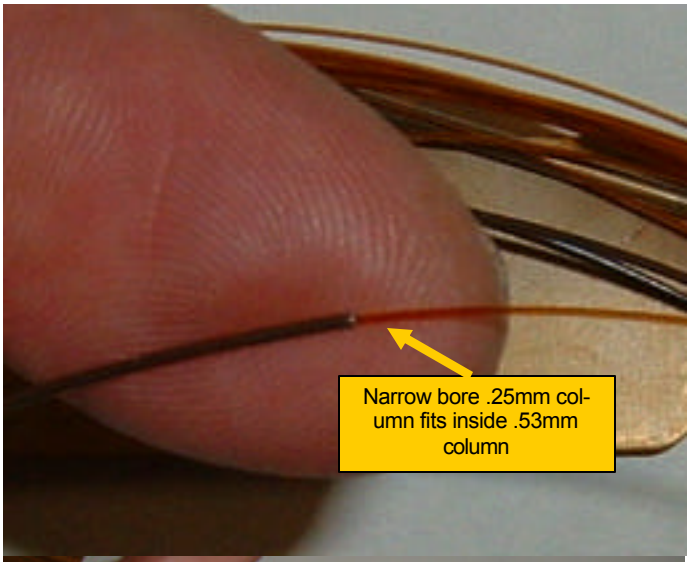
To install a narrow-bore capillary column in the on-column injection port, connect the narrow-bore to a 5 meter length of .53mm MXT pre-column. The pre-column is de-activated MXT tubing but there is no phase coated on the inside of the tubing like a normal column, so analytes are not retained.

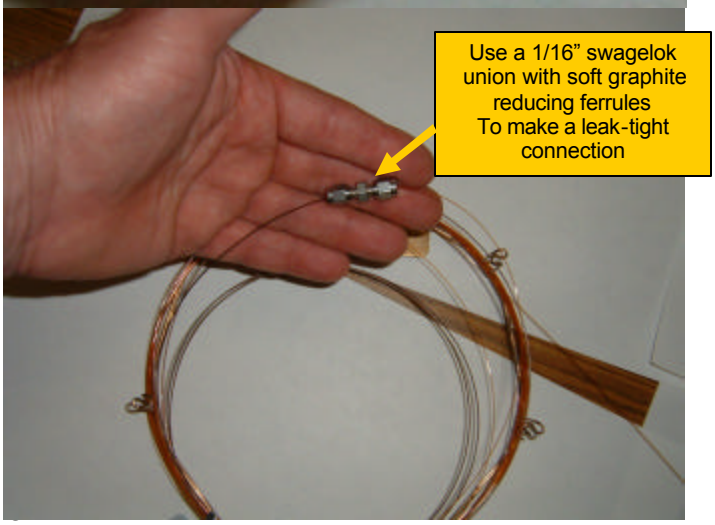
The narrow-bore column fits inside the .53 for a low dead volume connection. Make the connection leak-tight by securing the connected columns with a 1/16" swagelok union and soft graphite reducing ferrules.

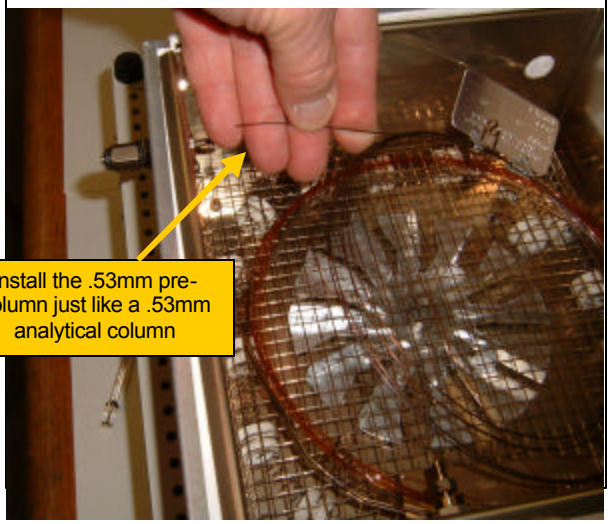
Then install the .53mm pre-column in the on-column injector just as if it were a .53mm analytical column.

Connect the narrow-bore column to the detector using the appropriate hard graphite reducing ferrule.





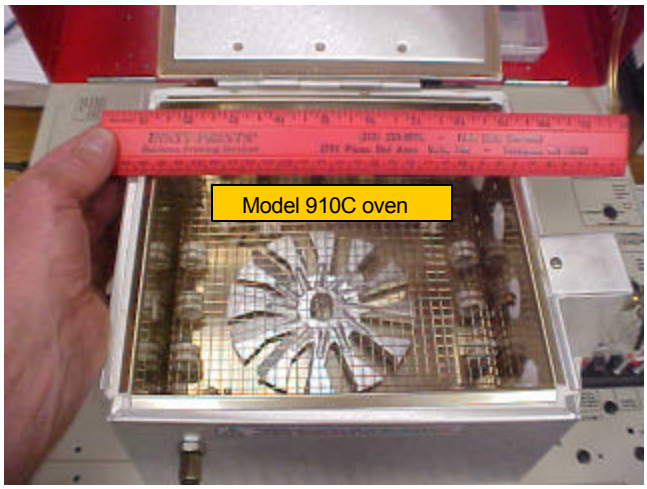




Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

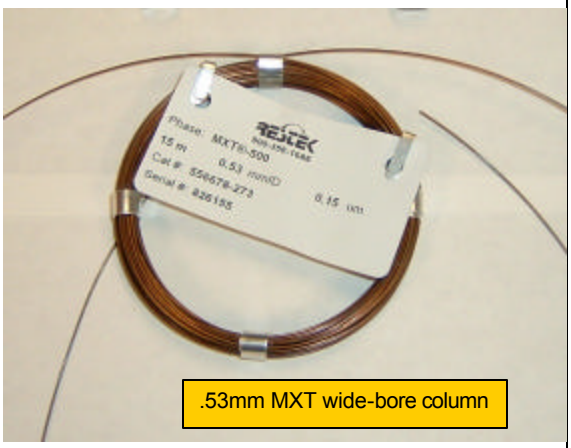
Buck Gas Chromatographs are designed to use both packed and capillary columns. The Model 910 has an oven which allows for columns coiled on a 7" (17.5 cm) diameter or smaller, while the Model 310 GC (which has a smaller oven) can fit columns coiled on a 5" diameter or smaller. The column installation procedure is identical on either GC since both GCs use the same injector and detector hardware. Only the oven size is different.

The split/splitless injector can be used with both wide-bore and narrow-bore capillary columns.

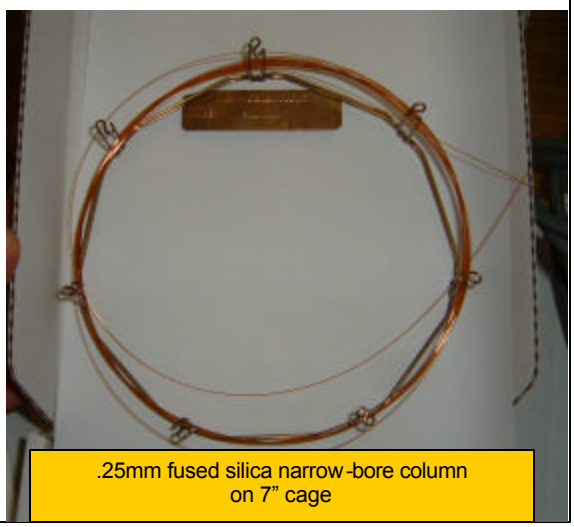


Model 910C oven

Model 310 oven



.53mm MXT wide-bore column



.25mm fused silica narrow-bore column on 7" cage

.25mm narrow -bore on 5" cage

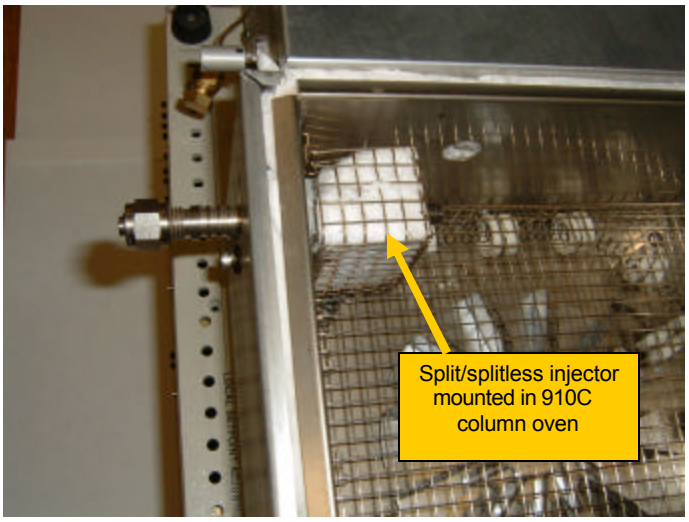
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

