

some SRI GCs Literature



**typical DATA as per CD-ROM
- with a GC Purchase**



**2011-13
Catalog**



SRI 8610V GC

***SRI 8610C GC
& External PD DS
any PC***

other FlipPAGE Collections
SRI Literature . . . by Chromtech



some popular GC Configurations
SRI Datta Sheets

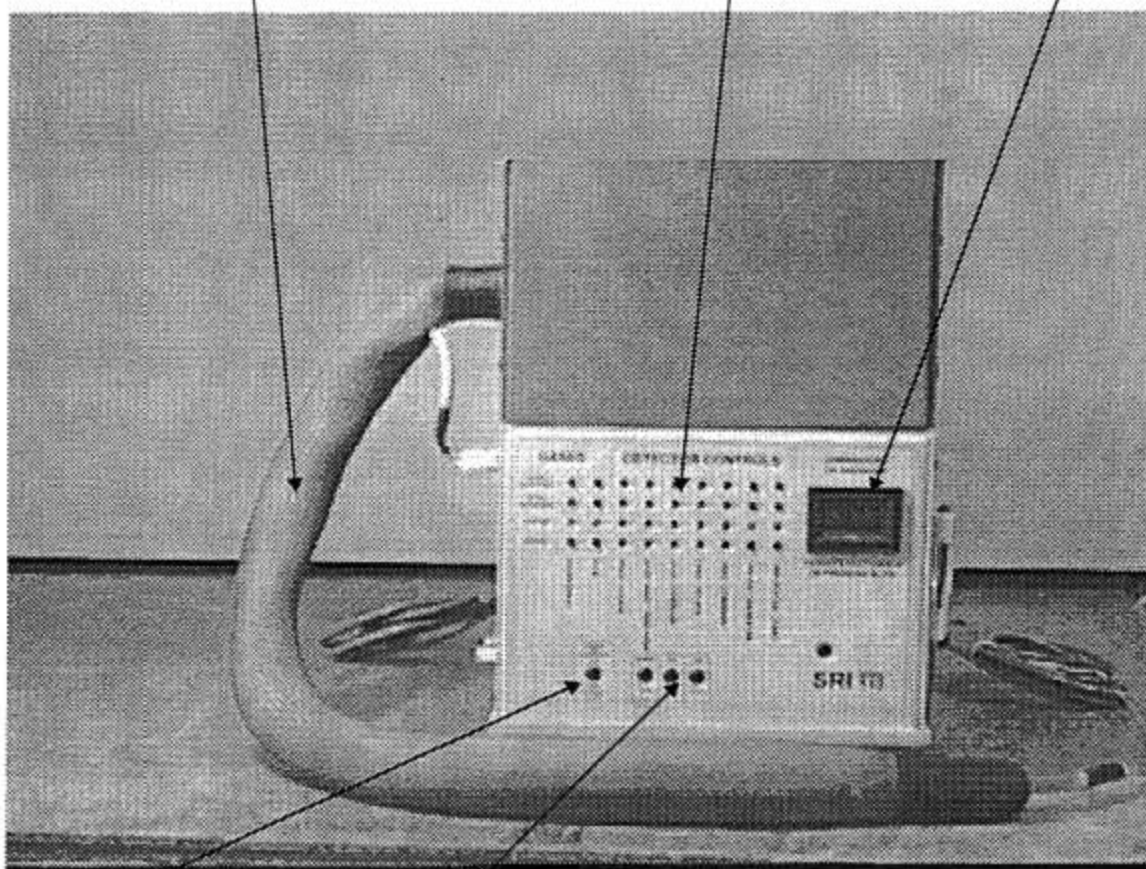
Chapter: MODEL 110 GC CHASSIS

Topic: FRONT PANEL ORIENTATION

Heated transfer line runs about 200 degrees C and has a thick layer of insulation covered by red colored woven tubing. A length of .53mm I.D. silco-steel tubing carries the carrier gas from the host GC to the detector mounted in the 110 chassis.

"At a glance" LED display shows status of all detector parameters

Digital panel meter reads out detector temperatures, voltages, etc. when a specific button on the front panel is depressed.



On/off switch for optional built-in air compressor

Detector parameter on/off switches for FID ignitor, NPD bead voltage, PID lamp current, etc.

The SRI Model 110 chassis is used primarily as a mounting platform for stand-alone GC detectors. The heated transfer line makes it easy to connect the detector to the host GC since only a small opening into the host GC's column oven is required. User's should note that because the heated transfer line operates at 200 C, some high boiling point analytes may condense before reaching the detector. Where high temperature analyses are envisioned, it makes sense to mount the detector on the GC itself instead of on the stand-alone chassis.

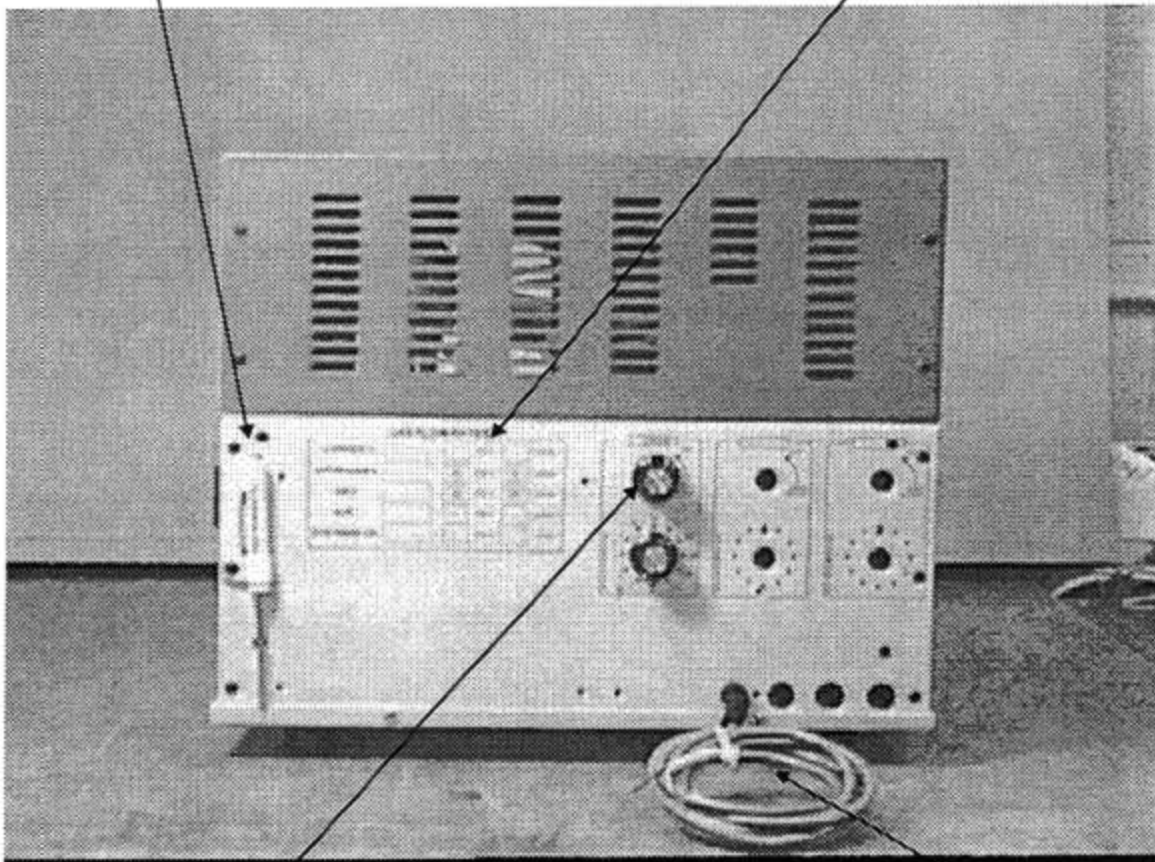
H014.doc

Chapter: MODEL 110 GC CHASSIS

Topic: RIGHT SIDE PANEL ORIENTATION

Screwdriver mounted in handy "holster" for adjusting detector parameters or temperature setpoints

Gas flow rate table is used to record the flow rates and pressures used for detector support gases. Factory technicians record typical flow rates and pressures used to test detectors before shipment during final test at the manufacturing facility.



Zero and attenuator controls for detector output signals. The zero control is a ten turn potentiometer which allows the output from the detector to be offset to 0.00. The attenuator divides the signal by selectable powers of 2 (1,2,4,8 etc.) so that the peak remains on scale when using a strip chart recorder with a fixed span (i.e. 10millivolts full scale). When used with a computer data system or integrator the attenuator control is normally set and left on maximum sensitivity (att=1).

Detector signal cable output wire. This cable containing two wires is hooked up to your strip chart recorder or data system.

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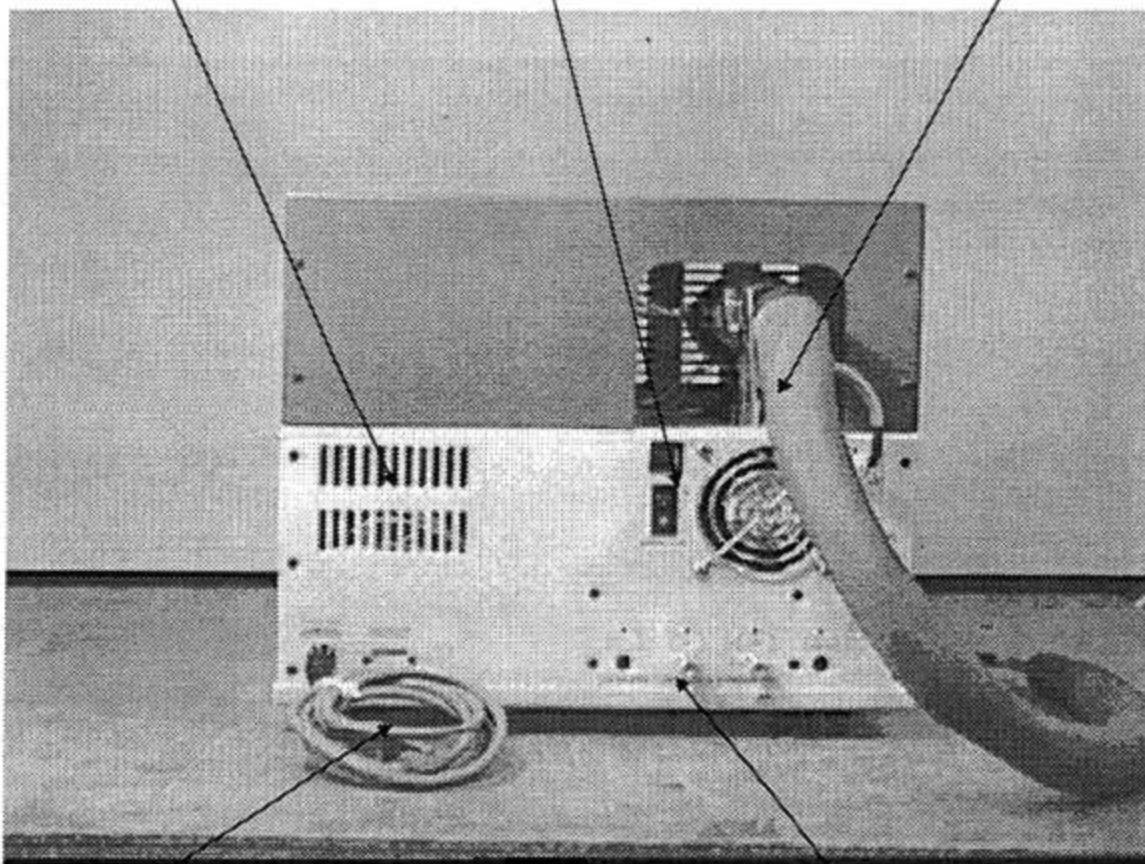
Chapter: MODEL 110 GC CHASSIS

Topic: LEFT SIDE PANEL ORIENTATION

Chassis cooling air exit slots. Air expelled from the chassis by the cooling fan exits through these slots. Do not obstruct the slot openings.

Main power switch, circuit breakers, and chassis cooling fan. This fan cycles on and off to maintain the selected interior chassis temperature.

Heated transfer line for connecting column outlet from host GC to stand-alone detector on Model 110 chassis. Transfer line operates at 200 degrees C. Take care to route transfer line away from heat sensitive surfaces.



Power cord. On 220 volt models it may be necessary to replace the plug on the end of this cord to match the plug type for the country or region.

Gas inlet bulkheads for connection of detector support gases (typically hydrogen and air). Use 1/8th inch O.D. copper tubing to connect gas cylinder to stainless steel bulkhead, not teflon or other plastic tubing types. Use brass ferrules for good sealing.

H016.doc

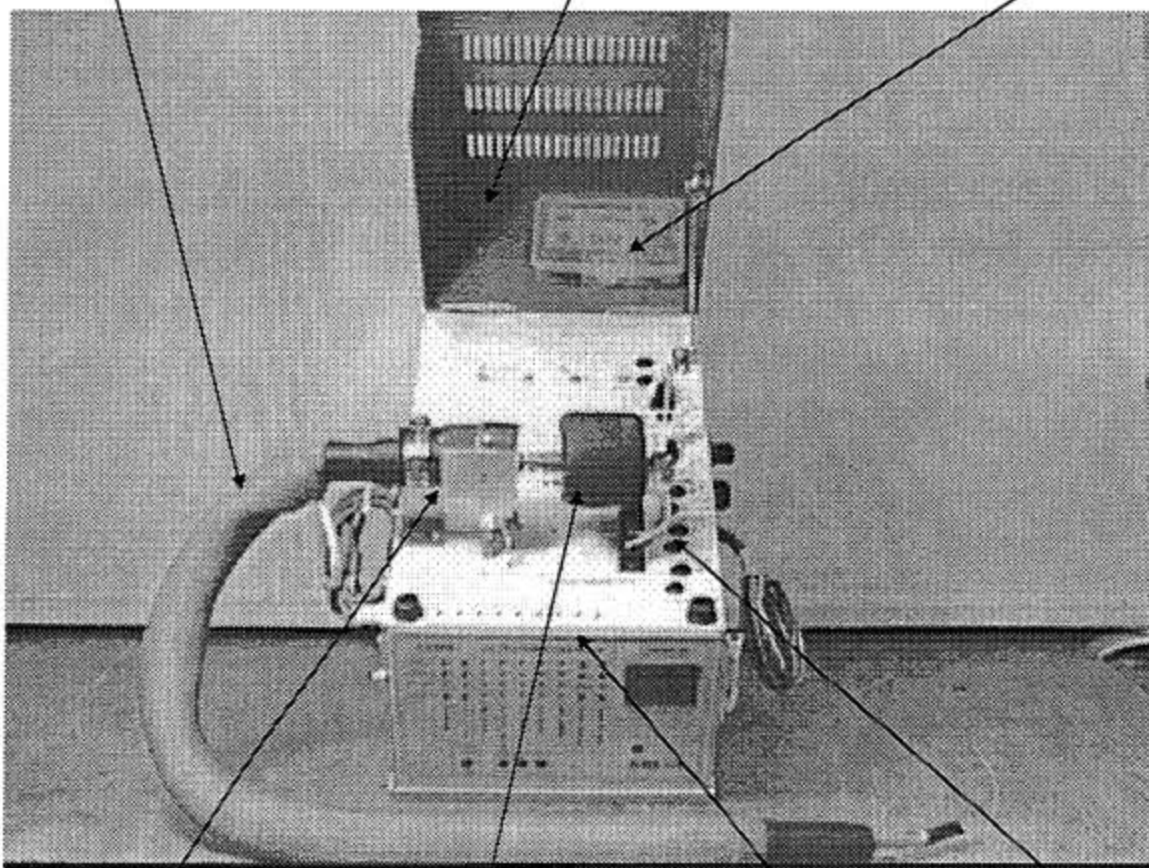
Chapter: MODEL 110 GC CHASSIS

Topic: TOP PANEL ORIENTATION

Heated transfer line connects from host GC to detector mounted on Model 110 chassis. .53mm I.D. silco-steel tubing runs inside heated transfer line so sample only contacts inert fused silica surfaces for most of the length.

Red lid hinges up to allow access to detectors

Spare parts storage container is convenient for keeping extra nuts, ferrules, etc.



Detector heated block and cover terminate transfer line in a hot location to avoid sample condensation

Detector shown above is the SRI DELCD detector, but any of 13 detector types or combinations of detectors may be mounted.

Detector parameter and temperature adjustments are done by using the provided screwdriver to adjust the setpoints through the holes in the forward edge of the chassis

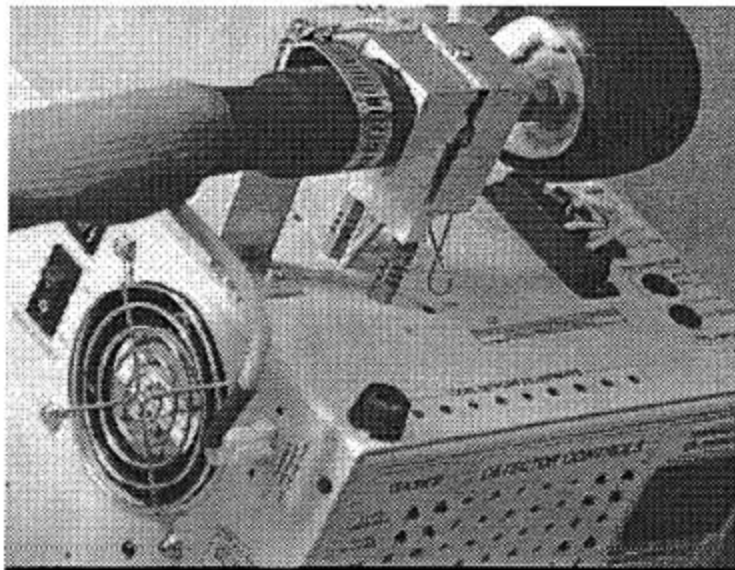
Detector gain controls are located here in the exact same layout as the 310 and 8610C GCs.

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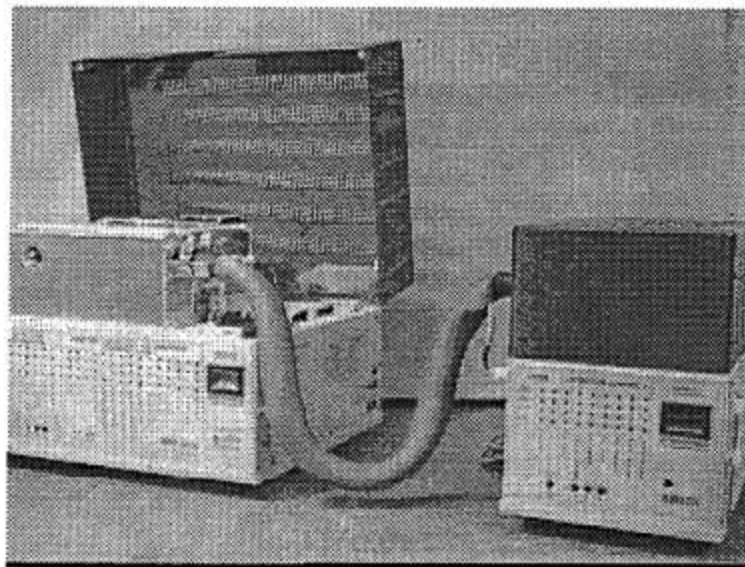
Chapter: MODEL 110 GC CHASSIS

Topic: HEATED TRANSFER LINE

This photo shows the detector end of the heated transfer line as it attaches to the heater block and enclosure. When removing and reattaching the heated transfer line be careful to eliminate any cold spots which could cause sample condensation.



This photo shows the typical installation of the Model 110 to the right of the GC with the heated transfer line connecting the two units. Be careful to route the transfer line so it does not rest on heat sensitive surfaces. In some cases, the lid of the GC may need to have a small notch cut-out of the right side panel to allow the transfer line to exit cleanly from the GC when the red lid is lowered.



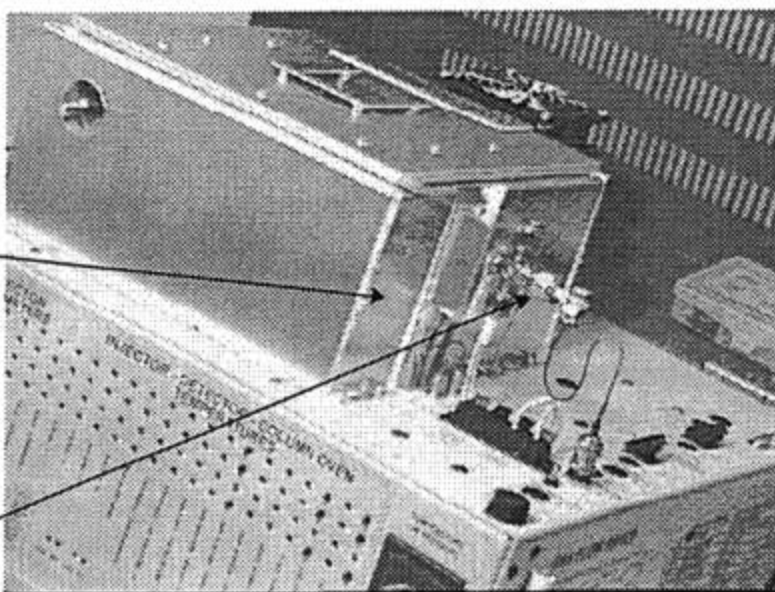
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Chapter: MODEL 110 GC CHASSIS

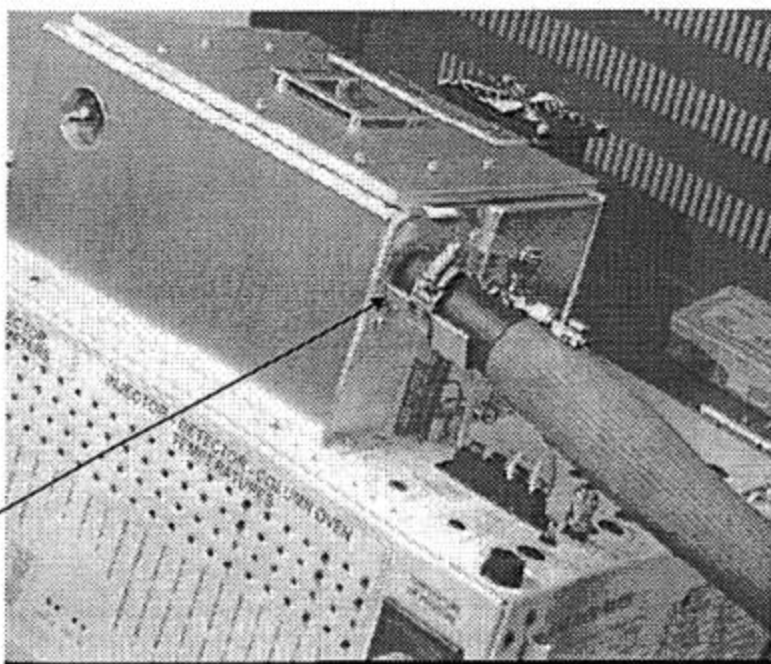
Topic: CONNECTING TRANSFER LINE TO GC

If you are connecting the Model 110 detector to a SRI Model 8610C or 310 GC the right hand side of the GC's column oven has 4 identical detector mounting locations. Locations where no detector is installed are supplied with blank cover plates.

FID detector installed



Replace one of the blank cover plates with the Transfer Line Mounting Plate (SRI part# 8670-9836) by removing the two screws at the base of the plate. The nuts on the underside of the chassis must be accessed by removing the bottom plate of the GC. The heated transfer line is then lightly secured to the plate with the hose clamp so that the heated portion of the line penetrates into the column oven so that cold spots are eliminated

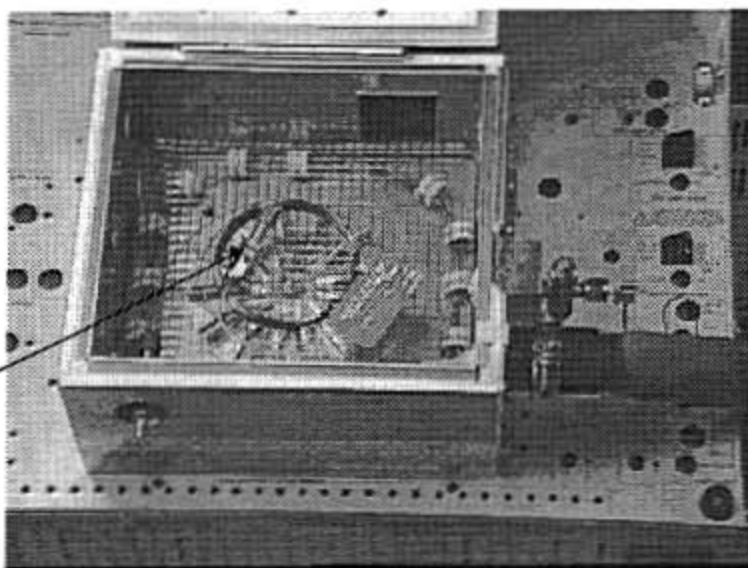


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Chapter: MODEL 110 GC CHASSIS

Topic: COLUMN/TRANSFER LINE CONNECTION

The .53mm I.D. silco-steel tubing which runs down the center of the transfer line is connected to the end of the analytical column inside the GC's column oven. A special 1/8th inch stainless steel bulkhead union and insert are provided to ensure a low dead volume butt type connection. The union may be mounted on a flange or bracket, or just left hanging in the oven.



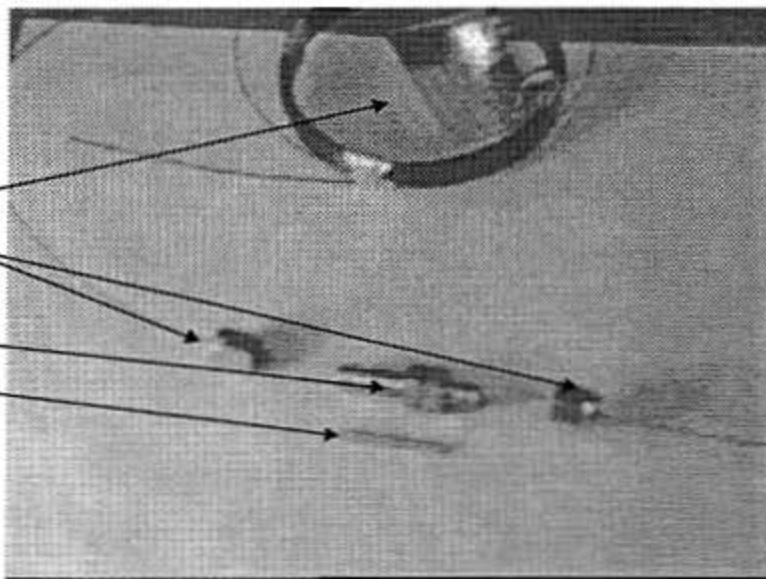
The separate parts of the union and column to transfer line connection hardware consist of:

GC column

Nut with graphite ferrule (2)

Stainless Steel bulkhead

Internal alignment guide which holds the transfer line and column butt to butt inside the bulkhead union

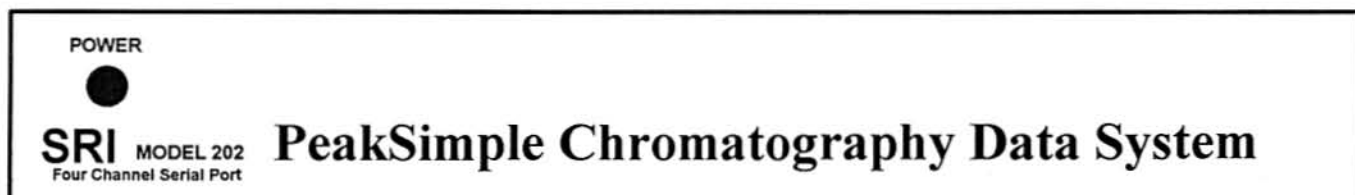


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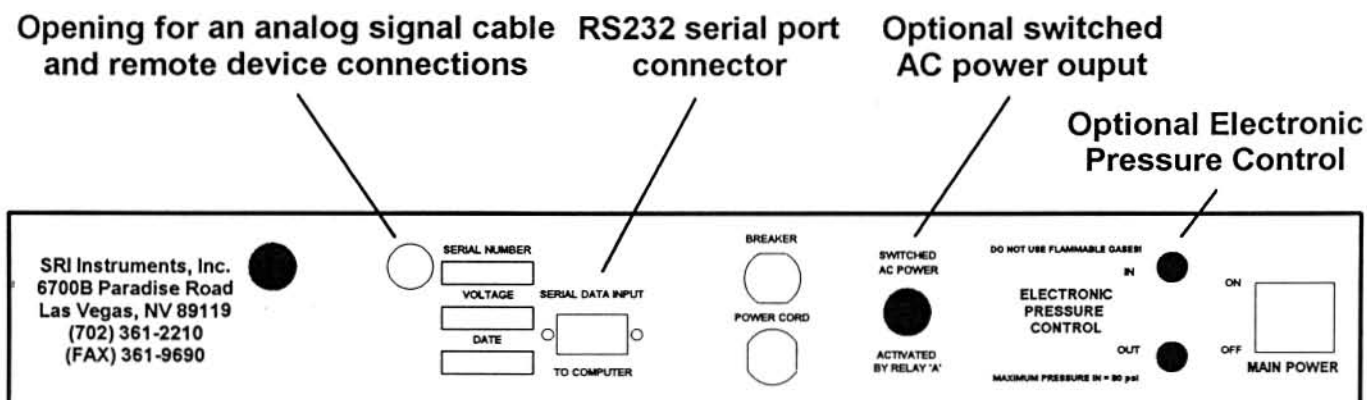
The SRI Model 202 PeakSimple Chromatography Data System is a four channel, analog to digital converter, controlled by our powerful PeakSimple Software. The Model 202 may be used with any brand or model of HPLC or gas chromatograph offering an analog detector output signal.

The Model 202 also features two independent, programmable controls which can be used for temperature and pressure ramping or HPLC gradient formation. There are also two Remote Start inputs that are compatible with two-wire switch closure signals typically output by GCs and LCs as a remote start signal.

Eight TTL outputs (0 to 5 volts) for computer control of external events come standard with the Model 202. If TTL outputs are not adequate for your application, the Model 202 also contains relay circuits offering normally open (NO) and normally closed (NC) switch closures. Electronic Pressure Control (EPC) and a switched AC power output may be ordered as an option for the Model 202.



(front view)



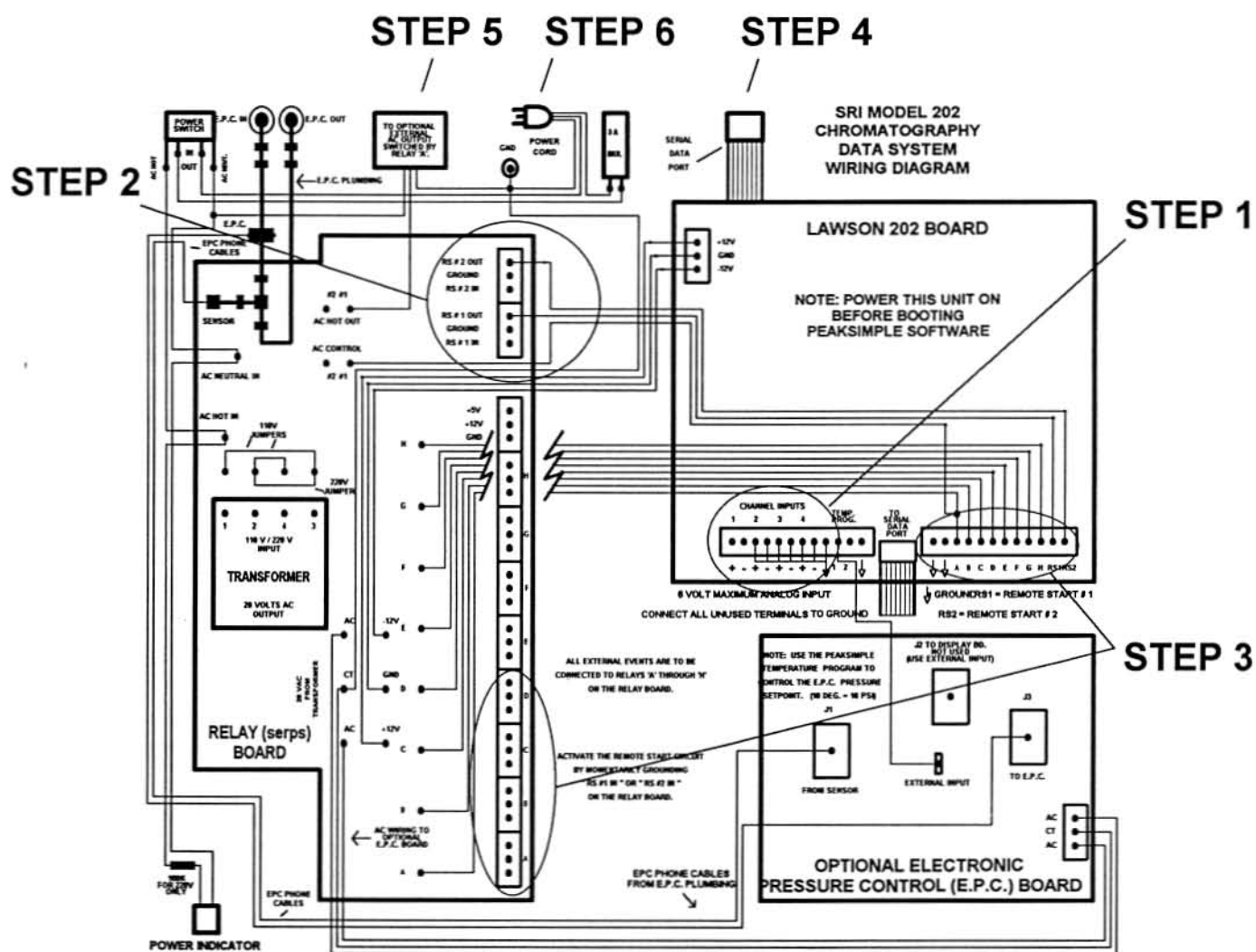
(rear view)

The Model 202 comes with a built-in serial interface for connection to your desktop or laptop computer's COM Port. (See the "PeakSimple For Windows" section in this manual for minimum system requirements.)

You should have received the following with your Model 202 purchase:

- (1) Model 202 PeakSimple Chromatography Data System Box
- (1) Serial Data Interface Cable for connection to your computer's COM Port
- (1) PeakSimple For Windows software package
- (1) PeakSimple Chromatography Data System Manual

To connect the Model 202 to your computer it will be necessary to access connection terminals inside the Model 202 Box.
Verify that **NO POWER** is applied to the unit before performing the following procedure!!
Remove the thumbscrews on either side of the Model 202 Box and carefully slide up the top cover and set it aside. Figure 1, (below), depicts the layout of the Model 202 circuit boards and all wiring connections. To connect your system to the Model 202 Data System; please complete Steps 1 Through 7 as shown below and described on the following pages.



STEP 7 — Install PeakSimple For Windows software.

FIGURE 1

STEP 1: Connecting the analog signal cable(s):

NOTE: The analog output from some GCs or LCs can have a range of up to 10 volts dc. Although the Model 202 will allow high voltage inputs such as this; be advised that signals above 6 volts will generate unwanted noise and signals above 5 volts will be "clipped". (The tops of the waveforms will be cut off.)

Route the analog signal cables from your instrument through the open hole in the back of the Model 202.

Strip 1/4" of insulation off of the 'signal +' and 'signal-' wires of your signal cables. Insert 'signal +' into the Lawson 202 board screw terminal marked 'CH1 sig +' and secure the connection using a small screwdriver.

Insert 'signal -' into the Lawson 202 board screw terminal marked 'CH1 sig -' and secure the connection using a small screwdriver.

Repeat the connection of signal cables for channels 2, 3 and 4.

Any unused channels MUST have both inputs jumpered to ground.

STEP 2: (OPTIONAL) Connecting the remote start cable(s):

NOTE: The Model 202 offers remote starting capability as a standard feature. Two separate remote start circuits permit the user to start the MAIN and ALTERNATE Trigger Groups of the data system by means of a switch closure, such as a footswitch. In some applications, the chromatograph being used with the Model 202 may offer a remote start signal output or switch closure output that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the Model 202.

Route the remote start cable from your instrument through the open hole in the back of the Model 202.

Strip 1/4" of insulation off of the '+' and '-' wires of your remote start cable. Insert '+' into the RELAY (serps) board screw terminal marked '#1 IN' and secure the connection using a small screwdriver.

Insert '-' into the RELAY (serps) board screw terminal marked '#1 G' and secure the connection using a small screwdriver.

NOTE: Be sure to check the "Remote Start" box in the PeakSimple For Windows EDIT - CHANNELS - DETAILS screen for the appropriate channels. Refer to the "PeakSimple For Windows" section of this manual.

STEP 3: (OPTIONAL) Connecting the external event relay wires:

The Model 202 features eight 0-5 volt TTL Level outputs that may be turned on and off individually and automatically by means of a timed event table.

Manual control is also available via the keyboard.

These outputs may be used to control external events or devices. If TTL level outputs are not adequate for your application, the Model 202 is also equipped with eight relay circuits offering normally open (NO) and normally closed (NC) contact closures.

STEP 3: (Continued)

Route the external event wires from your instrument through the open hole in the back of the Model 202.

Strip 1/4" of insulation off of each wire. Select which device should be connected to events 'A' through 'H' and insert the wire into the appropriate screw terminal and secure the connection using a small screwdriver.

Refer to the "PeakSimple For Windows" section of this manual for setting up event tables, keyboard activation, etc.

STEP 4: Connecting the Serial Data Interface cable to your computer:

The Model 202 is equipped with a RS-232 serial port. A DB-9 type serial cable (provided) connects the Model 202 to your personal computer through the PC's COM port. This simple interface permits the data system software to be loaded onto, and operated from, either a desktop or notebook PC for portability in field operations.

Secure one end of the Serial Data Interface cable to an available COM port on the Back of your PC. Secure the other end to the DB-9 connector on the back of the Model 202. (Refer again to Figure 1 for location of the Serial Data port.)

STEP 5: (OPTIONAL) Connecting to the switched AC output:

Connections for the switched AC output are pre-wired at the factory.

All you need to do is plug your device into the cord provided; activate relay 'A' using PeakSimple software and the outlet will be powered. (1 AMP maximum)

NOTE: The switched AC output must be specifically requested at the time you order the Model 202.

STEP 6: Connecting power to the Model 202:

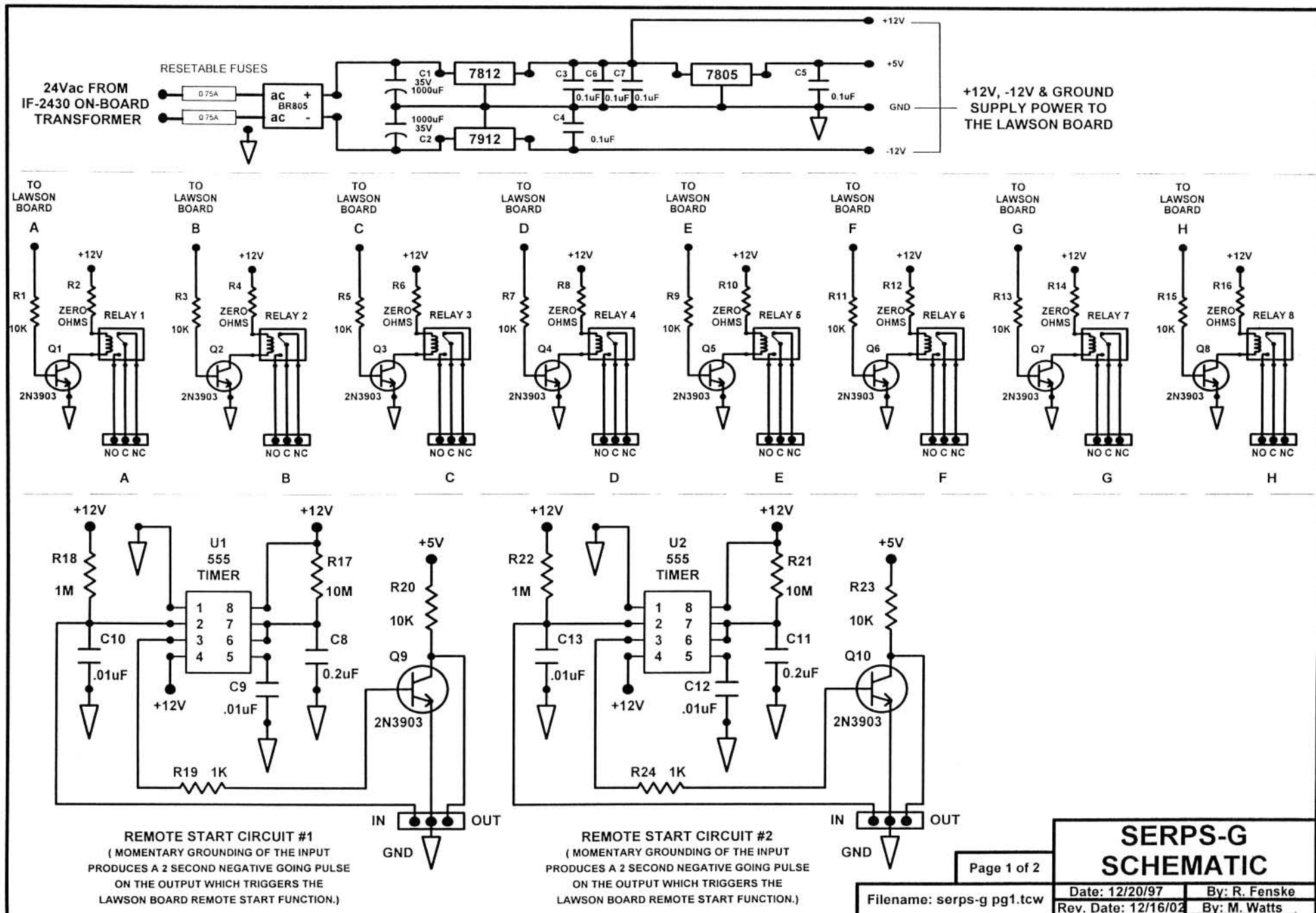
Slide the top cover back onto the Model 202.

Secure the cover with the two thumbscrews.

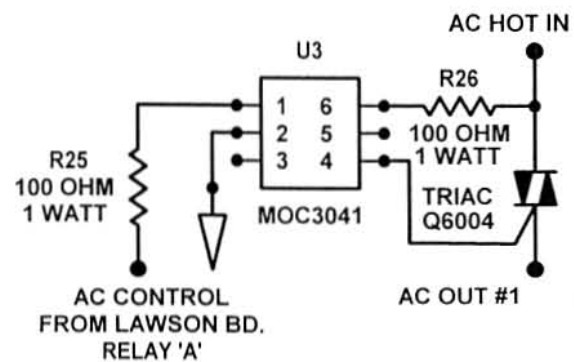
Model 202 units are equipped with a power cord which plugs into a standard 110 (or 220) volt outlet. Plug the Model 202 into the outlet and verify that the POWER indicator on the front of the Model 202 is lit.

STEP 7: Installation of PeakSimple Software:

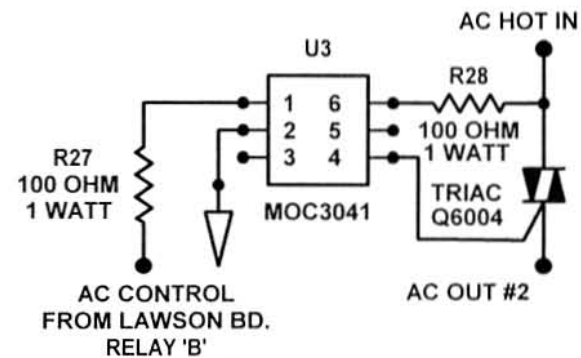
Refer to the "PeakSimple For Windows" section of this manual for details on proper installation and operation.



OPTIONAL AC OUTPUT CIRCUIT #1



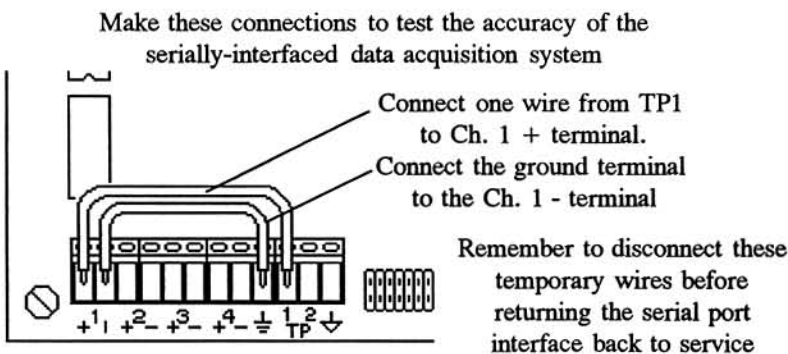
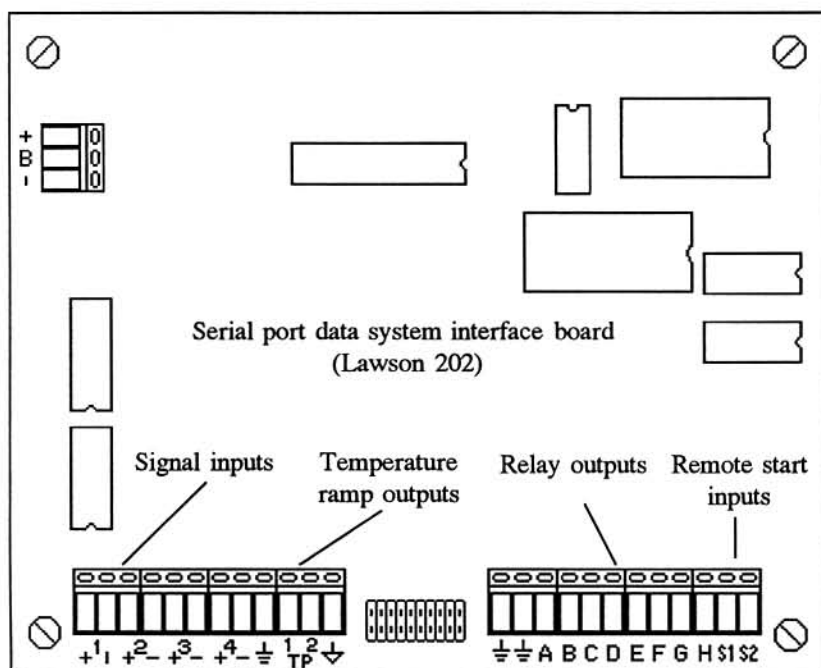
OPTIONAL AC OUTPUT CIRCUIT #2



The SRI serial port data system is equipped with four channels of precision data acquisition. In fact, the signal inputs available on the serial port data acquisition interface offer the precision of a digital voltmeter. Any 0 to 5VDC detector signal may be connected to any one of the channel inputs, and the millivolt reading will be displayed on-screen. This reading should match the readings of any precision meter connected to the same input. No special calibration of detector signal inputs is required.

If it does become necessary to verify the precision of the serially-interfaced signal inputs, the following procedure will permit easy confirmation.

In order to confirm the precision of the signal inputs, temporary electrical connections are made at the serial port interface's terminal strips. First, connect a wire from the temperature ramp output on the serial port interface labelled "TP1", to the + (positive) terminal of an unused channel input. Then connect another wire from the ground terminal to the - (negative) terminal of the same channel input, as illustrated in the diagram below.



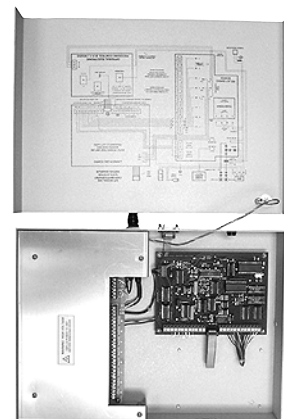
Whether the serial port interface board is built into the GC, or is installed in its own stand-alone case, these connections will permit the temperature program signal output by channel 1 to be fed back into the data acquisition circuitry for channel 1. The temperature program signal outputs 10mV for each degree.

When the two test jumpers are in place, an artificial signal is generated by loading the AREATEST.TEM file, included with the PeakSimple software, onto channel 1. When this temperature file has been loaded into the channel 1 temperature programming window, starting a run will send a ramped temperature program signal from TP1 into the channel 1 + input. This signal is plotted, and the user will see that the on-screen readings match those of a reliable digital voltmeter, down to the third decimal place.