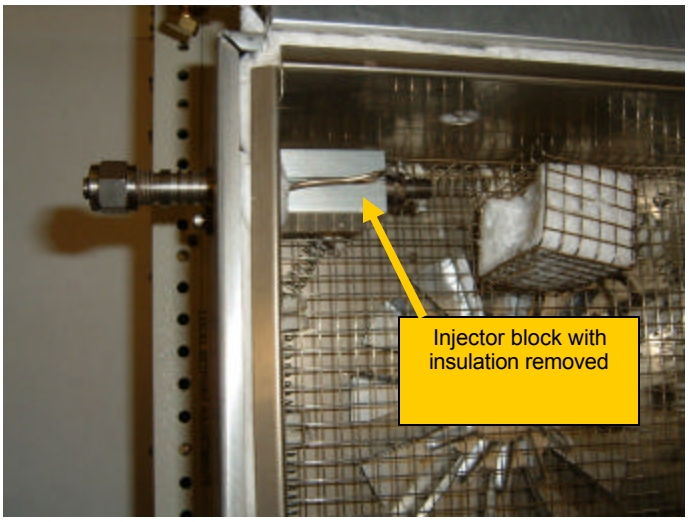
The split/splitless injector can be used for on-column injections onto .53mm capillary columns, split injections onto any column, or splitless injections.

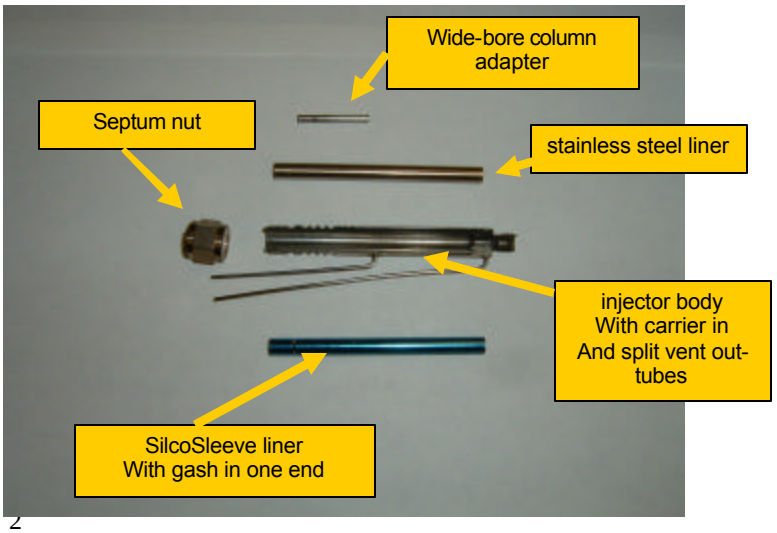
The injector has its own temperature control which is set from the front panel using the screwdriver provided with the GC. Set the temperature depending on the needs of the analysis.

The injector is supplied with both stainless steel and SilcoSleeve liners. The stainless steel liner supports the wide-bore adapter for use with .53mm column and on-column injections.

The SilcoSleeve liner is more inert (since it is lined with fused silica) and is used with narrow-bore columns in the split or splitless modes.





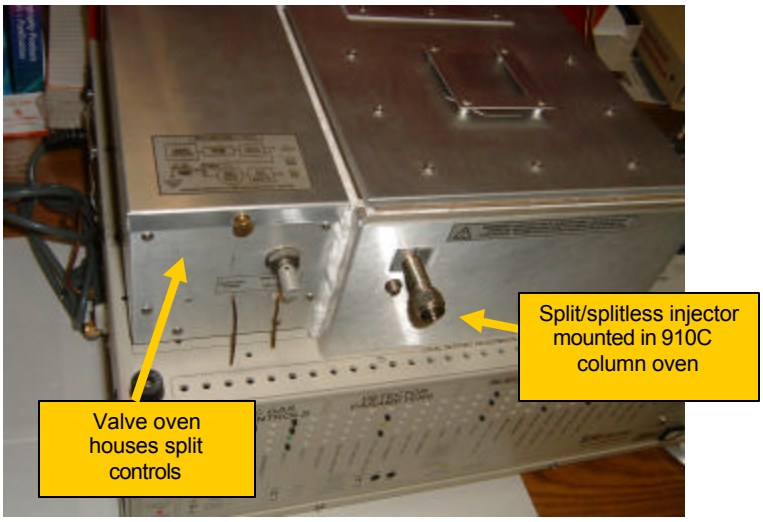


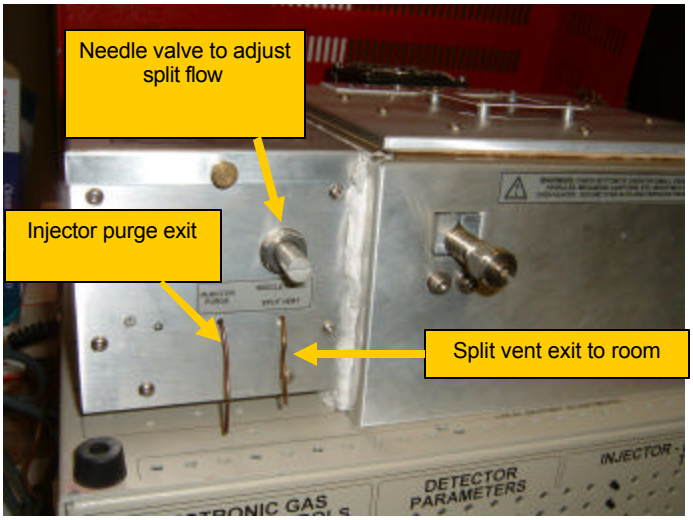
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

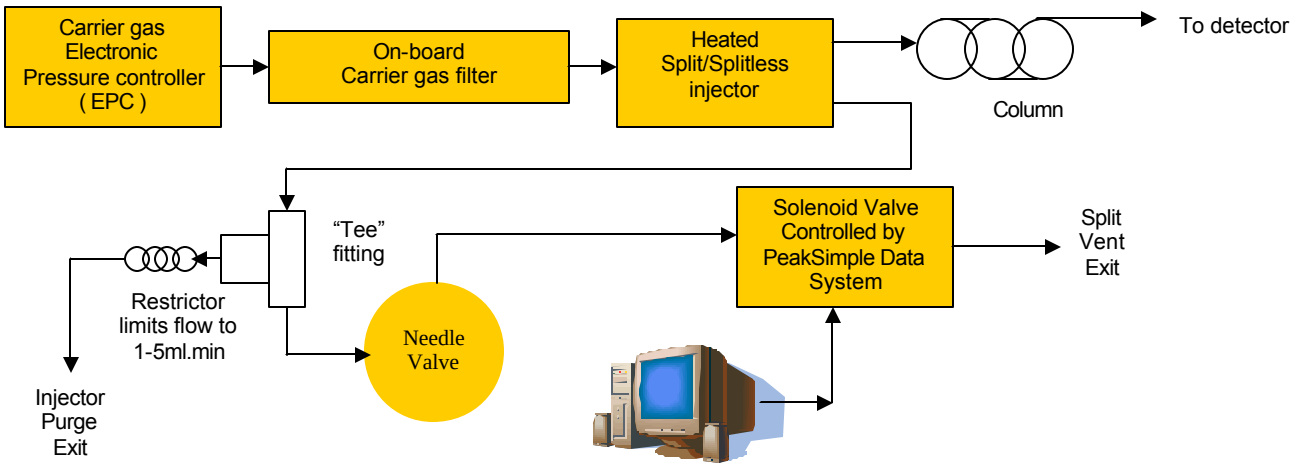
On the Model 910C GC, the needle valve, restrictor and “tee” fitting are mounted in the valve oven to the left of the column oven. The needle valve is temperature controlled to avoid solvent condensation.

The needle valve in the Model 310 GC is mounted in the GC chassis but is not temperature controlled.

As shown in the schematic diagram below, the injector vent line goes to a “tee” fitting where it splits into a restrictor tube which limits the flow to a few milliliters per minute and to a PeakSimple controlled solenoid valve which turns the main split flow on and off. The main split flow is controlled by a needle valve. The flow through the restrictor ensures that high boiling molecules from previous injections can not diffuse back into the injector body.







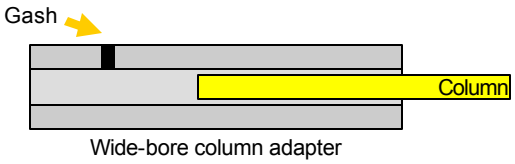
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

To install a .53mm MXT type wide-bore capillary column in the "on-column" mode:

1) Slide a 1/8" swagelok nut and 1/8" to .8mm soft graphite reducing ferrule over the end of the column. Then slide the column through the split/splitless injector so the column projects out the front of the oven.

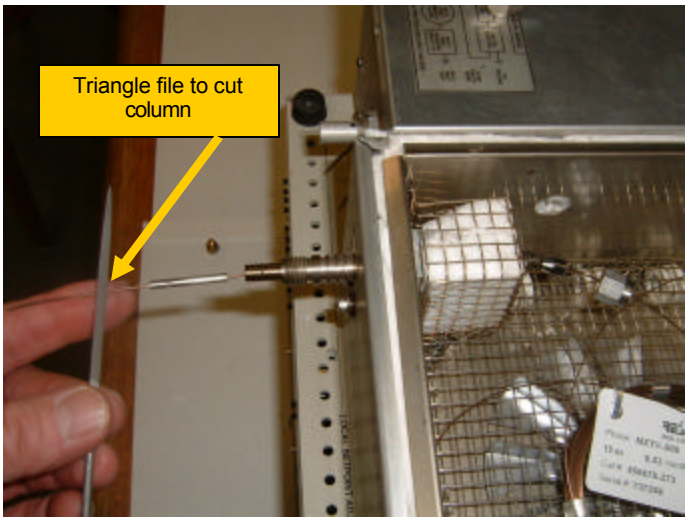
2) Slide the stainless liner and wide-bore adapter on the column, then cut 3 cm off the column using a triangle file. *Always trim the column after passing it though a graphite ferrule as the column's sharp edge may shave graphite particles into the bore of the column causing tailing peaks.*

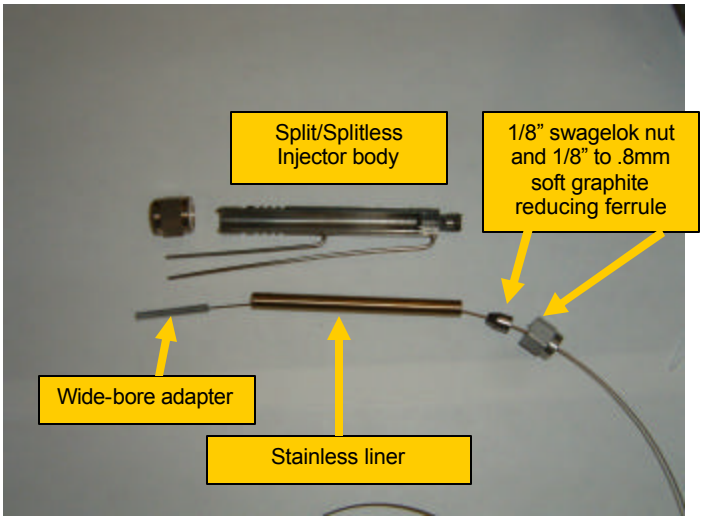
3) Pull the column back into the injector so the end of the .53 column is positioned about midway in the adapter.

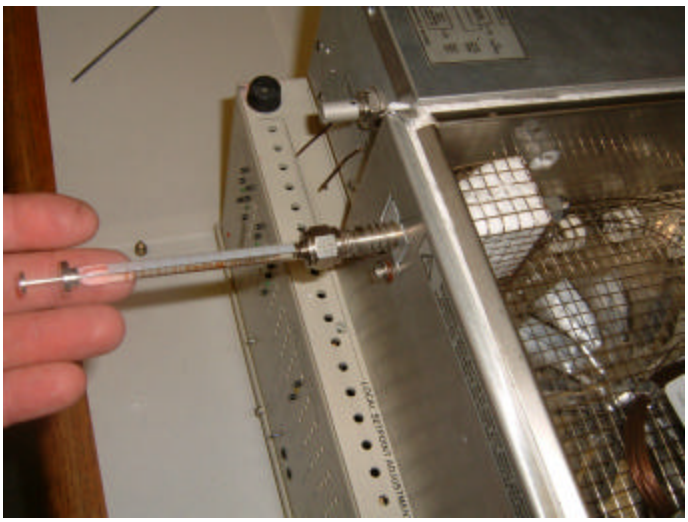


4) Tighten the nut and graphite ferrule to hold the column securely. The column should not move when you pull on the column tubing from the oven side

5) Test the installation by inserting a dry 26 gauge syringe with a 5 or 6cm long needle onto the column. The syringe should glide into the bore of the column itself without snagging or catching. If the syringe snags the lip of the column then re-position the column about 1 cm closer to the center of the oven. This will give the syringe a smoother entrance.

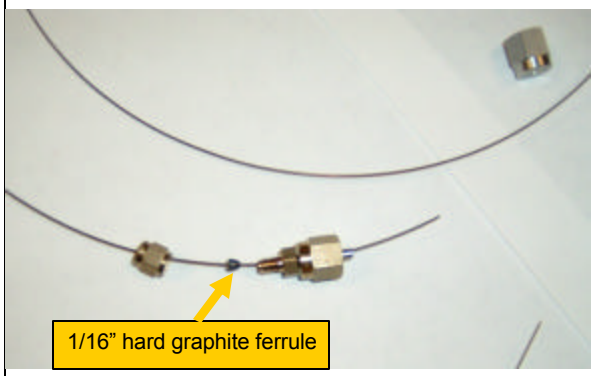






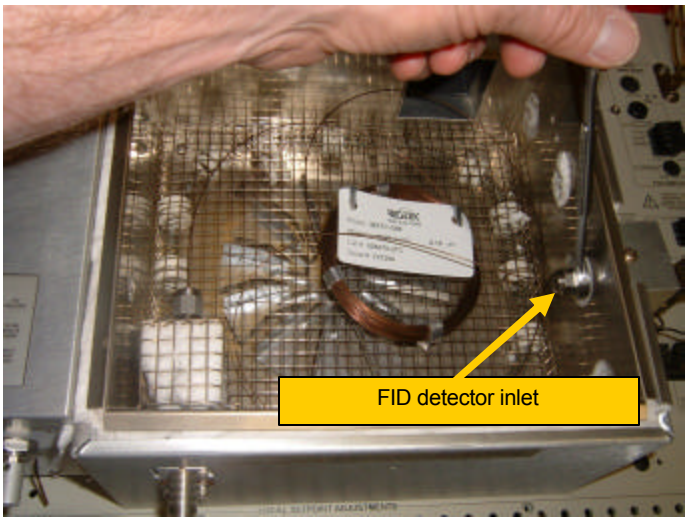
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

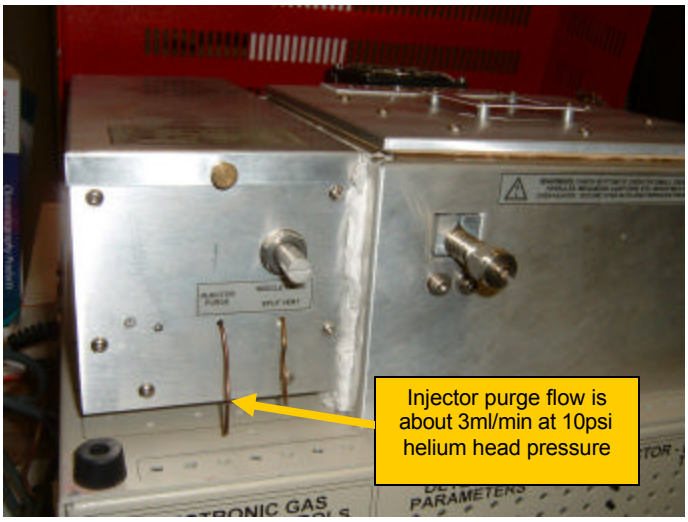
Connect the column to the detector using a 1/8” swagelok nut and 1/8” soft graphite ferrule or optionally a swagelok reducing fitting and 1/16” hard graphite ferrule.