Quick Start

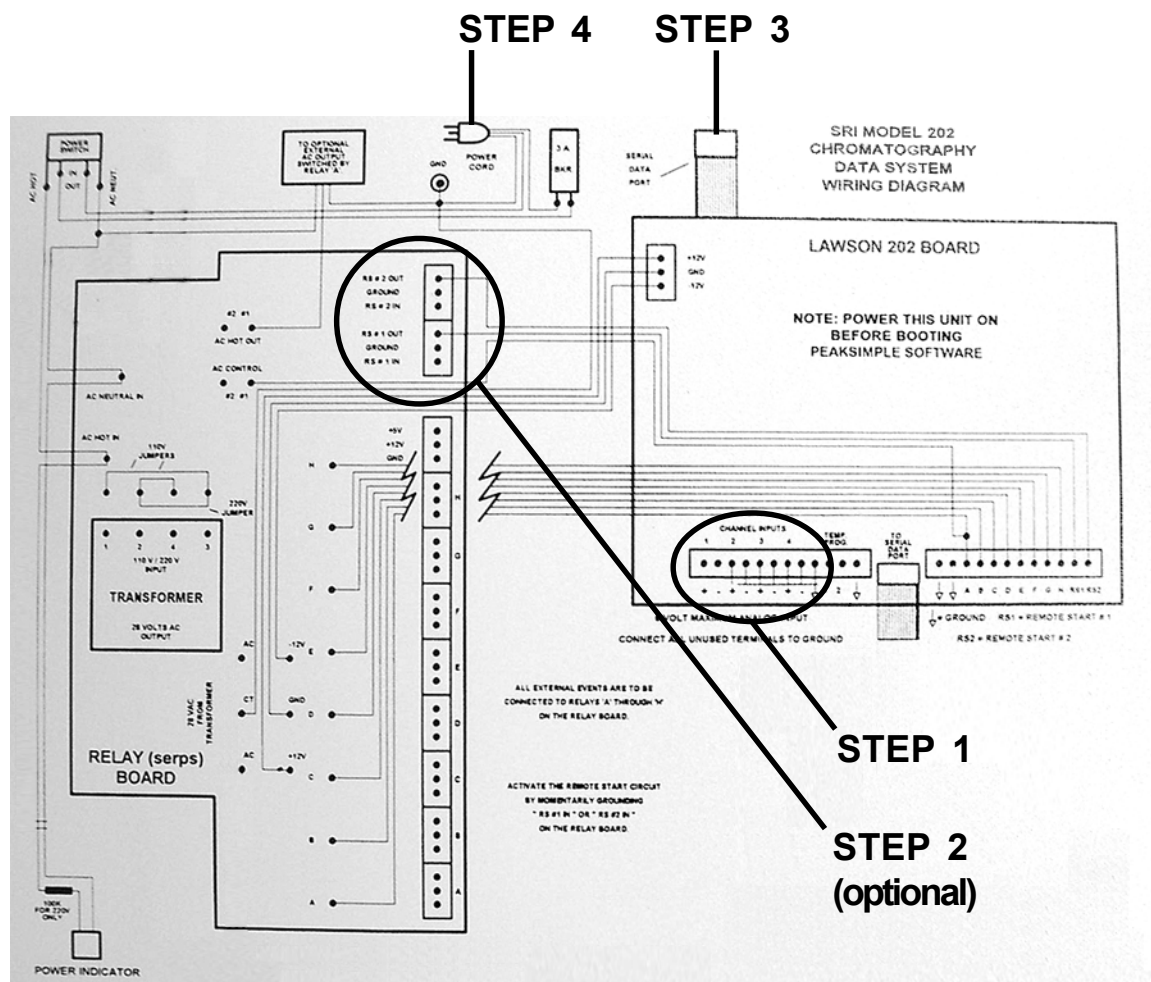
Model 202 Four Channel PeakSimple Data System

The **Model 202** may be used with any brand or model of GC or HPLC offering an analog detector output signal ranging from -5V to +5V. It includes two independent, programmable controls (-5V to +5V analog output) for temperature & pressure or HPLC gradient formation. The Model 202 has four channels, which can be split any way between the two time bases (TIMEBASE 1 and 2). Two remote start inputs compatible with 2-wire switch closures (typically output by GCs and HPLCs as a remote start signal) are also included for your optional use.



Open the Model 202

Verify that the Model 202 is powered OFF and unplugged. Remove the thumbscrews on both sides of the Model 202 box and slide the top cover up and off. It is connected to the bottom of the box by the ground wire, so just set it next to the bottom half of the box. There is a wiring diagram of the Model 202 circuit boards and all wiring connections on the inside of the top cover. Use this wiring diagram (shown below) to complete steps 1-4 as described on the following pages, then proceed to steps 5 & 6.



STEP 5: Install PeakSimple

STEP 6: Starting an Analysis

Quick Start

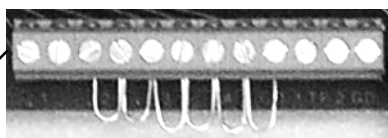
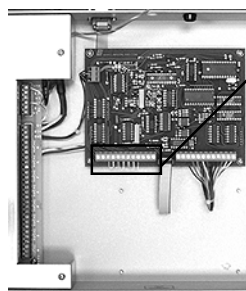
Model 202 Four Channel PeakSimple Data System

STEP 1: Connect the Analog Signal Cable(s)

1-1. Route the analog signal cables from your instrument through the open hole in the back of the Model 202.

1-2. Strip 1/4" of insulation from the "signal+" and "signal-" wires of your instrument's signal cables.

Route the signal cables through this hole



The Model 202 is shipped with jumpers in the Channel screw terminals

1-3. Remove the jumpers shown in the close-up picture to the left. Insert the "signal+" wire into the Lawson 202 board screw terminal marked "CH1 sig +" and secure the connection with a small flat-blade screwdriver.

1-4. Insert the "signal-" wire into the Lawson 202 board screw terminal marked "CH1 sig -" and secure the connection.

1-5. Repeat the connection of signal cables for channels 2, 3, and 4. Any unused channels MUST have both inputs jumpered to ground.

STEP 2: (OPTIONAL) Connect the Remote Start Cable

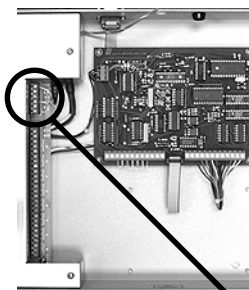
The Model 202 remote start capability allows you to start the data system by means of a switch closure. Two separate remote start circuits permit the user to individually start TIMEBASE 1 and 2 of the data system. In some applications, the chromatograph being used with the Model 202 may offer a remote start signal output or switch closure output that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the Model 202.

2-1. Route the remote start cable from your instrument through the open hole in the back of the Model 202.

2-2. Strip 1/4" of insulation from the "+" and "-" wires of your remote start cable.

2-3. Insert the "+" wire into the RELAY board screw terminal marked "#1 IN" and secure the connection.

2-4. Insert the "-" wire into the RELAY board screw terminal marked "#1 G" and secure the connection.



Quick Start

Model 202 Four Channel PeakSimple Data System

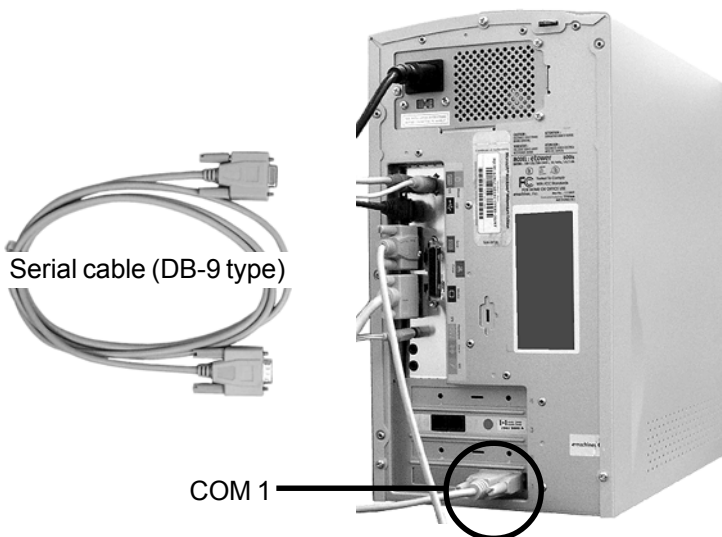
Slide the cover back onto the Model 202 and secure it with the thumbscrews.

STEP 3: Connect the Serial Cable to Your Computer

The Model 202 is equipped with a RS-232 serial port. A DB-9 type serial cable (provided) connects the Model 202 to your Windows™ computer through the PC's COM port. This simple interface permits the Model 202 to be operated from a desktop or laptop computer.

3-1. Secure one end of the serial cable to an available COM port on your PC.

3-2. Secure the other end to the RS-232 serial port on the back of the Model 202.



STEP 4: Connect Power to the Model 202

The Model 202 is provided with a power cord which plugs into a standard 110 (or 220) volt outlet. Plug the Model 202 into the wall outlet. Turn ON the power switch and verify that the POWER LED on the front of the Model 202 is lit.



The power LED is lit when the Model 202 is connected to a power source.

STEP 5: Install PeakSimple Chromatography Software

5-1. Locate your copy of PeakSimple, which is shipped inside the front cover of your SRI manual. Insert the CD or floppy disk(s) into your computer's appropriate drive.

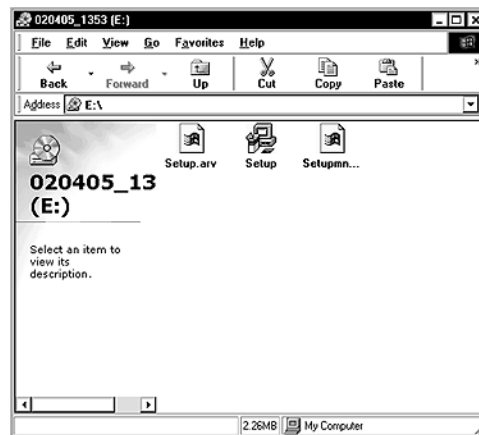


Quick Start

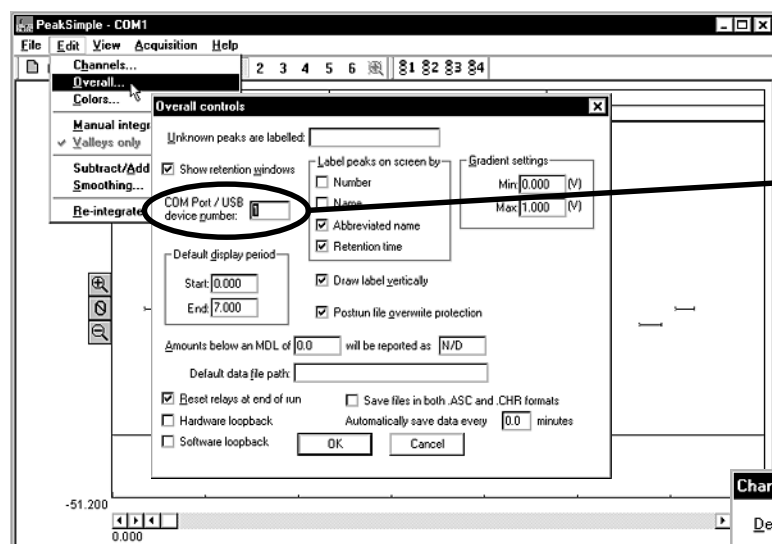
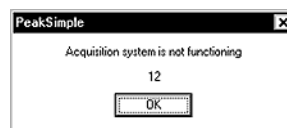
Model 202 Four Channel PeakSimple Data System

STEP 5 Continued

5-2. Open the appropriate drive through My Computer, then double click on “Setup.exe” and follow the instructions.

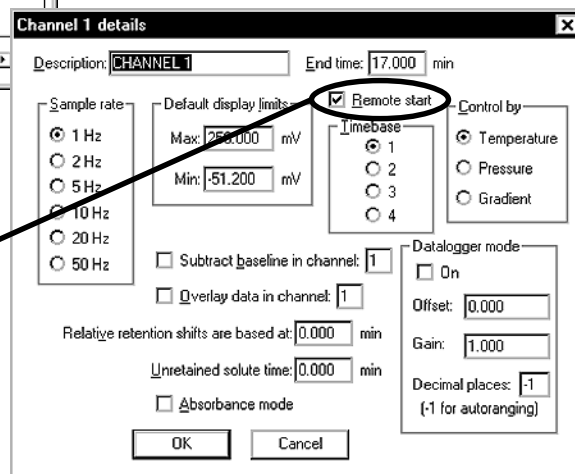


5-3. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between your computer and the Model 202. An error message will appear if communication is not established.



5-4. PeakSimple defaults to COM 1. If you did not connect the Model 202 to COM 1, you will get the error message. Open the Edit menu and choose Overall. In the dialog box that appears, enter the number of the COM port to which you have connected the Model 202. If you do not know the number of the COM port to which you connected the 202, use the process of elimination: try different numbers until you find one that works.

5-5. For the remote start option: Open the Edit menu and choose Channels. Click on the Details button for channel 1. Verify that Remote start is enabled (the box should be checked). Repeat this step for channel 2.

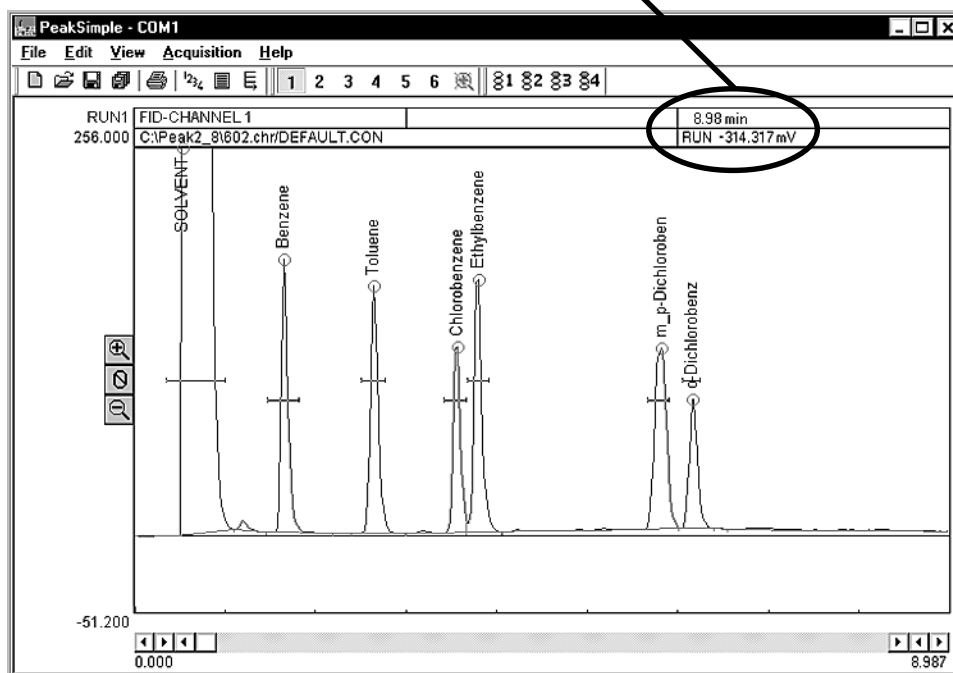


Quick Start

Model 202 Four Channel PeakSimple Data System

STEP 6: Starting an Analysis

6-1. The upper right corner of the PeakSimple chromatogram window contains real-time information pertinent to your analysis in progress. The status of the run (STAND BY, RUN) is displayed in capital letters next to the millivolt (mV) reading, underneath the amount of time into the run.



6-2. Hit your computer keyboard spacebar to begin the run, and the data is plotted onscreen in the chromatogram window.



Press the spacebar to begin the run

Press the End key
to stop the run

6-3. Hit the End key on your computer keyboard to stop the run.

More on PeakSimple:

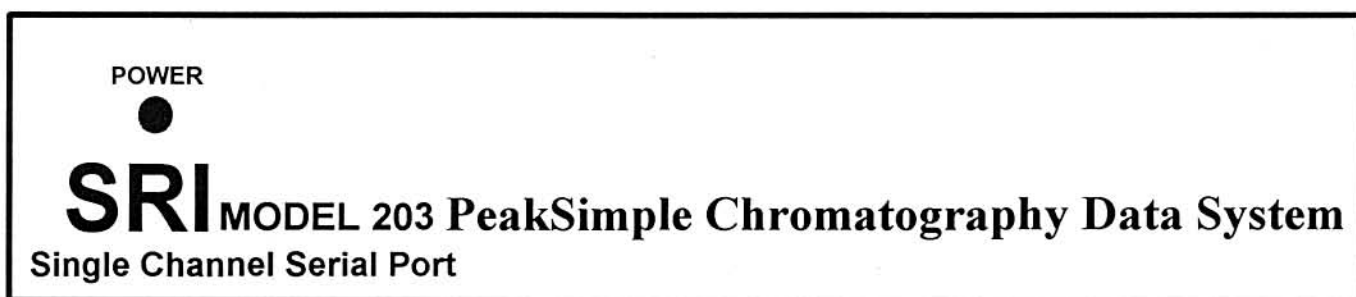
This Quick Start guide presents a very brief introduction to PeakSimple. There are tutorials in the manual and online at www.srigc.com (click on the "Download Our Documents" button) that will acquaint you with PeakSimple's basic functions.

If you have questions or problems, call SRI for free technical support at 310-214-5092, 8am - 5pm California time.

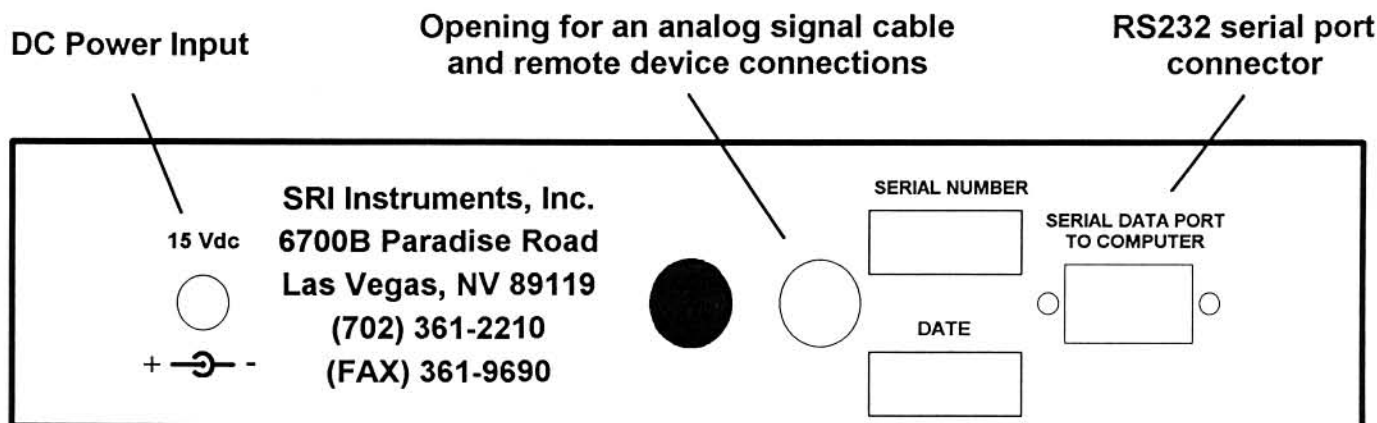
The SRI Model 203 PeakSimple Chromatography Data System is a single channel, analog to digital converter, controlled by our powerful PeakSimple Software. The Model 203 may be used with any brand or model of HPLC or gas chromatograph offering an analog detector output signal.

The Model 203 also features two independent, programmable controls which can be used for temperature and pressure ramping or HPLC gradient formation. There is also a Remote Start input that is compatible with two-wire switch closure signals typically output by GCs and LCs as a remote start signal.

Eight TTL outputs (0 to 5 volts) for computer control of external events come standard with the Model 203. If TTL outputs are not adequate for your application, the Model 203 can also be ordered with optional relay circuits offering normally open (NO) and normally closed (NC) switch closures.



(front view)



(rear view)

The Model 203 comes with a built-in serial interface for connection to your desktop or laptop computer's COM Port. (See the "PeakSimple For Windows" section in this manual for minimum system requirements.)

You should have received the following with your Model 203 purchase:

- (1) Model 203 PeakSimple Chromatography Data System Box
- (1) Serial Data Interface Cable for connection to your computer's COM Port
- (1) 15 Volt dc Wall Transformer
- (1) PeakSimple For Windows software package
- (1) PeakSimple Chromatography Data System Manual

To connect the Model 203 to your computer it will be necessary to access connection terminals inside the Model 203 Box.

Verify that **NO POWER** is applied to the unit before performing the following procedure!! Remove the thumbscrews on either side of the Model 203 Box and carefully slide up the top cover and set it aside. Figure 1, (below), depicts the layout of the Model 203 circuit boards and all wiring connections. To connect your system to the Model 203 Data System; please complete Steps 1 Through 6 as shown below and described on the following pages.

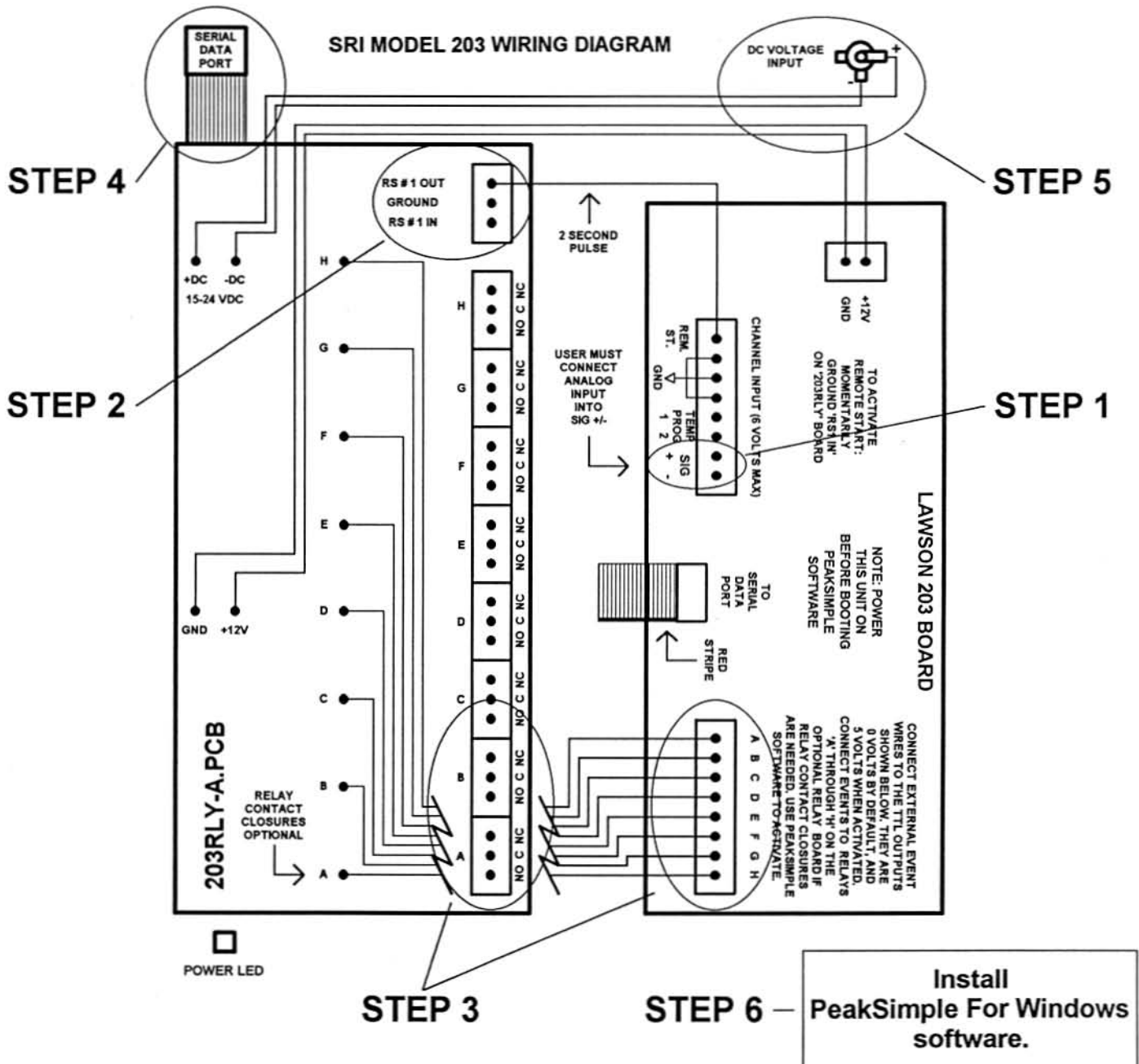


FIGURE 1

STEP 1: Connecting the analog signal cable:

NOTE: The analog output from some GCs or LCs can have a range of up to 10 volts dc. Although the Model 203 will allow high voltage inputs such as this; be advised that signals above 6 volts will generate unwanted noise and signals above 5 volts will be "clipped". (The tops of the waveforms will be cut off.)

Route the analog signal cable from your instrument through the open hole in the back of the Model 203.

Strip 1/4" of insulation off of the 'signal +' and 'signal-' wires of your signal cable. Insert 'signal +' into the Lawson 203 board screw terminal marked 'sig +' and secure the connection using a small screwdriver.

Insert 'signal -' into the Lawson 203 board screw terminal marked 'sig -' and secure the connection using a small screwdriver.

STEP 2: (OPTIONAL) Connecting the remote start cable:

NOTE: The Model 203 offers a remote starting capability as a standard feature. This permits the user to start the data system by means of a switch closure, such as a footswitch. In some applications, the chromatograph being used with the Model 203 may offer a remote start signal output or switch closure output that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the Model 203.

Route the remote start cable from your instrument through the open hole in the back of the Model 203.

Strip 1/4" of insulation off of the '+' and '-' wires of your remote start cable. Insert '+' into the 203RLY board screw terminal marked 'IN' and secure the connection using a small screwdriver.

Insert '-' into the 203RLY board screw terminal marked 'GND' and secure the connection using a small screwdriver.

NOTE: Be sure to check the "Remote Start" box in the PeakSimple For Windows EDIT - CHANNELS - DETAILS screen for channel 1. Refer to the "PeakSimple For Windows" section of this manual.

STEP 3: (OPTIONAL) Connecting the external event relay wires:

The Model 203 features eight 0-5 volt TTL Level outputs that may be turned on and off individually and automatically by means of a timed event table. Manual control is also available via the keyboard.

These outputs may be used to control external events or devices. If TTL level outputs are not adequate for your application, the Model 203 can be fitted with eight relay circuits offering normally open (NO) and normally closed (NC) contact closures. NOTE: Relay contact closures must be specifically requested at the time you order the Model 203.

STEP 3: (Continued)

Route the external event wires from your instrument through the open hole in the back of the Model 203.

Strip 1/4" of insulation off of each wire. Select which device should be connected to events 'A' through 'H' and insert the wire into the appropriate screw terminal and secure the connection using a small screwdriver.

Refer to the PeakSimple Software section of this manual for setting up event tables, keyboard activation, etc.

STEP 4: Connecting the Serial Data Interface cable to your computer:

The Model 203 is equipped with a RS-232 serial port. A DB-9 type serial cable (provided) connects the Model 203 to your personal computer through the PC's COM port. This simple interface permits the data system software to be loaded onto, and operated from, either a desktop or notebook PC for portability in field operations.

Secure one end of the Serial Data Interface cable to an available COM port on the Back of your PC. Secure the other end to the DB-9 connector on the back of the Model 203. (Refer again to Figure 1 for location of the Serial Data port.)

STEP 5: Connecting power to the Model 203:

Slide the top cover back onto the Model 203.

Secure the cover with the two thumbscrews.

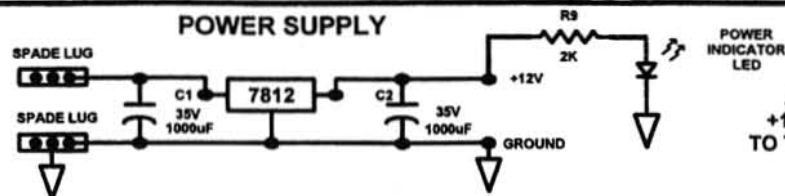
The Model 203 requires a minimum input of 14.8 V dc to operate.

110 volt units are provided with a 15 V dc transformer which plugs into a standard 110 volt outlet. To avoid damaging the unit; plug the transformer output plug into the back of the Model 203 first and THEN plug the main transformer into the wall outlet. Verify that the POWER LED on the front of the Model 203 is lit.

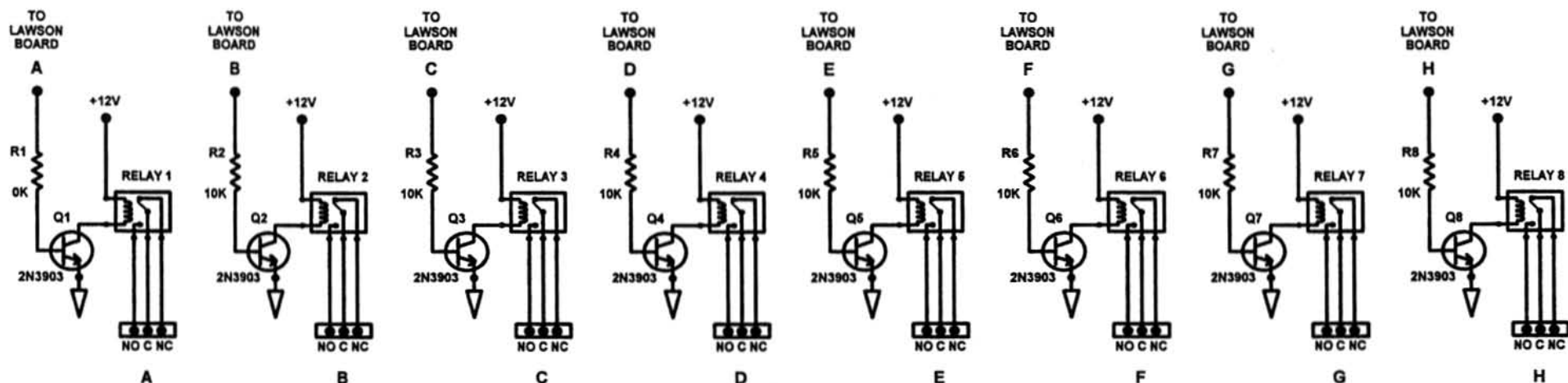
STEP 6: Installation of PeakSimple Software:

Refer to the "PeakSimple For Windows" section of this manual for details on proper installation and operation.

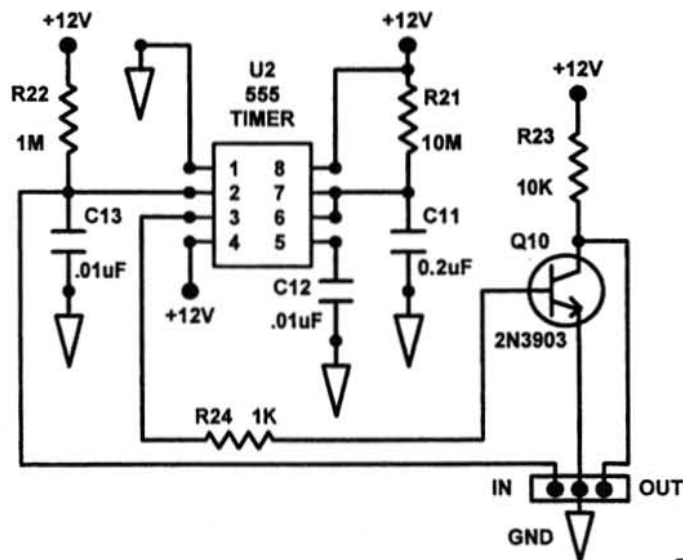
15 - 24 Vdc INPUT FROM
WALL TRANSFORMER



ALSO SUPPLIES
+12 V AND GROUND
TO THE 203 A/D BOARD



REMOTE START CIRCUIT
(MOMENTARY GROUNDING OF THE INPUT
PRODUCES A 2 SECOND NEGATIVE GOING PULSE
ON THE OUTPUT WHICH TRIGGERS THE
203 A/D BOARD REMOTE START FUNCTION.)



OPTIONAL RELAY CIRCUITS:
USED WHEN A CONTACT CLOSURE
IS NEEDED FOR ACTIVATION
OF EXTERNAL DEVICES.

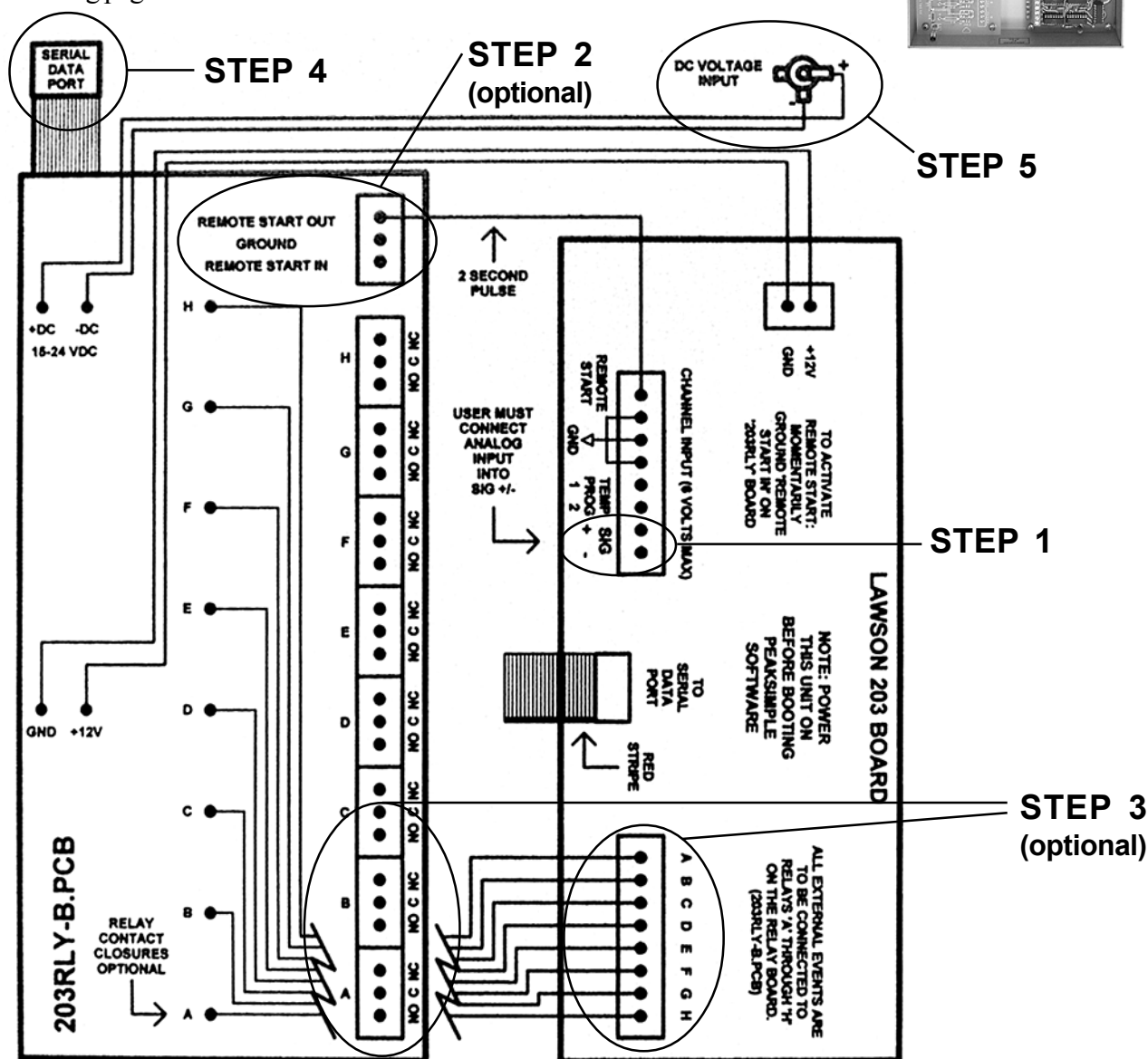
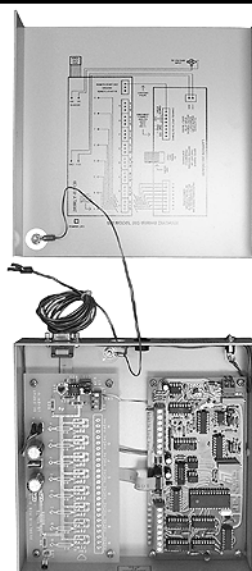
Model 203

Single Channel PeakSimple Data System

The Model 203 may be used with any brand or model of GC or HPLC offering an analog detector output signal ranging from 0-5V. It includes two independent, programmable controls (0-5V analog output) for temperature & pressure or HPLC gradient formation. A remote start input compatible with 2-wire switch closures (typically output by GCs and HPLCs as a remote start signal) is also included for your optional use.

Open the Model 203

Verify that the Model 203 is not plugged into a wall socket and is therefore powered OFF (no power switch). Remove the thumbscrews on both sides of the Model 203 box and slide the top cover up and off. It is connected to the bottom of the box by the ground wire, so just set it next to the bottom half of the box. There is a wiring diagram of the Model 203 circuit boards and all wiring connections on the inside of the top cover. Use this wiring diagram (shown below) to complete steps 1-5 as described on the following pages.



POWER LED

HROM*alytic* +61(0)3 9762 2034

ECHnology Pty Ltd

Australian Distributors
Importers & Manufacturers
www.chromtech.net.au

Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Quick Start

Model 203 Single Channel PeakSimple Data System

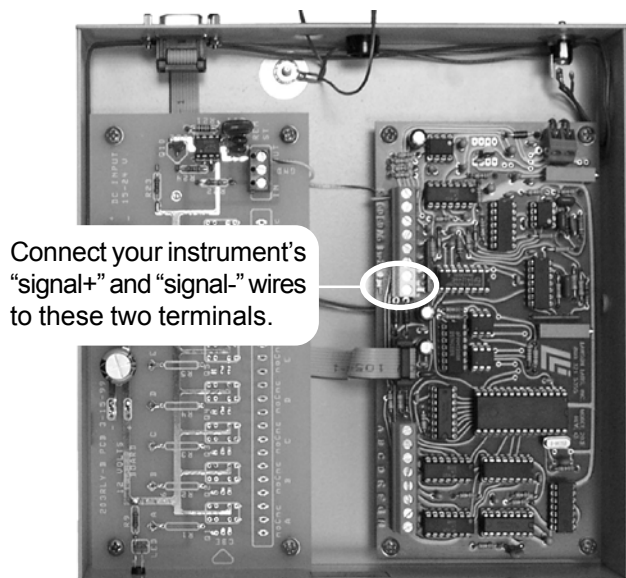
STEP 1: Connect the Analog Signal Cable

1-1. Route the analog signal cable from your instrument through the open hole in the back of the Model 203.

1-2. Strip 1/4" of insulation from the "signal+" and "signal-" wires of your instrument's signal cable.



Route wires through this hole



Connect your instrument's "signal+" and "signal-" wires to these two terminals.

1-3. Insert "signal+" into the Lawson 203 board screw terminal marked "signal+" and secure the connection with a small flat-blade screwdriver.

1-4. Insert "signal-" into the Lawson 203 board screw terminal marked "signal-" and secure the connection.

STEP 2: (OPTIONAL) Connect the Remote Start Cable

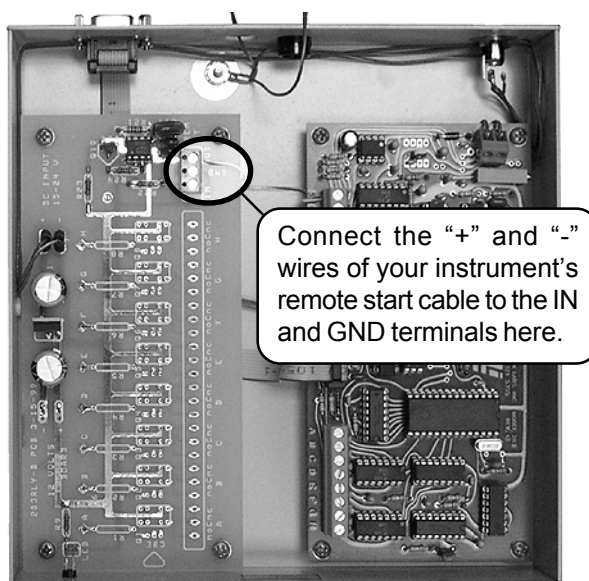
The Model 203 remote start capability allows you to start the data system by means of a switch closure. In some applications, the chromatograph being used with the Model 203 may offer a remote start signal output or switch closure output that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the Model 203.

2-1. Route the remote start cable from your instrument through the open hole in the back of the Model 203.

2-2. Strip 1/4" of insulation from the "+" and "-" wires of your remote start cable.

2-3. Insert the "+" wire into the 203RLY board screw terminal marked "IN" and secure the connection.

2-4. Insert the "-" wire into the 203RLY board screw terminal marked "GND" and secure the connection.



Connect the "+" and "-" wires of your instrument's remote start cable to the IN and GND terminals here.

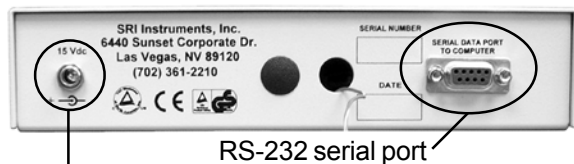
Replace the Model 203 cover and secure it with the thumbscrews.

Quick Start

Model 203 Single Channel PeakSimple Data System

STEP 3: Connect the Serial Cable to Your Computer

The Model 203 is equipped with a RS-232 serial port. A DB-9 type serial cable (provided) connects the Model 203 to your Windows™ computer through the PC's COM port. This simple interface permits the Model 203 to be operated from a desktop or laptop computer.



DC power input

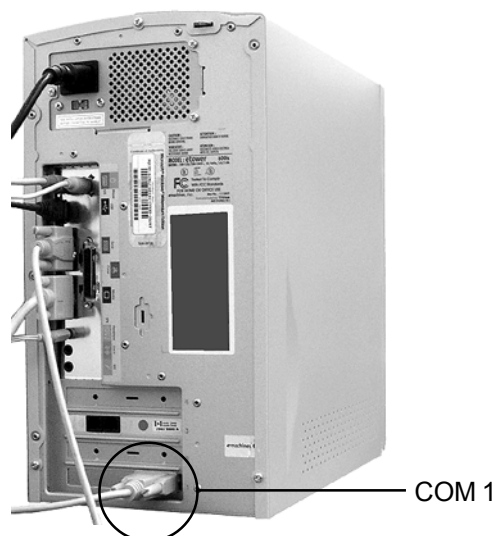
RS-232 serial port

Serial cable (DB-9 type)



3-1. Secure one end of the serial cable to an available COM port on your PC.

3-2. Secure the other end to the RS-232 serial port on the back of the Model 203.



COM 1

STEP 4: Connect Power to the Model 203

Model 203 units are provided with a 15 V DC power supply which plugs into a standard wall volt outlet. Plug the power supply output plug into the back of the Model 203 and plug the power supply into the wall outlet. Verify that the POWER LED on the front of the Model 203 is lit.

POWER LED



15 V DC
power supply

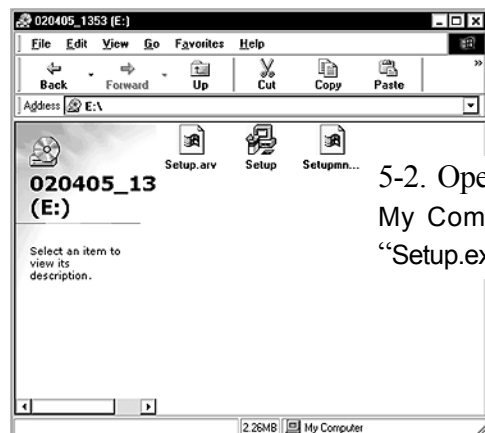


Quick Start

Model 203 Single Channel PeakSimple Data System

STEP 5: Install PeakSimple Chromatography Software

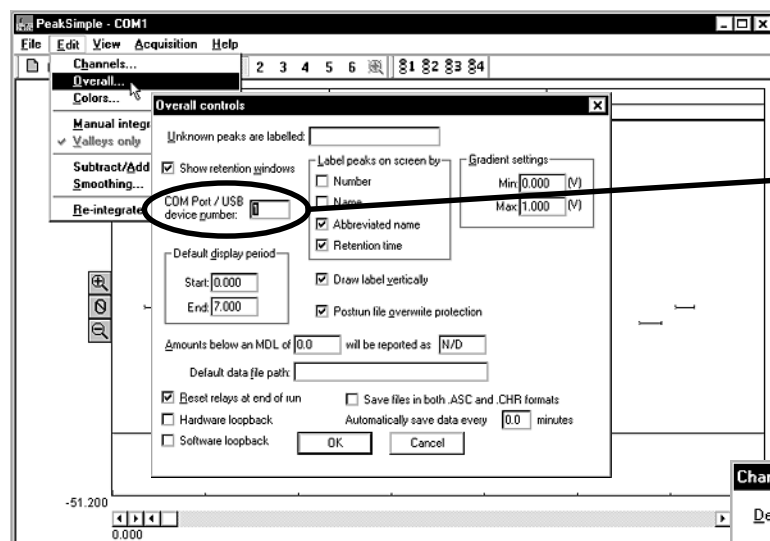
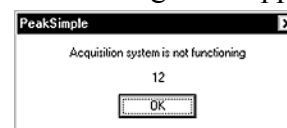
5-1. Locate your copy of the PeakSimple software, which is shipped inside the front cover of your SRI manual. Insert the CD or floppy disk(s) into your computer's appropriate drive.



5-2. Open the appropriate drive through My Computer, then double click on "Setup.exe" and follow the instructions.

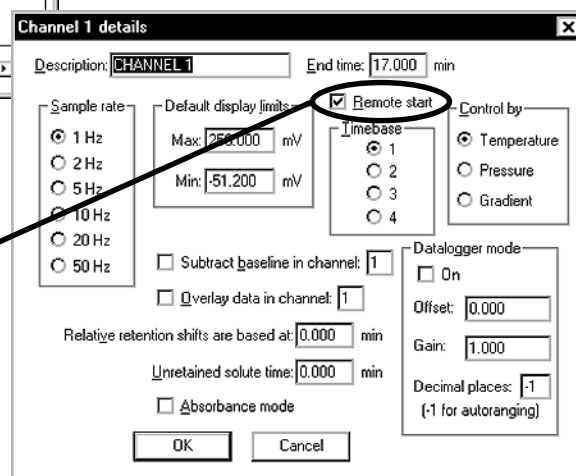


5-3. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between your computer and the Model 203. An error message will appear if communication is not established.



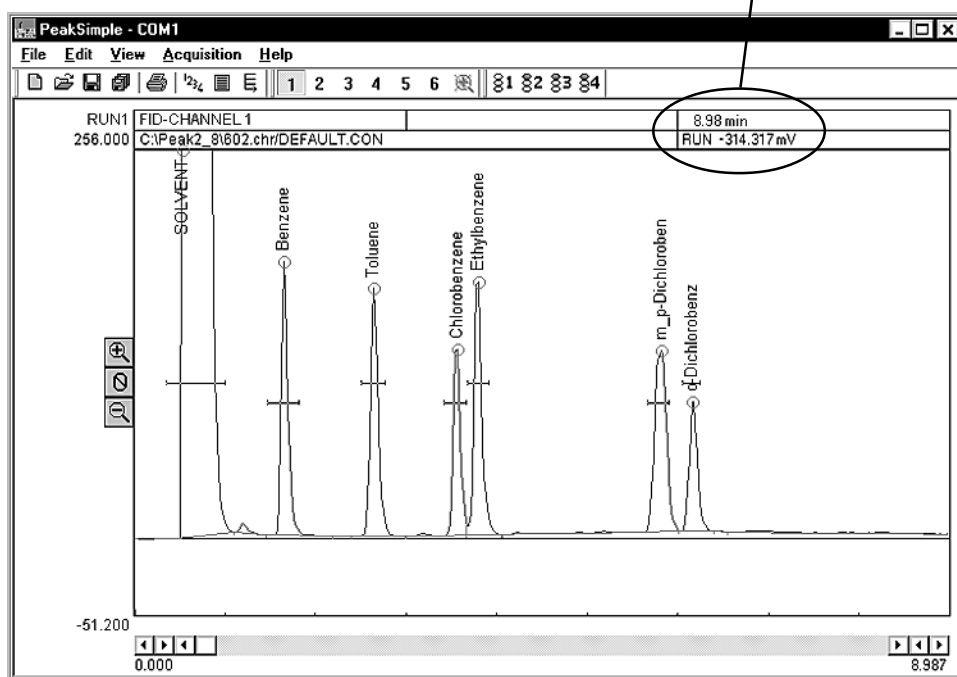
5-4. PeakSimple defaults to COM 1. If you did not connect the Model 203 to COM 1, you will get the error message. Open the Edit menu and choose Overall. In the dialog box that appears, enter the number of the COM port to which you have connected the Model 203. If you do not know the number of the COM port to which you connected the 203, use the process of elimination: try different numbers until you find one that works.

5-5. For the remote start option: Open the Edit menu and choose Channels. Click on the Details button for channel 1. Verify that Remote start is enabled (the box should be checked).



STEP 6: Starting an Analysis

6-1. The upper right corner of the PeakSimple chromatogram window contains real-time information pertinent to your analysis in progress. The status of the run (RUN, STAND BY) is displayed in capital letters next to the millivolt (mV) reading, underneath the amount of time into the run.



6-2. Hit your computer keyboard spacebar to begin the run, and the data is plotted onscreen in the chromatogram window.

6-3. Hit the End key on your computer keyboard to stop the run.

More on PeakSimple:

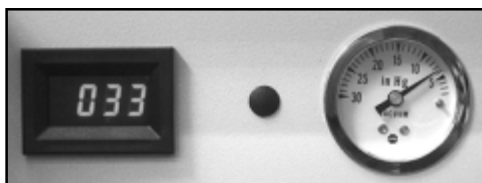
This Quick Start guide presents a very brief introduction to PeakSimple. There are tutorials in the manual and online at www.srigc.com (click on the "Download Our Documents" button) that will acquaint you with PeakSimple's basic functions.

If you have questions or problems, call SRI for free technical support at 310-214-5092, 8am - 5pm California time.