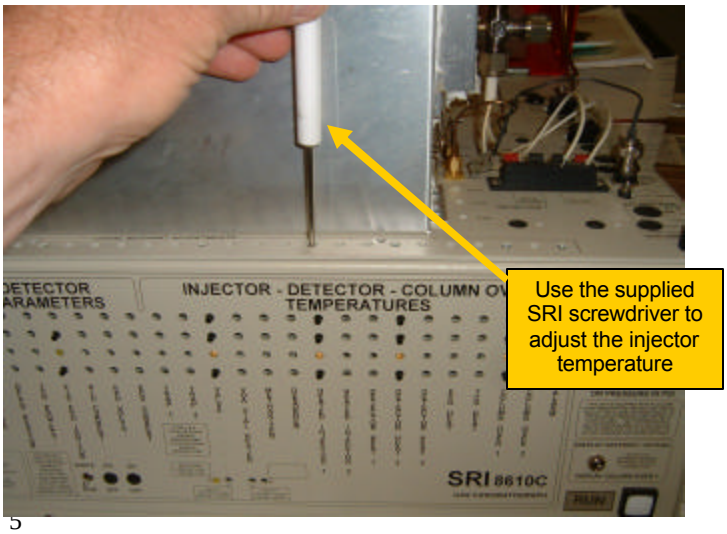


The needle valve is normally adjusted fully clockwise (off) since there is no split during an on-column injection, and the split solenoid is not actuated from PeakSimple. There will be some injector purge flow continuously from the injector purge exit tube (typically 3ml/min at 10psi head pressure), but this does not affect the on-column injection since the sample is deposited in the bore of the column by the syringe.

Set the injector temperature to about the boiling point of the analytes. Don’t set the temperature higher than required since this shortens the septum life, and makes it harder for the column oven to cool down close to ambient temperatures.







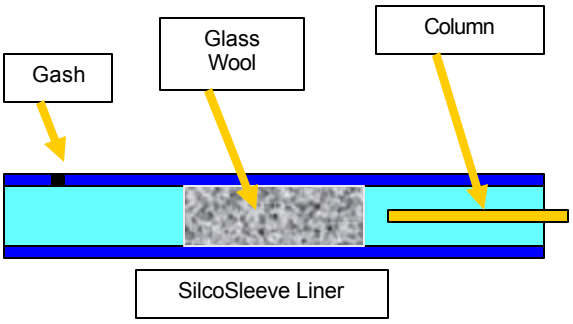
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

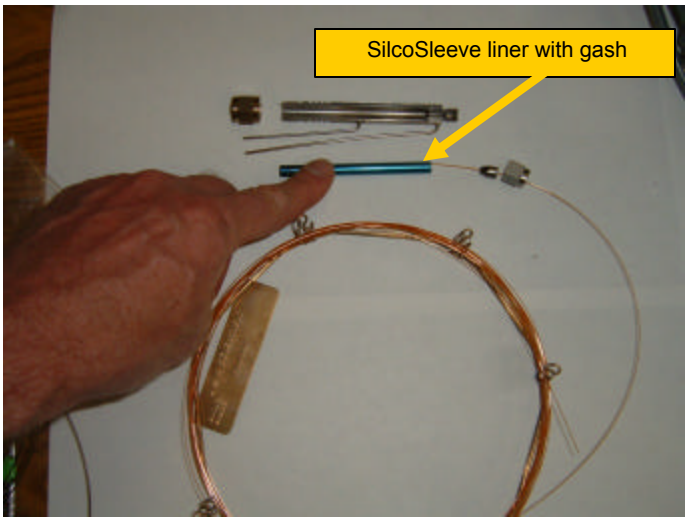
To connect a narrow bore column to the split\splitless injector:

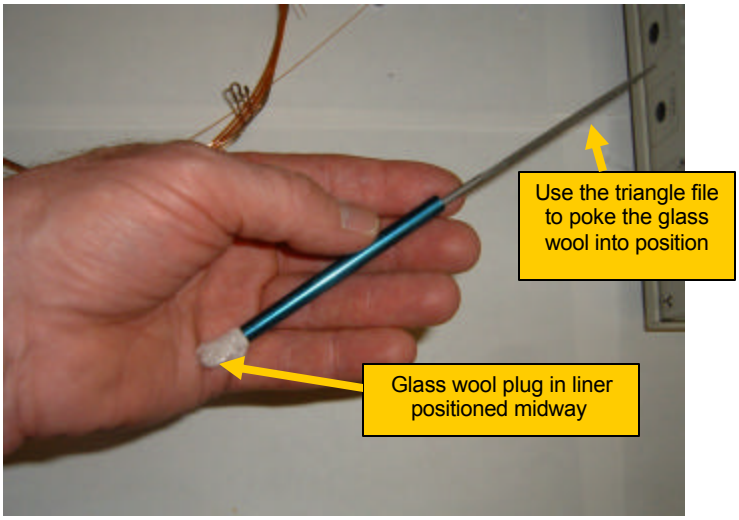
1) Locate the SilcoSleeve liner with a gash at one end. The GC is shipped with the SilcoSleeve liner in the parts box under the red lid. The regular stainless liner can also be used, but it is not as inert as the fused silica lined SilcoSleeve.

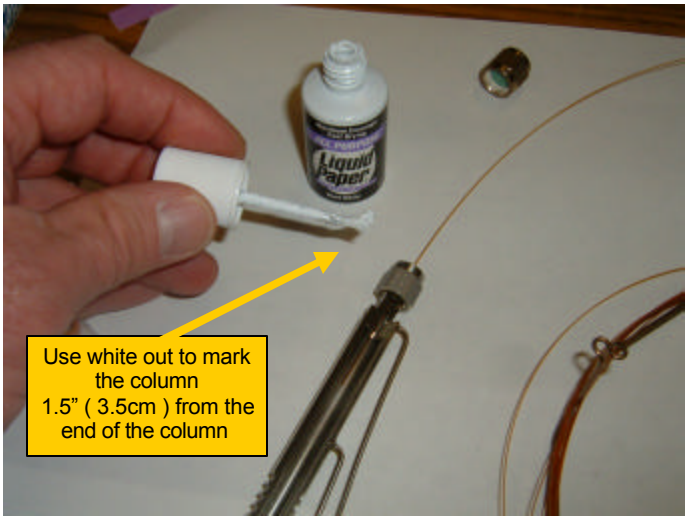
2) Verify that the liner has a plug of glass wool positioned midway. The glass wool gives the sample a surface from which to evaporate, so the syringe tip should deposit the sample directly into the plug of glass wool.

3) Mark the column with White-Out about 3.5 cm (1.5") from the end to position it in the liner just downstream of the glass wool plug.









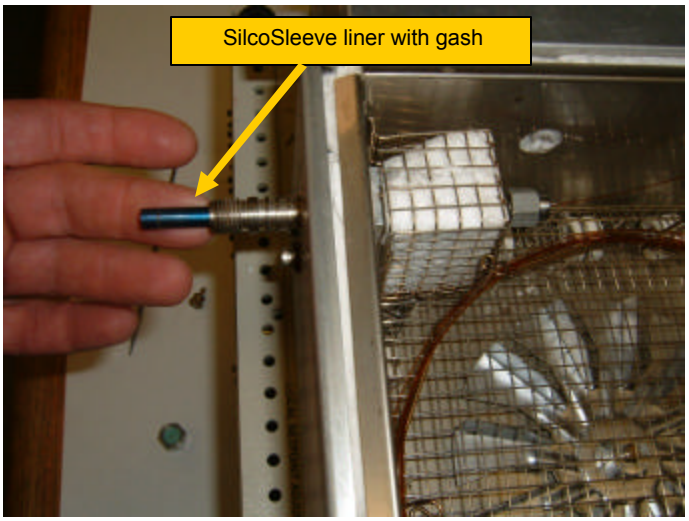
Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