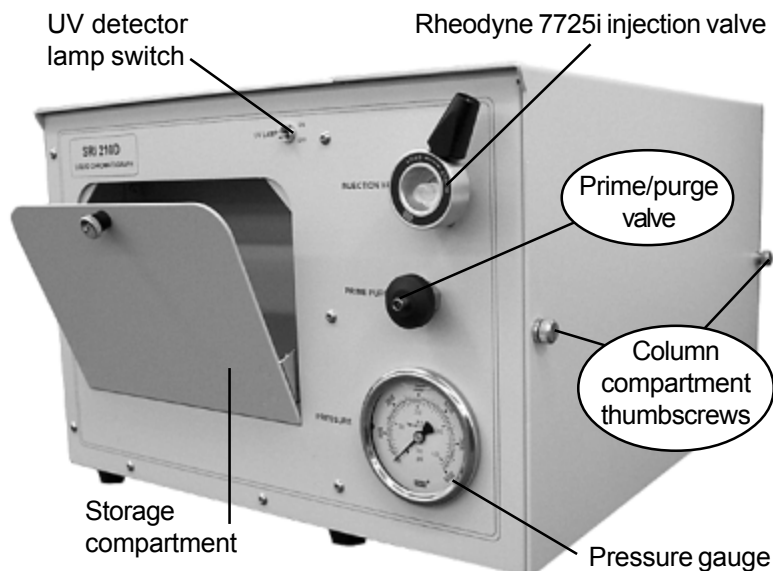
HPLC Model 210D

1. Overview

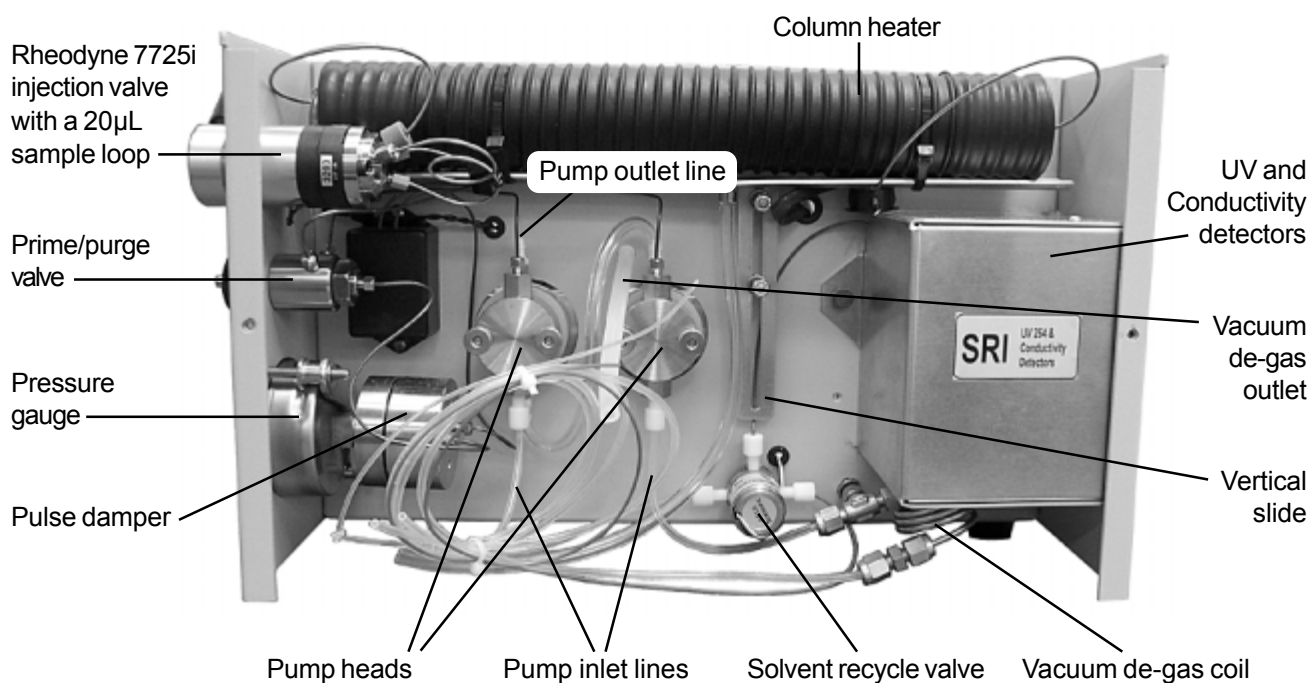
The Model 210D front panel houses the UV detector lamp switch, Rheodyne injection valve, prime/purge valve, pressure gauge, and the storage compartment. In the storage compartment are the LED display for the column temperature, and the vacuum degas pressure



gauge (shown above). An accessories kit is included with your 210D. In the kit are two PEEK finger tight nuts (Alltech part # 32233) for column connection, a spare 1/16" stainless steel fitting (Valco part ZN1) and ferrule (Valco part ZF1S6), a flange fitting (Upchurch part 203X) and ferrule (Upchurch part 240X) for the solvent recycle valve, a 20mL priming syringe (Sigma Aldrich part Z248037), and a 100µL glass injection syringe (SGE part 005300).



On the right hand side of the 210D is the column compartment, which gives you access to the column heater, pump head(s), solvent recycle valve, and the detector housing. Open the column compartment by loosening the two captive thumbscrews and lifting off the cover. Use the picture below to familiarize yourself with the interior of the column compartment.

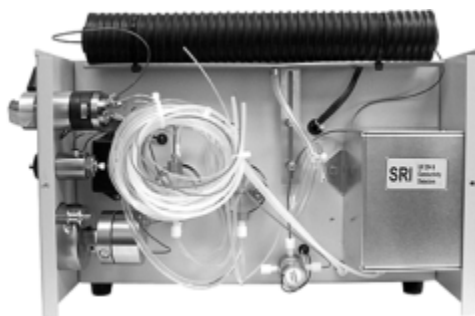


HPLC

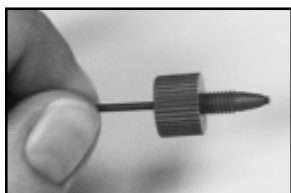
Model 210D

2. Installing a Column

Slide the column heater up.

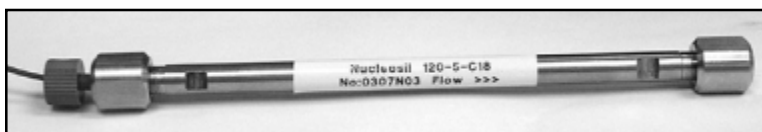


Slide the column heater up on its aluminum slider.

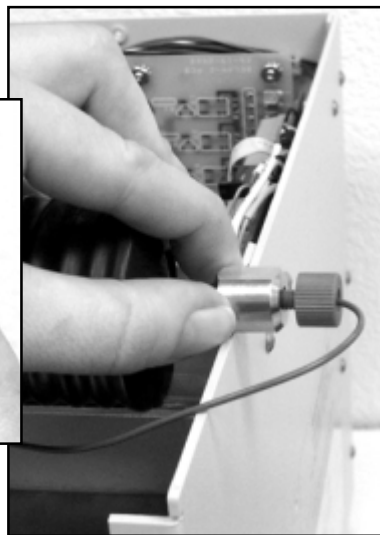
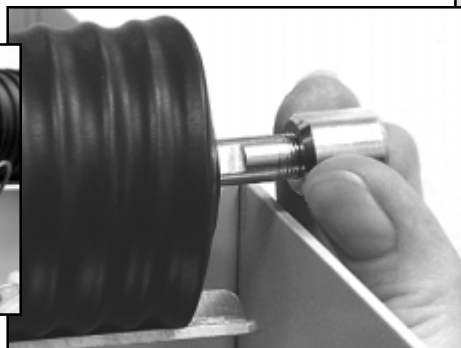
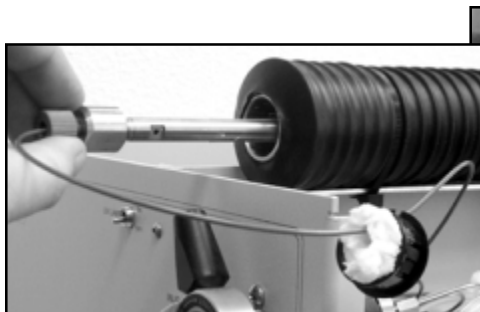


Install the column of your choice using the included PEEK finger tight nuts. Slide the nuts onto the PEEK tubing, letting about 1 mm of PEEK tubing protrude.

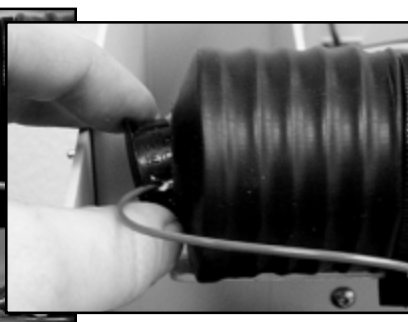
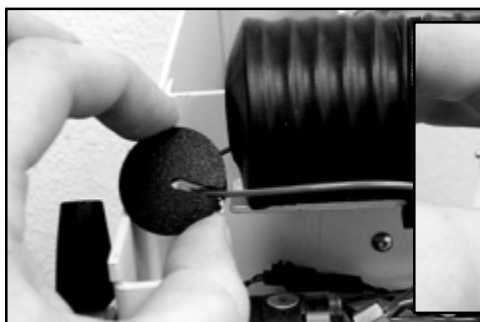
Secure one nut to the column.



Slide the column through the column heater so you can reach the other end, and attach the second nut. Roughly center the column in the column heater.

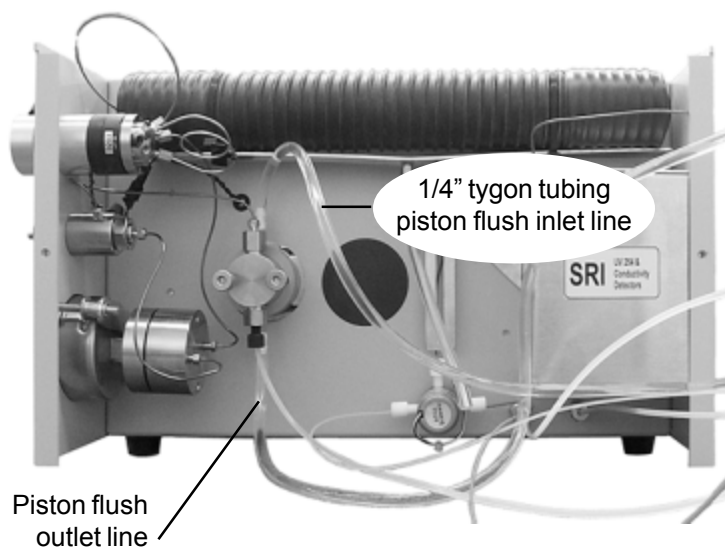
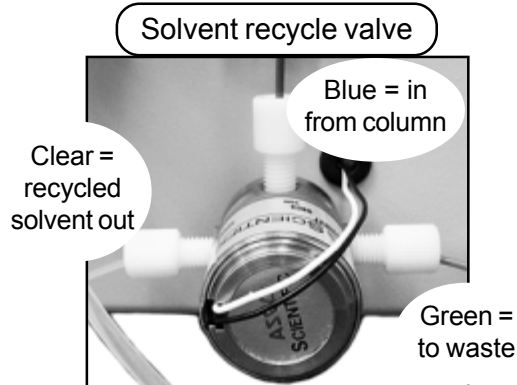


Replace the black column heater end caps, which have notches in them for the PEEK tubing. Pack in any loose insulation. When you have replaced both end caps as shown below, slide the column heater back down into the operating position.



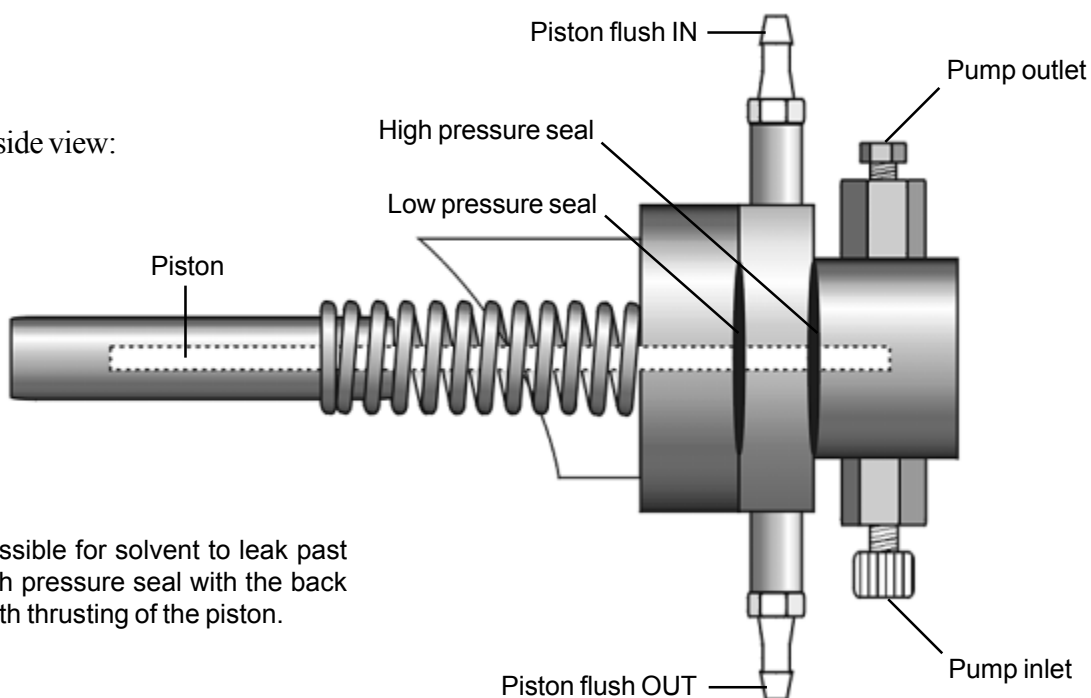
3. Solvent Set-up

The pump heads each have an 1/8" Teflon solvent inlet line. Place the ends of these lines in your solvent bottle. The solvent recycle valve has two outlet lines. The 1/16" clear line is recycled solvent. Put it into the solvent bottle, or into its own container if you wish to keep it separate from your fresh solvent. Place the end of the 1/16" green line into a waste bottle. A simplified way to remember: all the clear lines go to the solvent bottle. In default mode, the solvent recycling valve sends the effluent out the clear tube; when activated, it directs the effluent out the green waste tube.



The 1/4" Tygon tubing attached to the pump head(s) is the piston flush line. Piston flushing helps keep the high pressure seal(s) clean, minimizing wear and tear from crystallized and abrasive buffer deposits that leak through the seal, and would otherwise dry on the piston. There are inlet and outlet lines for piston flushing. Place the ends of both into a bottle containing at least 50% organic solvent (usually diluted with deionized water). Binary gradient pump head piston flush lines are daisy chained, and share the same in and out lines.

210D pump, side view:



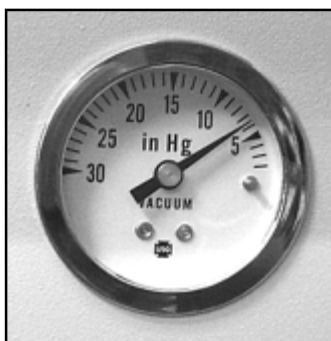
It is possible for solvent to leak past the high pressure seal with the back and forth thrusting of the piston.

HPLC Model 210D

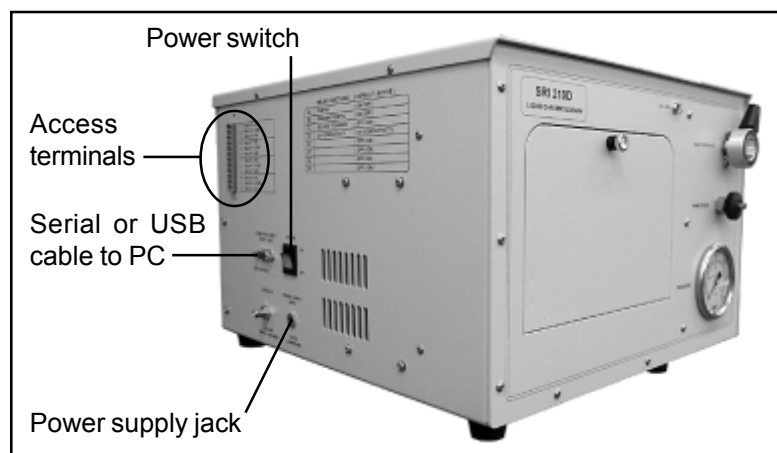
4. Vacuum De-gas

While not all HPLC users degas their solvent, it is a good idea to do so. The 210D is equipped with an air pump for vacuum degassing. A length of tygon tubing is provided to connect the pump to the degas fitting on the left-hand side of the HPLC. The tubing is equipped with a nut for easy connection to the degas fitting. With the 210D power ON, connect the pump tubing to the degas fitting, then plug the pump into a wall outlet. Open the storage compartment to see the vacuum degas gauge, which should read 7psi with the pump ON and solvent lines attached.

The vacuum degas gauge should read 7psi during operation.



Simply screw this nut onto the degas fitting to connect the vacuum pump to the 210D.



5. Computer Connection

Connect your 210D to your Windows™ computer with the provided serial or USB cable. The cable connection port is located on the left-hand side of the 210D.

6. Power Supply

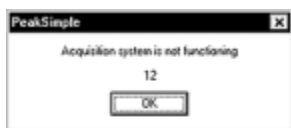
Your 210D comes with a universal (115V or 230V) benchtop 12 volt power supply (shown at right). Plug the small power source cord into the jack on the left hand side of the LC. Plug the larger power cord into a wall outlet. Turn the power ON with the power switch, located above the power supply jack. The 210D can be powered with any regulated or unregulated voltage in the range of 11-15 volts at 80 watts (approximately 7 amps), including a car battery via the cigarette lighter.



7. Installing PeakSimple software



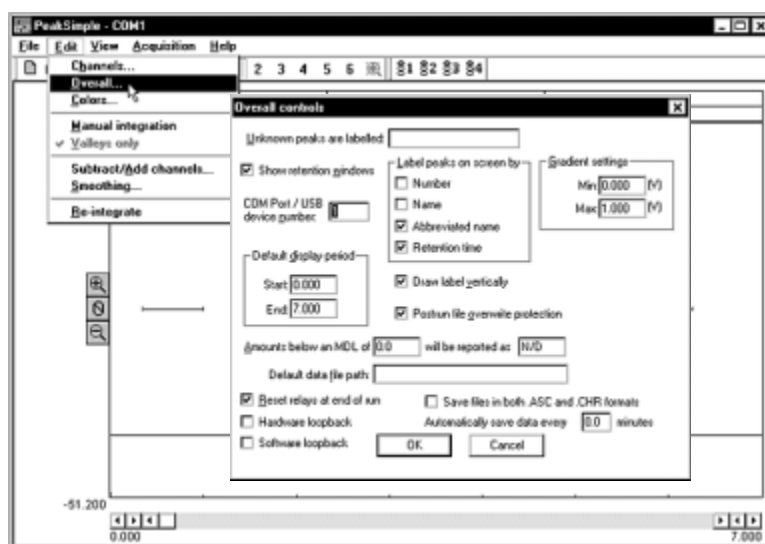
You will find your PeakSimple installation disk just inside the front cover of your manual. Install the PeakSimple software by inserting the PeakSimple CD into the CD-ROM drive of your Windows™ computer. Double-click on My Computer, then open the CD-ROM drive. Double-click on the “setup.exe” program icon and follow the instructions.



8. Double-click on the PeakSimple icon to launch the program. Verify that communication is established between the computer and the pump. An error message (shown at left) will appear if communication is not established.

9. Load the LC.CON control file from your PeakSimple program folder. This control file contains all the necessary information to configure PeakSimple for HPLC.

10. Verify that the correct I/O port is specified in the Edit/Overall screen. By default, COM 1 is entered in the Edit/Overall screen because many Windows™ computers have COM 1 designated as the serial port. Other computers may use COM 2, COM 3, or COM 4. You may have to examine the My Computer/System screen to determine what serial port numbers Windows has assigned to the hardware in your particular computer. If you upgraded to the 6 channel USB data system, enter the USB device ID number in the box instead. USB device ID numbers are unique to each instrument, and the number for your 210D is printed on the HPLC under the cable port, and also on your PeakSimple disk. Once you have successfully gotten the data system to sign on, click Save All so you don't have to repeat this step the next time you open PeakSimple.



HPLC

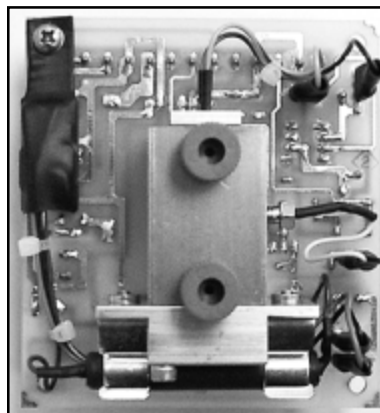
Model 210D

11. UV Detector

The UV detector signal is connected to channel 1. The detector uses a low volume flow-through cell. The cell temperature is factory set to 40°C, which is suitable for a wide range of common applications.

Note for those who have the 4 or 6 channel data system upgrade:
Unless you have connected an external detector to the 210D, the column temperature will display on channel 3, and the detector cell temperature will display on channel 4.

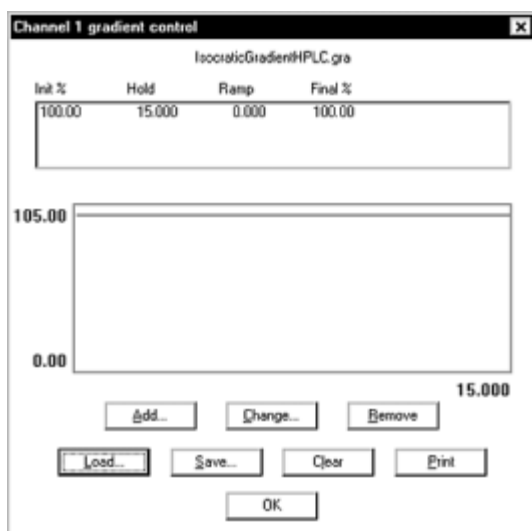
Both detectors are housed in the cell in the middle of this board.



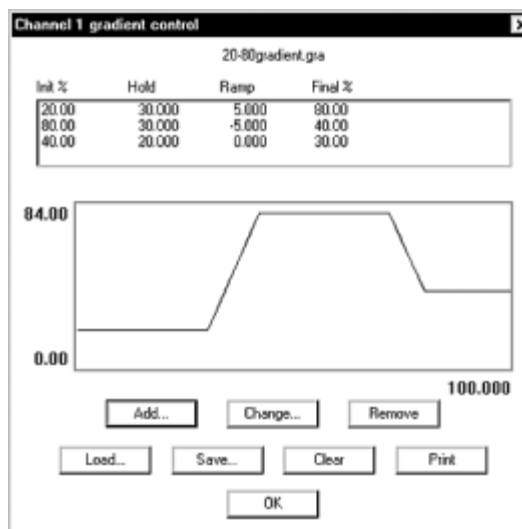
12. Setting a Gradient Program

Click on the Gradient button for channel 1. If your system is isocratic, the Initial and Final percentages are 100%. The only variable is the Hold time period, which is determined by the length of your analytical run. Setting a gradient program automatically starts the pump at a very low speed. For binary gradient systems, load a gradient program from your PeakSimple program folder (version 3.19 and later) by clicking on the Load button in the Channel 1 gradient control window. Use the Add button to create a new gradient.

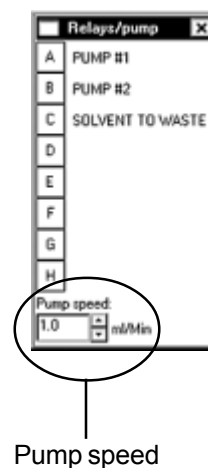
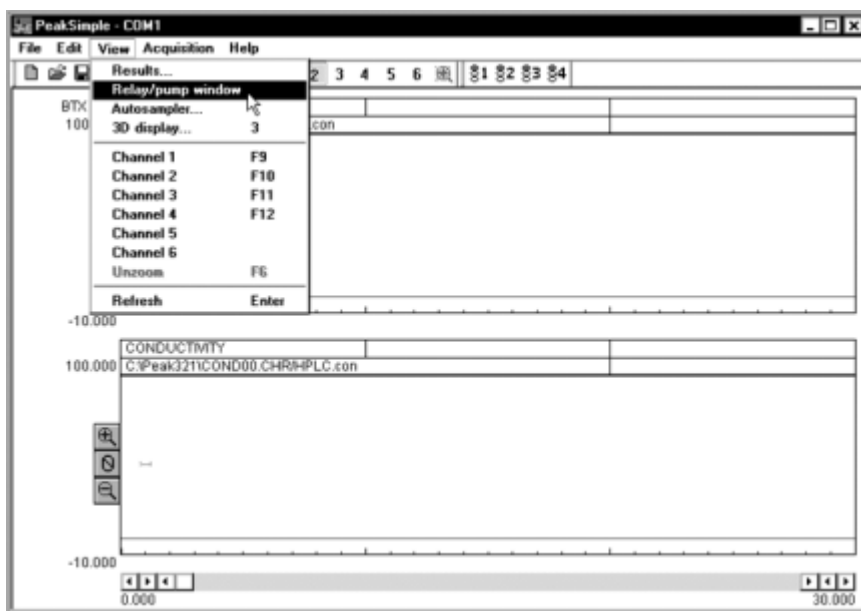
Isocratic gradient



Binary gradient



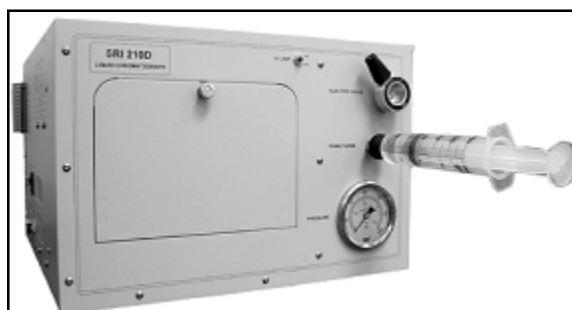
13. Click View and choose Relay/pump window. In the Relays/pump window, click on Relay A to turn the pump OFF, since you haven't primed it yet. The flow rate of the pump (pump A in a binary gradient system) can be adjusted from 0.1 to 5.0mL/minute, in increments of 0.1mL/minute, using the dialog box at the bottom of the Relays/pump window. In a binary gradient system, the gradient program determines how the flow is split between the two pumps.



14. Priming the Pump

Note: Do not open the prime/purge valve until pressure is at zero, or very close. Otherwise, system pressure could damage the pulse damper or cause the column adsorbent bed to shift.

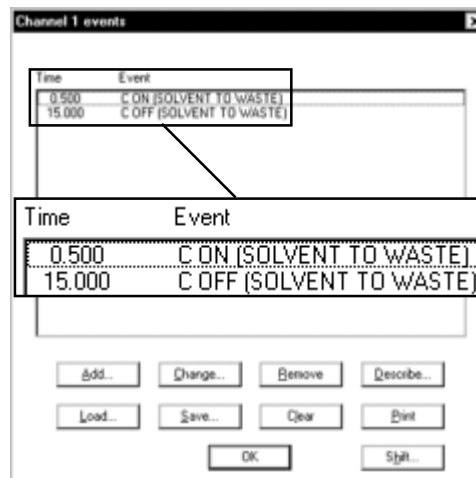
Open the prime/purge valve by turning it to the left, then start the pump by clicking on Relay A in the pump window. Insert the priming syringe into the prime/purge valve, then slowly and gently pull back the plunger. When you can pull a bubble-less stream out of the prime/purge valve, close the valve by turning it back to the right. The pressure should begin building to operating range, which is dependent upon the flow rate, solvent viscosity, and the back pressure of the column selected. When the system stabilizes at the operating pressure range, you may inject your sample. It is normal for the pressure gauge needle to fluctuate with the pump stroke.



HPLC Model 210D

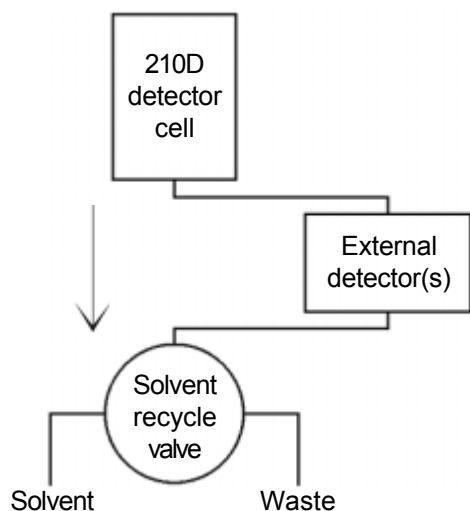
15. Solvent Recycle Control

Relay C controls the solvent recycle valve. When Relay C is ON, the solvent is directed to waste (green exit tube). Turn Relay C ON before any peaks appear onscreen, and turn it OFF at the end of the run. Click on Relay C in the Relays/pump window to turn it ON and OFF, or use an event table to do it automatically. The solvent recycle valve may also be used as a single peak fraction collector by turning it ON/OFF at specific times during the run.



Time	Event
0.500	C ON (SOLVENT TO WASTE)
15.000	C OFF (SOLVENT TO WASTE)

Example event table for the
solvent recycle valve



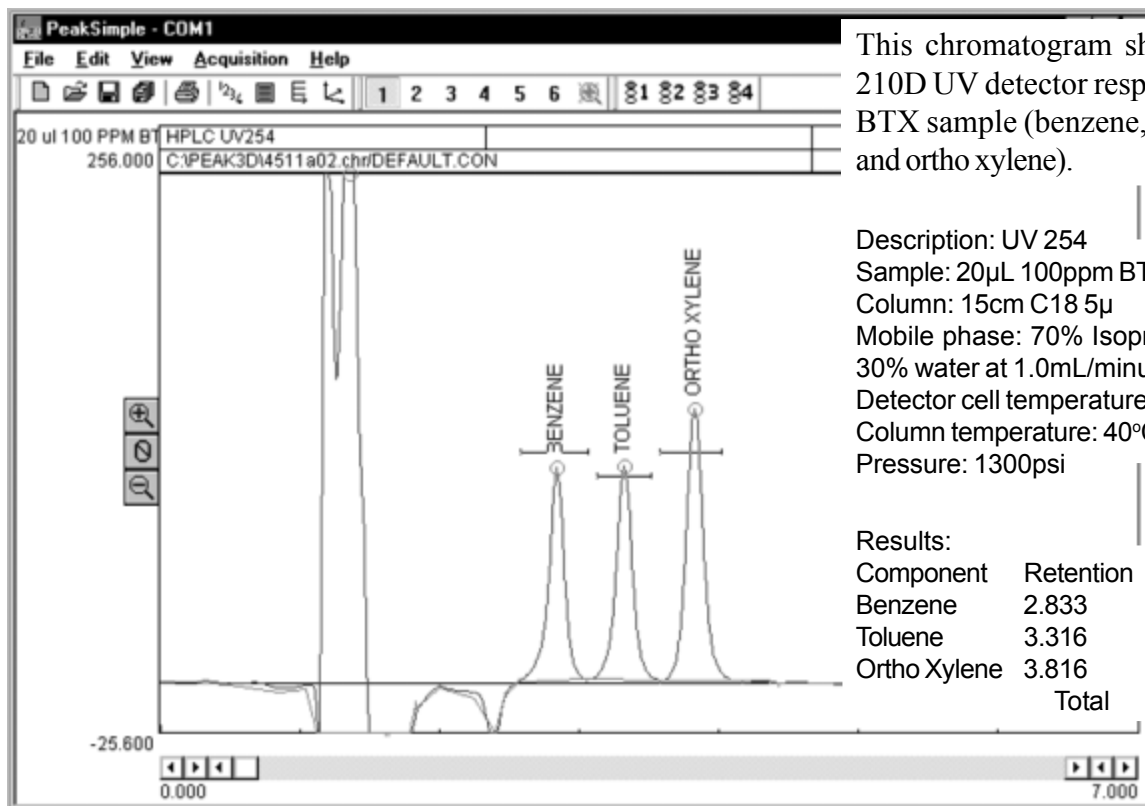
The detector exit tube that connects to the top of the solvent recycle valve may be disconnected, and connected to external detectors, like refractive index or fluorescence detectors. The external detector's exit tubes are then connected back to the solvent recycle valve.

16. Injecting a Sample and Starting the Run

The Rheodyne injection valve comes with its own blunt-tipped flushing needle, which is shipped inserted into the valve under the protective red cap. Remove the protective cap and tape, then pull out the needle. The provided 100µL glass injection syringe should suit most injection applications. For the best results, prepare your sample in the same solvent you are using for your mobile phase. Fill the syringe with sample, and eliminate any air bubbles. Insert the syringe into the Rheodyne injection valve, pushing gently until the needle hits the stop. Depress the syringe plunger to fill the 20µL sample loop. Leave the syringe in the while you turn the Rheodyne injection valve knob to the INJECT position. The run will automatically start. See the Rheodyne documentation in your SRI manual under HPLC Injectors for more information about operating the injection valve.



Expected Performance

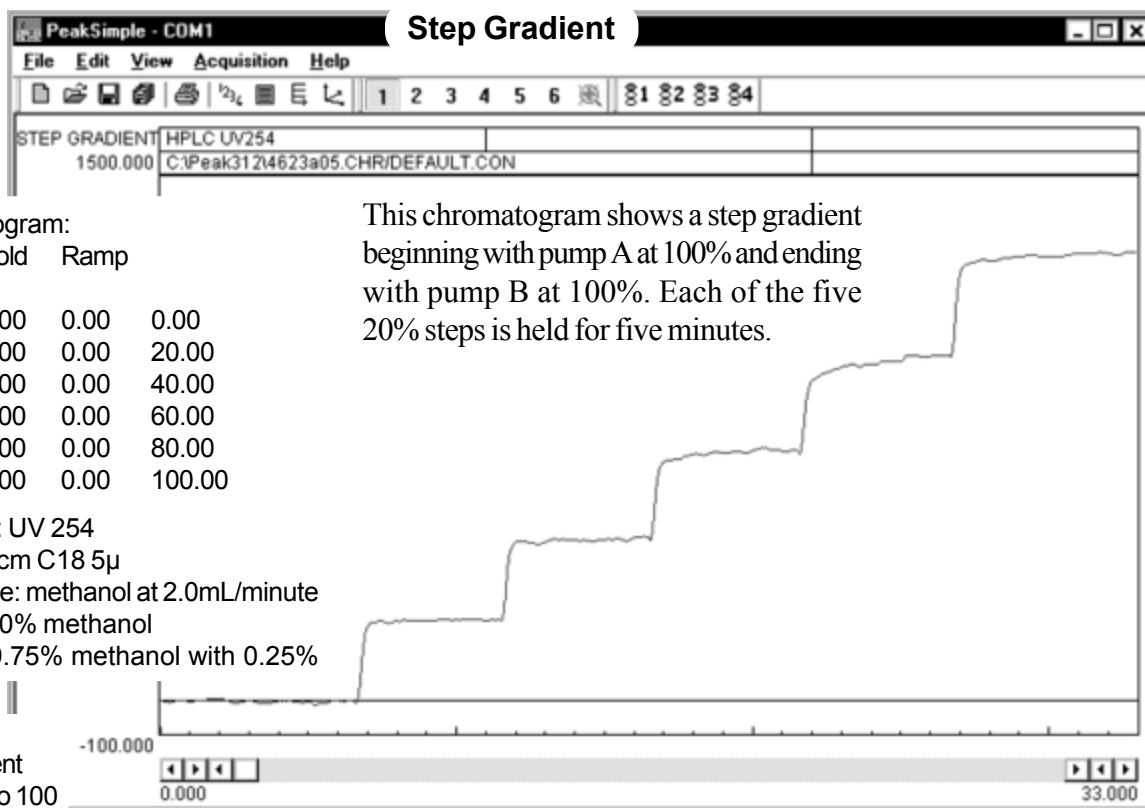


This chromatogram shows the 210D UV detector response to a BTX sample (benzene, toluene, and ortho xylene).

Description: UV 254
Sample: 20µL 100ppm BTX
Column: 15cm C18 5µ
Mobile phase: 70% Isopropyl Alcohol, 30% water at 1.0mL/minute
Detector cell temperature: 40°C
Column temperature: 40°C
Pressure: 1300psi

Results:

Component	Retention	Area
Benzene	2.833	851.1640
Toluene	3.316	912.9170
Ortho Xylene	3.816	1207.1795
Total		2971.2605



Gradient program:

%pumpA	Hold	Ramp	%pumpB
100.00	5.00	0.00	0.00
80.00	5.00	0.00	20.00
60.00	5.00	0.00	40.00
40.00	5.00	0.00	60.00
20.00	5.00	0.00	80.00
0.00	5.00	0.00	100.00

This chromatogram shows a step gradient beginning with pump A at 100% and ending with pump B at 100%. Each of the five 20% steps is held for five minutes.

Description: UV 254
Column: 15cm C18 5µ
Mobile phase: methanol at 2.0mL/minute
Pump A: 100% methanol
Pump B: 99.75% methanol with 0.25% acetone

Events:

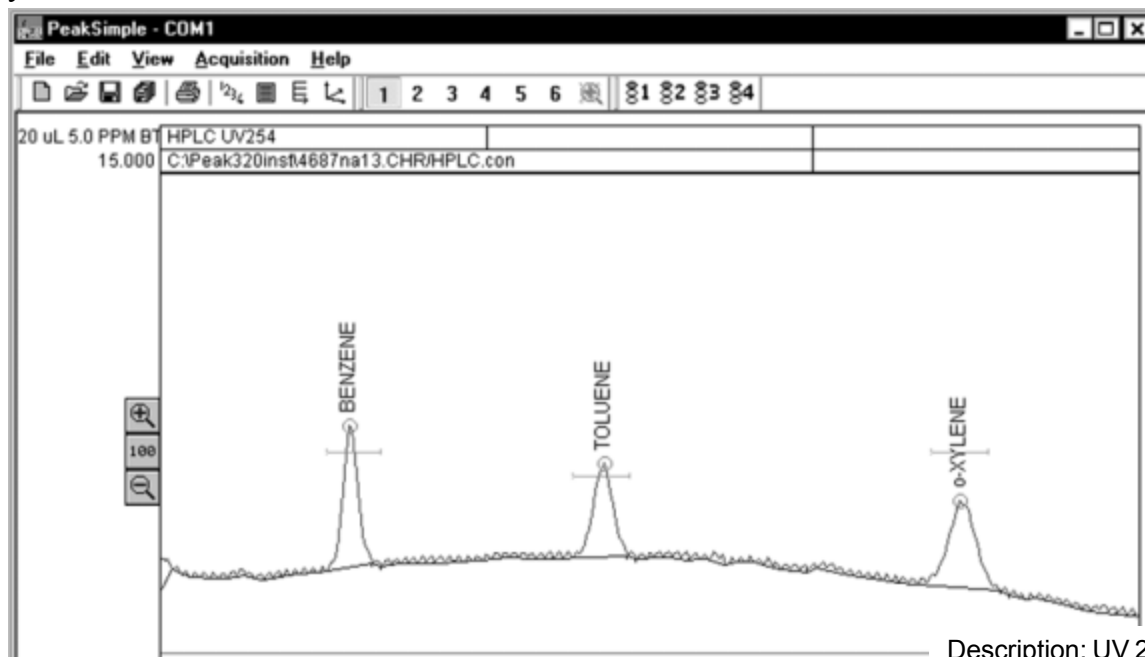
Time	Event
0.00	Zero 100

HPLC

Model 210D

Expected Performance

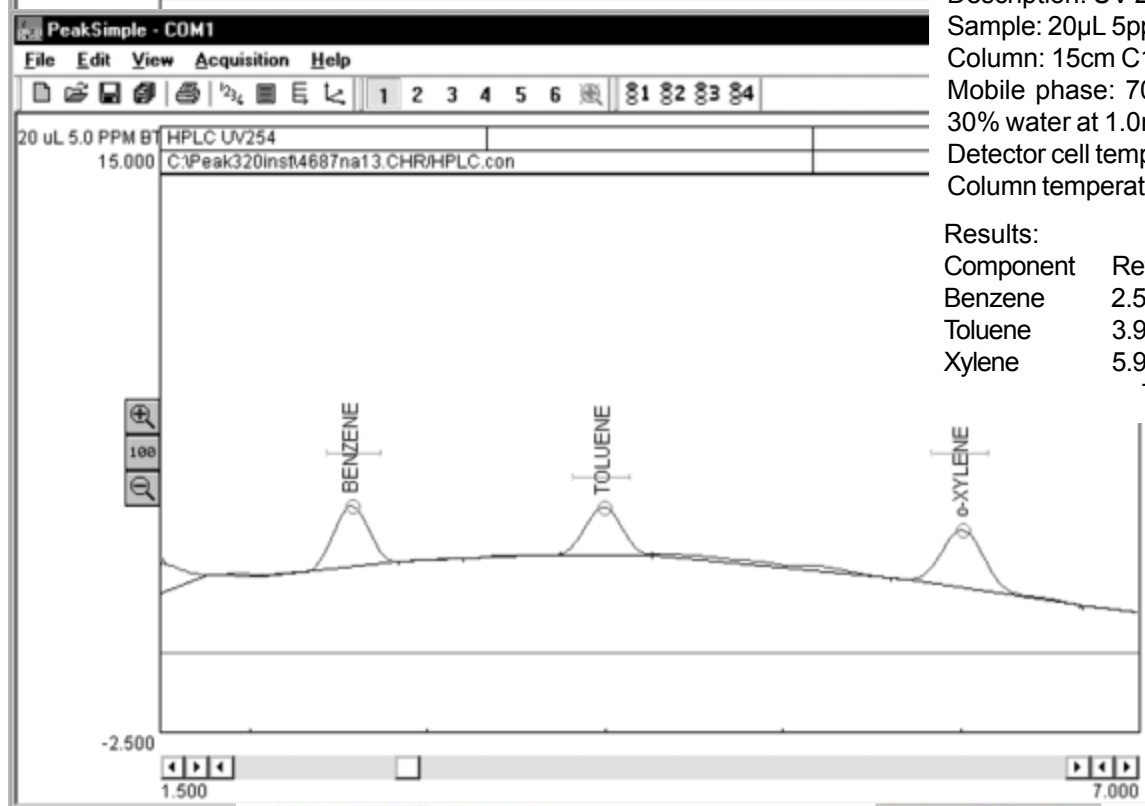
The first chromatogram is a UV separation of 5ppm BTX with an isocratic 210D system. The second chromatogram shows the same run after using PeakSimple's data smoothing function. Short term up/down noise is a function of pump refill. Binary systems are therefore noisier than isocratic systems. PeakSimple's smoothing feature can reduce the pump noise for a clearer picture of your data. The data in any channel can be smoothed automatically using Post-run actions for that channel. See the "PeakSimple Software" section of your manual for more information.



Description: UV 254
Sample: 20µL 5ppm BTX
Column: 15cm C18 5µ
Mobile phase: 70% Methanol,
30% water at 1.0mL/minute
Detector cell temperature: 40°C
Column temperature: 40°C

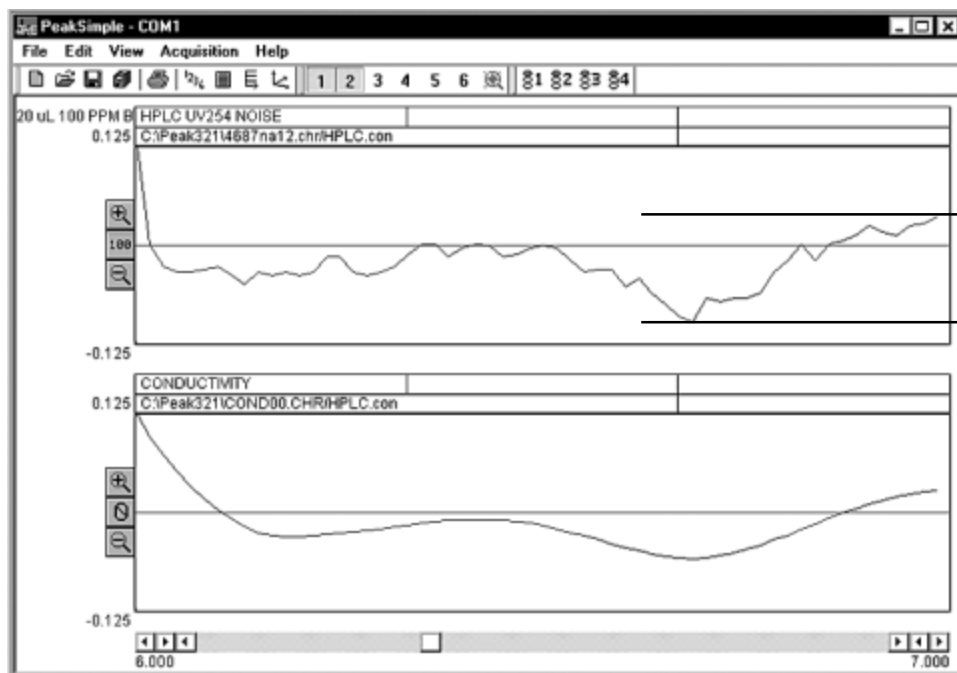
Results:

Component	Retention	Area
Benzene	2.550	26.01
Toluene	3.983	22.69
Xylene	5.983	29.90
Total		78.60



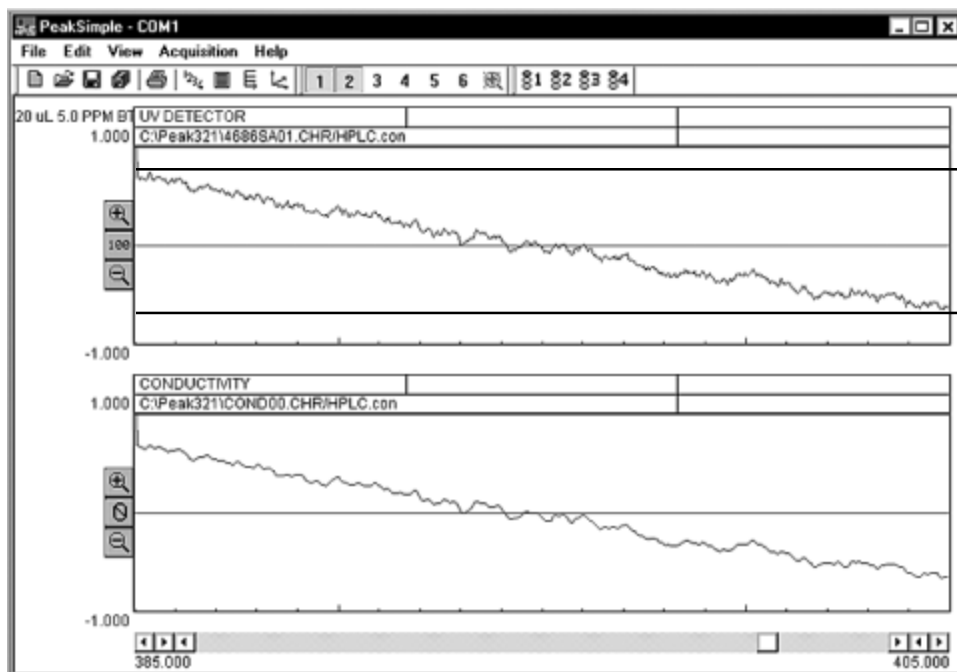
Expected Performance UV Detector Noise

This pair of chromatograms shows a short-term, one minute UV detector noise run on an isocratic 210D system. The first chromatogram is the raw data, and the second shows it after smoothing.



UV detector noise
averages
approximately
100 μV from peak to
peak, or 10^{-4} a.u.
(absorbance units).

This pair of raw and smoothed chromatograms shows 20 minutes of a UV detector noise run on an isocratic 210D system. In the longer time segment, the signal drift is visible.



This UV detector
signal drift
averages
approximately
1.3 mV in 20
minutes, or
0.0013 a.u.

HPLC Model 210D

General Information

Smoothing: Why Use It?

Short term up/down noise is a function of pump refill. Most other manufacturers smooth data inside detector. SRI does not, so pump noise is visible. Binary systems are therefore noisier than isocratic systems. PeakSimple's smoothing feature can reduce the pump noise so you can more clearly picture your data. Pump noise peaks are much narrower than data peaks, and do not obscure, esp after smoothing is applied.



To smooth data, open the chromatogram of your choice in channel 1. Any channel will work, this is just an example. Open the **Data smoothing** window by clicking on the **Edit** pull-down menu, and choosing **Smoothing...** Specify the source channel (the channel in which your chromatogram is open), then specify the channel in which you want the smoothed chromatogram to display. Even if you have a single channel data system, PeakSimple gives you 6 channel screens in which you can open, compare, and edit your chromatograms. Next, choose one of the three available smoothing methods by clicking in the radio button beside it. If you choose Savitzky-Golay, enter the order. Type in the desired filter width and iterations (you may need to experiment to find the best combination for your data). Click the Apply button, and your smoothed chromatogram will appear in the destination channel you designated.

Absorbance VS Transmittance

Other manufacturers make the conversion from transmittance to absorbance in the hardware. To help keep products affordable and portable, SRI does the conversion through PeakSimple software. With the UV detector lamp OFF, the data system signal should be at or very close to zero. Click on the Auto Zero button on the channel 1 chromatogram window to zero the signal at 0% transmittance (0%T). Turn ON the UV lamp switch. The signal should jump to 3-6mV. This signal is 100% transmittance, or 0 absorbance units. Click on the Zero 100 button to zero the signal with just the solvent flowing (this is 100% transmission). Keep in mind that light absorbance is exponential. For example, twice as much toluene does not equal twice as much absorbance. See the conversion chart below:

% Transmittance	Absorbance units	Millivolt signal readings
100%	0	0mV
10%	1	1000mV
1%	2	2000mV
0.1%	3	3000mV
0.01%	4	4000mV
0.001%	5	5000mV

Quick Start

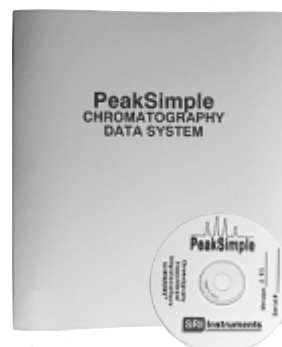
Model 302 Six Channel USB PeakSimple Data System

The **Model 302** may be used with any brand or model of GC or HPLC offering an analog detector output signal ranging from -5V to +5V. It includes three independent, programmable controls (0V to +5V analog output) for temperature & pressure or HPLC gradient formation. The Model 302 has six channels, which can be randomly assigned to one of four time bases, which allows independent start and stop times for four separate instruments. Four remote start inputs compatible with 2-wire switch closures (typically output by GCs and HPLCs as a remote start signal) are also included for your use. Two pulse stretchers are provided to accommodate instruments with remote start signals shorter than one second (such as Hewlett Packard GCs).

The computer to which you connect the Model 302 must support USB (it must have at least one USB port—rev 2.0 or higher—and use Windows™ 98, 98SE, ME, 2000, XP or newer).

With your purchase of the Model 302, you should receive the following items:

- 1 - Model 302 Data System box (front and rear views shown below)
- 2 - USB cable for connection to your computer's USB port
- 3 - Manual (either the PeakSimple Chromatography Data Systems or the SRI general product manual)
- 4 - PeakSimple for Windows™ software (inside the manual cover)



The Model 302 comes in a sturdy aluminum box consisting of top and bottom halves, secured together with two brass thumbscrews for easy interior access.



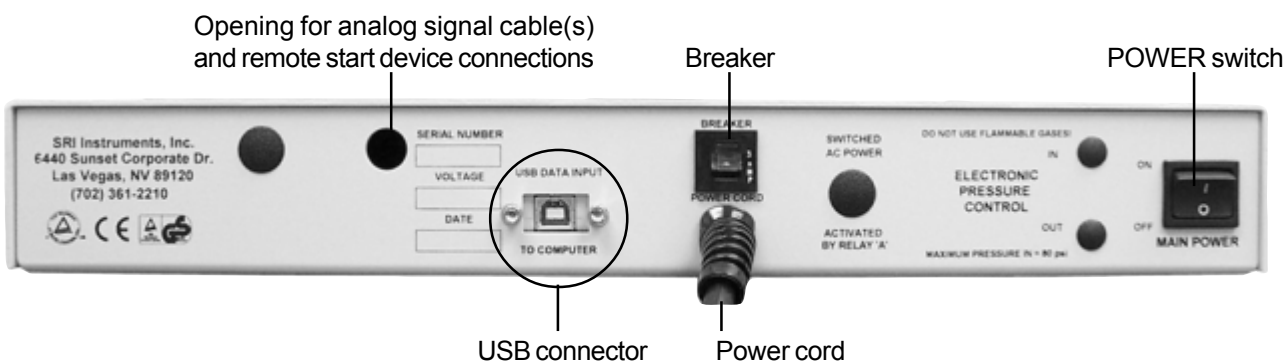
The brass thumbscrews are on the left- and right-hand panels of the Model 302 box.

Front View



POWER Indicator LED

Rear View

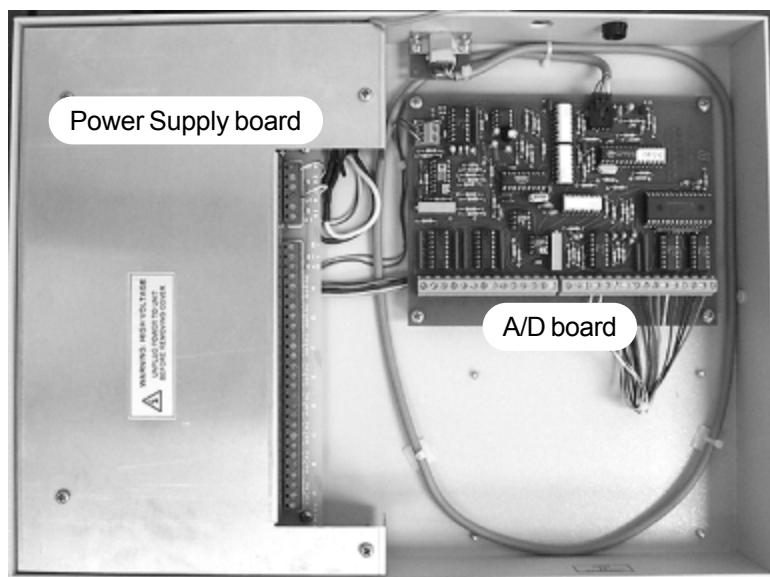


Quick Start

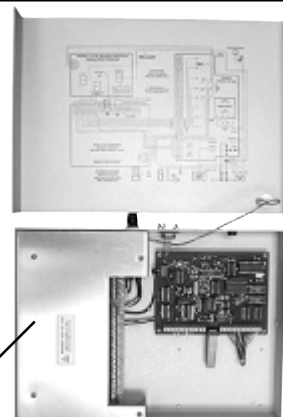
Model 302 Six Channel USB PeakSimple Data System

1. Open the Model 302

Verify that the Model 302 is powered OFF and unplugged. Remove the thumbscrews on both sides of the Model 302 box and slide the top cover up and off. It is connected to the bottom of the box by a ground wire, so just set it next to the bottom half of the box.



High voltage aluminum safety cover

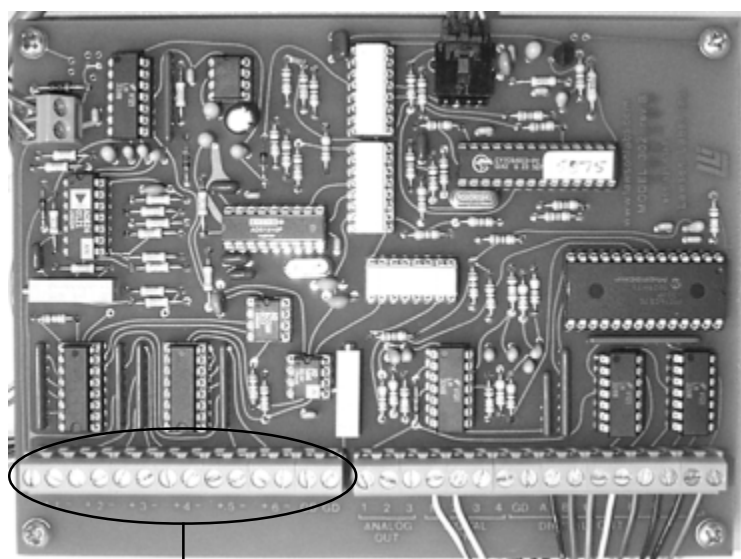


The Model 302 box contains two circuit boards. The board on the right-hand side is the A/D board. The board on the left-hand side under the removable high voltage aluminum safety cover is the Power Supply board. **If you need to remove the high voltage aluminum safety cover, ALWAYS unplug the Model 302 from the wall power outlet first** (you do not need to remove it for the wiring connections described here).

2. Connect the Analog Signal Cable(s)

NOTE: The analog output from some GCs and LCs can have a range of up to 10 volts DC. The Model 302 can tolerate this voltage input, but signals above 6 volts will generate unwanted noise and signals above 5 volts will be “clipped” (the tops of the waveforms will be cut off). Use the 1 volt output typically available on the back of your instrument.

2-1. Route the analog signal cables from your instrument through the open hole in the back of the Model 302.



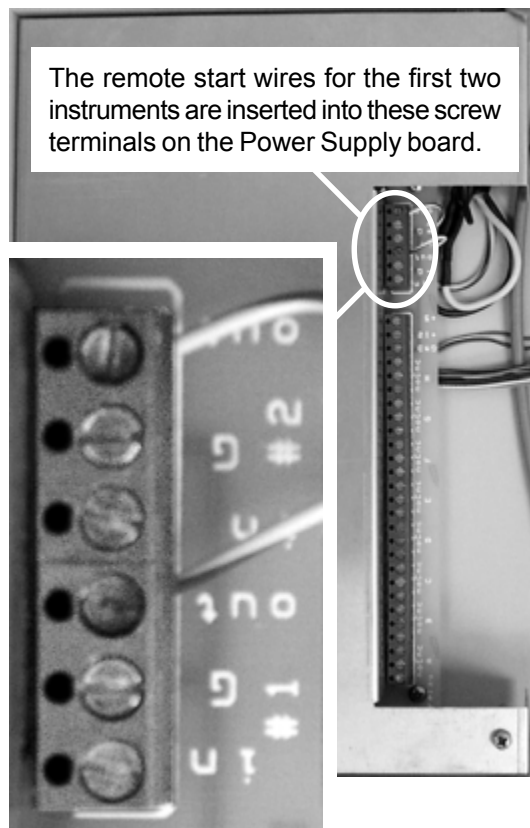
Channels 1-6 and GD (ground) screw terminals

2-2. Strip 1/4” of insulation from the “signal+” and “signal-” wires of your instrument’s signal cables.

2-3. Remove any jumpers placed in the Channels 1-6 screw terminals at the factory. Insert the “signal+” wire into the A/D board screw terminal marked “1 +” and secure the connection with a small flat-blade screwdriver.

2-4. Insert the “signal-” wire into the A/D board screw terminal marked “1 -” and secure the connection.

2-5. Repeat the connection of signal cables for channels 2, 3, 4, 5, and 6. Any unused channels **MUST** have both inputs jumpered to ground.



3. Connect the Remote Start Cables (OPTIONAL)

The Model 302 remote start capability allows you to start the data system by means of a switch closure. Four separate remote start circuits permit the user to individually start TIMEBASE 1, 2, 3, and 4 of the data system. In some applications, the chromatograph being used with the Model 302 may offer a remote start signal output or switch closure output that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the Model 302. TIMEBASES 1 and 2 are equipped with pulse stretchers.

3-1. Route the remote start cable from your instrument through the open hole in the back of the Model 302.

3-2. Strip 1/4" of insulation from the "+" and "-" wires of your remote start cable(s).

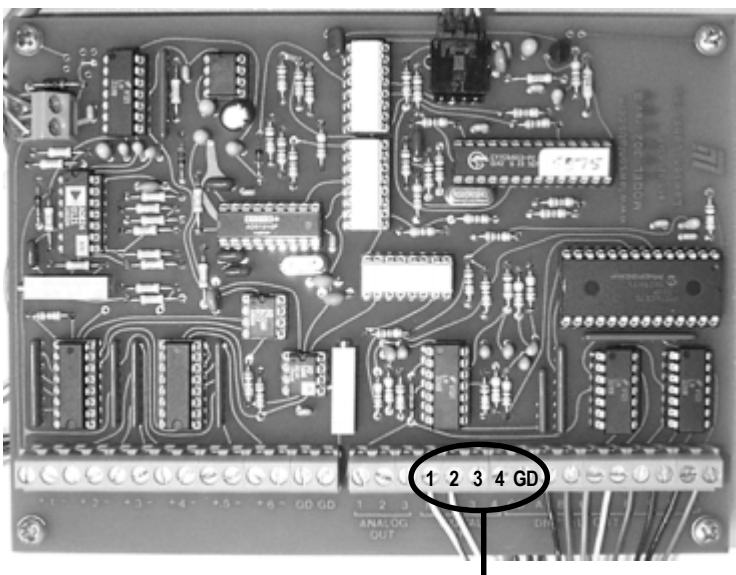
3-3. Insert the "+" wire into the Power Supply board screw terminal marked "#1 IN" and secure the connection.

3-4. Insert the "-" wire into the Power Supply board screw terminal marked "#1 G" and secure the connection.

3-5. For a second instrument, insert the "+" wire into the "#2 IN" terminal, and the "-" wire into the "#2 G" terminal.

3-6. The screw terminals for the third and fourth instruments' remote starts are on the A/D board. The bank of screw terminals is labeled "DIGITAL IN" under "1 2 3 4." Connect the "+" wires for the third and fourth instruments to screw terminals 3 and 4, respectively. Connect both "-" wires to the "GD" screw terminal next to the "4" screw terminal (on the right-hand side).

NOTE: TIMEBASES 3 and 4 require a remote start signal that persists longer for than one second. Check your instruments' specifications (for example, Hewlett Packard GCs produce a very short remote start pulse, so you should connect one of these to TIMEBASE 1 or 2, which are equipped with pulse stretchers).



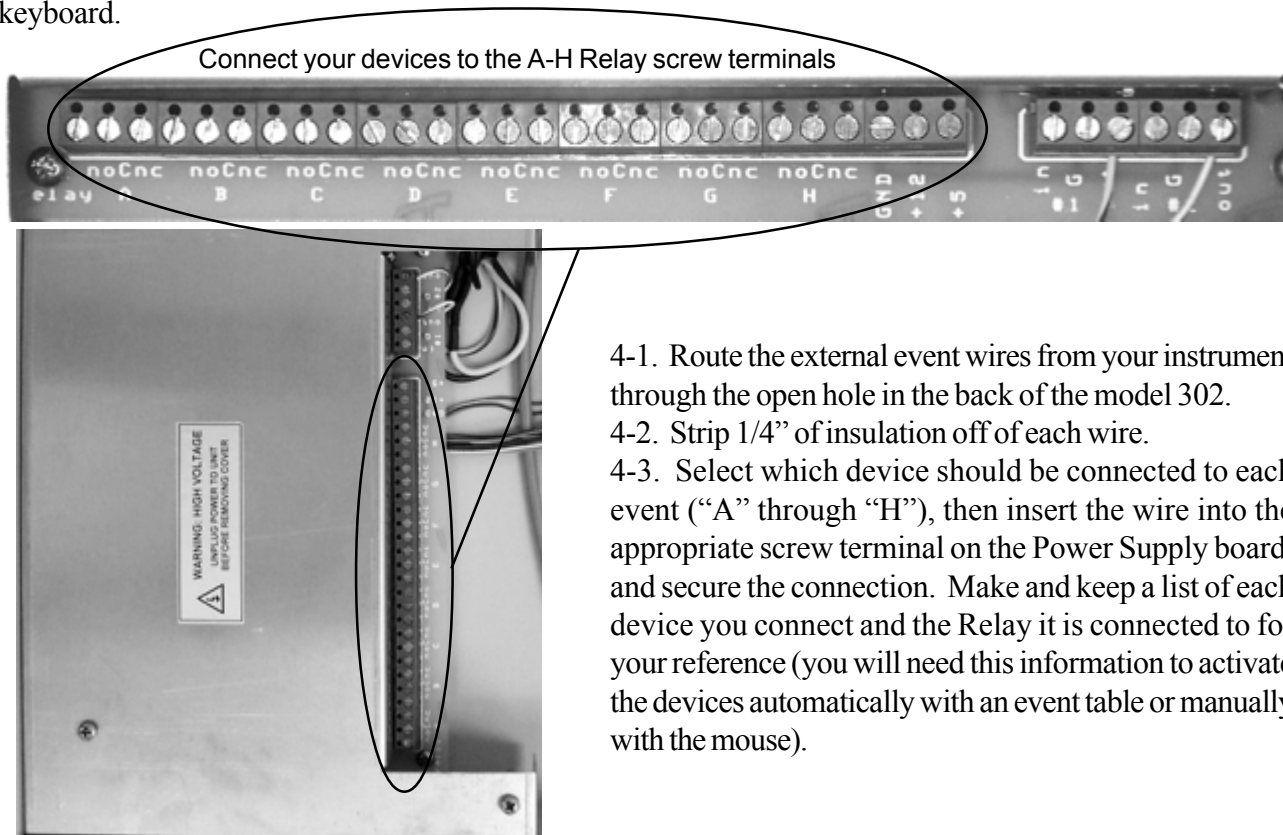
Connect the remote start "+" cables to screw terminals "3" & "4," and the "-" cables to "GD".

Quick Start

Model 302 Six Channel USB PeakSimple Data System

4. Connect the External Event Relay Wires (OPTIONAL)

The Model 302 has eight 0-5 volt TTL level outputs that are wired to a bank of mechanical relays with screw terminals for easy connection to any device which may be operated from a contact closure (normally open [NO] and normally closed [NC] contact closures). These relays may be turned ON and OFF individually and automatically through a PeakSimple timed event table. Manual control is also available via the computer keyboard.



4-1. Route the external event wires from your instrument through the open hole in the back of the model 302.

4-2. Strip 1/4" of insulation off of each wire.

4-3. Select which device should be connected to each event ("A" through "H"), then insert the wire into the appropriate screw terminal on the Power Supply board, and secure the connection. Make and keep a list of each device you connect and the Relay it is connected to for your reference (you will need this information to activate the devices automatically with an event table or manually with the mouse).

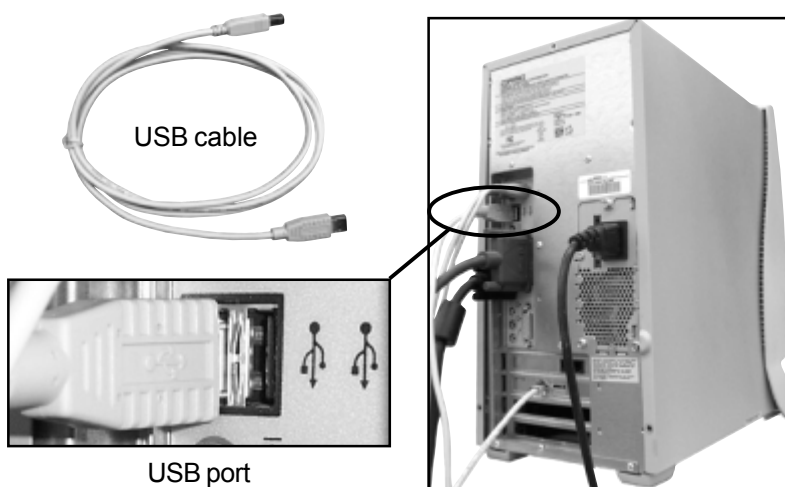
5. Replace the cover on the Model 302 and secure it with the thumbscrews.

6. Connect the USB Cable to Your Computer

The Model 302 is equipped with a USB connector. A USB cable (provided) connects the Model 302 to your Windows™ computer's USB port. This plug and play interface permits the Model 302 to be loaded onto and operated from a desktop or laptop computer that supports USB (rev. 2.0 or higher).

6-1. Secure one end of the USB cable to an available USB port on your PC.

6-2. Secure the other end to the USB connector on the back of the Model 302.



Quick Start

Model 302 Six Channel USB PeakSimple Data System

7. Connect Power to the Model 302

The Model 302 is provided with a power cord which plugs into a standard 110 (or 220) volt outlet. Plug the Model 302 into the wall outlet. Turn ON the power switch and verify that the POWER LED on the front of the Model 302 is lit.



The power LED is lit when the Model 302 is connected to a power source & switched ON.

8. Install PeakSimple Chromatography Software

8-1. Locate your copy of PeakSimple, which is shipped inside the front cover of your manual. Insert the CD or floppy disk(s) into your computer's appropriate drive.



8-2. Open the appropriate drive through My Computer, then double click on "Setup.exe" and follow the instructions. By default, the setup program places the PeakSimple application directory on the hard drive: c:\peak2000. If you put the application directory elsewhere, take note of the path as you may have to enter it in a dialog box during the USB driver installation procedure.

9. Install the USB Drivers

There are three important files saved to the PeakSimple application directory at the conclusion of the software installation: LL_USB.inf, LL_USB.sys, and LL_USB2K.sys. These files are required for Windows to recognize the A/D board connected to the computer's USB port.

9-1. Double-click on the My Computer icon on your desktop, then on Control Panel, then on Add New Hardware, which should open the Add New Hardware Wizard.

9-2. Click the Next button twice, until you get to the screen that gives you a choice between letting Windows find the new hardware, or selecting it yourself from a list. Click the radio button to choose the hardware from a list and click the Next button.



Quick Start

Model 302 Six Channel USB PeakSimple Data System



9-3. Scroll down the hardware list, click on Universal Serial Bus controllers, then click Next. From the following screen click the Have Disk button.



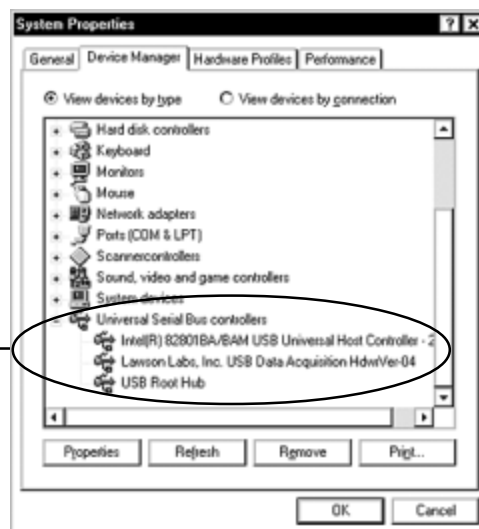
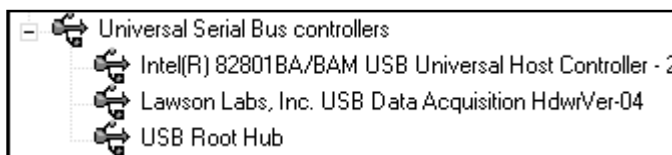
9-4. Click Browse and navigate to the PeakSimple application directory, or type in the path (“c:\peak2000” or the name you have chosen). The Wizard should find the LL_USB.inf file. When you click OK, the Wizard will verify that you want to copy files from the PeakSimple directory (“Copy manufacturer’s files from: c:\peak2000”).



9-5. When you click OK again, the Wizard will confirm that the drivers are for Lawson Labs. Click Next on this screen and the following screen, and Windows will finish installing the software for the Model 302. Click Finish.

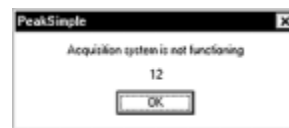



9-6. Restart your computer (you MUST restart your computer before the drivers will work). Open the Control Panel again, then System, then click on the Device Manager tab. If the USB drivers have been successfully installed, the Universal Serial Bus controllers section will list “Lawson Labs, Inc. USB Data Acquisition HdwrVer-04.”

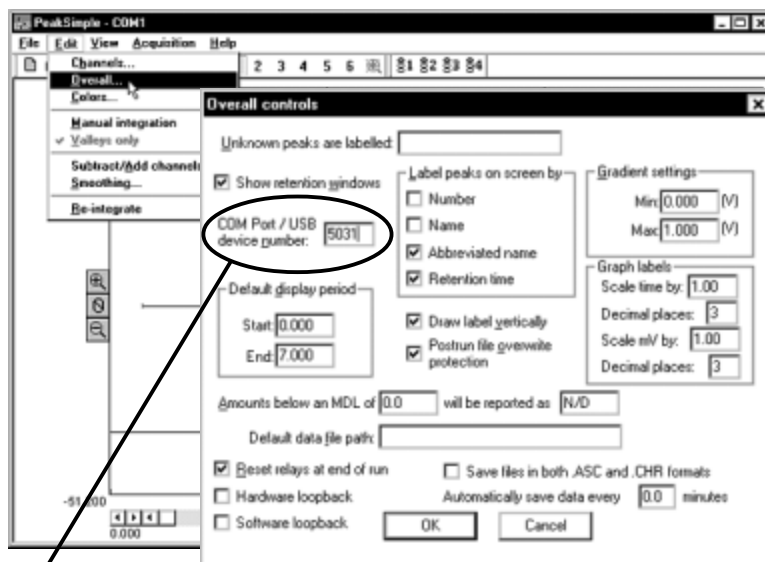


10. Launch PeakSimple

10-1. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between your computer and the Model 302. An error message will appear if communication is not established. This is normal until you complete the following step.

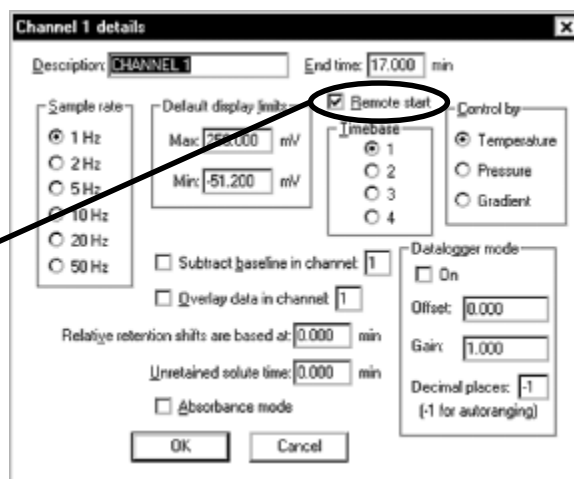


10-2. Each SRI USB data system has a unique 4-digit USB device number beginning with "5" (5031, 5032, etc.). This I.D. number is printed on the back of your Model 302, and on your PeakSimple disk. Open the PeakSimple Edit menu and choose Overall. Enter your Model 302 I.D. number in the box labeled "Com port / USB device number." Click OK, and PeakSimple will attempt to "wake-up" the data system. Click the Save All  icon so you don't have to re-enter the USB device number.



Enter the 4-digit USB device number here

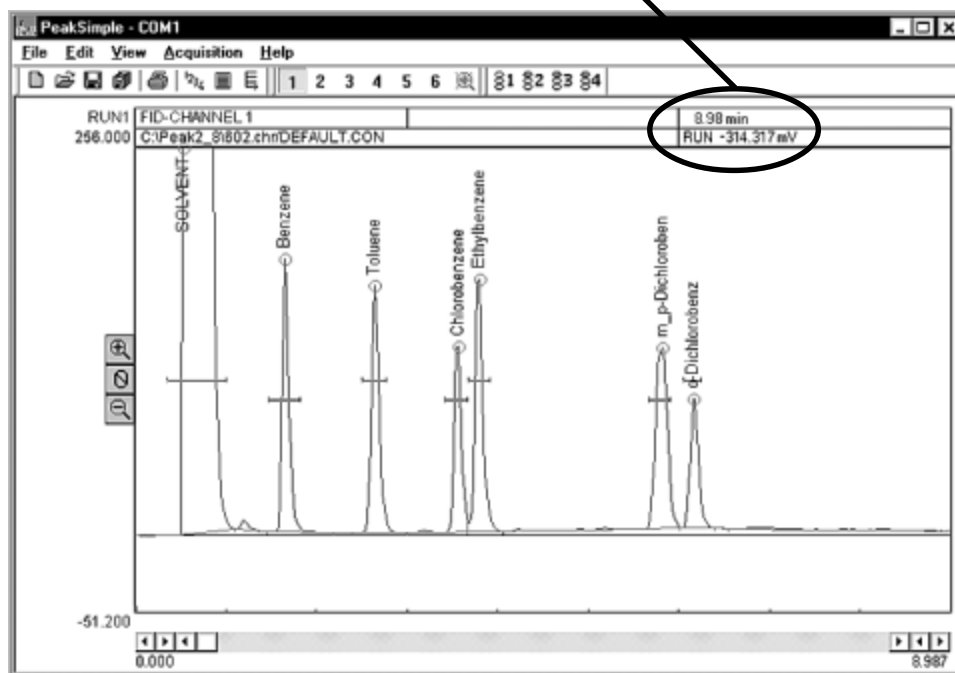
10-3. For the remote start option:
Open the Edit menu and choose Channels. Click on the Details button for channel 1. Verify that Remote start is enabled (the box should be checked). Repeat this step for channels 2-6 if necessary.



10-4. For information about using Event tables, manual Relay activation, etc., see the "PeakSimple Tutorials" and the "PeakSimple Software" sections in the manual (and online at www.srigc.com—click on the "Download Our Documents" button on the homepage).

11. Starting an Analysis

10-1. The upper right corner of the PeakSimple chromatogram window contains real-time information pertinent to your analysis in progress. The status of the run (STAND BY, RUN) is displayed in capital letters next to the millivolt (mV) reading, underneath the amount of time into the run.



11-2. Hit your computer keyboard spacebar to begin the run, and the data is plotted onscreen in the chromatogram window.



Press the spacebar to begin the run

Press the End key to stop the run

11-3. Hit the End key on your computer keyboard to stop the run.

Technical Support:

If you have questions or problems, call SRI for free technical support at 310-214-5092, 8am - 5pm California time.

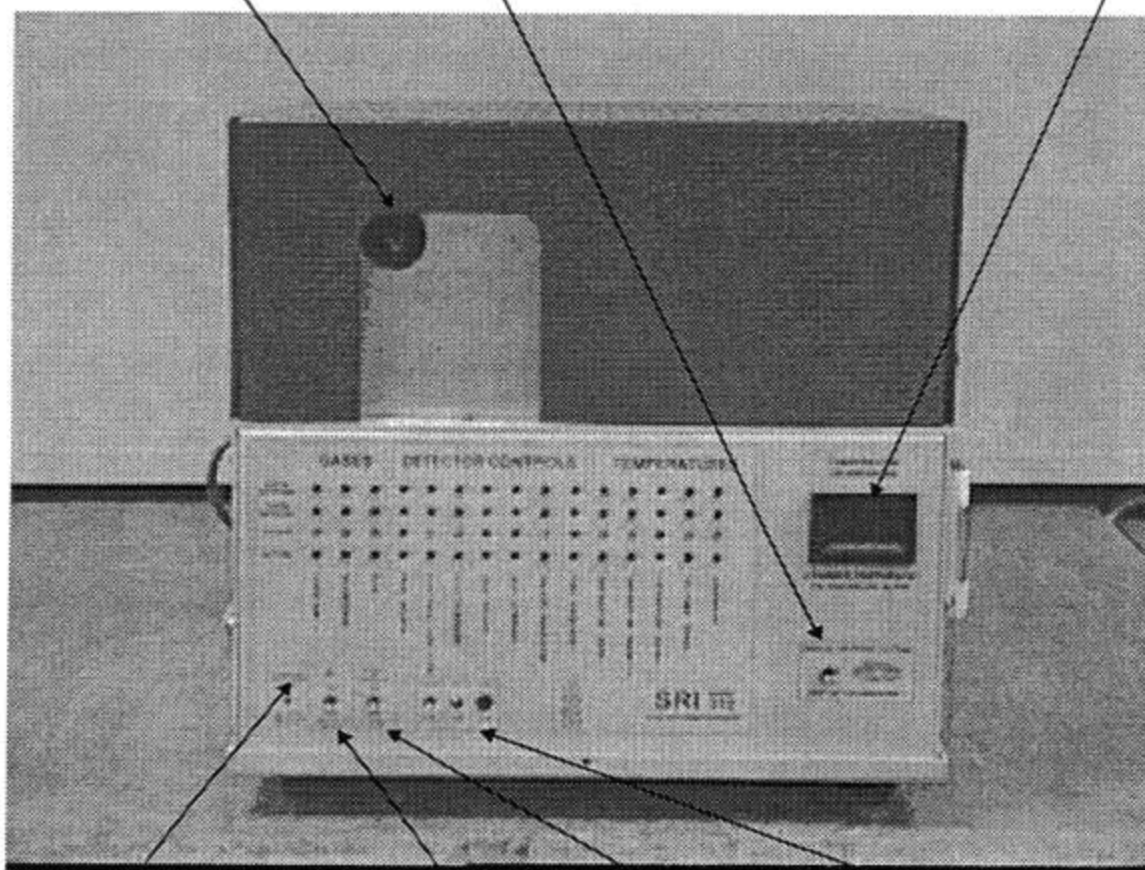
Chapter: MODEL 310 GC CHASSIS

Topic: FRONT PANEL ORIENTATION

Liquid or gas injection port for 26 gauge syringe needle

Meter selector switch allows constant display of column oven temperature (down position) or display of any zone setpoint or actual when a specific button on the front panel is pushed (up position).

Digital Panel Meter displays column oven temperature, detector temperatures, gas pressures, and detector parameters such as FID ignitor volts, PID lamp current, FPD PMT voltage, etc.



Thermo-couple out of range alarm LED indicates when any heated zone is reading less than 5 or more than 400 degrees Centigrade. When alarm is activated all AC power to heaters is shut off by de-energizing main power relay.

Polishing filter bake out switch heats built-in carrier gas filter for 5 minutes to eliminate contaminants.

Optional built-in internal air compressor on/off switch. Air compressor is used to supply air for FID, FPD and DELCD detectors

Detector control switches enable on/off of various detector parameters such as PID lamp current, FPD PMT volts or FID ignitor.

H008.doc

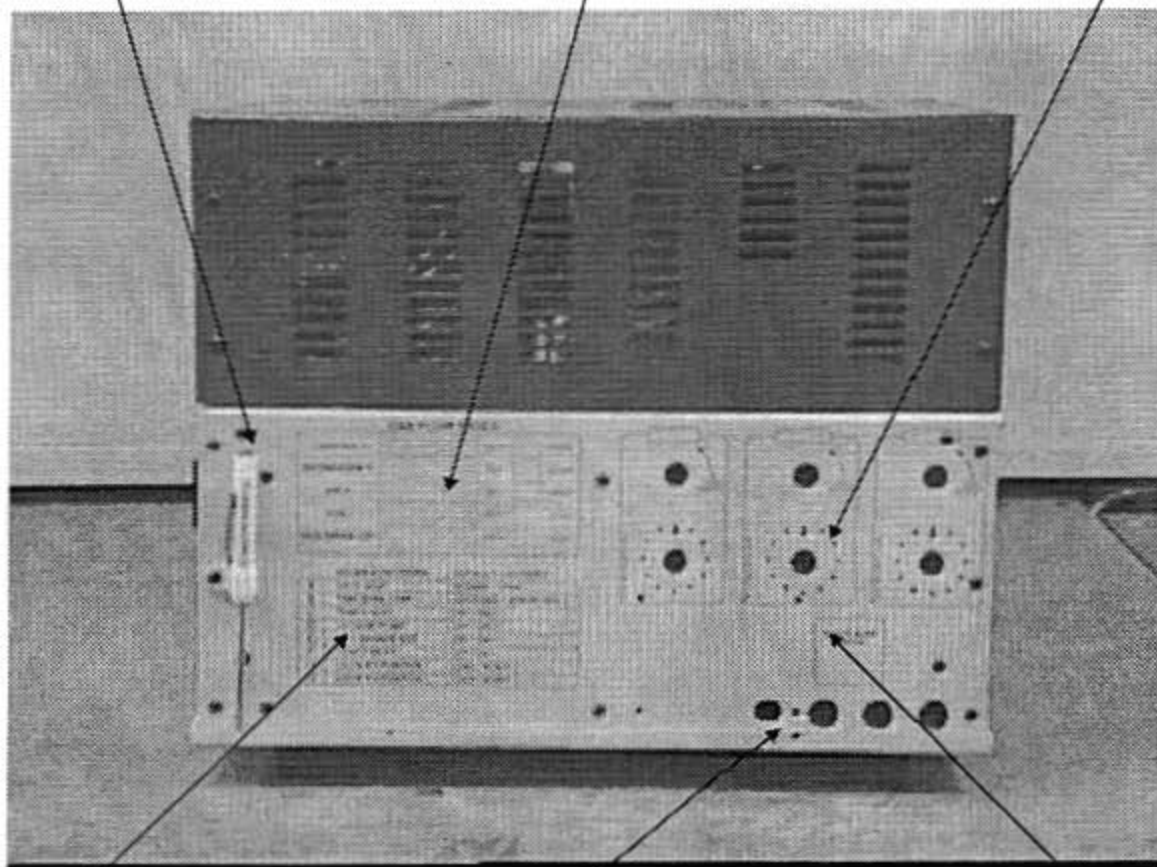
Chapter: MODEL 310 GC CHASSIS

Topic: RIGHT SIDE PANEL ORIENTATION

Screwdriver in convenient "holster" for adjustment of temperature and pressure setpoints

Gas flow rate table is used to record the flow rates and pressures used for detector support gases. Factory technicians record typical flow rates and pressures used to test detectors before shipment.

Mounting location for optional detector "zero" and attenuator knobs when such controls are installed. These controls are normally not required when the GC is supplied with the built-in PeakSimple Data system, but are installed when no data system is provided.



Relay function table shows which function each of the 8 (A-H) data system relays is assigned. Depending on GC configuration, some relays may have no function.

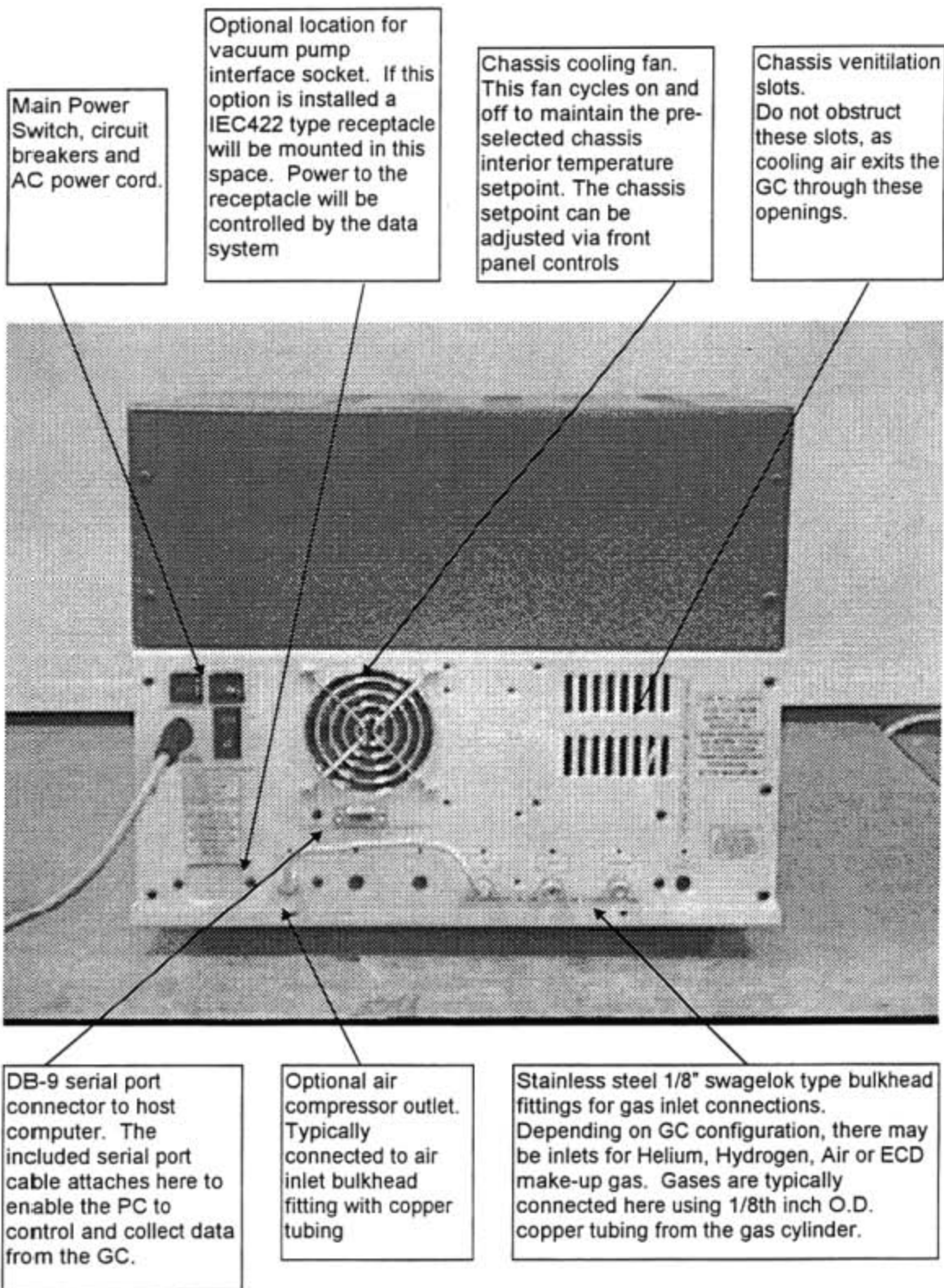
Signal cable access holes are provided for optional situations requiring wiring to exit GC.

Optional location for mounting of quick disconnect jack for remote start foot switch.

H009.doc

Chapter: MODEL 310 GC CHASSIS

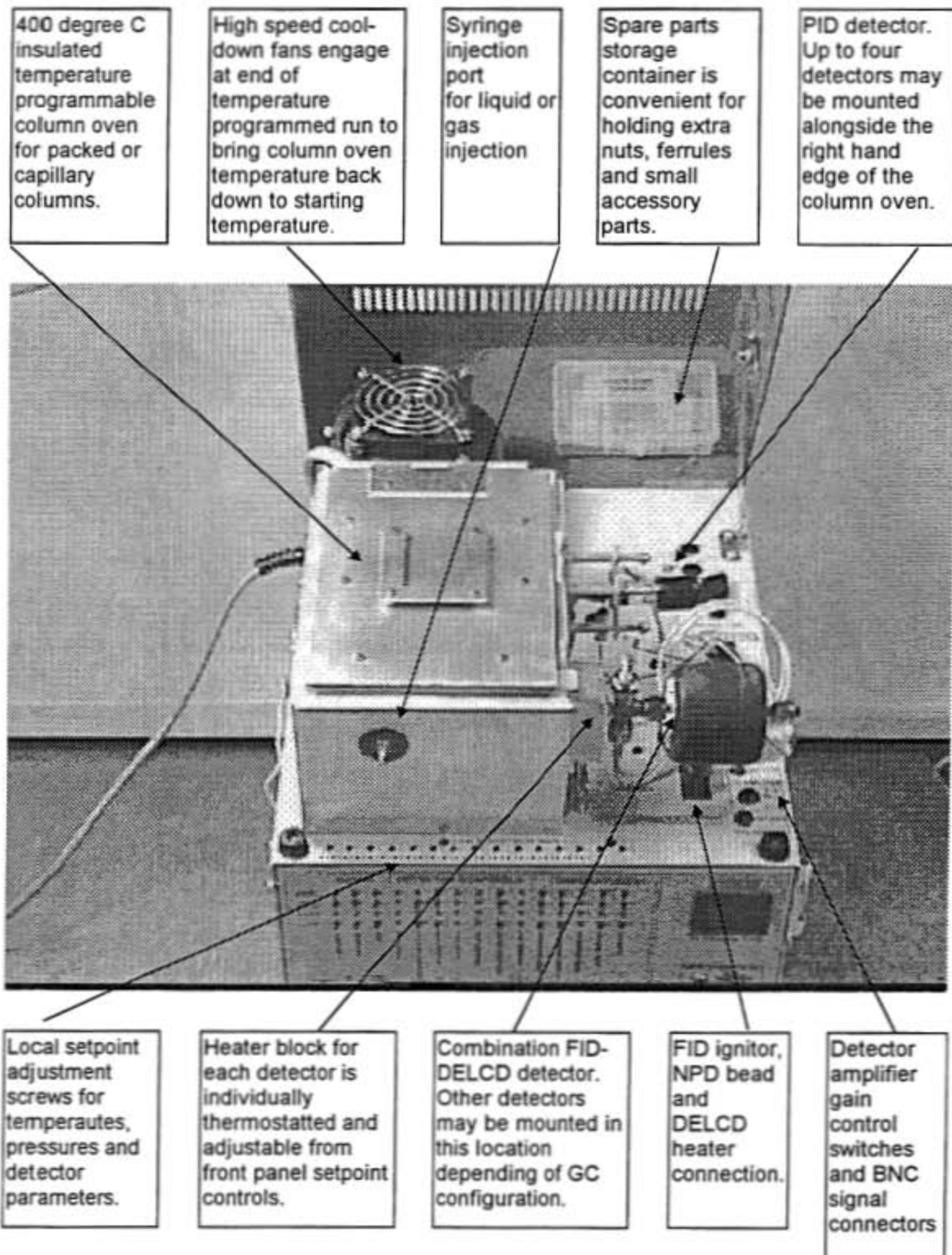
Topic: LEFT SIDE PANEL ORIENTATION



H010.doc

Chapter: MODEL 310 GC CHASSIS

Topic: TOP PANEL ORIENTATION



H012.doc

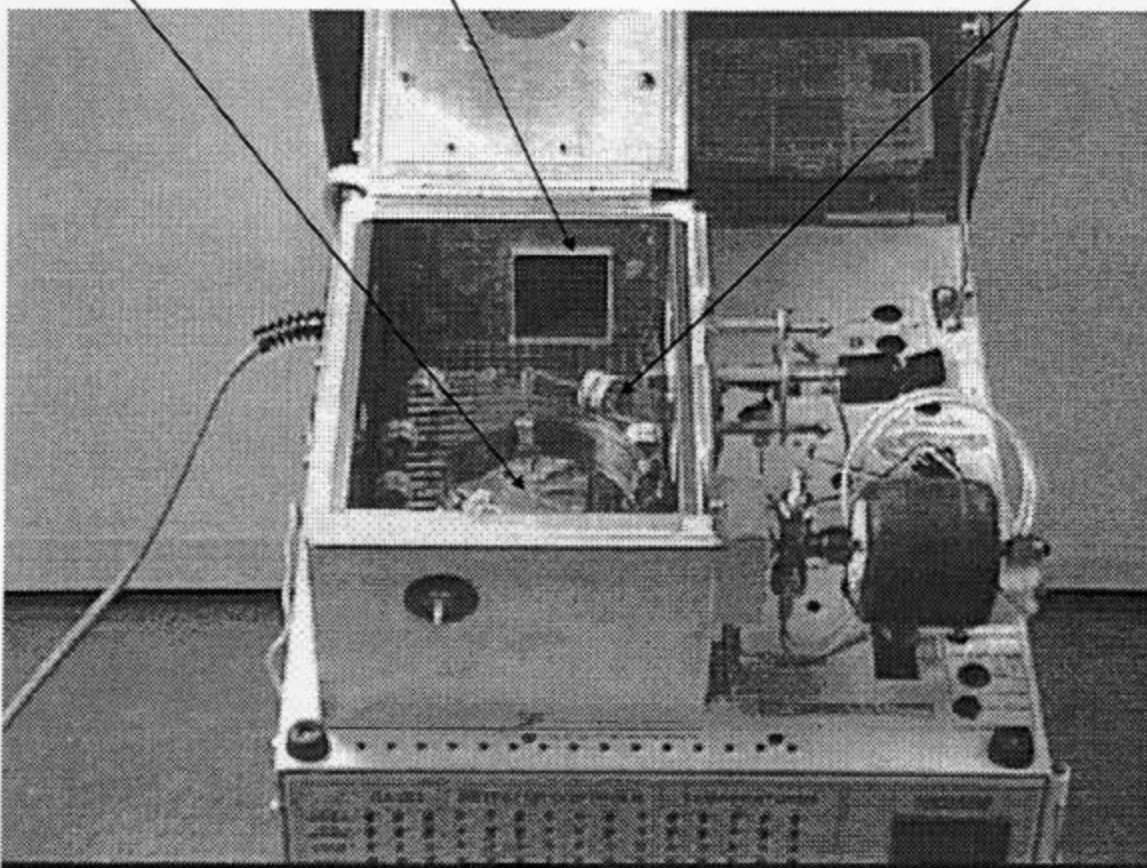
Chapter: MODEL 310 GC CHASSIS

Topic: COLUMN OVEN INTERIOR

60 meter .53 mm. I.D. capillary column shown mounted in column oven.

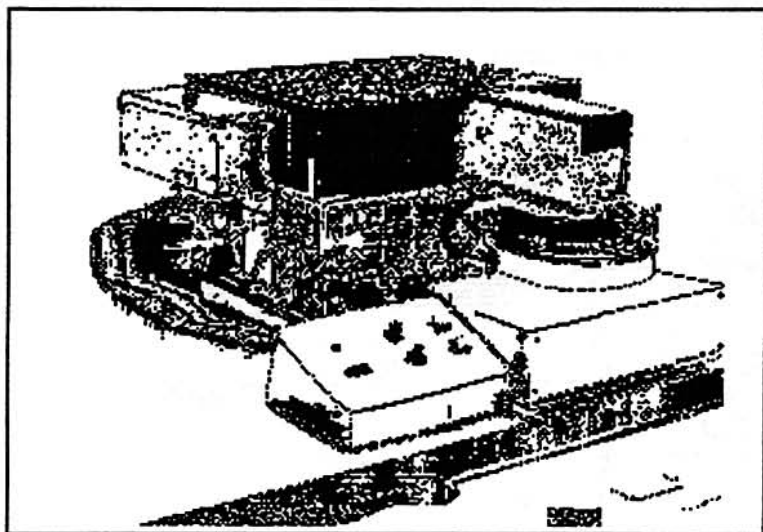
Duct for cooling air from oven cooling fans.

Circulation fan and heater coils on bottom of column oven. All heater circuits and circulation fan are disabled by interlock switch which is deactivated when red lid is raised.



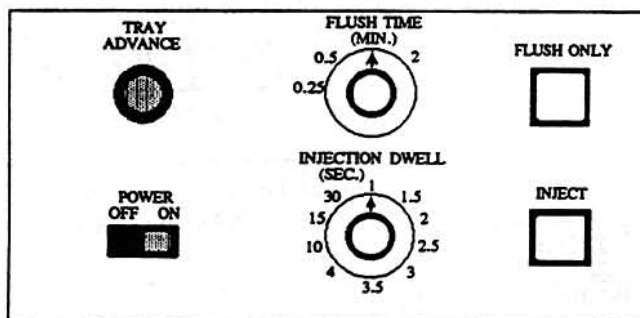
The column oven on the SRI Model 310 GC is designed for column diameters up to 4" (10 cm.). While this column diameter is smaller than average, most packed and capillary columns can be ordered with the recommended 3.5" coil diameter. Metal capillary columns are suggested because of their ruggedness and long life.

H013.doc



SRI liquid autosampler connected to SRI 8610 gas chromatograph

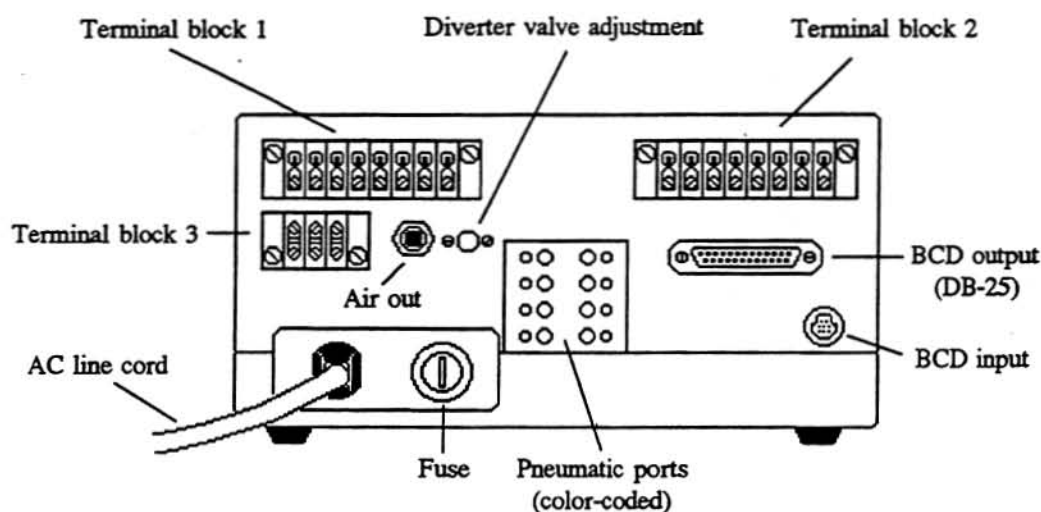
The SRI liquid autosampler is a multi-position sample injection system that permits the user to conduct unattended sampling, injection and analyses of multiple samples. Because the complete syringe rinse, load and inject sequence is mechanized and automated, the injection technique will be exact and identical from sample to sample, eliminating any variation in injection technique and sample delivery experienced between different operators when performing manual injections. This consistency will increase sample precision and reproducibility.



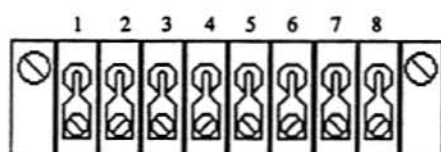
Front control panel of SRI autosampler controller

The autosampler is controlled automatically by PeakSimple software and/or manually at the autosampler control panel shown at left. Through software control, the autosampler is stepped through the sample vial positions until all samples inserted in the carousel have been injected and analyzed (without the need for operator intervention). A simple command in the event table (momentary activation of relay A) causes the autosampler to insert, flush, draw and inject the needle contents into the injection port. As soon as the needle has been withdrawn from the injection port, the autosampler is stepped to the next vial position to remain at the ready for the next actuation of relay A.