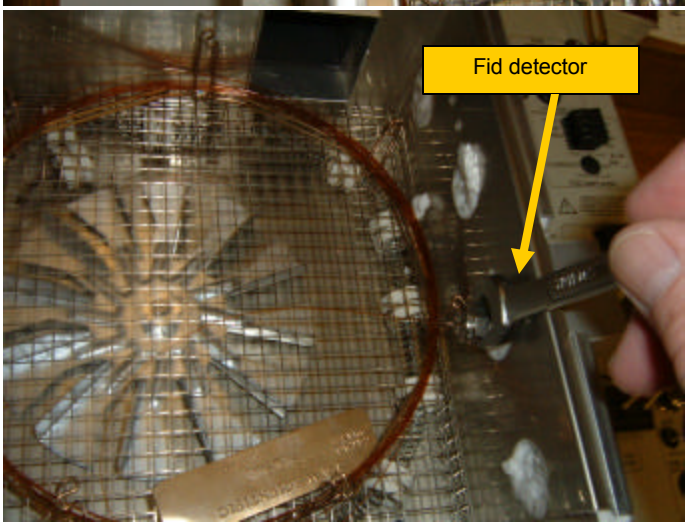
Place the liner in the injector with the gash side towards the operator. The gash allows carrier gas to flow though the liner. Without a gash, the carrier gas will flow around the liner instead of through it. Secure the column in place (so you can see the dab of White-Out just past the end of the 1/8" swagelok nut) using a swagelok nut and 1/8" to .4mm soft graphite reducing ferrule.

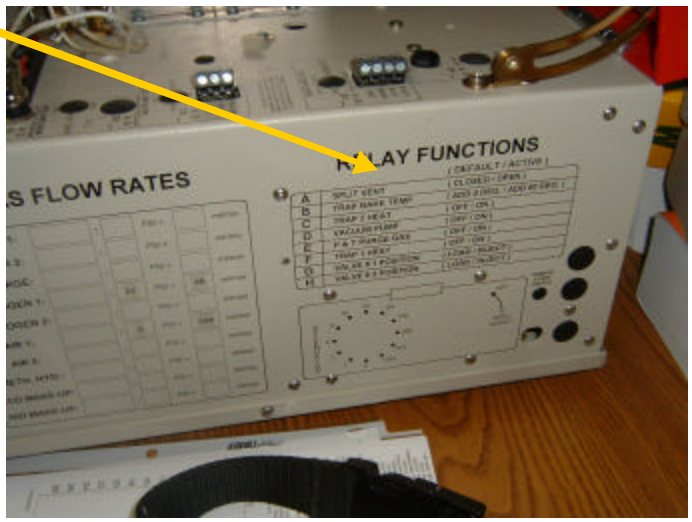
Connect the other end of the column to the detector using a 1/8" soft graphite ferrule or a fitting and a hard graphite 1/16" to .4mm reducing ferrule.

As shown on the right side of the GC, Relay A (typically) controls the split vent solenoid. *On some GCs, the split vent solenoid may be controlled by a different relay (B-H).*

In PeakSimple set the times you want the split vent to open and close. This will depend on whether you want a split or a splitless injection.

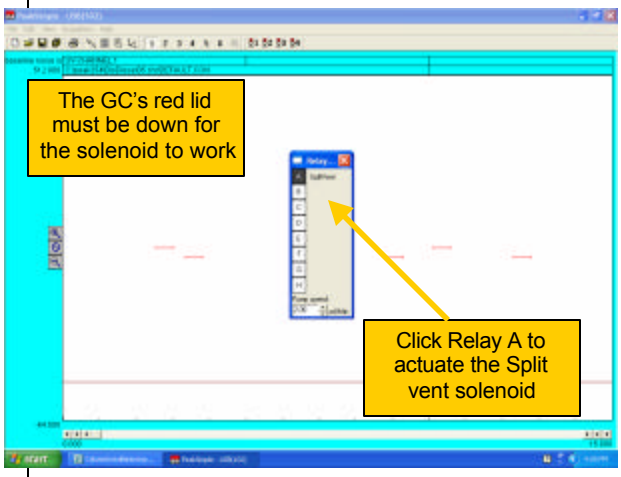


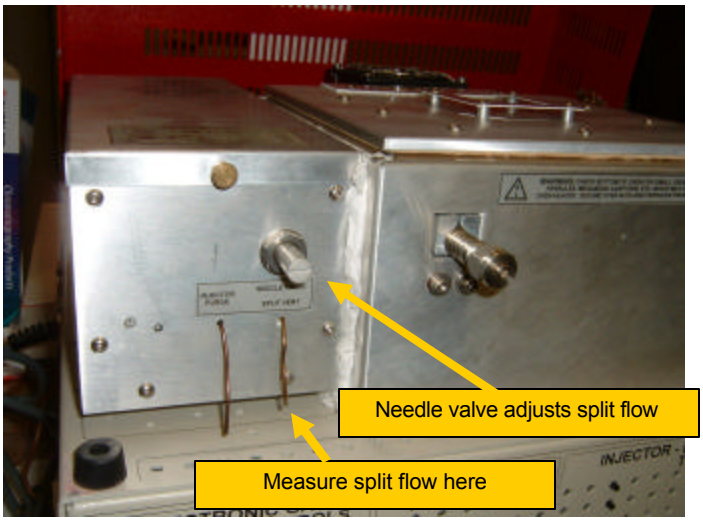


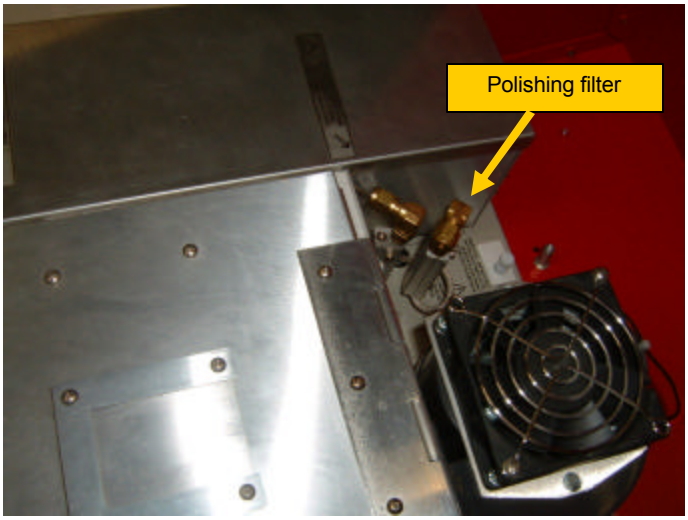


Column Installation in the Buck Model 910 and Model 310 Gas Chromatographs equipped with the split/splitless injector

For a split injection, measure the column flow at the end of the column. For a .25mm narrow-bore column this will be about 1 ml/minute. Measure the flow exiting the split vent tube with the split vent solenoid activated. The column flow divided by the split vent flow is the split ratio.