The sample vial tray may be manually advanced by hand or by pressing the TRAY ADVANCE actuator button. The amount of time (in minutes) that the syringe needle is flushed to clear the preceding sample is selectable using the FLUSH TIME control. The amount of solvent used to flush the syringe may also be varied by adjusting the sample pressurizing gas pressure. The actual volume of flush required will be dictated by the characteristics of the sample being injected. If not overly viscous, a sample flush volume of approximately 100 microliters should be adequate. If sample availability is limited to small volumes, then the flush may need to be reduced to economize on sample consumption. The amount of time that the needle remains in the injection port after the sample has been discharged from the syringe is also selectable using the INJECTION DWELL control. Selectable in seconds, the control permits the user to choose having the needle withdraw immediately upon having deposited its sample, or to maintain the needle in the injection port for an extended period, permitting any sample containing higher boiling or thermally labile components adequate time to exit the syringe and enter the injection port. High boiling components require longer needle-injection port dwell times than volatile components. A setting of 1 second signifies that the needle penetrates the septum, injects the sample and is withdrawn immediately, all within the duration of one second. A setting of 4 seconds permits the needle to dwell in the injection port for an additional 3.5 seconds.

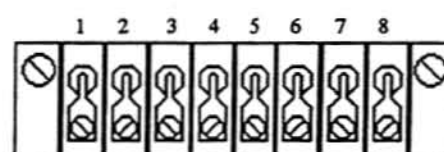


View of rear panel of SRI autosampler controller unit



Terminal block 1

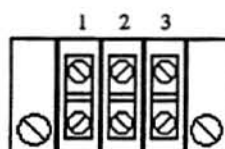
REAR PANEL
INTERFACE DETAILS



Terminal block 2

CONNECTIONS NECESSARY AT
TERMINAL BLOCK 1:

A jumper wire must be connected between terminals 7 and 8 of this terminal block (index enable position). Black wire provided.



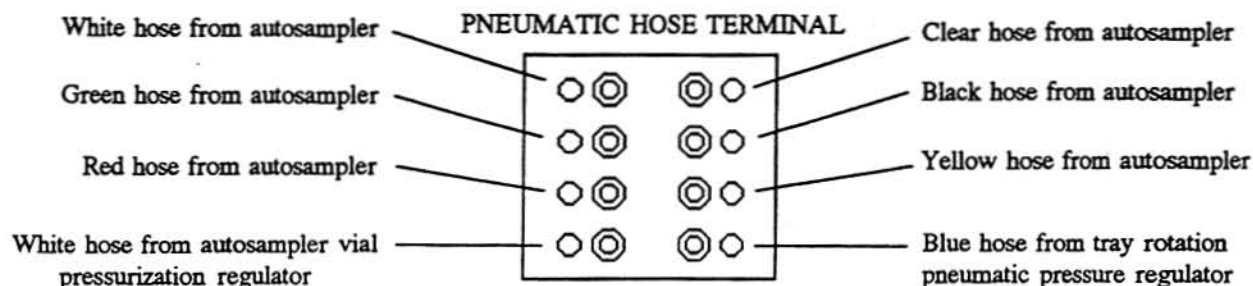
Terminal block 3

CONNECTIONS
NECESSARY AT
TERMINAL BLOCK 3:

Three-wire control cable from autosampler connects here. Black wire connects to terminal 1, red wire to terminal 2 and green wire to terminal 3

CONNECTIONS NECESSARY AT
TERMINAL BLOCK 2:

Purple wire must be connected from terminal 1 to RELAY A terminal on interface board. Black wire must be connected from terminal 2 to D.GND terminal on interface board.

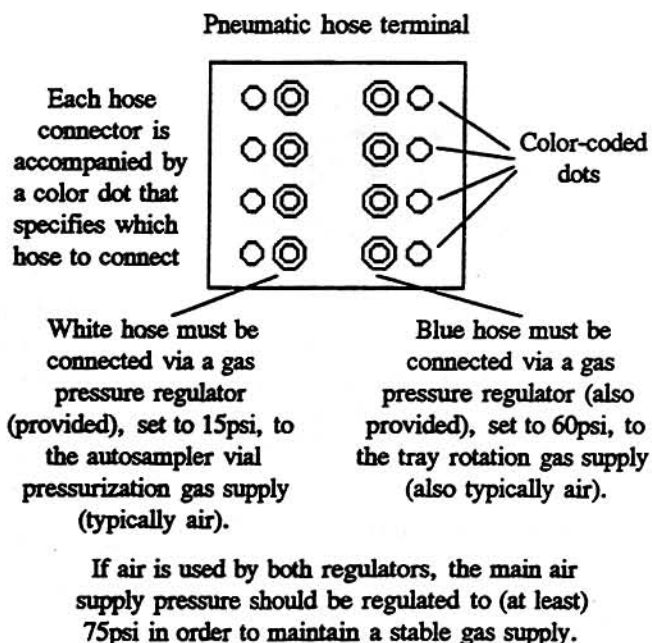


Terminal block is color-coded
to match hose colors

The procedure for installation of the autosampling carousel is simple and straight-forward. Pneumatic and electrical connections must be made at the controller unit and at the data system interface board (if in use by the system). Once these connections have been made, the autosampler is inspected for proper docking height with the chromatograph injection port. If the autosampler is intended for use with an early version of the model 8610 chromatograph (low-profile chassis with an injection port 6.5" above the countertop), the carousel will be equipped with three rubber feet that elevate the injection needle axis to exactly 6.5". Current production models are mounted on a platform that elevates the injection needle axis to exactly 10". This corresponds to the injection port height of the current production model 8610 chromatograph. When proper unit height has been verified at the injection port, the autosampler is mated to the chromatograph and operation may begin.

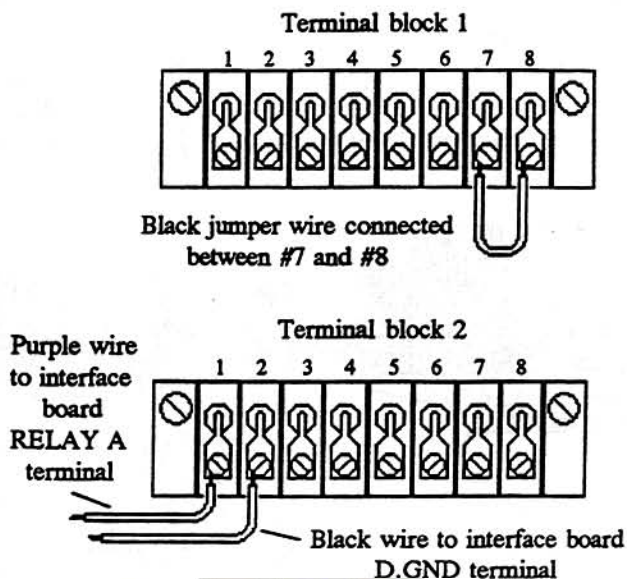
PNEUMATIC CONNECTIONS

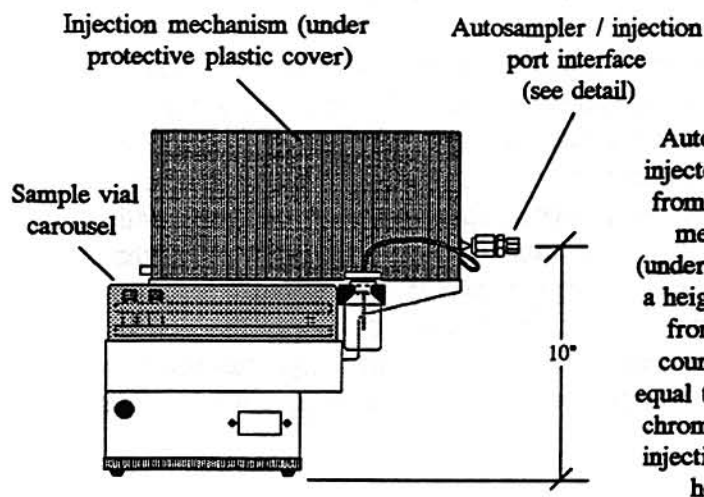
In addition to connecting the six color-coded hoses from the autosampler control harness to their respective pneumatic terminals on the rear of the autosampler controller unit, two gas connections must be made at the lower two pneumatic hose terminals on the bulkhead. The white hose provided must be connected to the autosampler vial pressurization gas supply, using the provided regulator. This regulator should be set to 15 psi. Gas (typically air or nitrogen) is injected into the vial by the outer sleeve of the concentric sampling needle (needle within a needle), forcing sample to flow out of the vial through the center needle and into the injection syringe. The blue hose should be connected to the tray rotation gas supply, set to 60psi with the other regulator provided with the unit. This gas enables the tray mechanism to rotate, advance the samples and operate the injection mechanism.



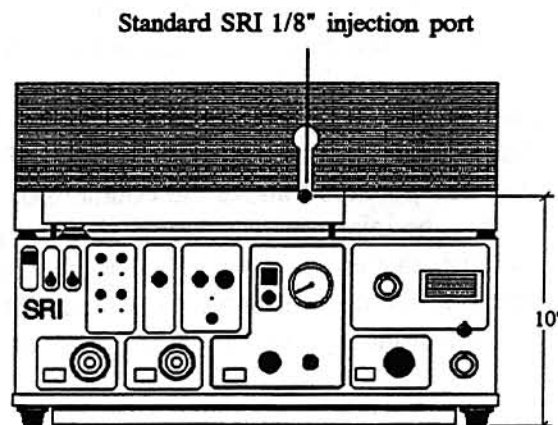
ELECTRICAL CONNECTIONS

There are only three connections to be made by the user. A black jumper wire (provided) must be connected between terminals 7 and 8 of terminal block 1. The purple and black wires (also provided) must be connected to the interface board terminals labeled RELAY A and D.GND, respectively. These two wires provide the remote activation of the autosampler advance and sample circuitry (same as INJECT on the controller unit) by the data system or other remote device.

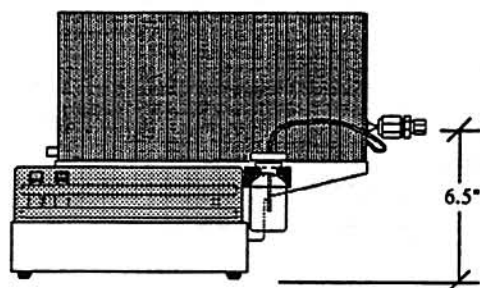




Right side view of SRI liquid autosampling carousel

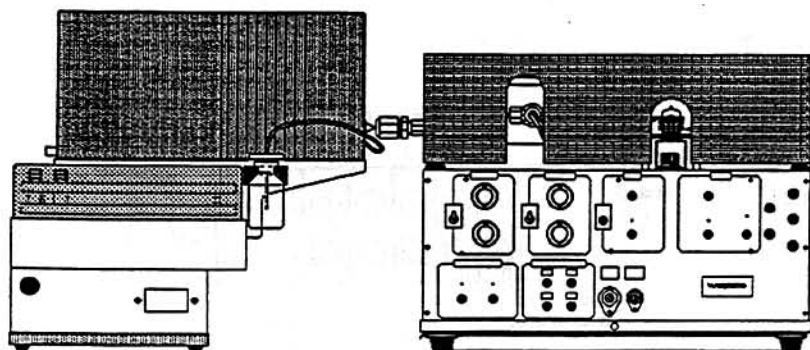
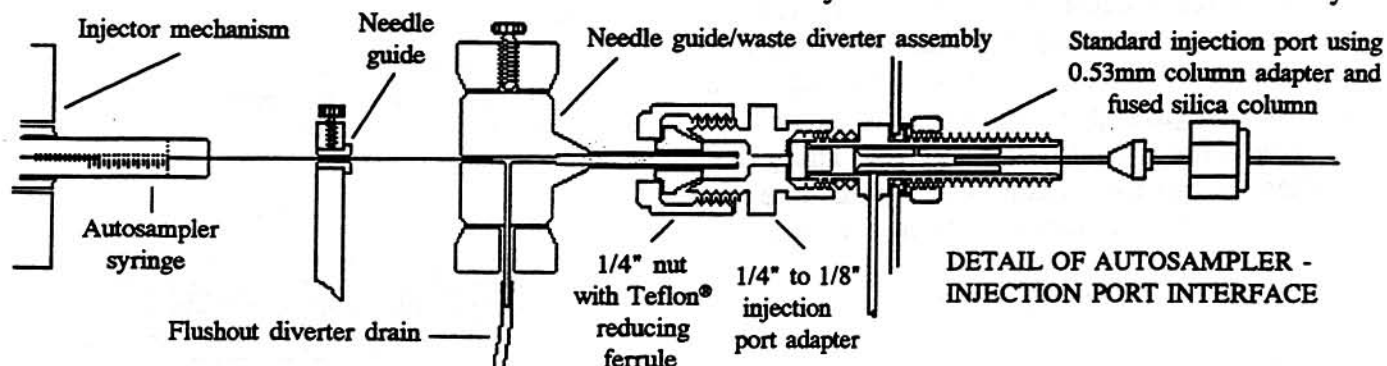


Front view of SRI model 8610 gas chromatograph



Autosampler configured for early production chromatographs with 6.5" high injection port

The SRI liquid autosampler injector mechanism is situated at a height equal to the injection port of the chromatograph. The injection port height of current production units is 10" (25.4cm). Previous models (pre-1992) employed an injector height of 6.5" (16.5cm). A special fitting is supplied with the autosampler that replaces the septum nut normally used to seal the injection port of the chromatograph. This fitting, also containing a septum, consists of a 1/8" to 1/4" adapter that is connected to the injection port. A special cylindrical brass fitting, employed as a needle guide and waste solvent diverter, is inserted into the needle end of the injector mechanism frame and secured by a



thumbscrew. From this cylindrical fitting, a 1/8" metal tube protrudes. This tube, an extension of the needle guide, is secured to the chromatograph's injection port adapter by means of a 1/4" nut with a 1/4" to 1/8" Teflon® reducing ferrule. Once both fittings are in place, the units are docked together and the 1/4" nut is secured. Then the autosampler controller unit is located in a convenient location and normal operation can begin.

410 RACK MOUNT GC CHASSIS Orientation

Overview

SRI's 410 Rack Mount GC Chassis is designed to mount in 19" shelf-equipped racks. The 410 can accommodate a single syringe injector, and one electrically actuated gas sampling valve in a heated valve oven.

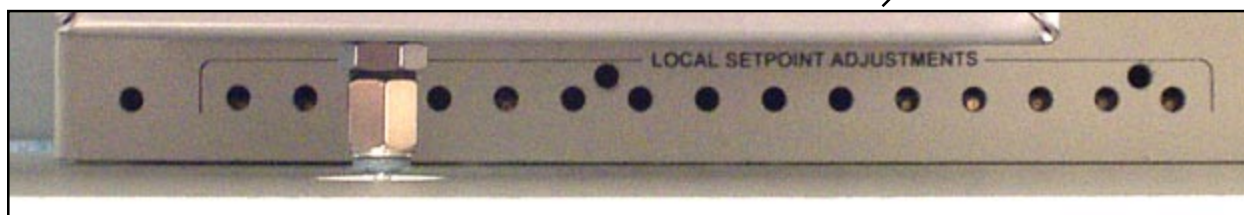
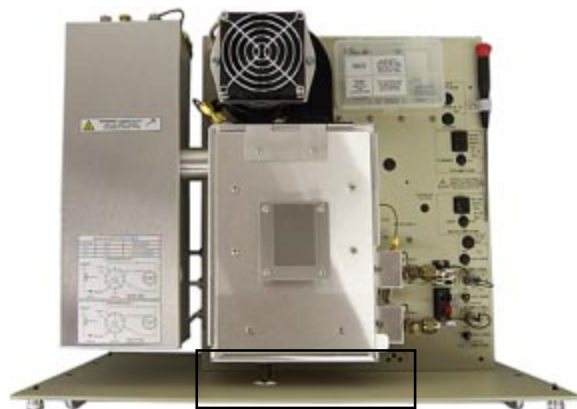
410 standard features:

- ambient to 400°C temperature programmable column oven
- single on-column injector with carrier EPC
- built-in, single channel PeakSimple data system



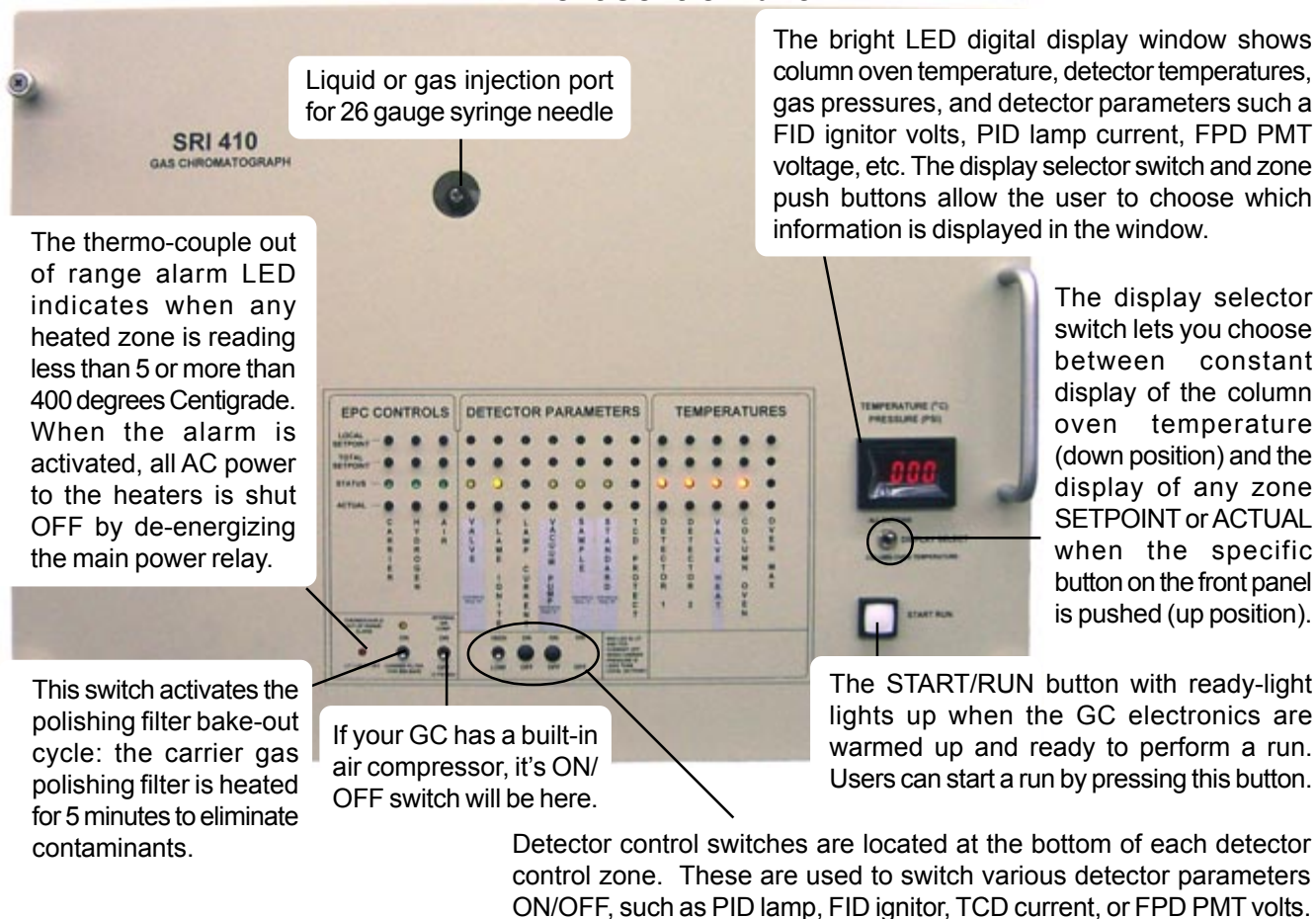
The front control panel is where you monitor gas pressures, detector parameters, and column oven temperatures.

The trimpots you turn to make adjustments to gas pressures, detector parameters, and column oven temperatures are located on the top control panel, between the column oven and the front control panel.



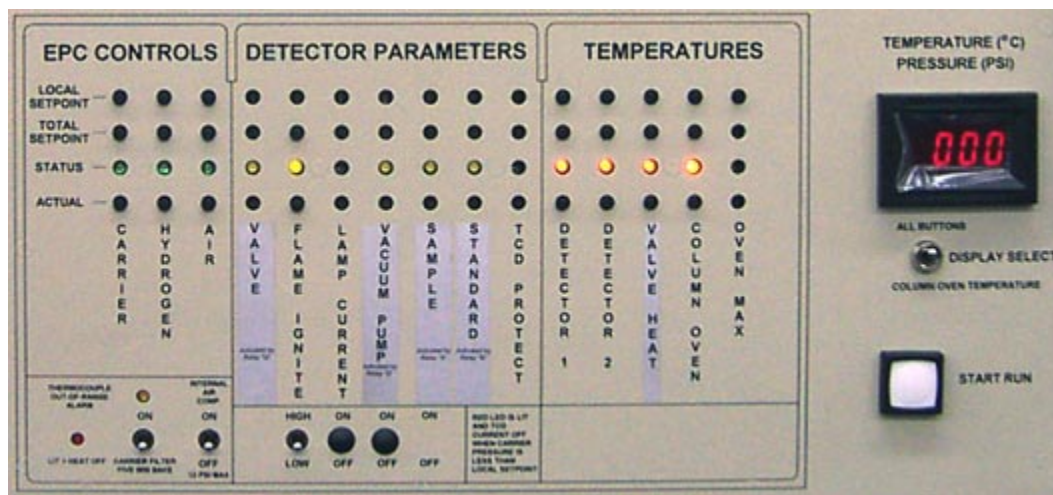
410 RACK MOUNT GC CHASSIS Orientation

Front Control Panel



The 410 front panel has 15 control zones. Each control zone has three black push buttons and one LED STATUS indicator, arranged in columns across the front control panel. The 410 has three gas pressure (EPC) control zones, seven detector parameter control zones (FID ignitor, PID lamp current, etc), and five temperature control zones. Each control zone is adjusted by a trimpot, located on the front edge of the top panel (immediately behind the front control panel). To adjust the local setpoint, press that button and turn the trimpot while observing the LED display until the desired setpoint appears. Ensure that the DISPLAY SELECT switch is on "ALL BUTTONS" (in the up position).

**LOCAL SETPOINT
TOTAL SETPOINT
STATUS LED
ACTUAL**



410 RACK MOUNT GC CHASSIS Orientation

Left Side Panel

The chassis cooling fan cycles ON and OFF to keep the chassis interior in the operable temperature range. The chassis setpoint can be adjusted via front panel controls.

Chassis ventilation; do not obstruct the slots!

Optional solenoid valves for sample stream switching are mounted on the underside of the optional valve oven.

The AC power cord, main power switch, and circuit breakers are on the left control panel (they are hidden by optional solenoid valves in this example).

Optional data system controlled, IEC422 type power receptacle for a vacuum pump or other device.

Optional air compressor outlet.

Valve actuator for optional valve

Stainless steel 1/8" swagelok type bulkhead fittings for gas inlet connections. Carrier and detector gases connect here, typically with 1/8" OD copper tubing from the cylinder.

Right Side Panel

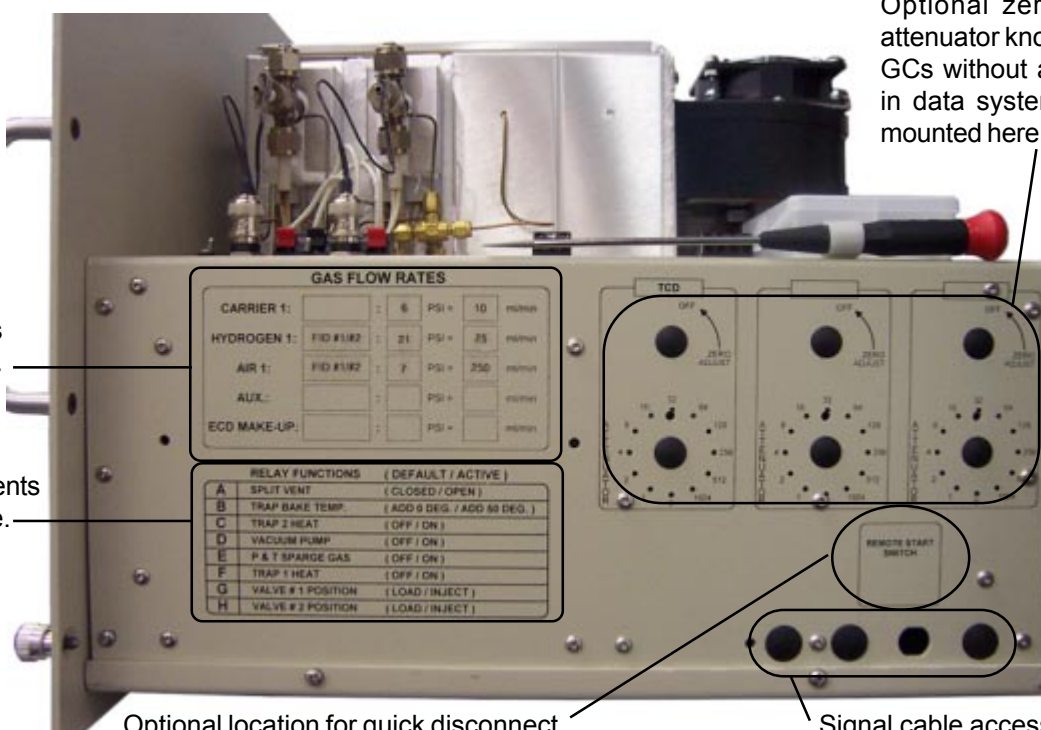
Optional zero and attenuator knobs, for GCs without a built-in data system, are mounted here.

Gas flow rates are printed here.

Relay assignments are printed here.

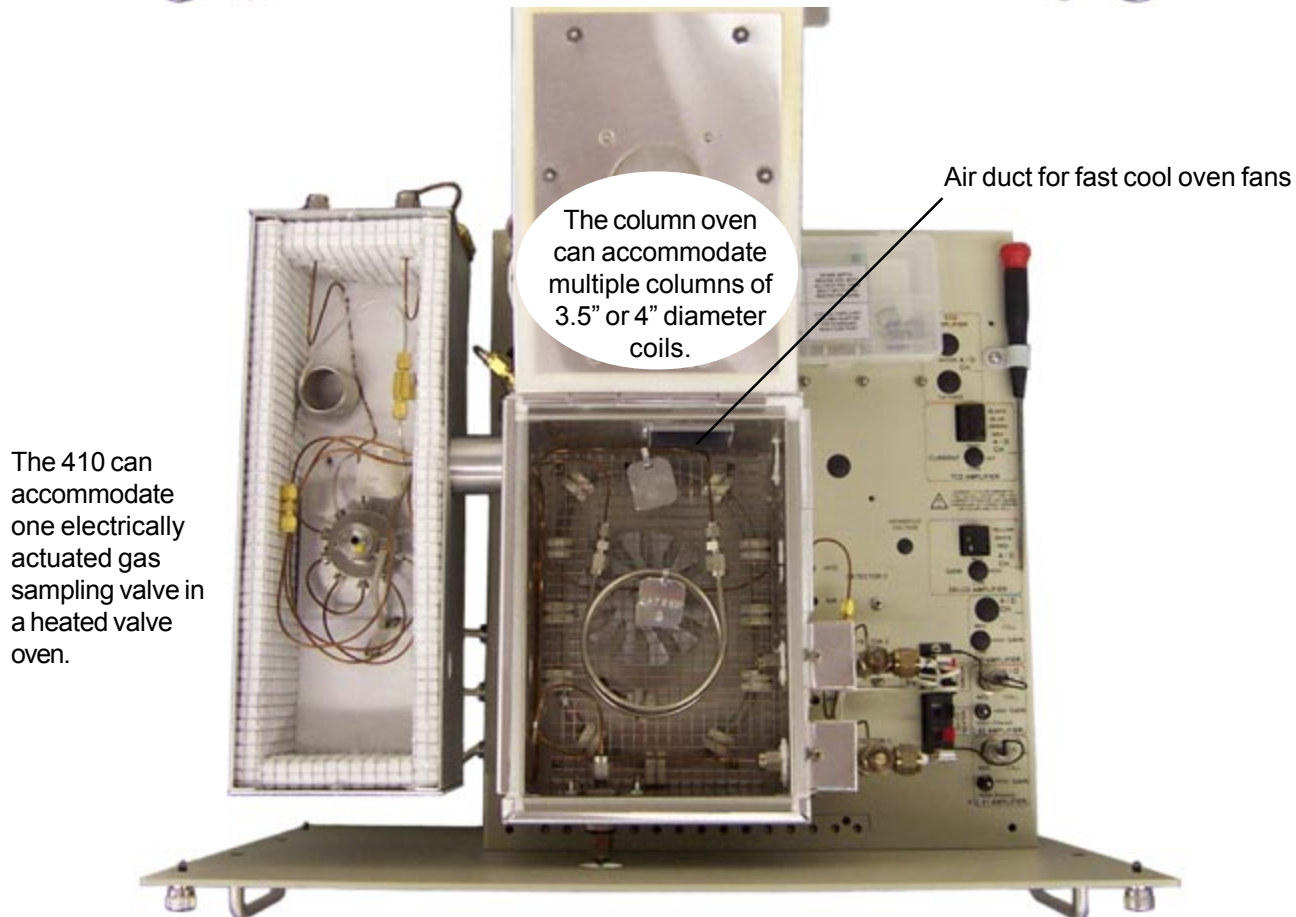
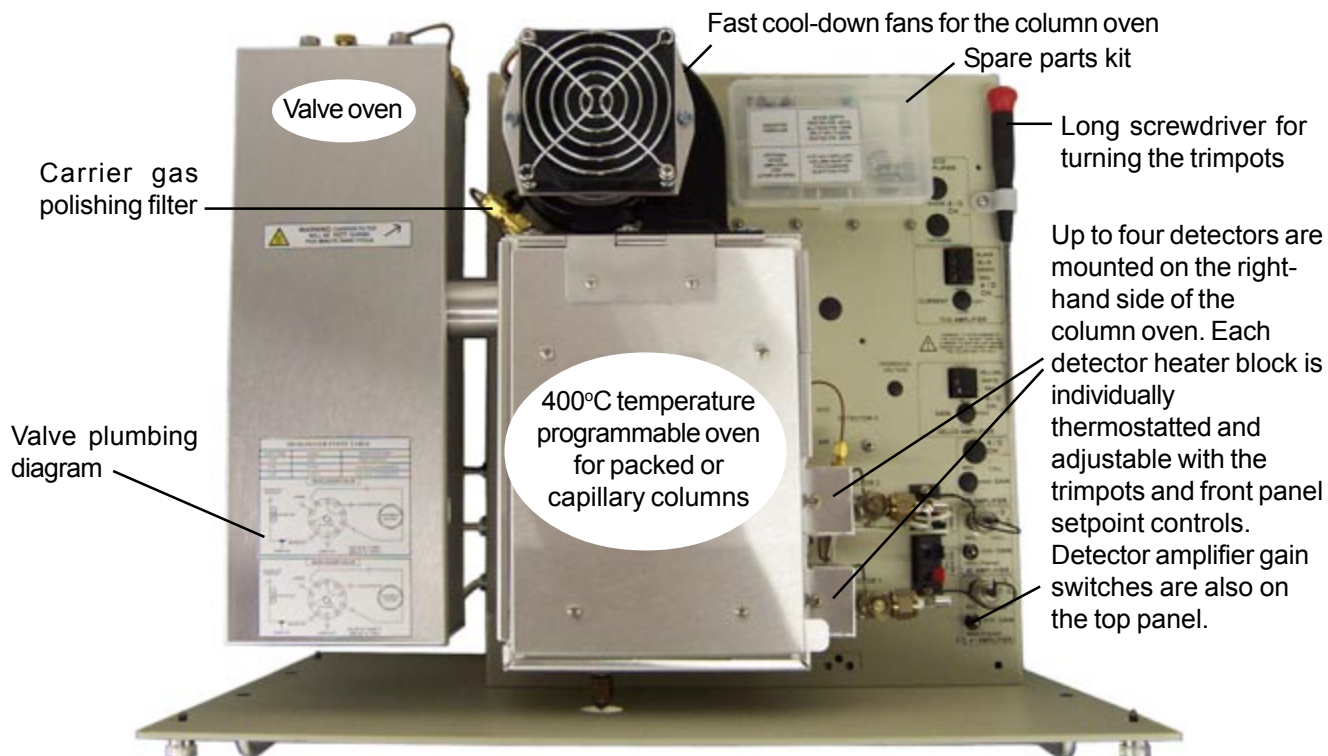
Optional location for quick disconnect jack for remote start foot switch

Signal cable access holes



410 RACK MOUNT GC CHASSIS Orientation

Top Panel



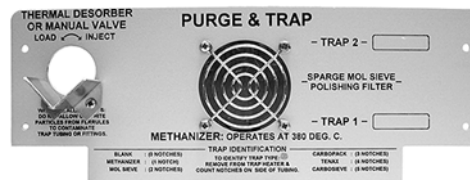
GC ACCESSORIES

10 Position Method 5030 Purge & Trap Autosampler Retrofit

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

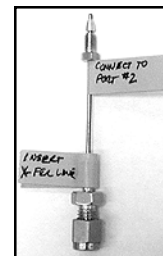
A. Purge & trap cover plate:

1. Remove the existing purge & trap cover plate from the GC by unscrewing the four thumbscrews that hold it in place.
2. Remove the fan from the existing purge & trap cover by unscrewing the four securing philips head screws and unplugging its white plastic 2-wire connector. Transfer the fan and its four screws to the new cover.
3. Attach the new purge & trap cover to the GC with the four screws from the old cover and re-connect the fan power.
4. Feed the autosampler's red heat transfer line through its cover plate hole, then secure it with the hose clamp included with the autosampler.



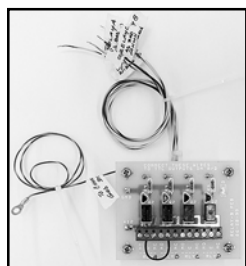
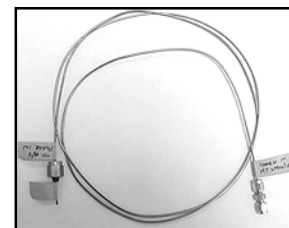
B. Transfer line to valve connection:

1. Remove the valve oven lid by unscrewing the brass thumbscrew on the front edge of the lid, tilting the front of the lid up, then sliding back slightly to free it from the screw in the back. Carefully remove the white insulation padding, and set it securely aside with the oven lid.
2. Connect the Valco fitting to PORT #2 of the gas sampling valve (the Valco fitting is labeled "CONNECT TO PORT #2").
3. Feed the transfer line completely through the Swagelok fitting and 1/16" tubing (this fitting is labeled "INSERT X-FER LINE").
4. Use a wrench to securely tighten the fitting until it is snug against the graphite ferrule; do not over-tighten.



C. Purge gas line:

1. Remove the glass test tube from the purge & trap.
2. Connect the brass union on the purge gas line to the purge gas tube (formerly inside the glass test tube). The durable Teflon™ ferrule in the brass union allows this operation to be performed many times.
3. Connect the 1/8" nut on the purge gas line to "PURGE IN" on the autosampler.



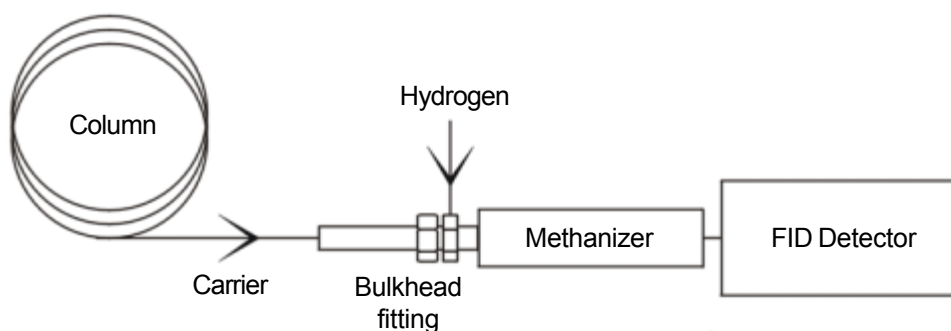
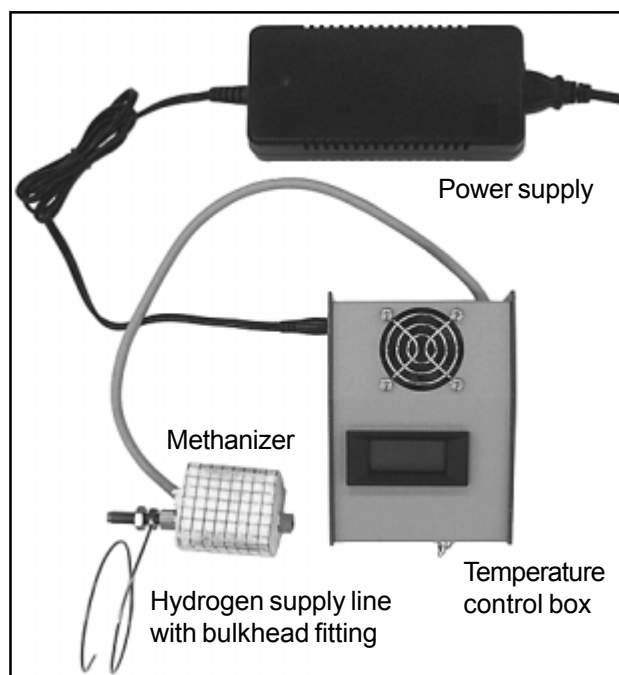
See the SRI manual for relay board installation instructions: "AUTOSAMPLERS; Installation of the Relay Board for the 10 Position Method 5030 Purge & Trap Autosampler (and other Autosamplers)"

GC ACCESSORIES

Methanizer

The Methanizer accessory enables any GC equipped with a Flame Ionization Detector to detect low levels of CO and CO₂. It comes complete with its own temperature control box and universal power supply, which can operate on any of the various voltages around the world (100-240V). Equipped with swagelok type fittings, the Methanizer can be connected anywhere between the column and the FID detector.

Like the FID detector, the Methanizer requires hydrogen for operation. The required flow rate of hydrogen through the Methanizer is 25mLs/minute. This can be a combination of carrier and make-up gases. Connect your hydrogen supply to the 1/16" make-up line on the 1/8" bulkhead fitting. The bulkhead fitting may also be used to mount the Methanizer body on your instrument.



GC ACCESSORIES Methanizer The Methanizer is packed with powder. During analysis, the Methanizer is heated to 380°C. This temperature is set at the factory, and should not normally require user adjustment. Once you turn the Methanizer ON, let it warm up for at least two minutes before verifying the temperature setpoint. When the column effluent mixes with the hydrogen carrier or FID supply and passes through the Methanizer, CO and CO₂ are converted to methane. Since the conversion of CO and CO₂ to methane occurs after the sample compounds have passed through the column, their retention times are unchanged. Hydrocarbons pass through the Methanizer unaffected.

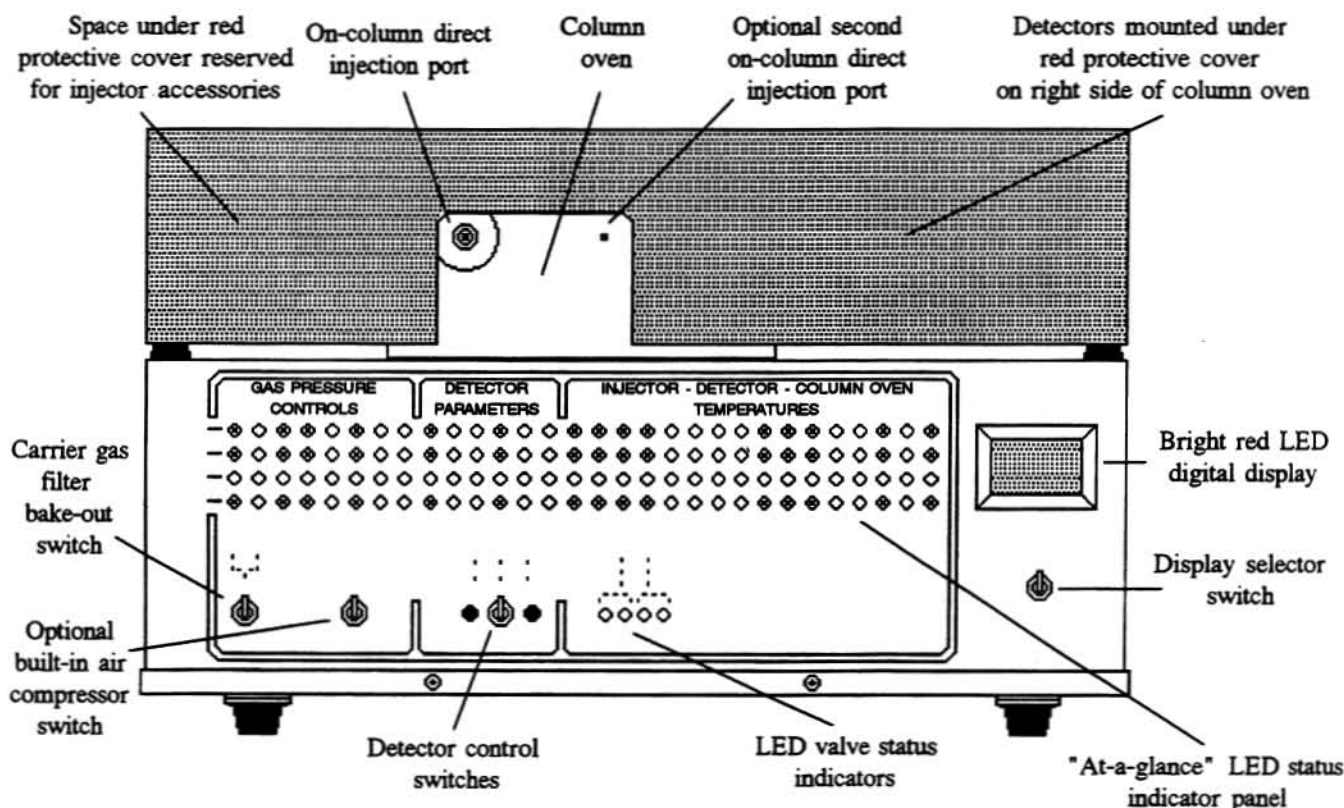
Actual temperature display

Trimpot for temperature setpoint adjustment

Temperature display selector switch

Heat ON/OFF switch





"AT-A-GLANCE" STATUS INDICATOR PANEL - All controlled zones on the gas chromatograph are displayable on this panel. Multicolored light-emitting diodes (LEDs) indicated when zones are active (on), or are being thermostatically-controlled (heated zones - pulsing).

DISPLAY SELECTOR SWITCH - This switch toggles between constant display of the column oven temperature, and display of zone setpoints and actual values when a specific button is pushed. Each "at-a-glance" status panel zone LED is accompanied by push-buttons that permit display of local and total setpoint values, and actual zone values on the digital LED panel meter.

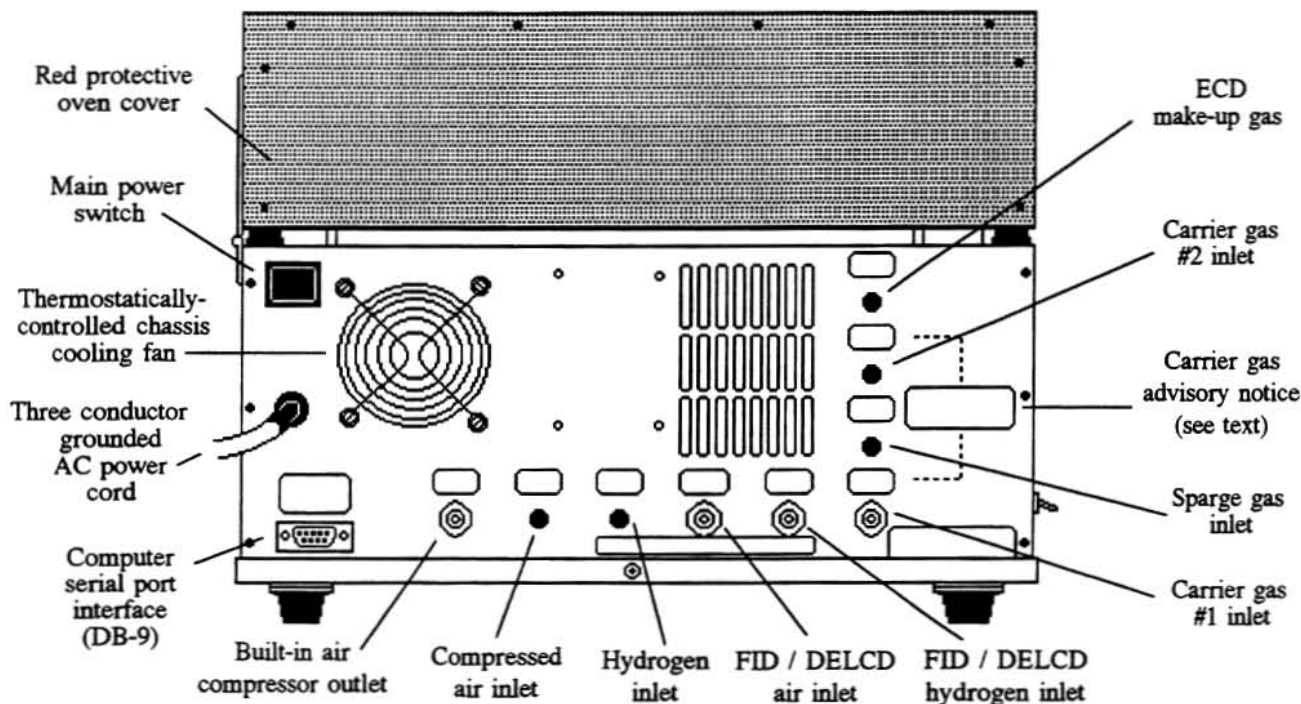
DIGITAL PANEL METER - A high-visibility, bright red 3-digit panel meter displayed either the current column oven temperature, or the temperatures, voltages, and pressures of all controlled zones. Zone value display is momentary, and is shown as long as a button is depressed.

VALVE STATUS INDICATORS - On gas chromatographs equipped with optional sampling valves, an LED glows to indicate the valve's current position. Up to two valves may be displayed.

INJECTOR PORT - A direct on-column inject port is provided, and supports the use of both packed and capillary analytical columns. A capillary column adapter is provided for installation of wide-bore capillary columns. Optional heated injection ports and heated split-splitless injection ports are available. A second injection port may be installed on the same column oven.

DETECTOR CONTROL SWITCHES - All detector control switches are located on the front control panel, including FID ignitor and PID current, and FPD voltage.

ADDITIONAL SWITCHES - A carrier gas filter bake-out switch is provided to bake impurities from the gas polishing filter. If the GC is equipped with an optional built-in air compressor for FID or DELCD use, a switch is also provided on the lower left corner of the front control panel.



MAIN POWER SWITCH: A rocker switch provides control of the AC power for the entire gas chromatograph. When the GC power switch is turned off, the built-in serial data acquisition interface is also inactive, and communications with the computer cease.

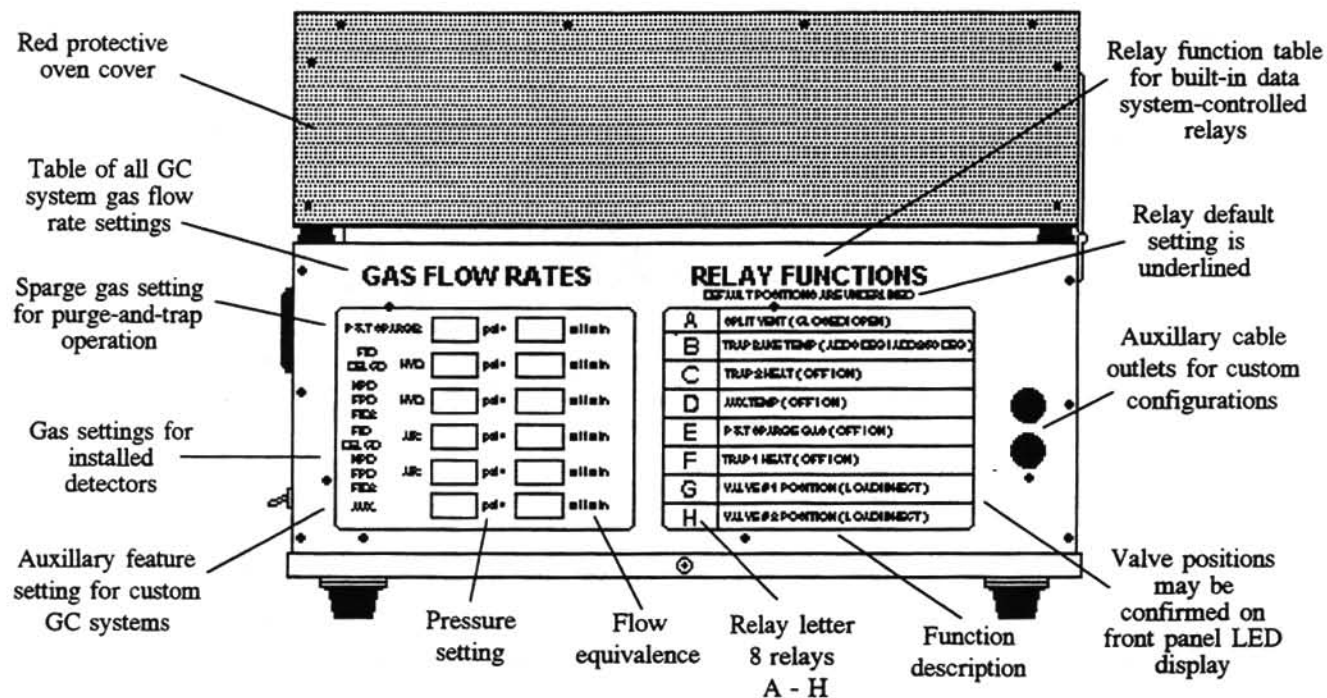
COMPUTER SERIAL PORT INTERFACE: This port, a standard RS-232 serial interface, connects the gas chromatograph to any IBM PC-compatible desktop or notebook computer serial port. The computer collects the data and controls the gas chromatograph. No data storage occurs in the chromatograph. A six-foot DB-9 type serial cable is provided for connection to the PC.