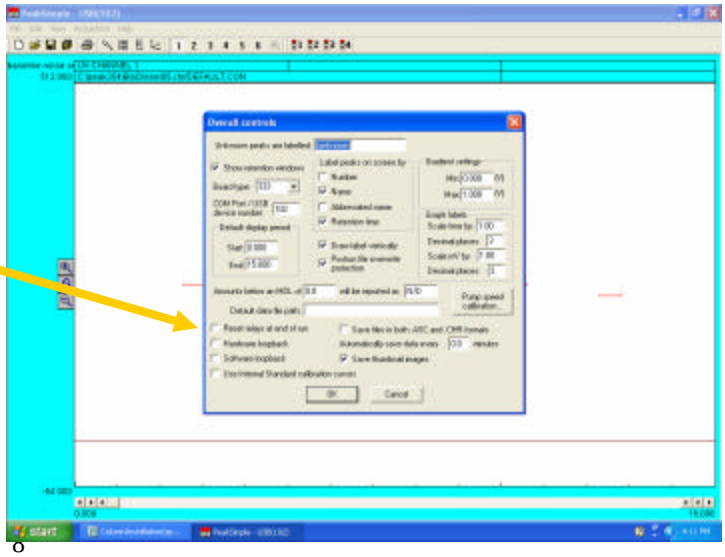






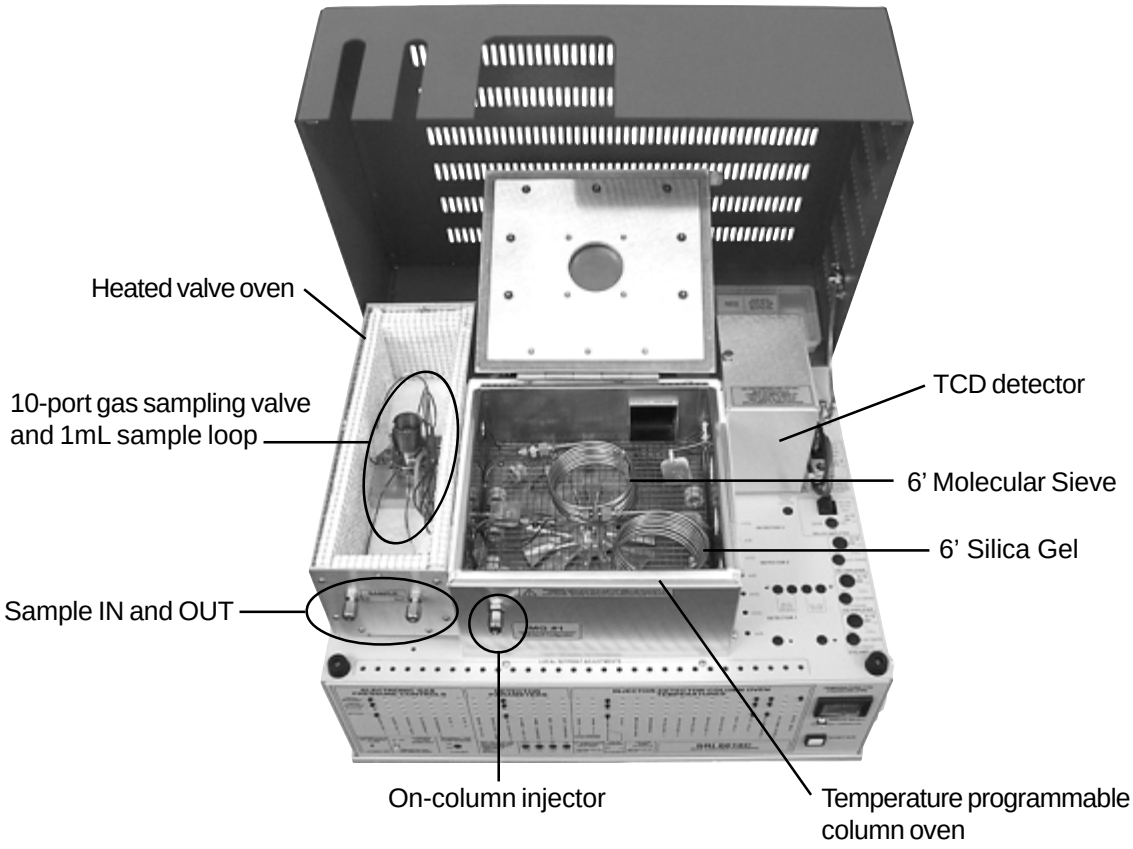
If the split ratio must be very large (more than about 100 to 1), you may have to remove the polishing filter. The polishing filter is located behind the column oven. The molecular sieve filled tube may limit the maximum split flow because it acts like a flow restrictor. It can easily be removed and replaced with a blank tube for higher split ratios.

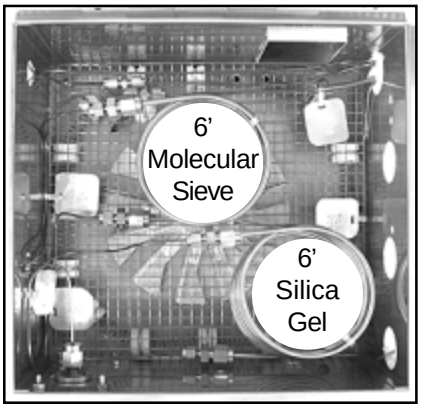
If you want the split vent to be open before the injection and to stay open, uncheck the box in the Edit/Overall screen labeled "reset relays at end of run".
If you want the split to shut off during the run, actuate Relay A before injecting. Then close the solenoid during the run by entering Relay A off in PeakSimple's event table.



System Overview

Your Buck Multiple Gas Analyzer #1 (MG#1) GC is pre-plumbed and ready to resolve H_2, O_2, N_2, Methane, CO, Ethane, CO_2, Ethylene, NOx, Acetylene, Propane, Butanes, Pentanes, and C_6 through C_8. The basic version of the MG#1 has a TCD detector An HID detector or an FID with the integrated Methanizer may be added.





The MG#1 allows you obtain complete analyses of the fixed and natural gases listed above with a single injection. The MG#1 achieves this using a 10-port gas sampling valve with a 1mL sample loop in the heated valve oven, and two columns in the temperature programmable column oven.

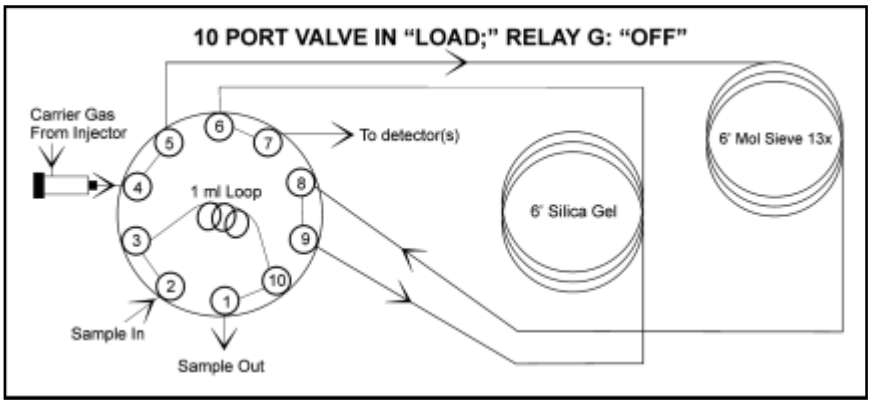
POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

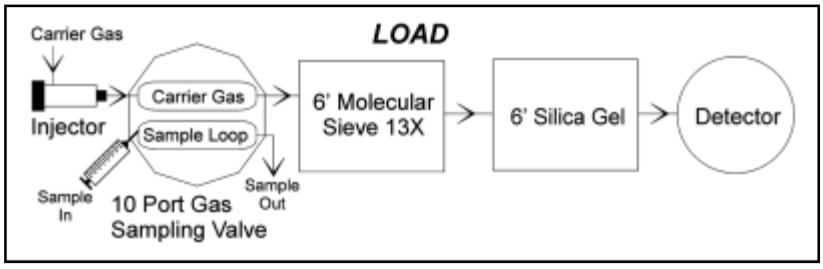
Theory of Operation
10-Port Gas Sampling Valve Plumbing Connections

The valve, sample loop, and column combination is plumbed in a specific way to allow the MG#1 to separate hydrogen, oxygen, nitrogen, methane, ethane, propanes, butanes, pentanes, carbon monoxide, and carbon dioxide with a single injection.

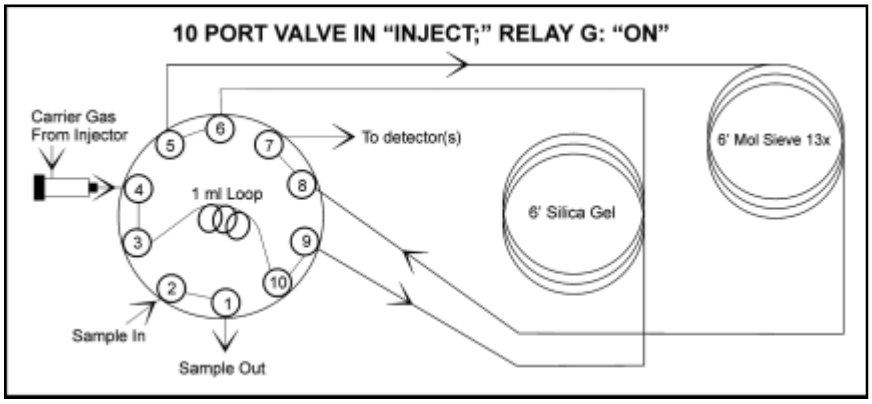
10-Port Gas Sampling Valve in the LOAD Position

A one-milliliter sample loop is connected to the 10-port gas sampling valve. When the valve is in the LOAD position, sample may be flowed through this loop until the moment injection occurs (when the valve switches to the INJECT position).