CARRIER GAS INLETS (1 AND 2): The 8610C GC may be equipped with up to two independent carrier gas systems for independent injectors, columns, and detectors. An important advisory message, regarding the use of helium carrier gas only, is printed on the chassis and refers to all 8610C models. A dangerous condition could occur if hydrogen carrier gas were being used and a leak (such a break in the column) occurred downstream of the pressure control circuitry. The leak would not be detected by the system, and gas would be continuously vented at the set pressure, permitting explosive gas to accumulate in the vicinity of the chromatograph.

GAS INLETS: Stainless steel gas bulkhead fittings are provided for connection of all system gases. Separate inlets are provided for spurge, FID, DELCD, and ECD gases. If the GC is equipped with a built-in air compressor, a compressed air outlet is also provided.

CHASSIS COOLING FAN: This fan is thermostatically-controlled and draws ambient air into the chassis electronics compartment to maintain the internal electronic and pneumatic components at a stable, controlled temperature. The temperature setpoint is pre-set at the factory.

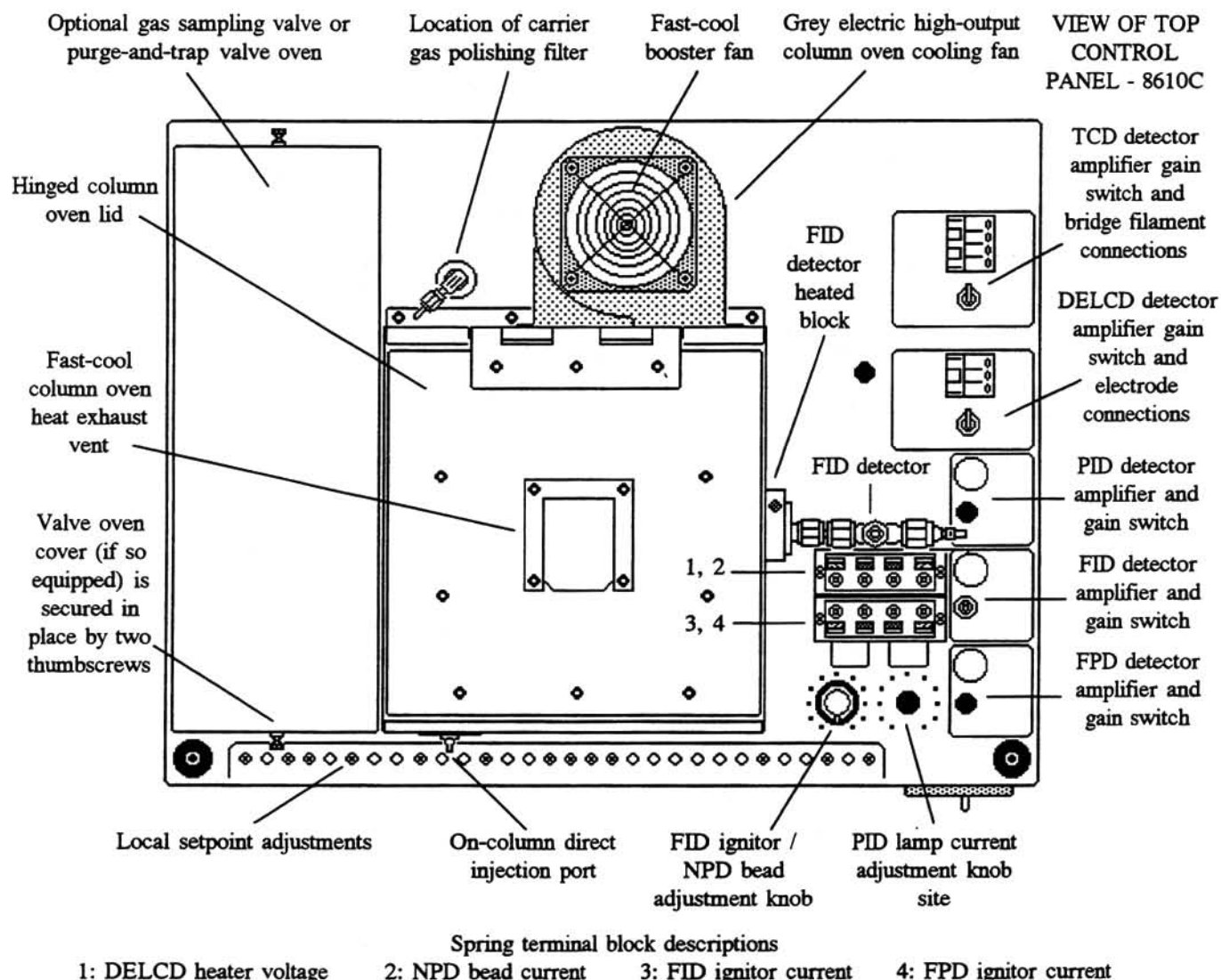
POWER CORD: A permanently-attached six foot, three-conductor cord is provided for connection to a grounded 110VAC power outlet. 220VAC models are supplied with the appropriate plug for standard grounded 220V outlets. Never defeat the safety feature inherent in the grounded cord by connecting it to a two-prong, ungrounded outlet.



GAS FLOW RATES TABLE: A table of all of the 8610C gas chromatograph's detector and special feature gas flow rates is provided on the right chassis panel of the GC. This table bears the recommended gas flow rate settings for every feature that the particular gas chromatograph is equipped with. All detectors requiring supportive gases, such as hydrogen and compressed air (in addition to carrier gas) for FID, FPD, NPD, and DELCD operation will have their suggested flows printed here for easy reference. Any special purpose gases requiring specific flow settings, such as the ECD make-up gas flow, will be printed here also.

A pressure figure is given adjacent to each gas, and this value should be used when initially setting up the chromatograph for operation. These settings will ensure proper operation. Once the detectors and other accessories are operating normally, the gas flow rates may be adjusted for optimization. The values printed on this table have been tested with the particular chromatograph in the SRI quality control laboratory. Flow equivalences for each pressure setting are also provided for your convenience. The indicated pressure setting should provide you with the flow rate shown to its right on the table. For precise flow measurements, a bubble or digital flowmeter should be used.

RELAY FUNCTIONS TABLE: Adjacent to the gas flow rate table, you will find a relay functions table that lists each of the eight data system-controlled relays (labeled A through H) available within the gas chromatograph. These relays may be operated by means of either a timed event table within any of the PeakSimple software programs, or directly by keyboard control. When using event table control, each relay called in the event table will activate or deactivate at the exact same time during each run. This makes these event table-controlled relays perfect for operation of solenoids, autosampler injector control, and rotation of automated gas sampling and stream selection valves. A description of the function of each relay is printed on the table. The default setting for each relay is identified by underlining of the descriptive text. Special purpose relays, such as the trap temperature toggle implemented via relay B, permit you to increase your trap temperatures from their normal desorption temperature, to a bake-out temperature fifty degrees above the desorb setpoint, when performing purge-and-trap analyses.



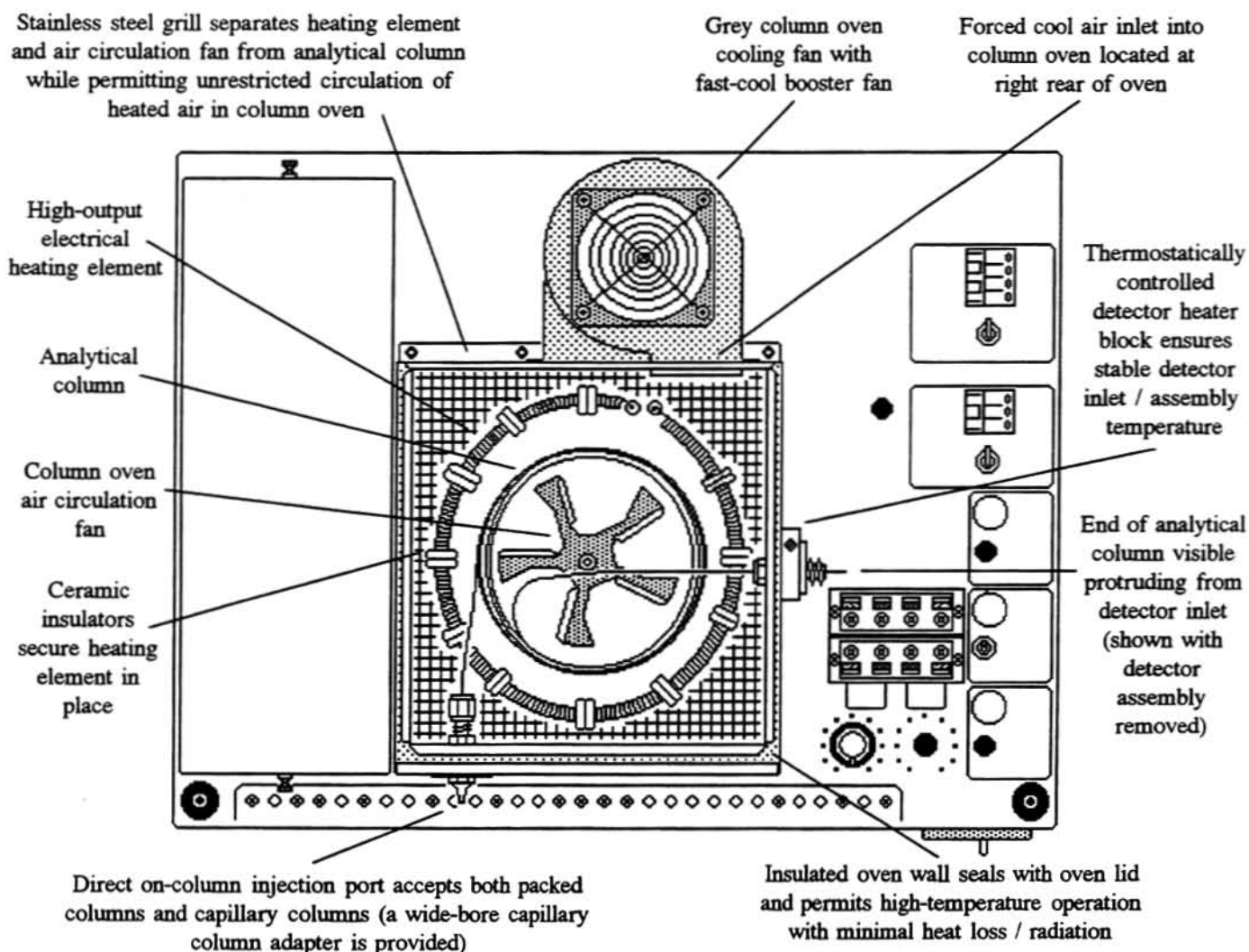
The 8610C top control panel is divided into four main areas:

COLUMN OVEN - The insulated column oven and associated cooling hardware is mounted in the middle of the top control panel. A direct, on-column injection port is located on the front left face of the column oven. The oven cover is hinged at the rear, and is equipped with an exhaust vent to facilitate evacuation of heat during operation of the high-output, fast cooling fans.

DETECTOR AMPLIFIER CONTROLS - All amplifier controls, including gain switches, current controls, and connectors, are located on the right side of the top control panel.

LOCAL SETPOINT ADJUSTMENTS - All user-selectable setpoint potentiometers are located on the front edge of the top control panel, immediately above the front panel "at-a-glance" display. A small blade screwdriver is needed to adjust these trimpots.

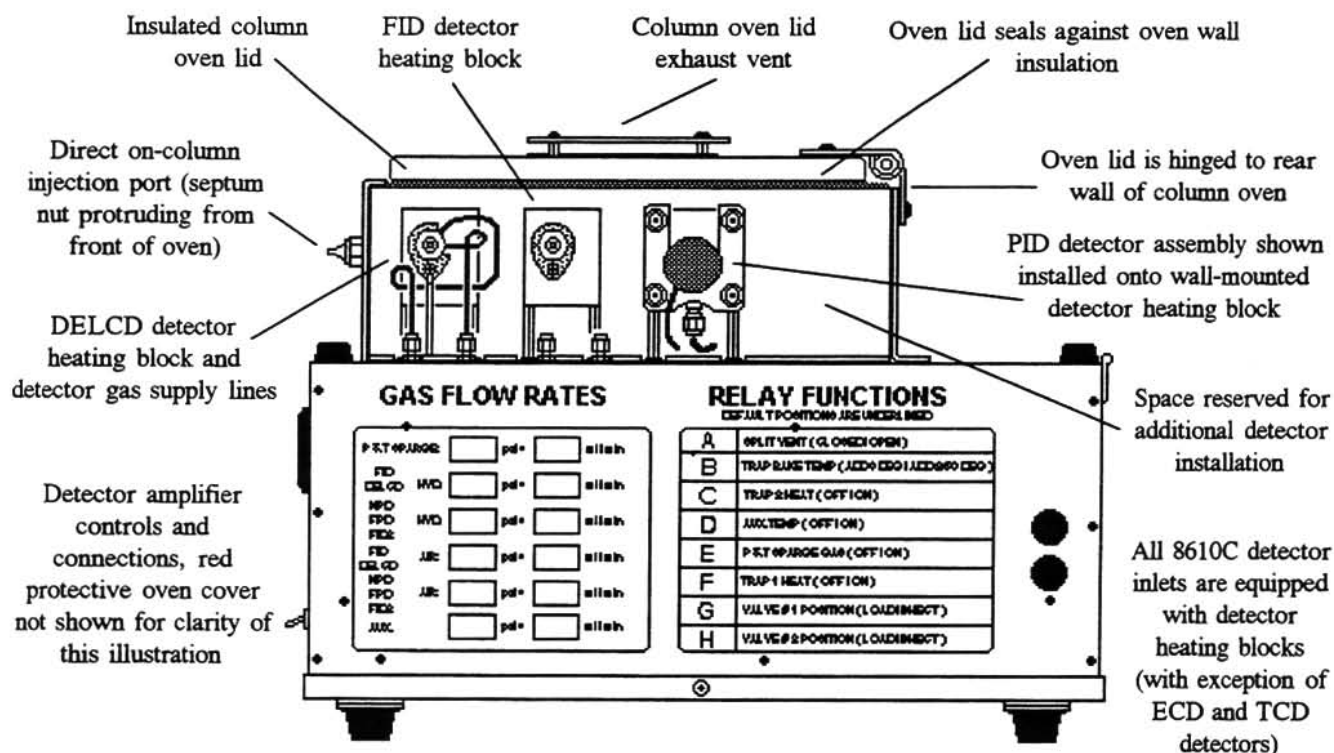
VALVE OVEN / PURGE-AND-TRAP ACCESSORY - Accessories, such as gas sampling valves, or the built-in purge-and-trap system, may be mounted to the left of the column oven, in a heated, insulated valve oven, which permits direct connection of enclosed hardware with the column oven.



TOP VIEW OF 8610C COLUMN OVEN (WITH COLUMN OVEN LID REMOVED FOR CLARITY)

The product of ten years of gas chromatograph design and manufacturing, the 8610C column oven is an insulated design that permits operation from ambient temperature to 400°C, with rapid ramping to maximum temperature and rapid cooling to initial oven temperature when operating in temperature-programmed mode. The high-output heating element permits heating at up to 40°C per minute, and the assisted cooling fan configuration permits return to 50°C from 250°C in five minutes or less. The oven lid is equipped with an exhaust vent that speeds the evacuation of heat from the oven during cooling. The oven may also be operated isothermally with excellent stability.

The open air circulation design eliminates gradients throughout the oven which could affect performance. Prepunched openings in both the left and right oven walls permit easy future implementation of accessories and detector additions. Up to four detectors may be mounted on the right oven wall for maximum analytical versatility. The outlet from non-destructive detectors, such as the PID, are routed within the column oven for convenient series detector operation. The column oven may be equipped with an optional second direct on-column injection port for use with a second analytical column, and also may be equipped with a heated injection port, with or without split-splitless capability.



RIGHT SIDE VIEW OF COLUMN OVEN WITH DETECTOR MOUNTING
HARDWARE VISIBLE (PID DETECTOR PRESENT)

All 8610C gas chromatographs are equipped with a thermostatically-controlled heating block mounted at the base (or inlet) of each detector. This new feature permits the user to preset the temperature of the detector inlet. This is convenient for methods prescribing a specific detector operating temperature, and ensures the temperature stability of each detector. Each detector heating block temperature may be accessed from the "at-a-glance" display panel on the front of the GC for viewing on the bright red, digital LED panel meter. The respective setpoint potentiometer, located on the top control panel immediately forward of the column oven, is easily adjusted using a small blade screwdriver. The TCD and ECD detectors, due to their enclosure in a temperature-controlled detector oven, do not require a heating block. These two detectors are mounted directly to the column oven wall, and the detector inlets and outlets are well-heated by the column oven.

The heated detector mounting blocks, or platforms, permit easy access to, and maintenance of the different detectors. The entire FID and DELCD detector assemblies may be removed for service in seconds. A new PID detector cell and platform design mount horizontally onto a heating block secured to the column oven, and the spring-loaded PID stage accepts compact O-I or Tracor-type PID lamps (a 10.2 eV lamp is standard equipment on SRI PID detectors).

A special electric heating cartridge is used in place of electrical heat ropes used on earlier models. The cartridges in use for detector heating blocks should provide years of service before requiring maintenance. The heating cartridge is installed in a well, drilled into the top of each cast aluminum heating block, and cartridge servicing and replacement is simple to perform, should it become necessary in the future.

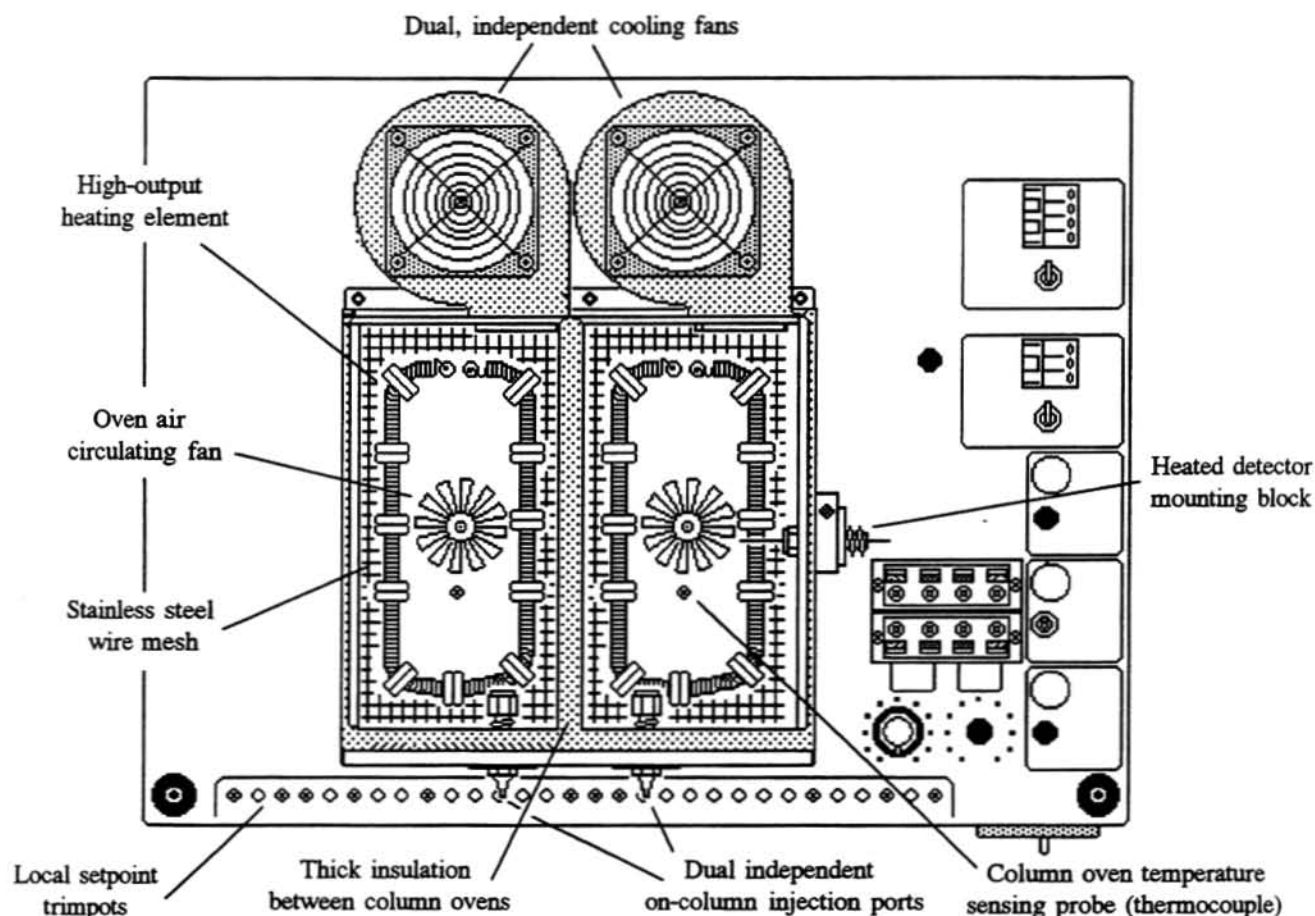


DIAGRAM OF DUAL COLUMN OVEN-EQUIPPED 8610C GC CHASSIS

For certain special applications, the SRI 8610C gas chromatograph chassis may be fitted with dual, independently-programmable column ovens. Dual column ovens permit a single 8610C gas chromatograph to perform two separate, unrelated analyses simultaneously with independent start times and temperature programs. The immediately apparent advantage to having a GC equipped with two column ovens is the ability, for instance, to perform a direct on-column injection of a BTEX sample onto a capillary column and flame ionization detector (FID) using a temperature program, such as 50°C to 200°C at a temperature ramp of 10°C per minute, while also performing a gas analysis by direct on-column injection at either an isothermal temperature or at a low-level temperature ramp, onto a packed column connected to a thermal conductivity detector (TCD) in one column oven. By placing one temperature program on channel 1 for the FID, and a different temperature program on channel 2 for the TCD, two separate column operating conditions may be simultaneously controlled.

A more sophisticated method to employ dual column ovens is multidimensional gas chromatography. Briefly, multidimensional GCs permit one sample to be analyzed normally on one column in a main column oven (connected to a dedicated detector), with the ability to "slice" a timed segment of the sample elution and place it onto the second column in the second column oven, to analyze it "under a magnifying glass", of sorts. The first column effluent is directed momentarily onto the second column and oven, where this "injection" is separated by a much longer, lower temperature column and second detector, providing a well-separated close-up of the time segment slice.

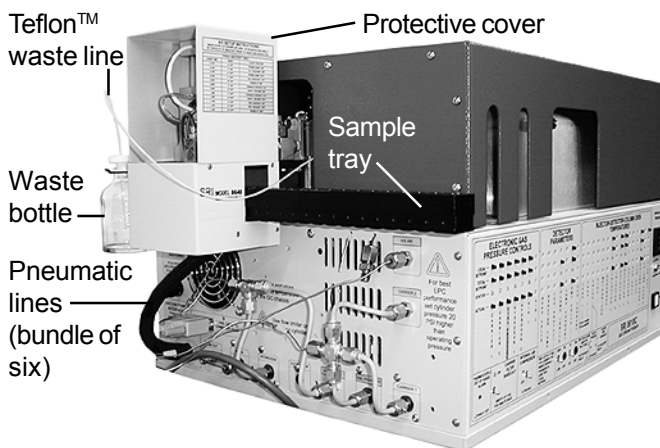
GC INJECTORS

Model 8640 20-Vial Integrated Liquid Autosampler

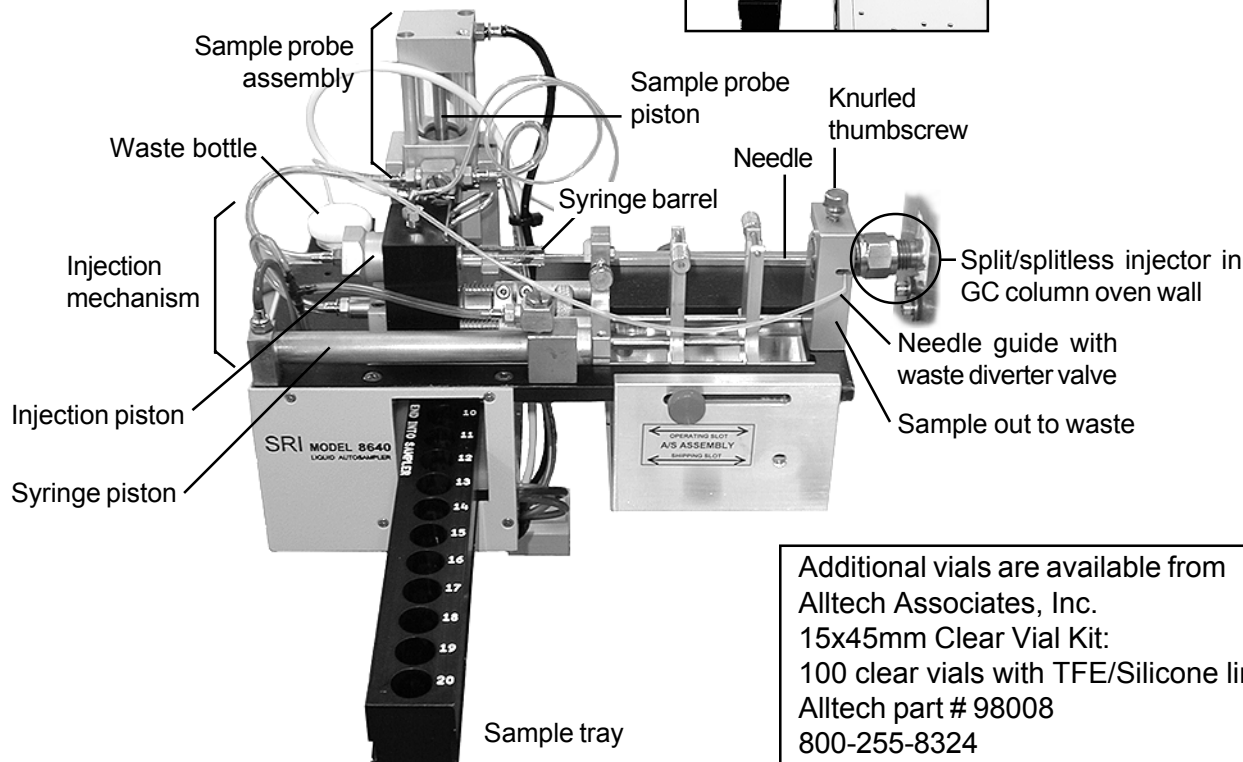
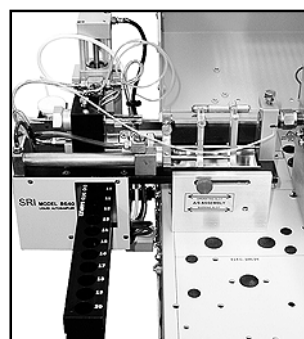
Overview

The SRI Model 8640 20-vial liquid autosampler is installed on the left-hand side of the SRI 8610C GC. The 8640 connects to an additional injector on the left side of the column oven. This additional injector may be on-column, heated, or split/splitless. It uses a sample tray to hold up to twenty 2mL vials, a sample probe to transfer the sample from the vials into the syringe barrel, and an injection mechanism to deliver the sample from the syringe barrel, through the needle, into the injector in the GC column oven wall. The 8640 uses 60psi of air or nitrogen to actuate its moving parts. The 8640 functions are assigned relays so that the autosampler may be operated automatically using a PeakSimple event table.

The 8640 is shipped with 100 screw-top vials and septa, replacements for which are available from a variety of suppliers. Extra sample trays and cooled sample trays are available. The cooled sample trays require an external refrigerated lab circulator.



8640 with the protective cover removed



Additional vials are available from
Alltech Associates, Inc.
15x45mm Clear Vial Kit:
100 clear vials with TFE/Silicone liners
Alltech part # 98008
800-255-8324
www.alltechweb.com

GC INJECTORS

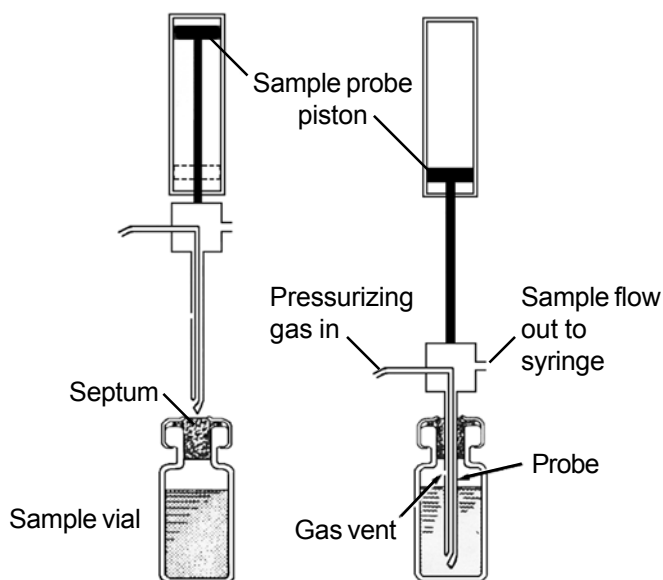
Model 8640 20-Vial Integrated Liquid Autosampler

Theory of Operation

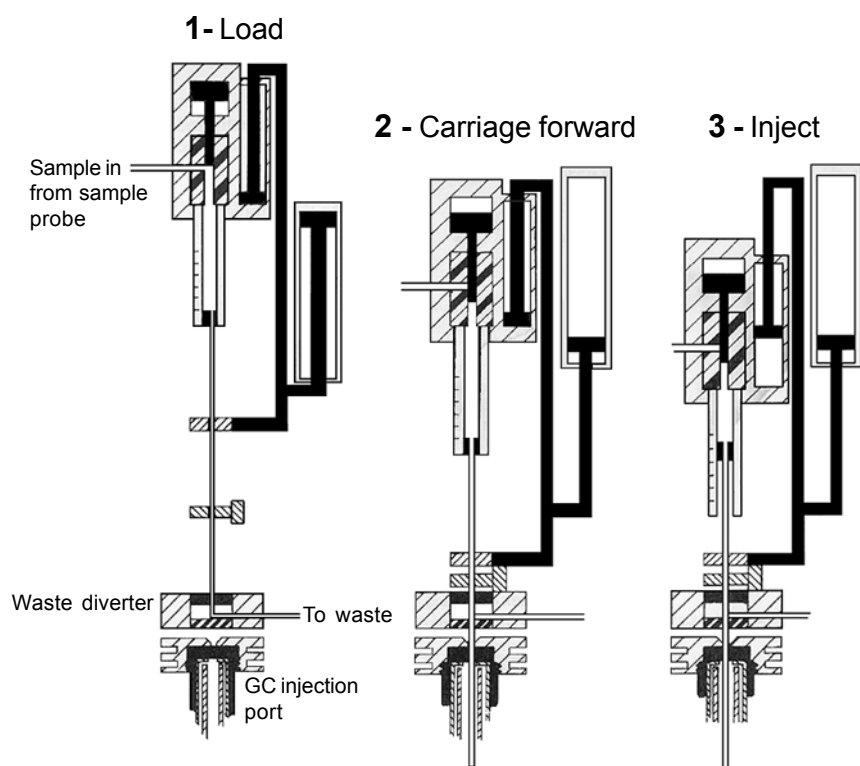
Liquid sample is sealed into vapor-tight vials which are inserted into the sample tray. The tray is then inserted into the Model 8640 assembly and positioned for the sampling sequence. The 8640 uses 60psi of air or nitrogen to actuate its moving parts.

The sampling probe, a concentric needle, is moved down by a piston to pressurize the vial with helium or other gas, causing sample to flow through the injection syringe and out the Teflon™ tubing into the waste bottle. The sample probe pressurizes the sample vial for a period of time long enough to rinse the previous sample to waste and fill the syringe with sample.

Sampling Probe Sequence



Injection Sequence



The syringe mechanism begins the process in the fully retracted position (1). In this position, the sample flows through the barrel of the injection syringe, through the needle to the waste diverter valve and into the waste bottle. After approximately 0.5mL of sample is flushed to waste, the syringe barrel is filled with sample. The syringe mechanism is then moved forward by a piston, so that the syringe needle penetrates the waste diverter seal, then the GC injection port septum (2). Once the injection needle has penetrated the GC injection port to the full depth, the syringe body is pushed forward over the needle, displacing the sample into the injection port (3). Once the sample has been injected, the syringe mechanism retracts, withdrawing from the GC injection port.

GC INJECTORS

Model 8640 20-Vial Integrated Liquid Autosampler

General Operating Procedures

1. Fill each 2mL vial at least 75% full with liquid sample. Close the vials so that they are vapor-tight, with the Teflon side of the vial septa facing downward into the vial.
2. The sample tray is inserted and removed from the 8640 in one direction only. To remove the sample vial tray, push it away from you, toward the back of the GC, until it is free of the autosampler assembly. Place the filled sample vials in the tray. Reinsert the sample tray into the 8640 assembly from the front. Push it gently toward the back of the GC until the white lines at the tip of the white arrow on the sample tray are aligned with the front edge of the 8640. The sample tray is then in the ready position, with vial number one in place under the sample probe. The sample tray shown below, right is almost in the ready position (it was left partially out for visibility of the lines and arrow).



3. Activate and heat the GC detector(s).
4. Load or create a column oven temperature program.
5. Load or create an event table. Version 2.74 (and higher) of the PeakSimple software includes an event table file called "8640as.evt" as a general event table for use with the 8640 autosampler. When you load this event file, the default relay descriptions will not match the actual 8640 autosampler relay descriptions. These autosampler-specific descriptions must be entered by you, the user. The relays assigned to the autosampler are as follows:

- Relay A - moves the sample probe DOWN
- Relay B - moves the sample probe UP
- Relay C - moves the syringe carriage FORWARD
- Relay D - INJECTS the contents of the syringe
- Relay E - ADVANCES the tray one position
- Relay F - PRESSURIZES the sample vial

WARNING!
To avoid injury, keep your hands clear of the 8640 during operation.

See the event table shown at right for appropriate descriptions. The 8640 relay descriptions are also labeled on the right-hand side of the GC.

6. Set the autosampler air or nitrogen tank to 60psi. Set the carrier gas to 10mL/minute (the equivalent psi setting for your machine is labeled on the right panel of the GC). The amount of sample used to flush the needle can be adjusted by varying the pressure of the gas used to force the sample from the vial. This gas pressure is adjusted with the EPC trimpot on the top edge of the GC's front control panel, located directly above the vertical label "VIAL PRESSURE" on the front control panel. Using the event table at right, you should count 25 drops during the time that the gas is pressurizing the sample (0.600 minutes).

8640.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero data system signal
0.050	A ON	Sample probe DOWN
0.100	F ON	Vial pressure ON (pressurize the sample vial)
0.650	A OFF	Release pressure holding sample probe DOWN
0.700	F OFF	Vial pressure OFF
0.750	B ON	Sample probe UP
0.800	C ON	Syringe carriage FORWARD
0.850	D ON	Sample syringe INJECT
1.000	C OFF	Syringe carriage RETRACT
1.050	D OFF	Sample syringe RETURN
1.100	E ON	Tray advance ON
1.200	E OFF	Tray advance OFF
1.300	B OFF	Release pressure holding sample probe UP

7. The injection volume is factory set at 1µL, but is adjustable to 0-3µL. Loosen the 2 hex-head lock nuts, then turn the knurled nut while observing the needle in the syringe barrel to achieve the desired injection volume (please see the picture on the **Changing the Needle** page to locate the lock nuts and knurled nut).

GC INJECTORS

Model 8640 20-Vial Integrated Liquid Autosampler

Changing the Needle

In the course of normal operation, the 8640 sample injection needle may become bent or otherwise damaged and require replacing. Make sure the syringe mechanism is fully retracted before starting; this is the default position to which it should return after a sample injection sequence.

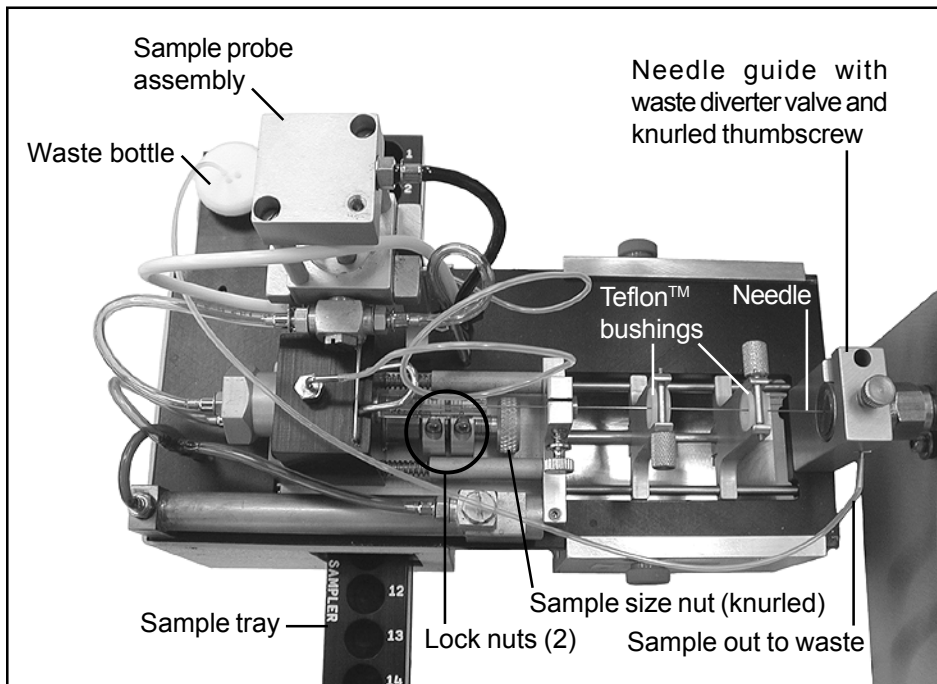
Replacement needles are available from Central Instruments under part number 502743. Syringe barrel and needle sets are available under part number 503188. Call Central Instruments at:

225-261-1917

Or write to:

P.O. Box 337

Greenwell Springs, LA 70739
USA.



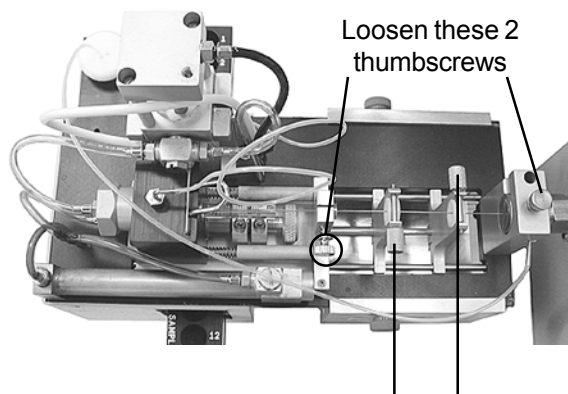
1. To remove the needle, loosen the thumbscrew on the top of the waste diverter and the thumbscrew on the needle guide closest to the syringe barrel. Loosen and remove the two bushing retainers. Carefully lift out the needle, the two Teflon™ bushings and the waste diverter valve together. You will have to push the waste diverter valve out of the needle guide, and angle the needle tip out through the slot in the side of the waste diverter needle guide as you pull the needle from the syringe barrel.

2. Slide the waste diverter valve and the two Teflon™ bushings off the old needle and onto the replacement needle.

3. Place the needle into the thumbscrew needle guide and the syringe barrel, and carefully angle the needle with the bushings and waste diverter valve into place, using the slot in the waste diverter needle guide to get the tip of the needle into alignment with the syringe barrel.

4. Position the two bushings in their cradles, then replace and tighten the bushing retainers. Tighten the thumbscrews on the needle guide and waste diverter.

5. Adjust the sample injection volume by loosening both hex-head lock nuts, then turning the knurled thumbscrew to achieve the desired volume. Tighten the lock nuts.

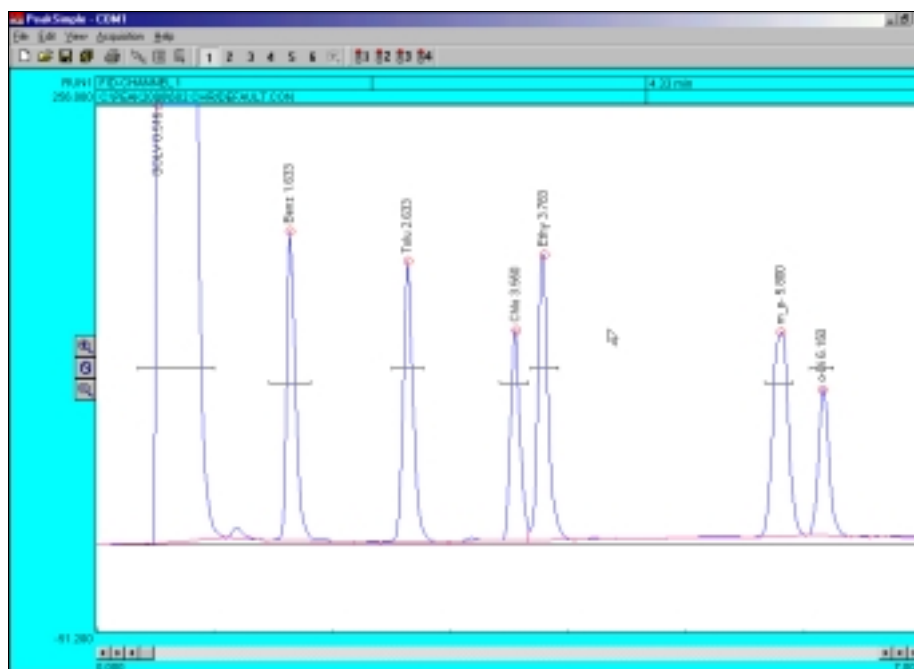


Loosen and remove these 2 bushing retainers, then remove the 2 bushings

PeakSimple 2000

Chromatography Integration Software

Advanced Tutorial



Installing PeakSimple 2000 from floppy disk or CD-Rom

- Start the Windows operating system in use on your computer. (Windows 95, 98, ME, 2000)
- Insert the PeakSimple 2000 disk or CD into your disk drive.
- Go to the **Start** menu in the bottom left hand corner of the windows screen and select **Run** from the set of icons.
- From the run menu, type **X:\setup** (where **X** is the letter of your computers disk drive).
- Now click on the **Continue** button with your mouse cursor or press the enter key on your keyboard to begin installation.
- To complete installation follow the onscreen instructions during the installation wizard.

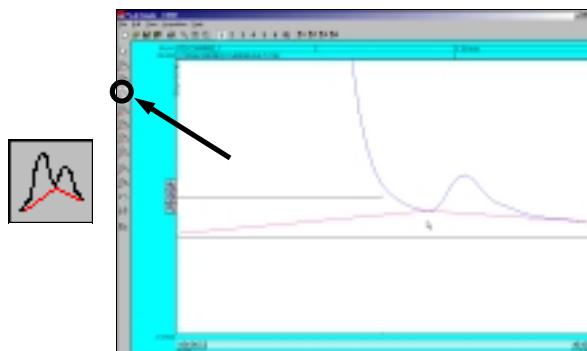
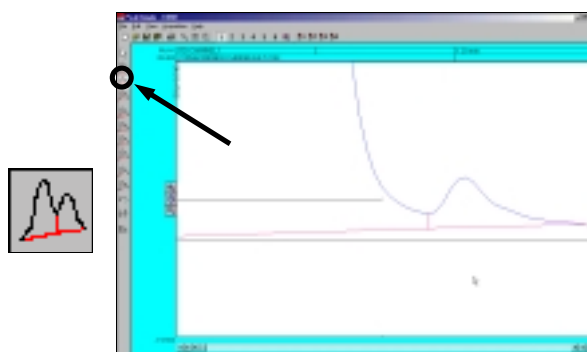
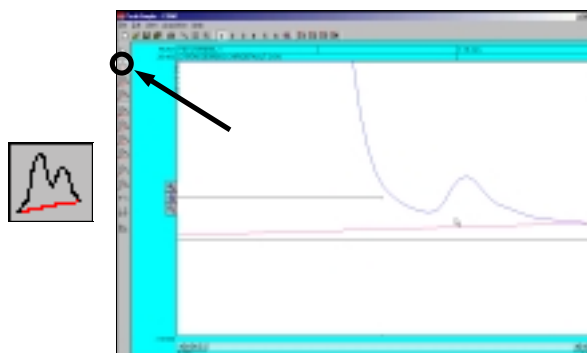
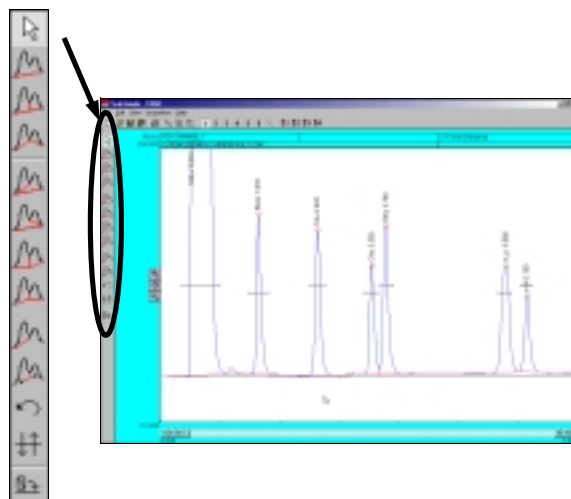
Installing PeakSimple 2000 from software download

- Start the Windows operating system and use an online browser to access www.srigc.com.
- From the menu on the left hand side of the screen select **Download our Software** and then download PeakSimple 2000 from the following page.
- Save the file to a temporary folder and then double click on it from My Computer to allow the program to self-extract.
- Once all the files have been extracted successfully double-click the install file and press the **Continue** button when prompted.
- Follow the onscreen instructions to complete the installation of PeakSimple.

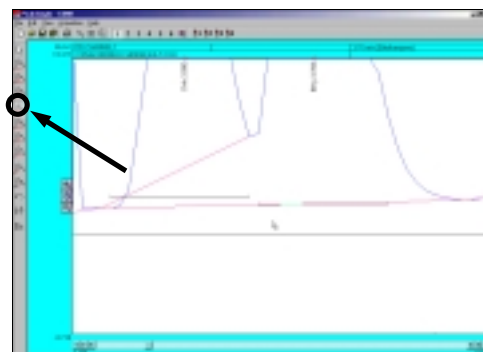
SRI Instruments 20720 Earl Street Torrance, CA 90503 U.S.A
 Telephone: (310) 214-5092 Fax: (310) 214-5097 sales@srigc.com www.srigc.com

Manual Integration

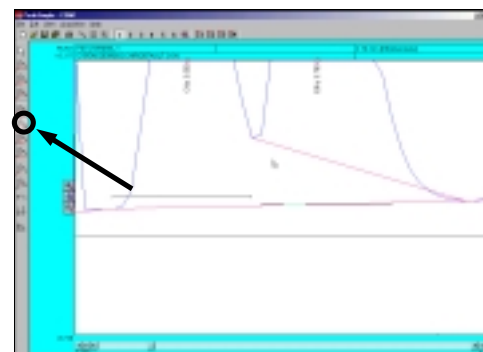
1. To manually integrate the PeakSimple baseline in a chromatogram use the manual integration tools found in the manual integration toolbar. To open the manual integration toolbar first have chromatogram 602.CHR loaded and then select **Edit** from the PeakSimple menu bar. From the drop down menu select **Manual integration** with the mouse cursor. The manual integration toolbar will now be displayed to the right of the PeakSimple toolbar in the left most part of the screen.
2. Use the None integration tool to add the area of the smaller peak to the area of the Solvent peak. First, zoom in on the solvent peak, the smaller peak to its right, and their baselines. Once the chromatogram is zoomed in select the **None** integration tool from the manual integration toolbar. With the None integration tool selected click once, using the left mouse button, on the valley between the solvent peak and the smaller peak.
3. Use the Drop integration tool to drop the baseline from the valley of the two peaks to an existing baseline. To drop the baseline select the **Drop** integration tool from the manual integration toolbar. Using the mouse cursor, click on the valley between the solvent peak and the smaller peak to drop the baseline.
4. The Based integration tool raises the baseline to the valley between two specified peaks. With the baseline dropped, click on the **Based** integration tool button and then click on the valley between the solvent peak and the smaller peak to its right to raise the baseline to the valley.



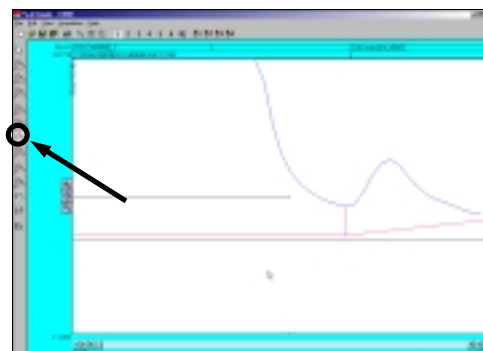
5. The Lead skim integration tool allows a peak's area to be skimmed off of the leading edge of another peak. To use the Lead skim tool first unzoom off of the solvent peak and the other smaller peak and then zoom in on the Chlorobenzene peak, the Ethylbenzene peak, and the baseline. After the chromatogram is zoomed click on the **Lead skim** integration tool button and then click on the valley between the two peaks with the mouse cursor.



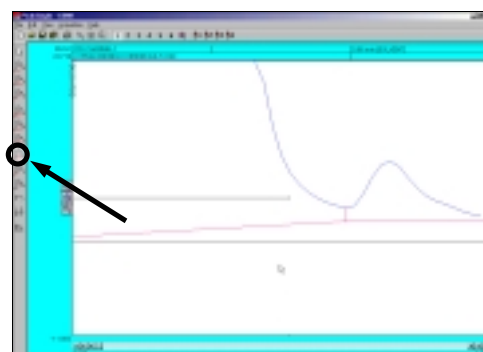
6. The Trail skim integration tool is similar to the Lead skim tool except a peak's area is now skimmed off of the trailing edge of another peak. Select the **Trail skim** tool button from the manual integration toolbar and then click on the valley between the Chlorobenzene and Ethylbenzene peaks with the mouse cursor to see the Ethylbenzene peak skimmed off of the Chlorobenzene peak.