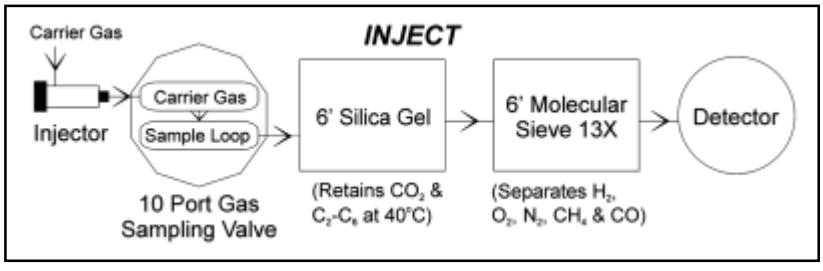


10-Port Gas Sampling Valve in the INJECT Position



At the beginning of the chromatographic run, the valve is actuated to the INJECT position, depositing the sample loop contents into the carrier gas stream and directing it to the two analytical columns, which are connected in series through the 10-port valve.

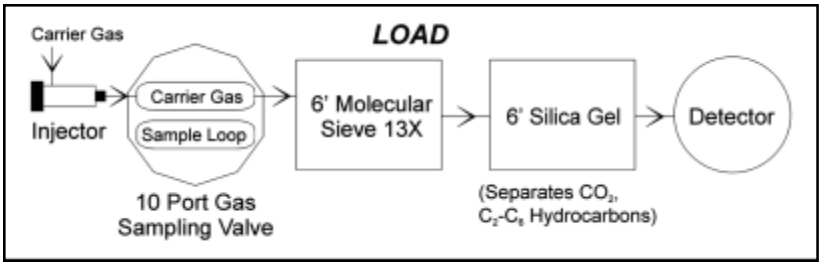
The column sequence is reversed while the flow direction remains the same.



Theory of Operation

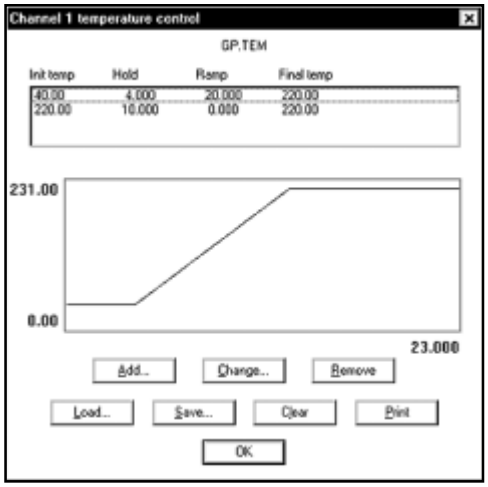
10-Port Gas Sampling Valve Plumbing Connections

The sample is deposited by the carrier gas stream first into the Silica Gel column, with the column oven holding at 40°C, where the ethane, propane, butanes, pentanes, and carbon dioxide are retained. The remainder of the sample containing H_2 (or helium, whichever is not being used as a carrier), O_2, N_2, methane, and CO, continues on to the Molecular Sieve column. During a chromatographic run with the sampling valve in the INJECT position, the H_2 or helium, O_2, N_2, and methane components are the first to elute through the columns and into the detector. This is due to the Silica Gel's long retention of C_2, CO_2 and higher hydrocarbons at 40°C. The sampling valve is actuated back into the LOAD position immediately following the elution of the CO peak. This reverses the sequence of the columns prior to the detector, and sends the components preparing to elute from the Silica Gel packed column (ethane, propane, etc.) to the detector without passing them through the Molecular Sieve packed column. At the same time, the Silica Gel packed column is temperature ramped to promote the rapid elution of the remaining components.



After the elution of the CO peak, the valve is switched back into the LOAD position, and the C_2, CO_2, and higher hydrocarbons come off the Silica Gel column.

The built-in data system automates the process: the column oven temperature is controlled through a PeakSimple temperature program, and the sampling valve is controlled through a PeakSimple event table.



Temperature program:

Initial	Hold	Ramp	Final
40°C	4.00	20.00	220°C
220°C	10.00	0.00	220°C

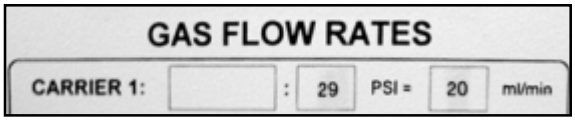
Event table:

Time	Event
0.000	ZERO
0.050	G ON (valve in INJECT)
6.000	G OFF (valve in LOAD)

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

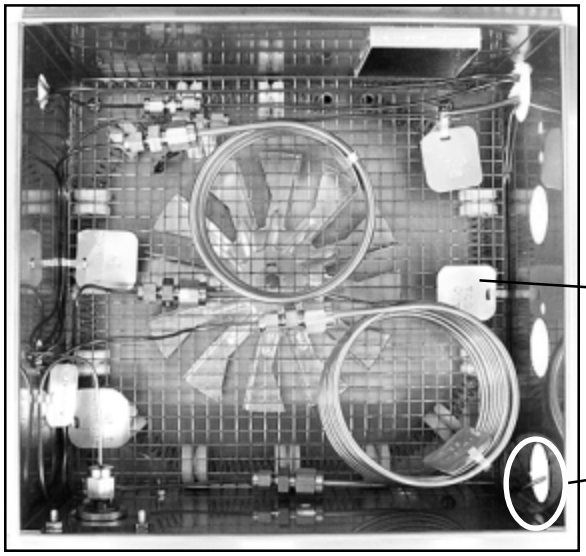
General Operating Procedures

1. Set the cylinder head pressure 15-20psi higher than the head pressure. The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC.

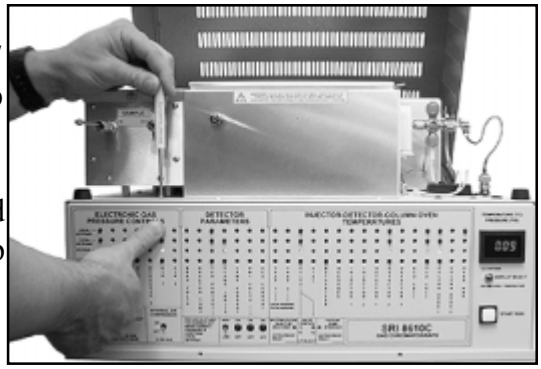


For this particular TCD-equipped MG#1, the head pressure required for a 20mL/min flow is 29psi.

2. *IMPORTANT:* 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 turning the TCD current ON. Tagged for identification, the TCD outlet tubing is located in the column oven. The end of this tubing will be protruding from the column oven wall on the detector side, unless there is also an FID or HID installed. In this case, the TCD outlet tubing will be connected to the FID or HID detector bulkhead fitting in the column oven wall. Place the end of the TCD outlet tubing in some liquid and observe. 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 shuts OFF the TCD current if the column head pressure drops below 3psi, but it cannot prevent filament damage under all circumstances. Any lack of carrier gas flow should be corrected before proceeding. If necessary, reconnect the TCD outlet tubing to the FID or HID when you are finished testing the carrier flow.



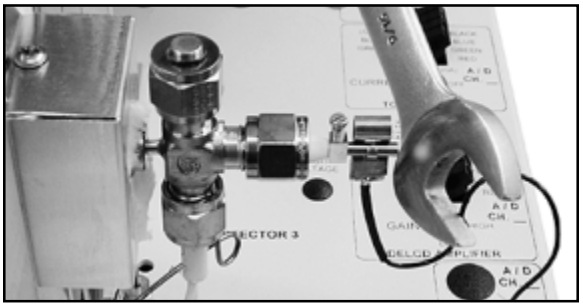
3. If your MG#1 has an FID/Methanizer, set the FID hydrogen flow to 25mL/minute, and the FID combustion air to 250mL/minute. If your MG#1 has an HID, set the helium make-up flow to 40mL/minute and the helium carrier to 10mL/minute. Again, check the "GAS FLOW RATES" printed on the right-hand side of your GC for its flows and the approximate required pressures. Gas flows are adjusted using the trimpots on the top edge of your GC's front control panel. Turn each trimpot while pressing its LOCAL SETPOINT button until the LED display shows the same pressure (in psi) as that printed under GAS FLOW RATES.



4. Set the valve oven temperature to 90°C. If present: set the FID/Methanizer temperature to 380°C; set the HID temperature to 200°C.

General Operating Procedures continued

5. Turn the TCD current ON to LOW. Ignite the FID, if present, by holding up the ignitor switch (lableled "FLAME IGNITE") for a couple of seconds until you hear a small POP. The ignitor switch is located on your GC's front control panel under the heading "DETECTOR PARAMETERS." Verify that the FID flame is lit by holding the shiny side of a chromed wrench directly in front of the FID exhaust vent. The flame is lit when condensation is visible on the wrench surface.



If present, switch on the HID current and set it to 100 using the trimpot and LOCAL SETPOINT button. You should be able to see a purple arc between the two HID electrodes.

Please see the DETECTORS section in your Buck manual for more information.

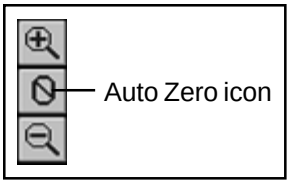
6. Ethane is the first peak to elute from the Silica Gel column after the H_2, O_2, N_2, CH_4, and CO, which are separated by the Molecular Sieve column. The ethane and CO_2 will get stuck in the Molecular Sieve column if the gas sampling valve is not rotated back into the LOAD position (by turning Relay G OFF) prior to the ethane elution. Therefore, you must determine the elution time of ethane, so that you can set an event program that will rotate the valve at the right time during the run. Type in an event program as follows:

Time	Event
0.00	Zero
0.1	G ON
0.3	G OFF

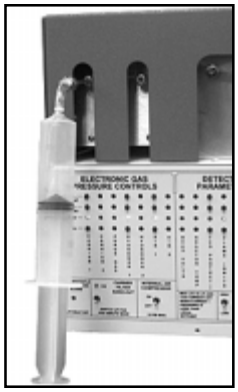
This event program will inject the sample loop contents into the Silica Gel column, then immediately reverse the columns so the sample will not enter the Molecular Sieve column. Since ethane is the first peak off the column, it is easy to determine its elution time.

7. Set the column oven temperature program as follows:

Initial	Hold	Ramp	Final
40°C	6.00	10.00	200°C



8. Zero the data system signal by clicking on the Auto Zero icon on the left side of the chromatogram window. Inject a sample containing ethane into the gas sampling valve through the sample inlet on the front of the valve oven. Start the run by pressing the computer keyboard spacebar, or by pressing the START button on the front of your GC. Note the elution time of ethane.



Injection by syringe of gas sample into valve

9. Revise the event program so that Relay G turns OFF just before the ethane peak begins to rise from the baseline. A typical event table for the MG#1 GC system is shown at right.

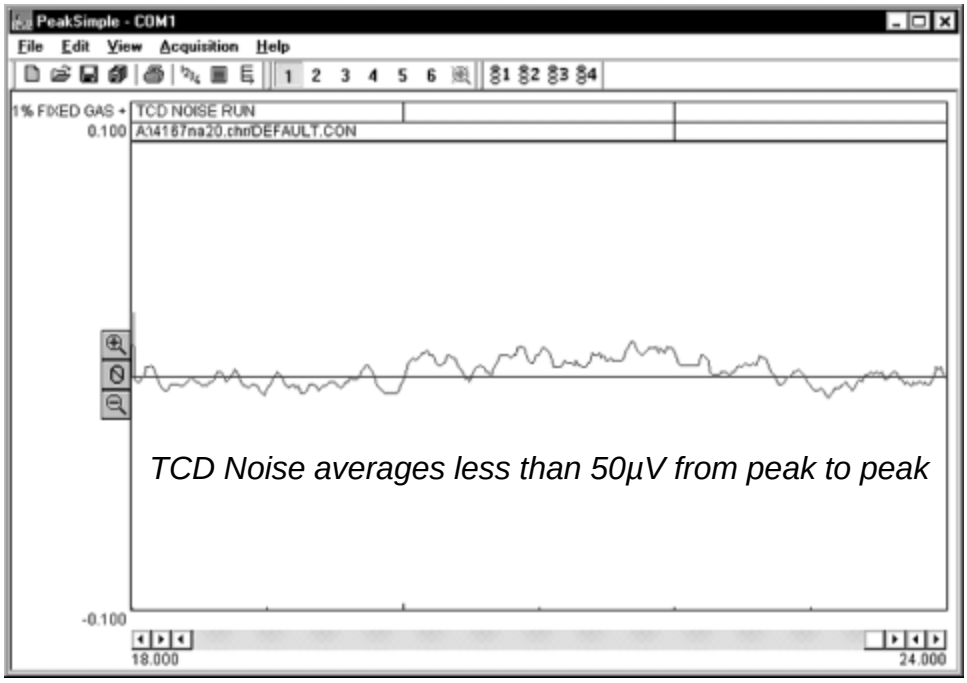
Time	Event
0.00	Zero
0.1	G ON
6.0	G OFF

10. Revise the temperature program if necessary. The temperature program used for the test chromatogram on the Expected Performance page works well with the above event program.

Initial	Hold	Ramp	Final
40°C	4.00	20.00	220°C

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

Expected Performance



TCD Noise Run

Columns: 6' Silica Gel, 6' Molecular Sieve
Carrier: Helium @ 20mL/min
TCD gain: LOW
TCD temperature: 150°C
Valve temperature: 90°C

Temperature program:

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

Factory Test Analysis of 1% Fixed Gas Standard + Ethane

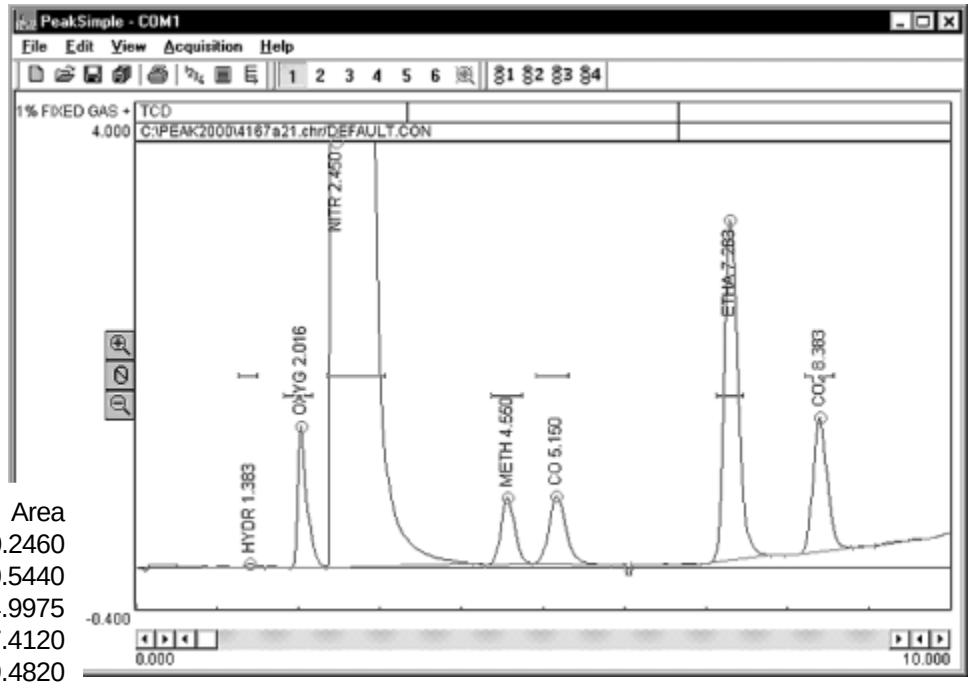
Sample: 1mL 100% ethane + 49mL 1% Fixed Gas Mix
Columns: 6' Silica Gel, 6' Molecular Sieve
Carrier: helium at 20mL/min
TCD gain: LOW
TCD temperature: 150°C
Valve temperature: 90°C

Temperature program:

Initial	Hold	Ramp	Final
40°C	4.00	20.00	220°C

Events:

Time	Event
0.00	Zero
0.050	G ON (valve INJECT)
6.000	G OFF (valve LOAD)



Results:

Component	Retention	Area
Hydrogen	1.383	0.2460
Oxygen	2.016	10.5440
Nitrogen	2.450	924.9975
Methane	4.550	7.4120
CO	5.510	9.4820
Ethane	7.283	38.2725
CO_2	8.383	15.4100
	TOTAL	1006.3640