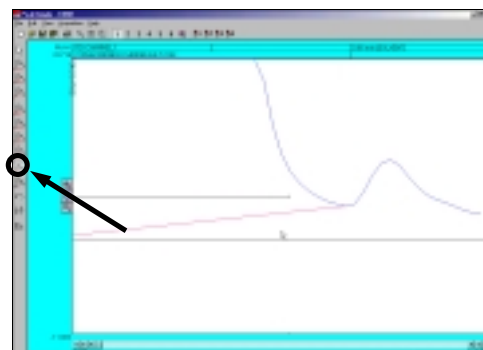
7. The Lead horizontal tool constructs the baseline horizontally for the leading peak while the trailing peak's baseline stretches from the horizontal line to the next valley. Unzoom off of the Chlorobenzene and Ethylbenzene peaks and instead zoom in on the Solvent peak, the smaller peak to its right, and the baseline. Click on the **Lead horizontal** integration tool in the manual integration toolbar and then click, using the left mouse button, on the valley between the solvent peak and the other smaller peak.



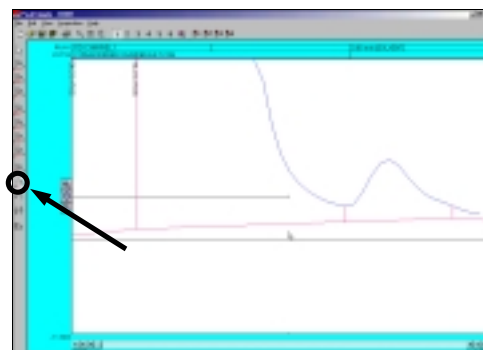
8. The Trail horizontal integration tool drops the baseline horizontally for the trailing peak while the lead peak's baseline stretches from the horizontal line to the previous valley in the chromatogram. After selecting the **Trail horizontal** tool in the manual integration toolbar click with the mouse cursor on the valley between the two zoomed in peaks.



9. The Inhibit tool ends the baseline after a valley effectively inhibiting a peak's area from being counted with the rest of the chromatogram. To use the Inhibit integration tool select the **Inhibit** tool button from the manual integration toolbar and click on the valley of the Solvent peak and the smaller peak to its right.



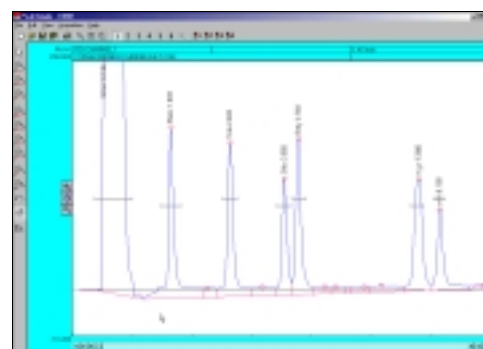
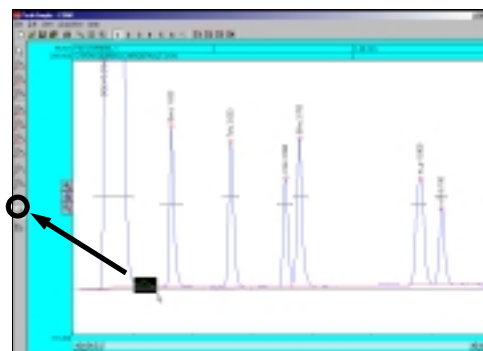
10. The Rubber Band tool is used to manually draw the baseline in a chromatogram. To use the Rubber Band tool first scroll the X-axis scrollbar all the way to the left to **0.000**. Select the **Rubber Band** tool from the manual integration toolbar and draw a line from the valley between the Solvent peak and the small peak to its left to the valley between the smaller peak to the right of the Solvent peak and the peak to its right.



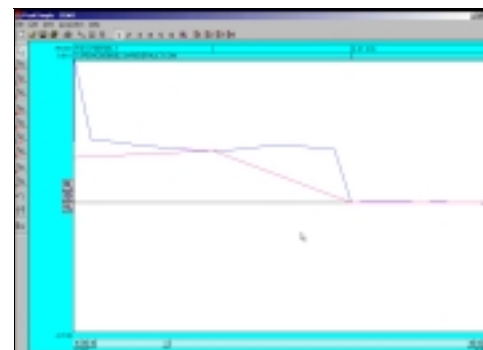
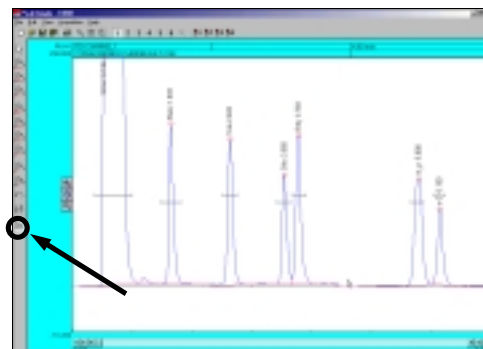
11. To undo a change made to the baseline of a chromatogram with the manual integration tools use the Undo button found in the manual integration toolbar. To undo the changes made to the baseline using the Rubber band tool click on the **Undo** button with your mouse cursor. All changes made to the baseline will now be undone.



12. The Reverse tool allows the inverting of a peak in a chromatogram. **Note:** To reverse the orientation of the X-axis in real time go to the Events table. First unzoom off of the Solvent peak and the smaller peak to its right and then select the **Reverse** tool from the manual integration toolbar and click and hold the left mouse button while the area of the chromatogram you want to reverse is dragged over with a black box. Let go of the mouse button when the desired area is selected to reverse the orientation.

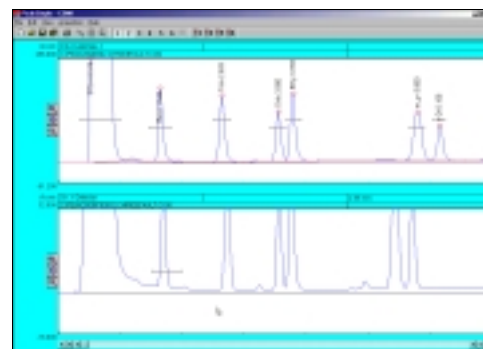
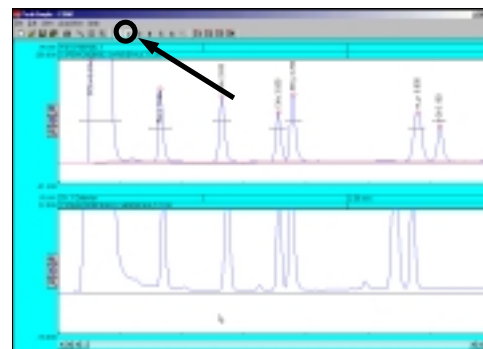


13. The Zero tool is used to set the value of the data line at a selected point and following in the chromatogram to zero. First undo the changes done to the chromatogram by the Reverse tool by reopening 602.CHR in the PeakSimple menu bar. **Note:** Changes made to a chromatogram by the Reverse tool and the Zero tool cannot be undone with the Undo tool. Once the file is reopened click on the **Zero** tool and click anywhere on the baseline between the Ethylbenzene peak and the two peaks to its right with the mouse cursor to set the data line at zero.

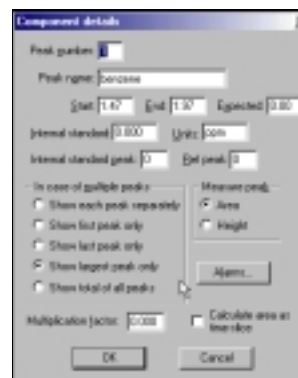


Creating Component Tables

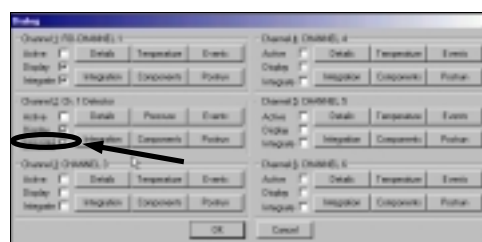
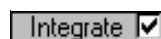
1. To create a component table from scratch open up a second channel in the PeakSimple window by clicking on the Display Channel 2 button in the PeakSimple toolbar. Once the second channel is open click on **File** and then **Open** to get to the Load chromatogram file window. Select file **FID602.CHR** from the list of files and select the Channel 2 radio button to open the file in channel 2. Click **OK** with the mouse cursor to load the file.
2. In channel 2 locate the second tall peak from the left and right click on it with the mouse cursor. From the resulting menu select **Add component** to add a retention window bar to the peak. Once again right click on the peak and select **Edit component** from the menu to open up the Component details window.



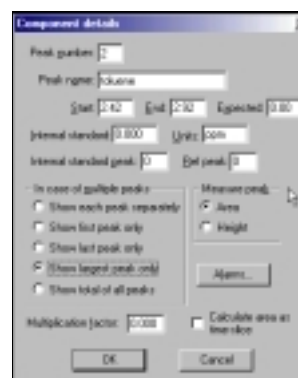
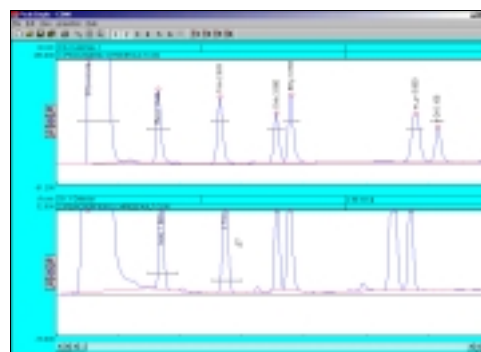
- Once the Component details window is open locate the Peak number dialogue box and add the number **1**. Immediately underneath the Peak number box is the Peak name dialogue box. In the Peak name dialogue box input **benzene** to name it. Locate the Units box and put **ppm** to make the units parts per million. Locate the In case of multiple peaks options box and select the radio button for **Show largest peak only**. Click on **OK** with the mouse cursor to close the window.



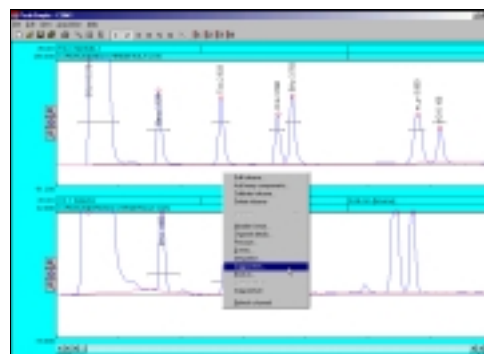
- Go to **Edit** in the PeakSimple menu bar and then **Channels** from the resulting menu. The Channel controls window is now open. Locate the Channel 2 options box and the Integrate checkbox. Check the **Integrate** checkbox and then click on **OK** with the mouse cursor to close the window. The peak in the second channel should now identify itself as benzene.



- Locate the large peak to the right of the benzene peak in the second channel. Right click and then select **Add component** to add a retention window bar to the peak. Right click again and go to **Edit component** to open up the Component details window. Change the Peak number to **2**, the Peak name to **toluene**, the Units to **ppm**, and the In case of multiple peaks options box to **Show largest peak only**. Click on **OK** with the mouse cursor to exit the window.



- Right click anywhere on the second channel and select **Components** from the list of options. Once the Channel 2 components window is open make sure all the data is correct and then click on **Save** to save the Component data to disk. Name the file **Ctable** and then click on **OK** to close the window. An unlimited number of component windows may be added to the component table.



Channel 2 components

Peak	Name	Start	End	Calibration
1	benzoin	1.000	1.500	
2	salicin	2.000	2.500	

Buttons: Add, Change, Remove, Calibrate, Load, Save, Use, Exit, OK

Temperature Programming

- To modify the temperature programming in PeakSimple first open chromatogram 602.CHR and then right click anywhere on the chromatogram. From the drop down menu select **Temperature** to open up the Temperature control window.
- In the Temperature control window click using the mouse cursor on the set of numbers in the box and select **Change** from the group of buttons below. The Temperature segment details window will open allowing the modification of the temperature programming. Locate the Hold for dialogue box and insert a **2** in the box. Click on **OK** to close the window and go back into the Temperature control window.



Temperature segment details

Initial temperature: 40 deg

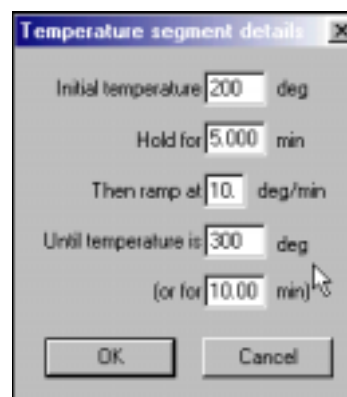
Hold for: 2 min

Then ramp at: 10. deg/min

Until temperature is: 200 deg
(or for 16.00 min)

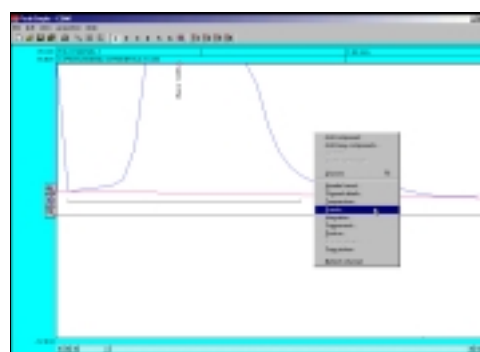
Buttons: OK, Cancel

3. Select the **Add** button from the Temperature control window to open up the Temperature segment details window once again. Leave the Initial temperature at 200 and insert a **1** in the Hold for dialogue box. Change the Then ramp at dialogue box to **5** and the Until temperature is box to **250**. Click on **OK** to close the window and to see the new temperature data added to the temperature box. Click on **OK** to close the window.



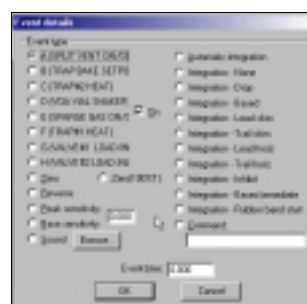
Events Table

1. To modify up the Events table in PeakSimple open up chromatogram 602.CHR and zoom in on the benzene peak, the smaller peak to its right, and the baseline. Right click anywhere on the chromatogram and select **Events** from the drop down menu. Doing this will open up the Events window where specific events can be added to the chromatogram.



2. Click using the mouse cursor on the **Add** button to view the Event details window. A list of event types are available with their radio buttons to either select or deselect the event.

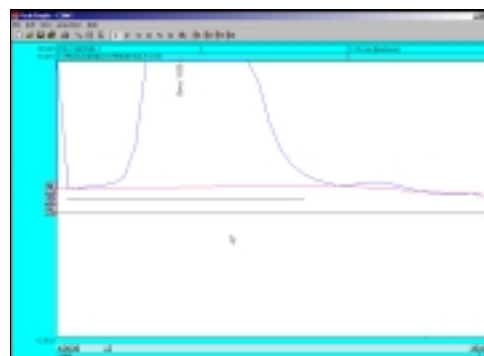
Note: The event types to the left of the window are real-time and thus will only affect the chromatogram when A/D hardware is connected. The event types to the right are concerned only with integration and their changes will be immediately evident after returning to the main screen and selecting **Reintegrate** from the **Edit** menu bar.



3. In the Event details window locate and select the relay **G** radio button with the mouse cursor and then locate the Event time dialogue box and enter **.1** in the box. Click on **OK** to exit the window. **Note:** The relay might be used to actuate a valve when hardware is connected. The event type will now be added to the Events table. Select the **Add** button and now locate and select the **Zero** event type radio button. Leave the Event time box at 0.000 and once again click on **OK** to exit the window and add the event to the Events table. **Note:** The Zero event auto-zeros the detector signal at the beginning of the run. Click on the **Add** button again and select the **Integration-Based immediate** radio button in the Event details window and input **1.86** in the Event time dialogue box. Select **OK** to exit the window.

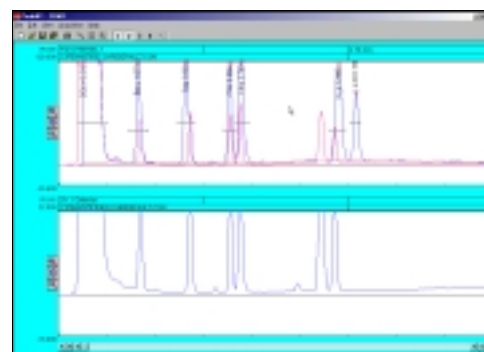
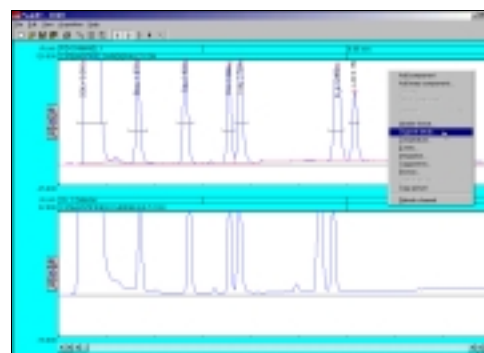


4. There are now three events in the Events table. Click on **OK** to exit the Events window and then hit the **Enter** button on the keyboard to reintegrate the baseline according to the events in the Events table. Notice that the baseline is connected to the data line at 1.86 minutes.

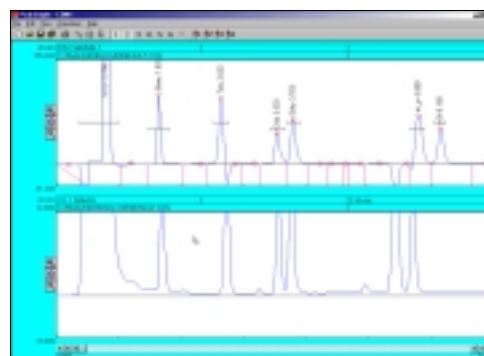
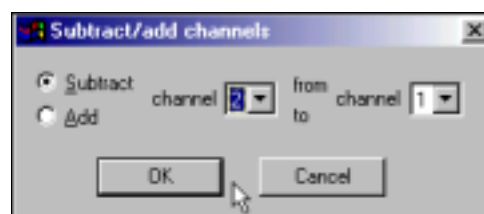
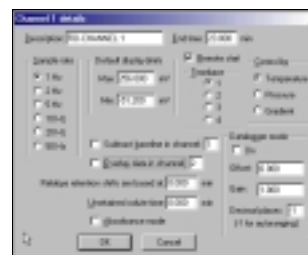


Overlay and Subtract

1. To overlay one PeakSimple chromatogram on top of another chromatogram open up a second channel in the main screen and load chromatogram 602.CHR in the first channel and chromatogram FID602.CHR in the second channel. Right click anywhere in the first channel and select **Channel details** from the drop down menu.
2. In the Channel 1 details window locate the Overlay data in channel checkbox and check it and then input a **2** in the dialogue box to the right. The chromatogram in channel 2 is now overlaid on top of the chromatogram in channel 1. The overlay appears in a different color.

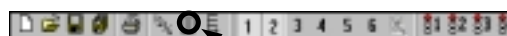


- Right click anywhere on the first channel and select **Overlay adjustment** from the drop down menu. In the Overlay adjustment window locate the Factor scroll box in the X box. Experiment scrolling the X factor up or down to shift the overlaid chromatogram to its right or left. Locate the Factor scroll box in the Y box and experiment scrolling the Y factor up or down to move the overlaid chromatogram up or down. Click on the **Close** button to close the window.
- To subtract a chromatogram in one channel from another channel, right click using the mouse cursor on channel 1 and select **Channel details**. From the Channel 1 details window deselect the Overlay data in channel checkbox and then click on the **OK** button to exit the window.
- Go to the **Edit** menu bar and select **Subtract/Add channels** from the drop down menu. In the Subtract/add channels window make sure the Subtract radio button is selected and that channel 2 is being taken from channel 1. Click on the **OK** button to make the changes take effect and have channel 2 subtracted from channel 1. The normal way to use this feature is to subtract a drifting baseline from a chromatogram.

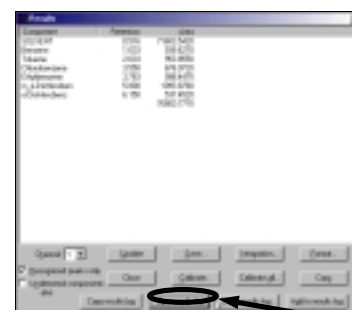


Results Log

- Open chromatogram 602.CHR in the PeakSimple main screen and then select the **Results** button from the PeakSimple toolbar. In the Results window click on the **Clear results log** button at the bottom of the window. Click on **Yes** from the resulting window to clear the results.
- Locate the **Add to results log** button and click on it three times to add the results on the screen to the Results log three times. Click on the **Show results log** button to view the results log in the Windows Notepad. Exit the Windows Notepad program by selecting **File** from the menu bar and then **Exit**.



Clear results log



3. In the Results window locate the **Copy results log** button at the bottom of the window and click on it with the mouse cursor (don't confuse the Copy button with the Copy results log button). Open up Microsoft Excel (or if Excel is not loaded Microsoft Word or Power-Point) and select **Edit** from the menu bar and then **Paste** to copy the results log to Excel.
4. Go back into PeakSimple and close the Results window by selecting the **Close** button. Right click using the mouse cursor on the chromatogram and select **Postrun** from the drop down menu to open the Post-run actions window. From the window locate the Add to results log checkbox and add a check to the box. By selecting the Add to results log checkbox all results from data analysis will automatically be added to the results log after the run is done. Click on **OK** to exit the window. In this way a summary of many analyses can be automatically created and then exported from PeakSimple.



This concludes the PeakSimple 2000 Advanced Tutorial

Further documentation can be obtained by going to:
www.srigc.com online

If you have questions or would like to place an order call:
 (310) 214-5092

CHAPTER: MAINTENANCE

Topic: Installation of the optional Air Compressor in SRI 8610C GC

Parts List:	Quantity
TOGGLE SWITCH	1
FLAT WASHER	4
LOCK NUT	4
6" COPPER CONNECTING TUBING	1
1/16" to 1/8" SS BULKHEAD FITTING	1
AIR COMPRESSOR with extended leads and extended output tubing	1

1. Turn off all gas supplies to the GC and remove AC power plug from outlet.
2. Remove the BOTTOM COVER of the GC by removing the 6 retaining screws.
3. Mount the AIR COMPRESSOR to the existing studs (see accompanying diagram) with supplied WASHERS and 6/32" LOCK NUTS. Secure all wires and make sure no wires are contacting the AIR COMPRESSOR. AIR COMPRESSOR will become hot when operating.
4. Remove the hole plug from the GC front panel hole marked as internal air compressor on/off switch.
5. Install and secure the TOGGLE SWITCH through the CIRCUIT BOARD with wires facing towards the bottom of the GC.
6. Solder the 2 wires from the TOGGLE SWITCH to the 2 bottom connector holes on the CIRCUIT BOARD to the left of the TOGGLE SWITCH.
7. Remove the hole plug from the GC front panel hole marked as

AIR COMP.
OUT

.
8. Mount the STAINLESS STEEL BULKHEAD FITTING in the opening marked as

AIR COMP.
OUT

 on the left side of GC.
9. Connect the extended gas tubing from the AIR COMPRESSOR to

AIR COMP.
OUT

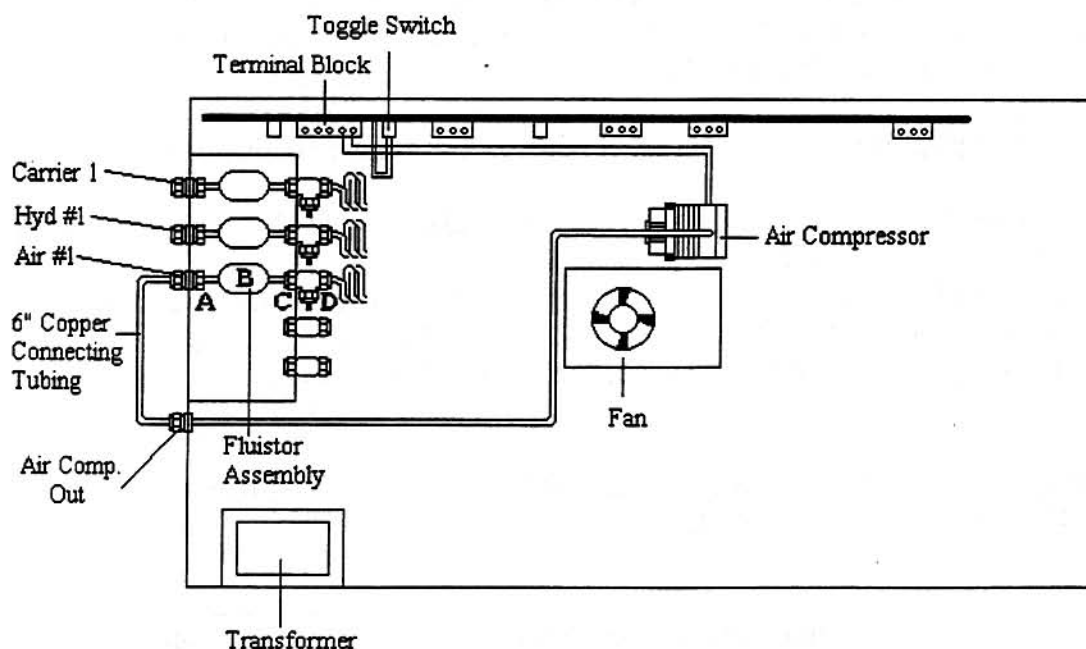
 marked on the left side panel of the GC. Route the tubing away from electrical components.
10. Connect the 2 wires from the AIR COMPRESSOR to first 2 positions of the blue terminal block, which is next to connector holes for the TOGGLE SWITCH wires on the CIRCUIT BOARD.
11. Disconnect the FLUISTOR ASSEMBLY (B in diagram) by loosening nut C in diagram with a 7/16" wrench and a 7/16" wrench on point D to prevent the T-fitting from roating.

CHAPTER: MAINTENANCE

Topic: Installation of the optional Air Compressor in SRI 8610C GC

Warning! Excess torque or stress on FLUISTOR ASSEMBLY can damage the delicate device.

12. Disconnect FLUISTOR ASSEMBLY by loosening nut A with a 7/16" wrench.
13. Remove the STAINLESS STEEL BULKHEAD FITTING from the GC.
14. Remove the frit, if present, inside the STAINLESS STEEL BULKHEAD FITTING by inserting a 1/16" diameter rod into the BULKHEAD FITTING and carefully tap the end of the rod on a desk. Without removing the frit, air supply pressure can be reduced from approximately 12 psi to 7 psi, which may result in low FID sensitivity.
15. Re-install the STAINLESS STEEL BULKHEAD FITTING and FLUISTOR ASSEMBLY.
16. Connect **AIR COMP. OUT** and **AIR #1** BULKHEADS, which are marked on the left side panel of the GC, with supplied 6" COPPER CONNECTING TUBING.
17. Re-secure the BOTTOM COVER of the GC.



Chapter: PeakSimple

Topic: Using the Windows Scheduler program to trigger PeakSimple's Autosampler queue

The Windows Task Scheduler program is supplied with the Windows operating system. It is found under Programs/Accessories/System Tools. The Scheduler allows you to trigger a PeakSimple Autosampler Queue or a specific control file at a scheduled time and date, or on a regular repeating basis using the computer's system clock (time/date).

When you click on the Add Scheduled Task icon in the Scheduler program, a Wizard will guide you through the process. When you get to the screen where you specify which program to start, enter the following line modified for your particular situation:

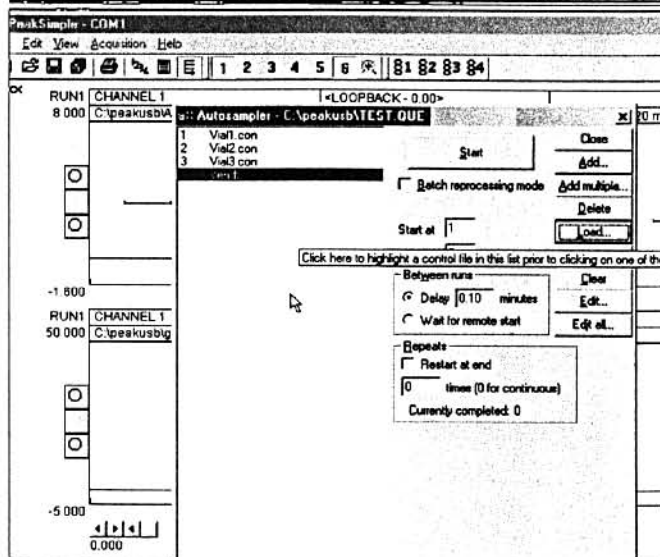
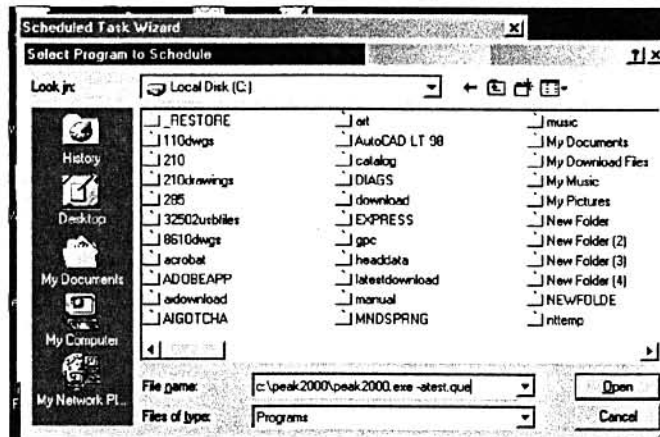
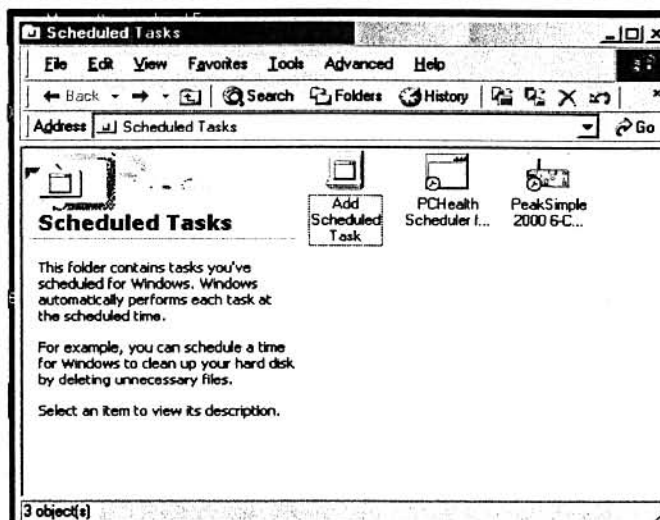
`c:\peak2000\peak2000.exe -atest.que`

C:\peak2000 is the folder or directory where the PeakSimple software has been installed. If you have installed PeakSimple in a different folder, substitute the name of your PeakSimple folder.

peak2000.exe is the name of the actual PeakSimple software program. If you have installed PeakSimple under a different name (later versions of PeakSimple may in fact have a different name) use the name of the PeakSimple program as it exists on your computer.

test.que is the name of the Autosampler queue file which you must have previously created in PeakSimple. Note that the -a must precede the name of the .que file. When you create the .que file in PeakSimple you can save the que under any name you want. The -a is a Windows programming convention and must precede the name of the que file you want to run.

When the Scheduler starts PeakSimple, the specified queue will begin. At the end of the queue, PeakSimple will wait for the delay time specified in the queue, and then PeakSimple will Close automatically.

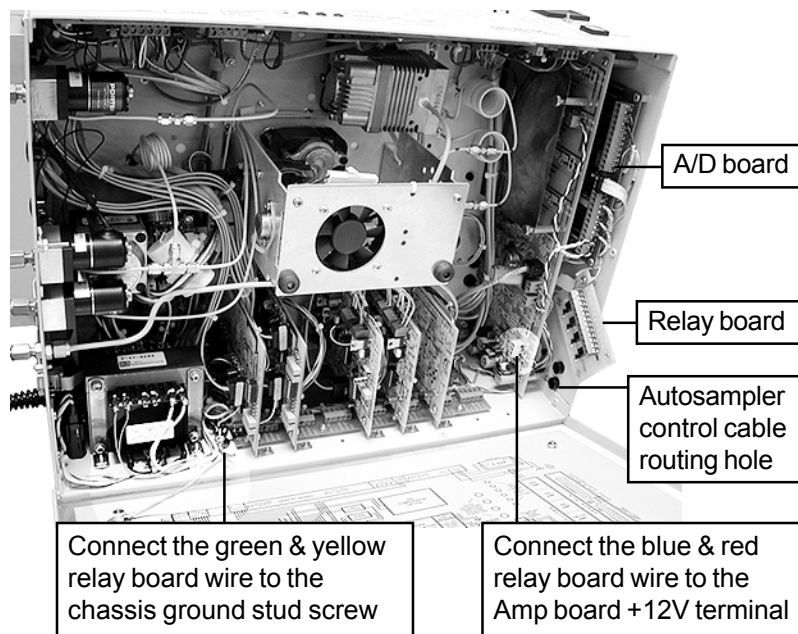


AUTOSAMPLERS

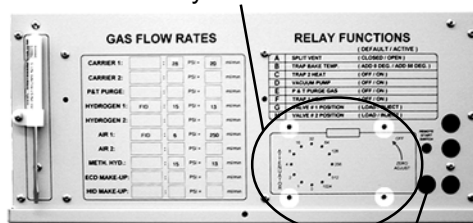
Installation of the Relay Board for the 10 Position Method 5030 Purge & Trap Autosampler (and other Autosamplers)

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

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



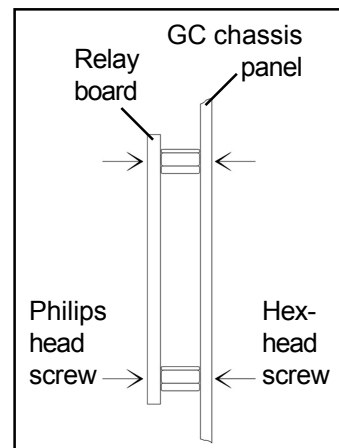
4 holes for Relay Board screws



Hole for autosampler control cable

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

2. Secure the four aluminum stand-offs in the relay board holes. Use the four hex-head screws provided, and secure the stand-offs from the outside of the



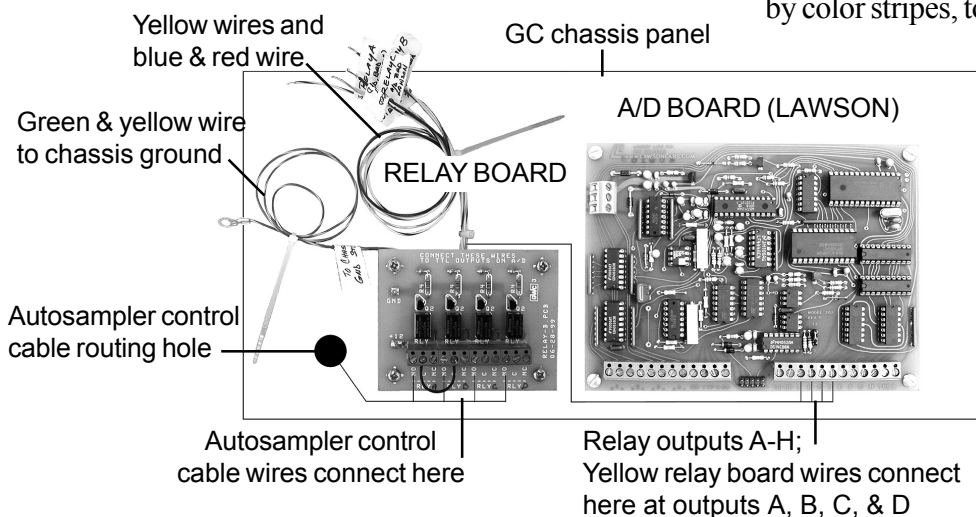
GC panel. Insert the relay board into position so that the component side faces outward. Secure it in place on the aluminum stand-offs with the four philips head screws provided.

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

4. Connect each of the four yellow wires, differentiated by color stripes, to the appropriate TTL relay outputs

on the A/D (Lawson) board. All eight of the TTL outputs are identical; use any available ones. Connect the blue and red wire to the Amp board 12V terminal.

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



PEAKSIMPLE SOFTWARE

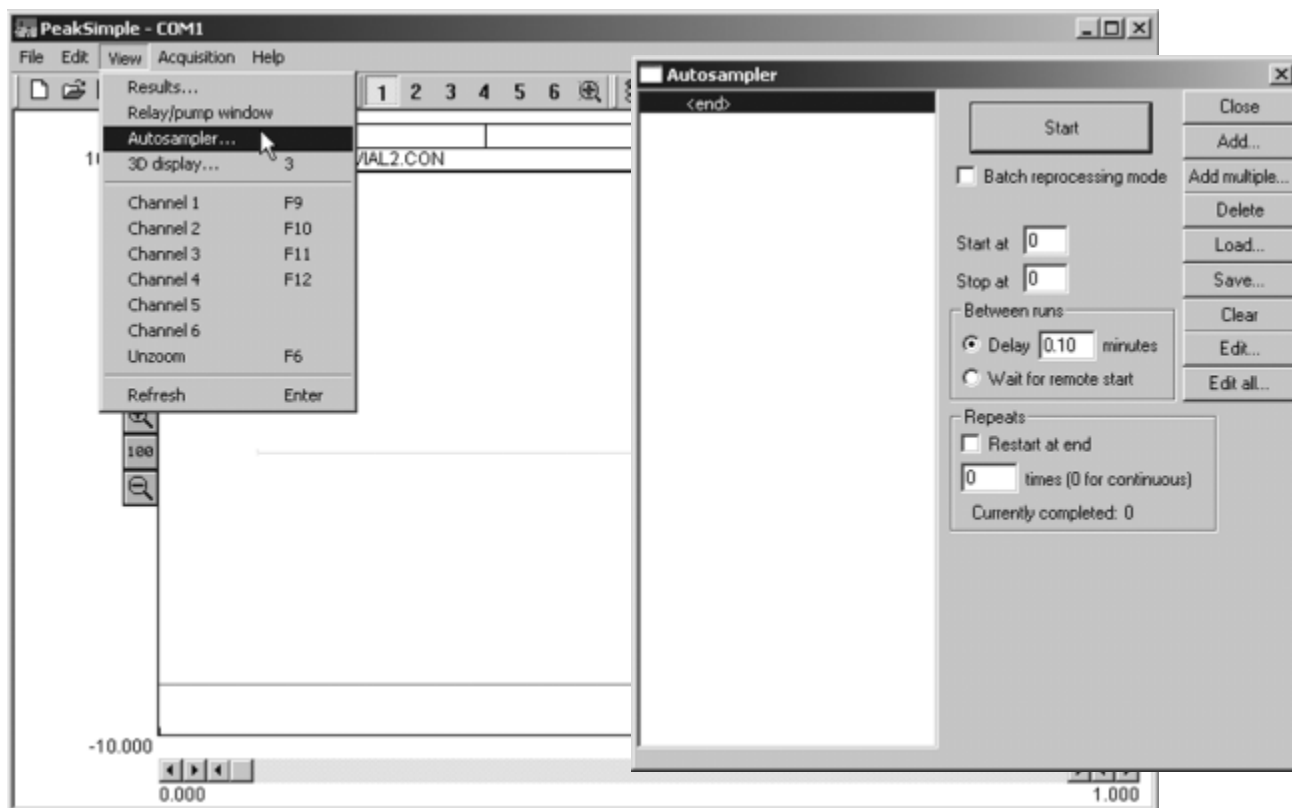
Autosampler Queue

The Autosampler Queue is the most powerful and complex function of PeakSimple, and requires a thorough understanding of the software's features. If you are at the beginner level, you should not attempt to use this feature.

The Autosampler Queue allows a list of control files to be run automatically in sequence. PeakSimple uses control files to save the operating settings of specific methods. Once all the user-definable parameters have been set, they can be saved in a control file for future use. Control files contain .CAL, .CHR, .CPT, .EVT, .GRA, and .TEM files, and every other user-modifiable parameter. You must begin with control files already made, to load into the Autosampler Queue. You must create or modify a control file for each vial.

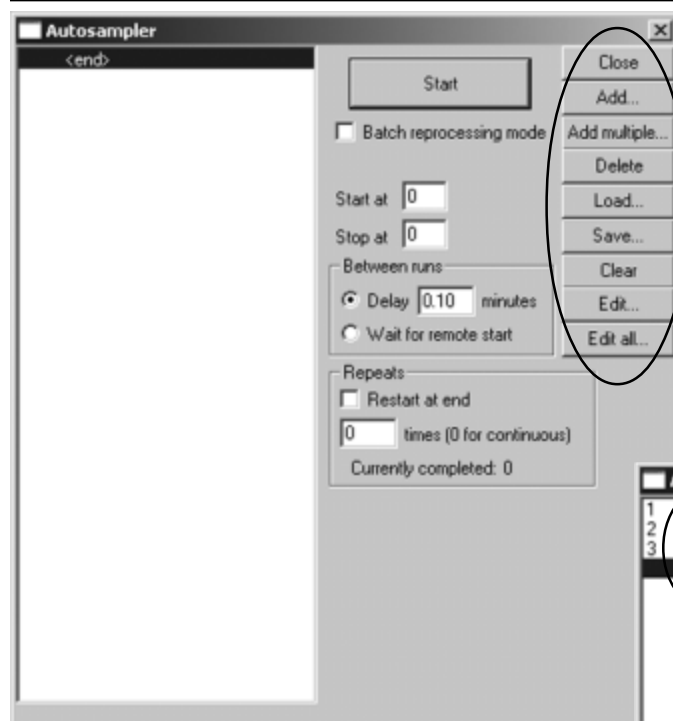
Each control file in the list must have a unique name, even if it is identical to others. It is helpful to include a number in the name of each control file that corresponds with its place in the list; for example: Vial01.con, Vial02.con, Vial03.con, etc. The control file(s) you wish to use in the Autosampler Queue must be saved in the PeakSimple application directory.

To open the Autosampler window, click View, then choose Autosampler.



PEAKSIMPLE SOFTWARE

Autosampler Queue

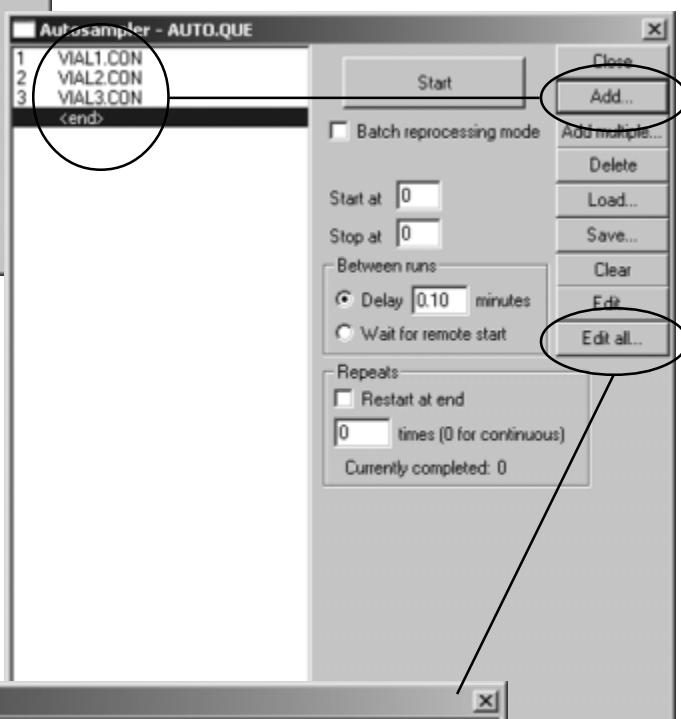


Autosampler Queue Buttons

There are several buttons on the right-hand side of the autosampler window. The **“Close” button** is for closing the Autosampler window. The **“Add” button** is for adding control files to the queue. Click the “Add...” button. Browse to the PeakSimple application directory, and select a control (.CON) file to place in the queue. PeakSimple will place the file in the queue above the .CON file you have highlighted, or at the end of the list if “<end>” is highlighted (default).

Since you can only add one control file to the queue at a time, it can get tedious, especially if you are using multiple instances of the same control file.

When this is the case, use the “Add...” button to add your master control file to the queue, then click on the “Edit all” button to open the control file attributes spreadsheet, shown below.



Autosampler queue					
Num	Control file	Temperature file	Standard weight	Sample weight	Recalib. le
1	VIAL1.CON		1.000000	1.000000	-2
2	VIAL2.CON		1.000000	1.000000	-2
3	VIAL3.CON		1.000000	1.000000	-2

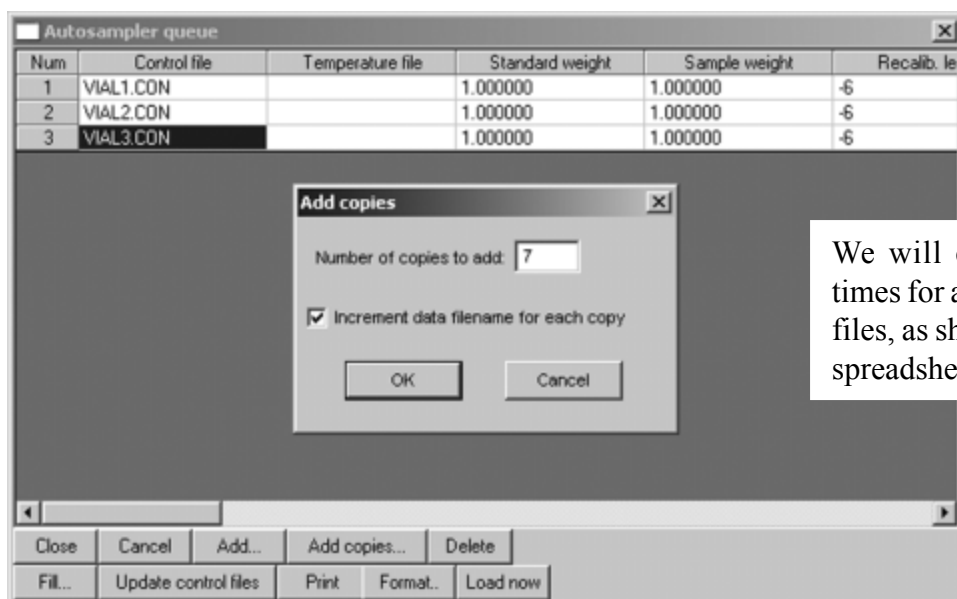
Buttons at the bottom: Close, Cancel, Add..., Add copies..., Delete, Fill..., Update control files, Print, Format..., Load now

PEAKSIMPLE SOFTWARE

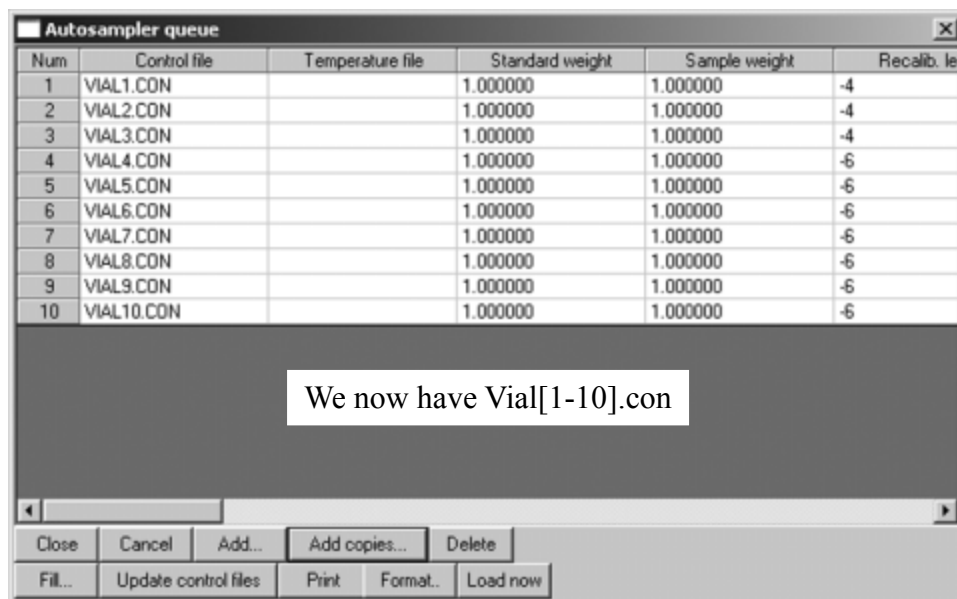
Autosampler Queue

Highlight the control file that is your master, or the one that is to be repeated, and click the “Add copies” button. In the window that pops up, enter the number of copies you wish to make, and check the “Increment data filename for each copy” box. PeakSimple will save the copied control files in the application directory.

The following example is for a 10 vial liquid autosampler for GC. In this example, we have three control files. The third control file is the one we will copy.



We will copy Vial3.con 7 times for a total of 10 control files, as shown in the Edit all spreadsheet below.

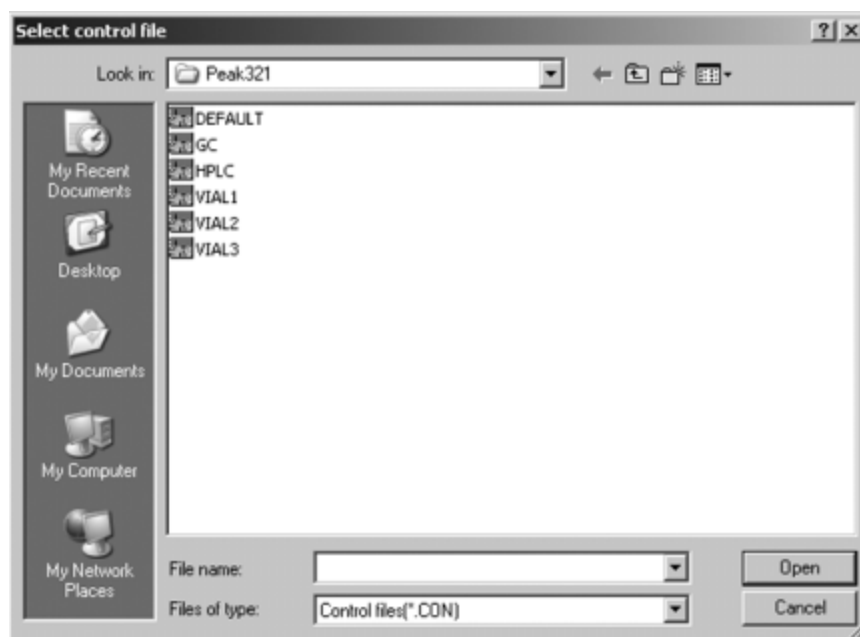


We now have Vial[1-10].con

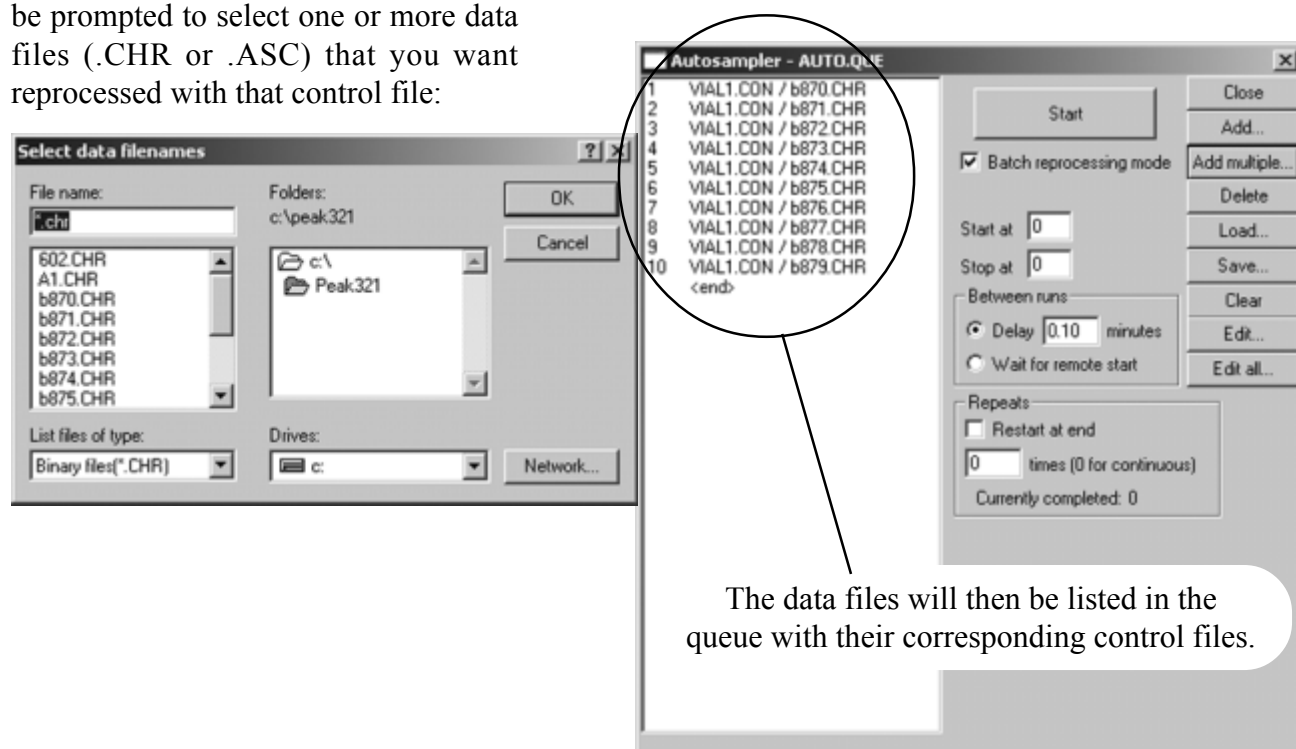
PEAKSIMPLE SOFTWARE

Autosampler Queue

The “**Add multiple**” button is for batch reprocessing. Batch reprocessing allows you to reprocess the raw data through a designated control file. This is useful in the event of a mistake or omission, or when header/comment info needs updating. The “Batch reprocessing mode” checkbox to the left of the “Add multiple” button must be selected. Then, when you click the “Add multiple” button, you will be prompted to select a control file:

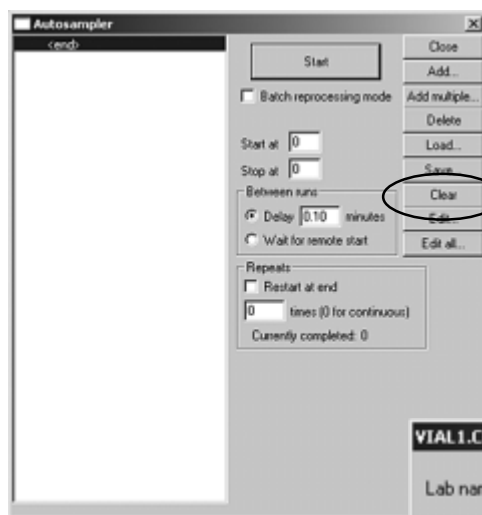
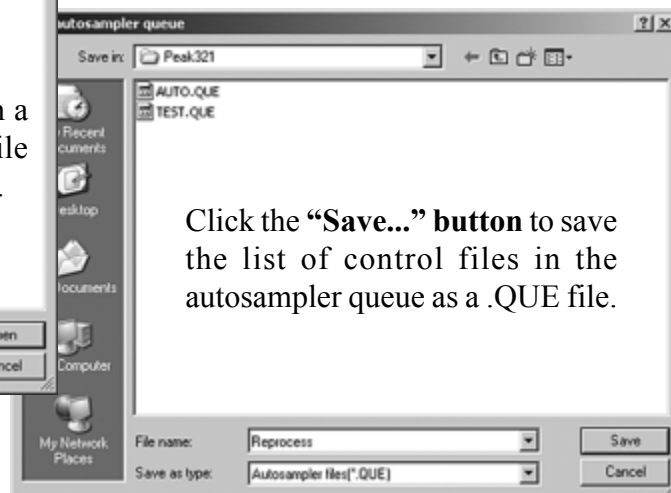
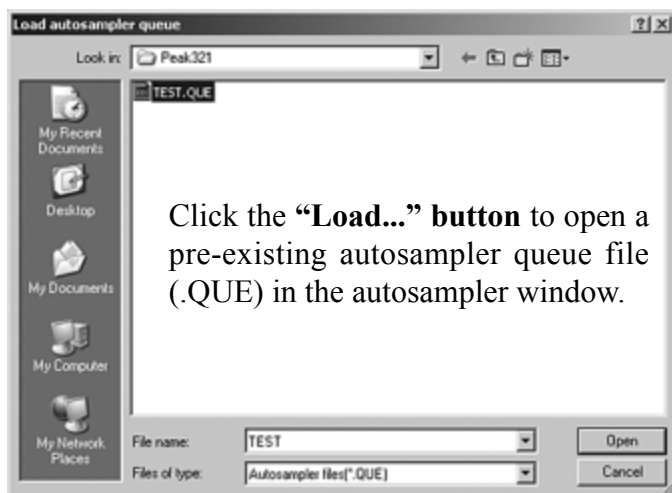


When you select a control file, you will be prompted to select one or more data files (.CHR or .ASC) that you want reprocessed with that control file:



PEAKSIMPLE SOFTWARE Autosampler Queue

To delete a control file from the autosampler queue, highlight it and click the **“Delete”** button. If no control file is highlighted, the last file in the list will be deleted when this button is clicked.



When you click the **“Edit...”** button after highlighting a control file in the queue, the control file is loaded and a screen opens in which you can edit common parameters. To edit other parameters of the control file, just close the “Edit” screen, then click on the PeakSimple window behind the Autosampler window to make the changes. Click File>Save control file when you are finished editing.

Lab name	SRI RUN#1	Data file	C:\Peak321\vb874.CHR
Client		Component file	QUEUE.CPT
Client ID		Event file	
Collection date		Temperature 1	
Holding time		Sample	VIAL1
Method		Operator	
Lab ID		QC batch number	
Description	1ST AUTOSAMP RUN	Standard weight	1.000
Column	RESTEK 15METER MXT-1	Sample weight	1.000
Carrier	HELIUM AT 300 ON DIAL	Recalibration level	0
		Add to results log	<input type="checkbox"/>

Channel: 1 (selected), 2, 3, 4, 5, 6

OK Cancel

PEAKSIMPLE SOFTWARE

Autosampler Queue

Click on the **“Edit all...”** button to open the control file attributes spreadsheet. The Edit all spreadsheet is a powerful tool for managing your control files; it allows you to edit any or all of the control files in the autosampler queue.

You can highlight an entire column to change...

...Or make changes in individual cells.

Num	Control file	Lab name	Client	Client ID	Collect
84	Sample80.CON	SRI RUN#2			
85	Sample81.CON	SRI RUN#2			
86	Sample82.CON	SRI RUN#2			
87	Sample83.CON	SRI RUN#2			
88	Sample84.CON	SRI RUN#2			
89	Sample85.CON	SRI RUN#2			
90	Sample86.CON	SRI RUN#2			
91	Sample87.CON	SRI RUN#2			
92	Sample88.CON	SRI RUN#2			
93	Sample89.CON	SRI RUN#2			
94	Sample90.CON	SRI RUN#2			
95	Sample91.CON	SRI RUN#2			
96	Sample92.CON	SRI RUN#2			
97	Sample93.CON	SRI RUN#2			
98	Sample94.CON	SRI RUN#2			
99	Sample95.CON	SRI RUN#2			

Num	QC batch	Standard weight	Sample weight	Lab name(2)	Clier
84		1.000000	1.000000	SRI Instruments	
85		1.000000	1.000000	SRI Instruments	
86		1.000000	1.000000	SRI Instruments	
87		1.000000	1.000000	SRI Instruments	
88		1.000000	1.000000	SRI Instruments	
89		1.000000	1.000000	SRI Instruments	
90		2.000000	1.000000	SRI Instruments	
91		1.000000	1.000000	SRI Instruments	
92		1.000000	1.000000	SRI Instruments	
93		1.000000	1.000000	SRI Instruments	
94		1.000000	1.000000	SRI Instruments	
95		1.000000	1.000000	SRI Instruments	
96		1.000000	1.000000	SRI Instruments	
97		1.000000	1.000000	SRI Instruments	
98		1.000000	1.000000	SRI Instruments	
99		1.000000	1.000000	SRI Instruments	
100		1.000000	1.000000	SRI Instruments	

Buttons: Close, Cancel, Add..., Add copies..., Delete, Fill..., Update control files, Print, Format..., Load now

There are ten buttons on the lower left corner of the “Edit all” spreadsheet.

Click on the **“Close”** button to close the spreadsheet window with a prompt to save your changes.

To close the spreadsheet window without saving any changes, click the **“Cancel”** button.

Click the **“Add...”** button to add a control file to the queue and spreadsheet.

Use the **“Add copies...”** button to add copies of a selected control file to the queue.

Click the **“Delete”** button to remove a highlighted control file from the queue. If no control file is highlighted, the last file in the list will be deleted when this button is clicked.

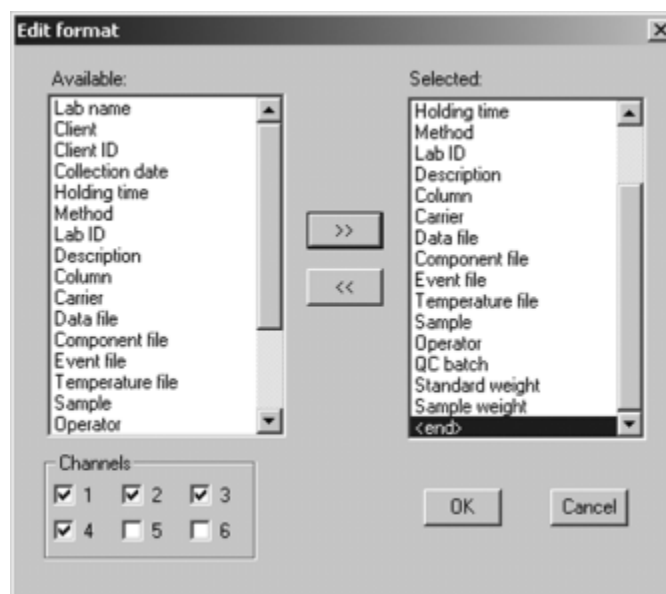
PEAKSIMPLE SOFTWARE Autosampler Queue

Use the **“Fill...”** button after highlighting a vertical column in the spreadsheet to fill the cells with new text. To highlight a vertical column, click in the uppermost cell you want to include, then drag your cursor down to the last cell you want included.

Num	Control file	Lab name	Client	Client ID	Collectio
84	Sample80.CON	SRI RUN#2			
85	Sample81.CON	SRI RUN#2			
86	Sample82.CON	SRI RUN#2			
87	Sample83.CON	SRI RUN#2			
88	Sample84.CON	SRI RUN#2			
89	Sample85.CON	SRI RUN#2			
90	Sample86.CON	SRI RUN#2			
91	Sample87.CON	SRI RUN#2			
92	Sample88.CON	SRI RUN#2			
93	Sample89.CON	SRI RUN#2			
94	Sample90.CON	SRI RUN#2			
95	Sample91.CON	SRI RUN#2			
96	Sample92.CON	SRI RUN#2			
97	Sample93.CON	SRI RUN#2			
98	Sample94.CON	SRI RUN#2			
99	Sample95.CON	SRI RUN#2			
100	Sample96.CON	SRI RUN#2			

Click the **“Update control files”** button to save all the changes made to the control files in the spreadsheet.

Click the **“Format...”** button to specify which control file parameters are displayed in the spreadsheet. Some commonly edited control file parameters are included by default, but the user may format the “Edit all” spreadsheet as desired. For example, if you have one detector, you can format the spreadsheet to display the parameters for one channel instead of all six (regardless of your GC and data system specs, PeakSimple always has six channel capability). To add a column to the spreadsheet, highlight the header in the list on the left, then click the double right arrow (>>). The header will appear in the list on the right, which is the list of selected features/column headers. To remove a header and column from the spreadsheet, highlight it in the “Selected” list, then click the double left arrow (<<).



Highlight a control file and click the **“Load now”** button to open it in the main PeakSimple window for editing.

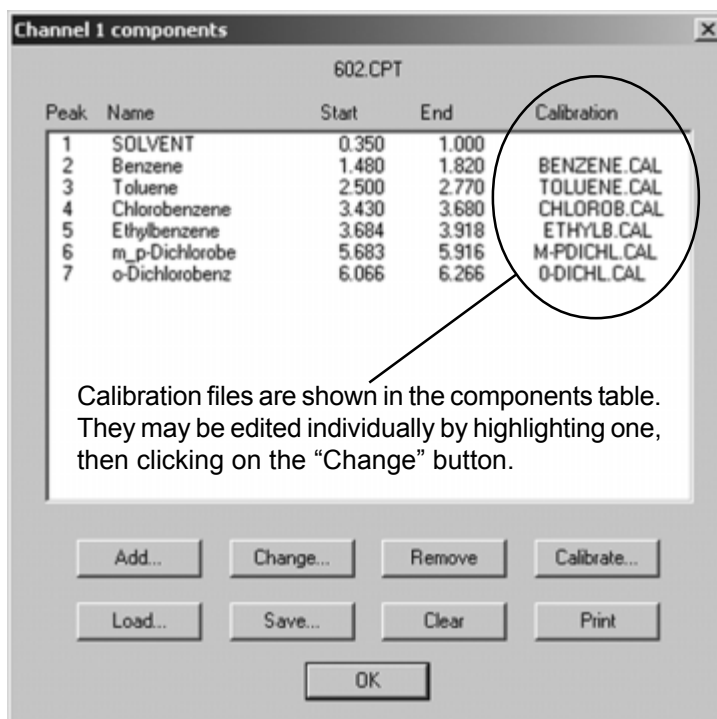
PEAKSIMPLE SOFTWARE

Autosampler Queue

Calibration

The calibration curve is calculated from user-generated results obtained at several different concentrations that span the expected range to be encountered in actual samples. Calibration is required for each component you expect to be in your sample, and for each detector you will be using in your analysis. Once calibration curves have been completed and calibration files saved, they will be included in the .CPT file.

It is typical to calibrate samples at the beginning of an autosampler queue. Remember, the calibration curve is calculated from user-generated results obtained at several different concentrations that span the expected range to be encountered in actual samples. You must have already created your calibration curves before using the Autosampler queue.



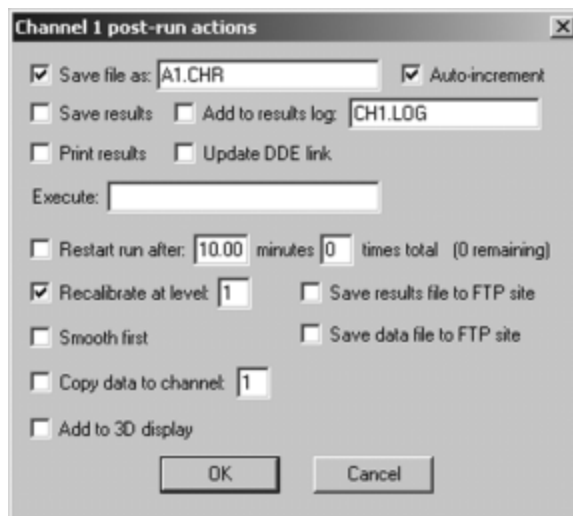
To make the first 3 vials calibration standards:

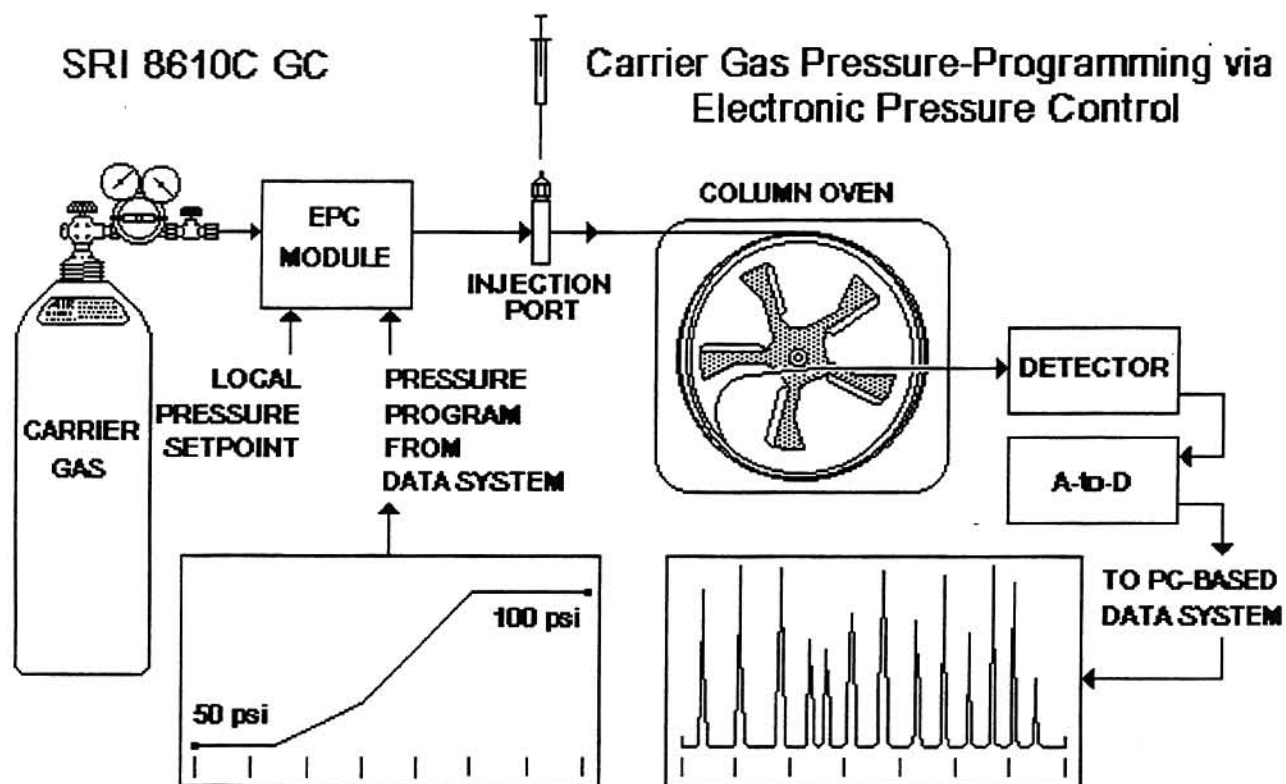
1. In PeakSimple, click “File,” then choose “Open control file.” Browse to the application directory, where you should already have saved the control files you want to use in the Autosampler queue. Select the control file that you want to run first. For each channel, open the post-run actions window, and select the “Recalibrate at level” box. Enter “1” in the box. Click “File” then choose “Save control file.”

2. Open the second control file in the Autosampler queue and do the same for each channel, except enter a “2” in the box. Save the second control file.

3. Repeat the steps for the third control file, except enter a “3” in the box. You can also edit the control files through the “Edit all” spreadsheet in the Autosampler queue.

In the post-run actions window, the “Recalibrate at level ____” check box recalibrates all identified peaks at the end of a run. The level of recalibration, from 1 to 7, is typed in by the user.





FLOW CHART ILLUSTRATING CARRIER GAS PRESSURE PROGRAMMING ON THE 8610C GC

All SRI 8610C gas chromatographs are equipped with Electronic (or Pneumatic) Pressure Control (EPC) of all system gases. Each gas, from the carrier gas, to the specific detector gases, such as FID hydrogen and FID compressed air, in the case of an FID detector, are controlled by a dedicated solid-state EPC module that electronically monitors and instantaneously adjusts the pressure being supplied to the particular feature. This electronic control facilitates extreme precision of gas flows to the various functions. Each EPC module features a local, user-adjustable setpoint accessed by a trimpot (variable potentiometer) located just above the particular function on the "at-a-glance" panel display. The carrier gas is among these adjustable setpoints. The term "local" refers to the fact that the "local" setpoint is set manually at the trimpot on the GC chassis. As in the case of the column oven temperature setpoint, the carrier gas pressure setpoint may be set "locally" (manually on the GC chassis), or from the computer via a pressure program. Created in the same format as a PeakSimple temperature program, the program signal is sent to the data system interface and converted to a control voltage that can increase, maintain, or decrease the carrier gas pressure automatically at the user's command.

The PeakSimple serial data system interface offers two rampable voltage outputs - one to program the column oven, and the other to program carrier gas pressure. Outputting a 0 to 5VDC variable signal, the EPC module will permit an output pressure of from 0 to 100psi (the carrier pressure shown is actually the column head pressure). Please note that any local setpoint value will be summed to this signal, resulting in the "total" setpoint value on the panel display. The carrier gas pressure regulator at the gas cylinder should be set 10psi higher than the highest programmed carrier gas head pressure desired for proper control. Ramping permits the head pressure to be varied, to speed or slow the elution of peaks from the analytical column as needed by the application or user.

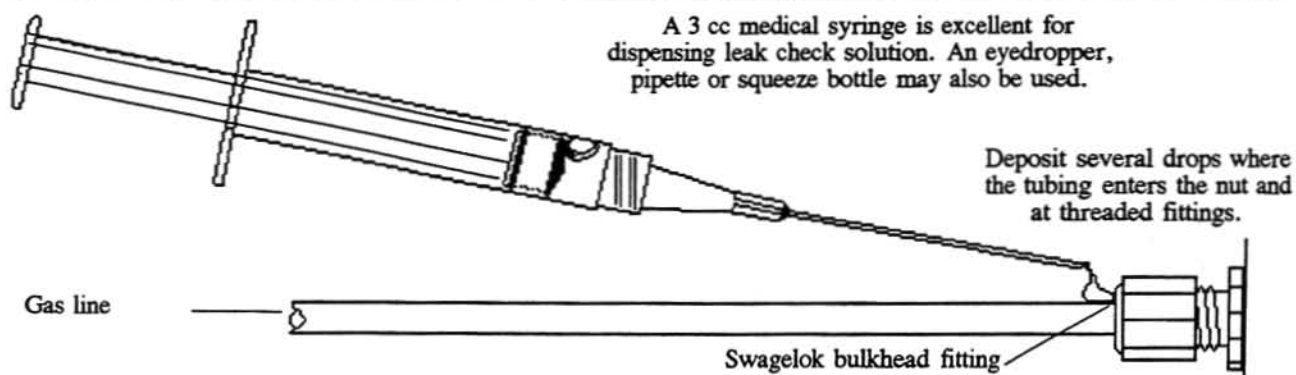
Once all of the appropriate gas supply sources and lines have been properly installed, along with all other GC columns and connections, the entire system should be systematically pressurized and checked for possible leaks. Begin by opening all of the compressed gas cylinder valves and setting exit pressures to the appropriate value for each cylinder regulator. Remember that cylinder exit pressures should never exceed the required GC pressure settings by more than 20 psi and 80 psi is the maximum pressure that the GC can safely handle.

First check for leaks in the lines and connections between the compressed gas cylinder and the GC flow control fluistors. With the system pressurized and the GC power turned off, close each of the compressed gas cylinder valves one at a time and closely watch the pressure indicator on the cylinder regulator to see if pressure decreases. If the system is leak free between these two points, the cylinder pressure indicator should not noticeably decrease for at least five minutes. If pressure does noticeably decrease over this time period, then it indicates a significant leak somewhere between the cylinder output and the GC fluistor. Any leak, especially with flammable gases, must be immediately located and repaired. The best way to check specific connections for leaks is with a leak check solution (see section below on Using Leak Check Solution). If pressure test indicates that the system is leak free from the cylinder to the fluistor, then proceed to check the rest of the carrier gas system for leaks. If the system does have a leak, locate and repair prior to proceeding.

Next check for leaks between the fluistor and injection port. Begin by disconnecting the column from the back side of the injection port. Next insert some type of pressure blocking fitting on the injection port where the column was attached. A standard Swagelok nut with an injection septum in place of the ferrule will work quite well. Turn the GC power and gas supply back on. Use the control panel to see what the **actual** carrier pressure value is and write it down. Now turn off the carrier gas supply at the cylinder once again. Wait 5 minutes and then use the GC control panel to view the **actual** carrier pressure once again. If this value has decreased in the 5 minute time frame and the previous test results were negative, it indicates that there is a significant leak somewhere in the internal GC carrier gas lines between the fluistor and the injection port. Once again immediately locate and repair any leaks using a leak check solution as described below.

After all of the leaks upstream from the column have been eliminated and confirmed by the two pressure tests described above, properly attach your column to the injection port. Use leak check solution to check all of the fittings within the column oven for leaks and repair any that you find.

Following all the instructions above will assure the operator that the system is leak free. Any time fittings are changed or the GC is relocated, the system should be rechecked for leaks. Failure to properly repair leaks can cause safety risks as well as operational malfunctions.



Leak Checking Solution

SRI recommends that a solution of 50% water and 50% alcohol (methanol, ethanol, or propanol) be used as a leak check solution. The water-alcohol mixture leaves no residue which could leak through the fittings and cause system contamination. Furthermore, water, when used alone and due to its high surface tension, tends to bead rather than flow into spaces between the tubing and the connectors where leaks may occur. A leak will show up as a stream or froth of tiny bubbles. Inspect any leaking fitting for damaged threads and reversed, missing, or damaged ferrules.

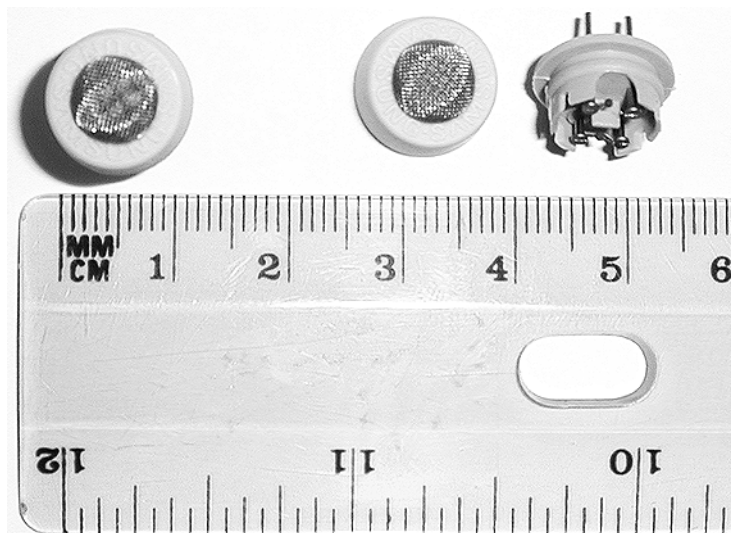
DETECTORS

Catalytic Combustion Detector - CCD

Overview

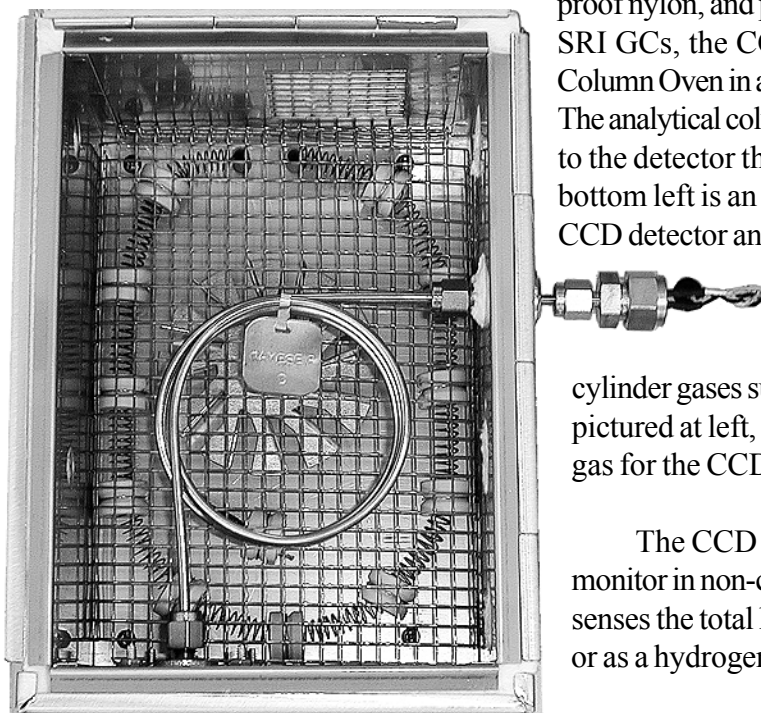


CCD on Column Oven



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

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



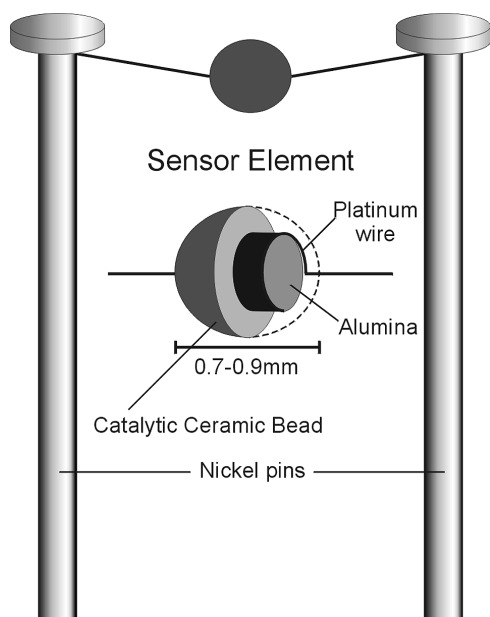
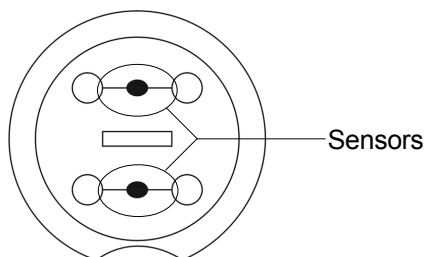
The CCD detector can also be used as a hydrocarbon monitor in non-chromatographic applications where the CCD senses the total hydrocarbon content of a flowing air stream, or as a hydrogen/hydrocarbon leak detector.

DETECTORS

Catalytic Combustion Detector - CCD

Theory of Operation

Top View of CCD Detector

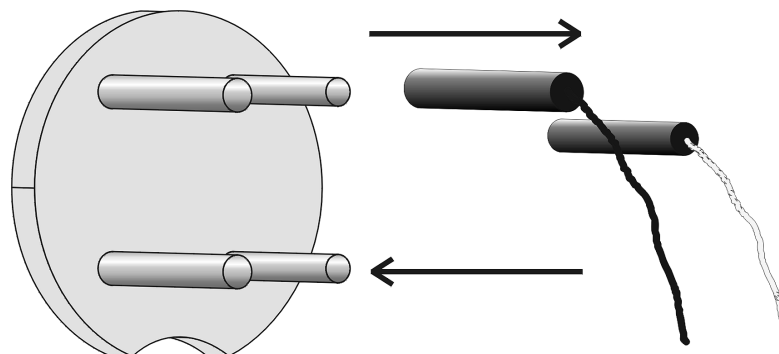
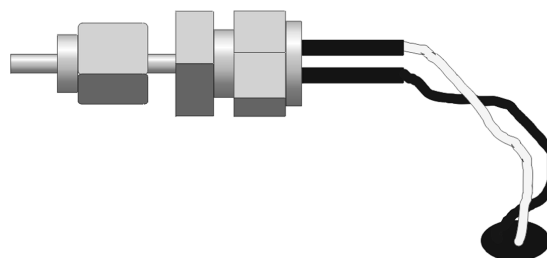


Side View of Sensor Element

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

To prolong the life of your CCD detector, use it in strict accordance with your GC system's operating instructions. For instance, if you have an SRI Mud-Logger GC, you should connect your sample streams at 10psi so that no more than 5mL/min of pure hydrocarbon flow reaches the CCD. In the event of a sensor burn-out, simply remove the white and black wires from the top two nickel pins, and move them to the bottom pair of

nickel pins to connect them to the second sensor. It does not matter which wire goes on which pin. To replace the CCD detector, unscrew its brass fitting after removing the wires from the nickel pins. Pull out the old one and remove the protective cap from the replacement. Sensor-side first, insert the replacement into the fitting with its half-moon shaped cut-out on the bottom. Replace the fitting and **HAND TIGHTEN** it. If the detector fitting is screwed on too tightly, the detector will not receive proper gas flow. Next, slip the black and white wire plugs over the pins, and your replacement CCD detector is ready to use.



DETECTORS

Catalytic Combustion Detector - CCD

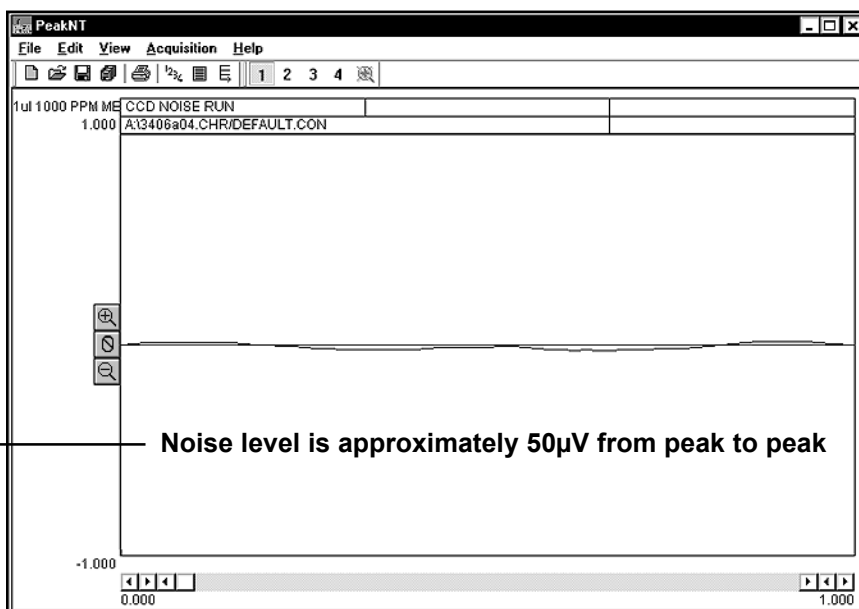
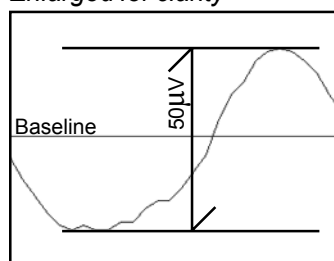
Expected Performance

CCD Detector Noise Run

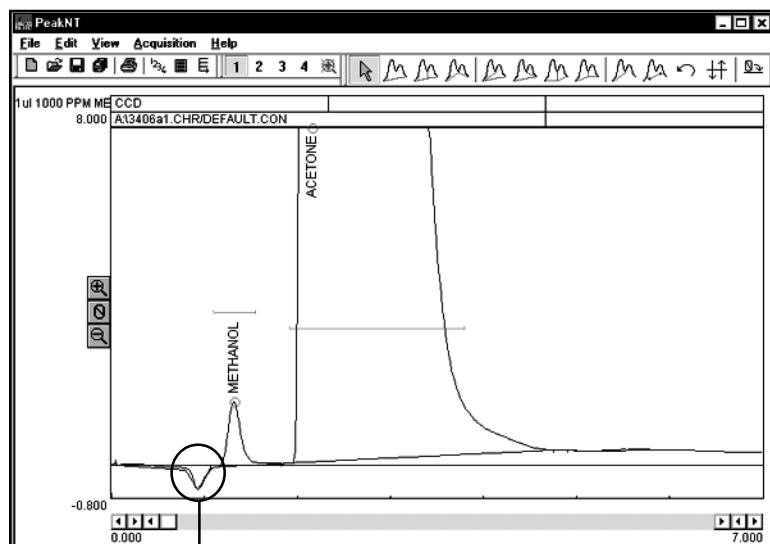
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:
Initial Hold Ramp Final
80°C 15.00 0.00 80°C

Enlarged for clarity



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

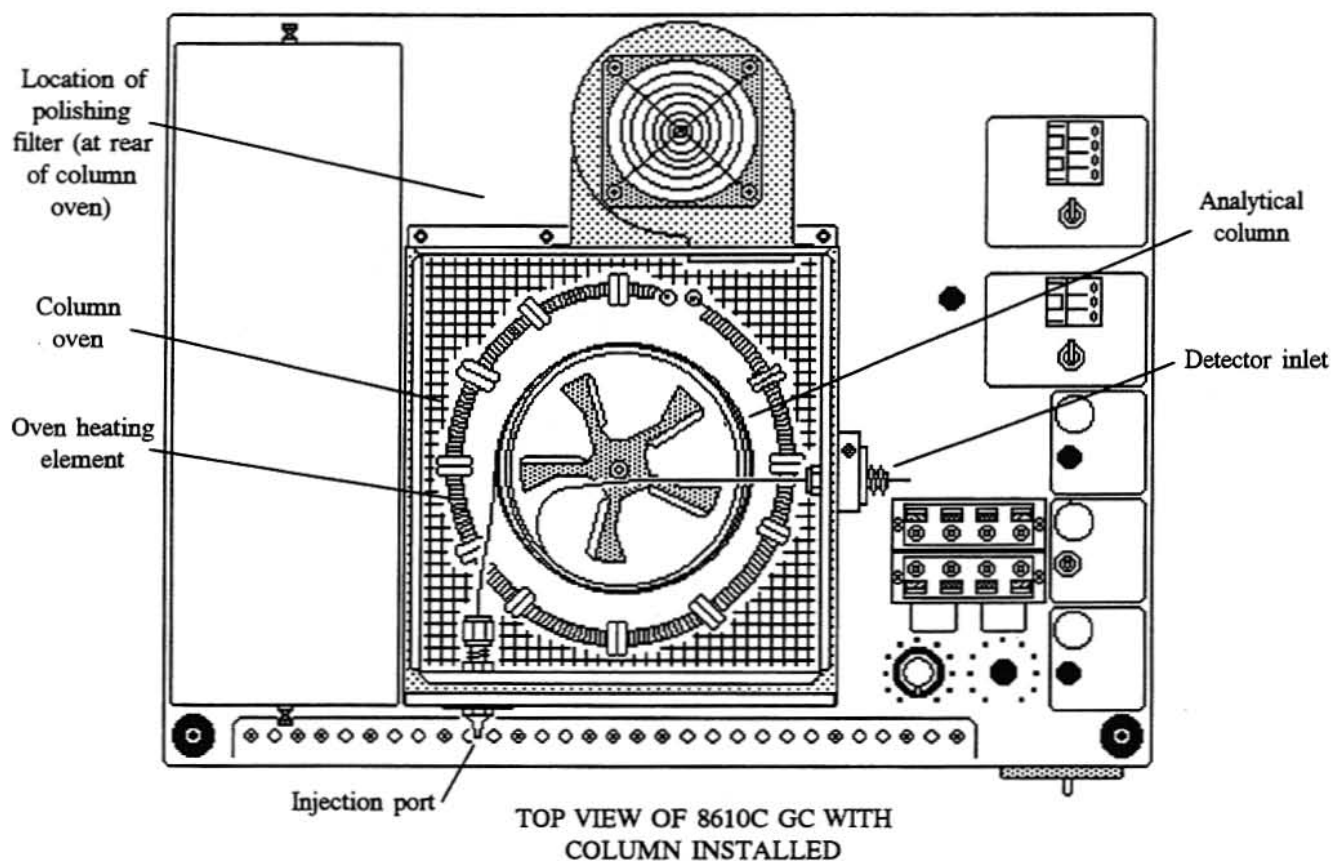


Column = 1m Hayesep D
Flow = 37mL/min
Sample = 1µL 1000ppm Methanol/Acetone mix; direct injection

Isothermal Temperature Program:
Initial Temp Hold Ramp Final Temp
130°C 10.00 0.00 130°C

RESULTS:		
Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
Total		6958.5600

Negative water peak



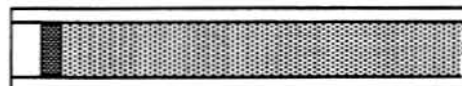
The column oven in the SRI 8610C GC measures approximately 7.8" x 8.0" x 3.0" (19.8 x 20.3 x 7.2cm). A column wound into a coiled form with a maximum diameter of 7" and a height of 3" may be installed in the interior space available. Standard 6" diameter or 3" diameter SRI-wound columns are installed with ease. Either capillary type (0.25 to 0.53mm I.D.) or packed columns (1/8" to 1/4") may be used, dependent on the application. Capillary columns may be made of either fused silica or stainless steel, and are coated on the inside with a fine film of stationary phase between 0.1 and 5.0 microns thick. This phase, specific to the application, permits the sample components to be properly separated for analysis. The packing material in a packed column serves the same purpose. For wide-bore capillary applications, metal capillary columns are recommended, as they are virtually indestructible and can withstand much physical abuse, unlike the fused silica variety, which can be broken with ease if handled improperly. SRI recommends the use of metal capillary columns when available for the application.



0.25 to 0.32mm I.d fused silica tubing coated on inside surface with stationary phase film 0.1 to 1.0 microns thick

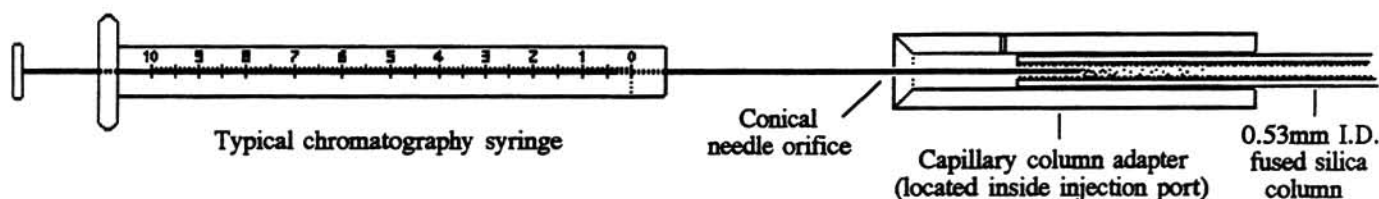


0.53mm I.D. fused silica or fused silica-lined stainless steel tubing coated on the inside surface with a stationary phase film 0.1 to 5.0 micron thick



1/8" to 1/4" O.D. stainless steel or glass tubing packed with granular support particles. These support particles may have a stationary phase coating. Glass tubing is specified for pesticide analysis, as some pesticide components react with stainless steel. A metal frit or glass wool plug retains the packing inside the tubing

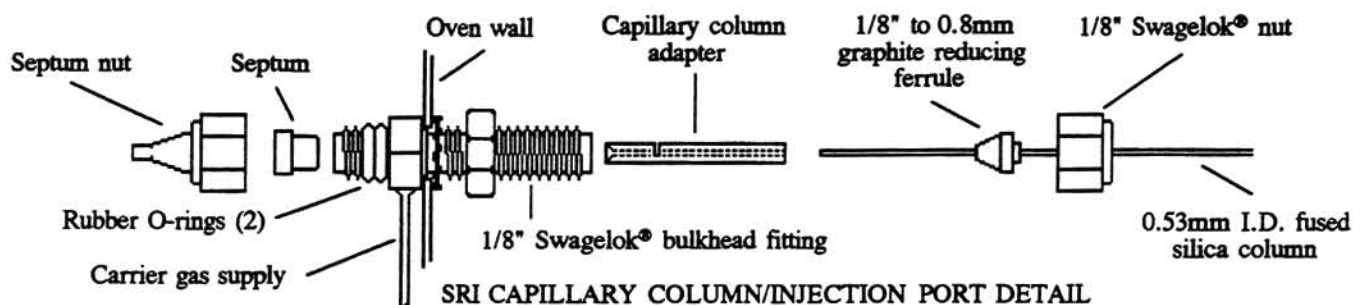
The injection port of the SRI chromatograph is designed specifically for direct injection onto a 0.53mm I.D. wide-bore capillary column. A sample, injected using a chromatography syringe equipped with a 26 gauge needle, is deposited directly into the column. The injector is supplied with a 1/8" O.D. stainless steel 0.53mm capillary column adapter that guides the syringe needle into the capillary column entrance. The sample is then injected onto the column. The user's sample injection technique (sample loading, needle insertion and injection) should be quick, precise and reproducible.



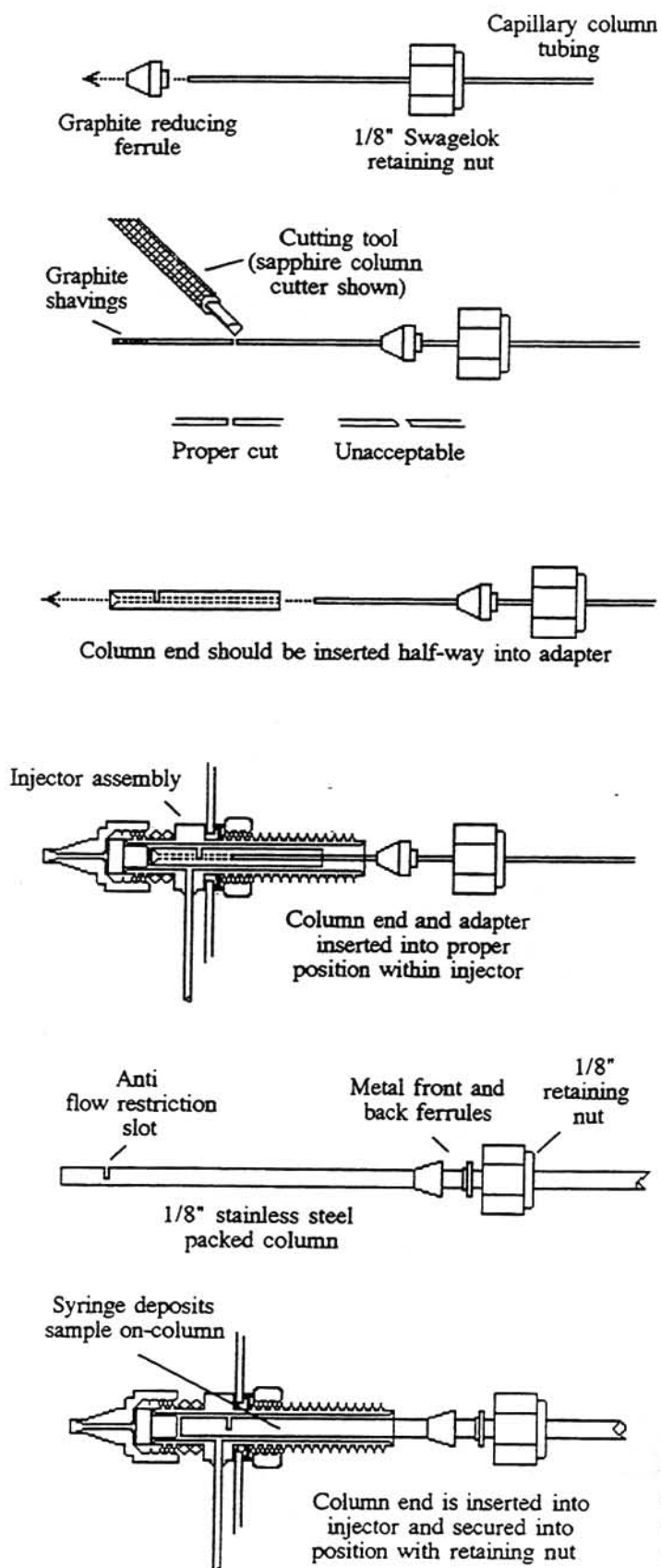
DIRECT INJECTION INTO A CAPILLARY COLUMN

The wide-bore capillary column adapter is machined from 1/8" stainless steel and accepts the insertion of 0.8mm O.D. tubing (the outer dimension of 0.53mm I.D. capillary column tubing). The injection end of the adapter is conical and "funnels" the needle into the column tubing inserted into the adapter from the column end. A slot cut in the adapter prevents carrier gas flow restrictions caused by overtightened septa. By guiding the injection needle well into the analytical column tubing, the sample may be deposited as a liquid onto the stationary phase of the column without exposing the sample to contact with hot metal or glass surfaces.

The capillary column adapter is located within the assembly that forms the injection port when a 0.53mm I.D. column is in use. The injection port is constructed from a 1/8" stainless steel Swagelok® bulkhead fitting that has been modified to permit the connection of a gas source directly into the fitting through the hexagonal flange at the bulkhead. This modification permits the introduction of carrier gas into the injector. The end of the injector bulkhead fitting located in the oven compartment accepts a 1/8" Swagelok® nut and graphite reducing ferrule (Alltech RF200/0.8-G) used to secure the capillary column in the injector. At the other end of the bulkhead fitting, facing the user, a 1/8" septum nut is used to secure a formed silicone septum in place in front of the column, sealing the injection port. The septum nut should be finger-tightened. Two rubber O-rings are installed on the injector where the septum nut is attached. The septum nut should never be tightened beyond the point where the nut contacts the outer O-ring.



The injection port is compact and has a low thermal mass. Since most of the injector body is located within the column oven, the injector and oven temperatures are always equal (the standard injector is not supplied with any provision for independent heating. Heated on-column injection is available as an option). Resultant sample component peaks are sharp and exhibit minimal or no tailing. This is due to the injection of the sample directly onto the column and at a temperature below the sample solvent boiling point. Decomposition of thermally-sensitive sample compounds does not occur and artifact formation is minimized because the sample is not subjected to vaporization and recondensation, as occurs in high temperature injectors.

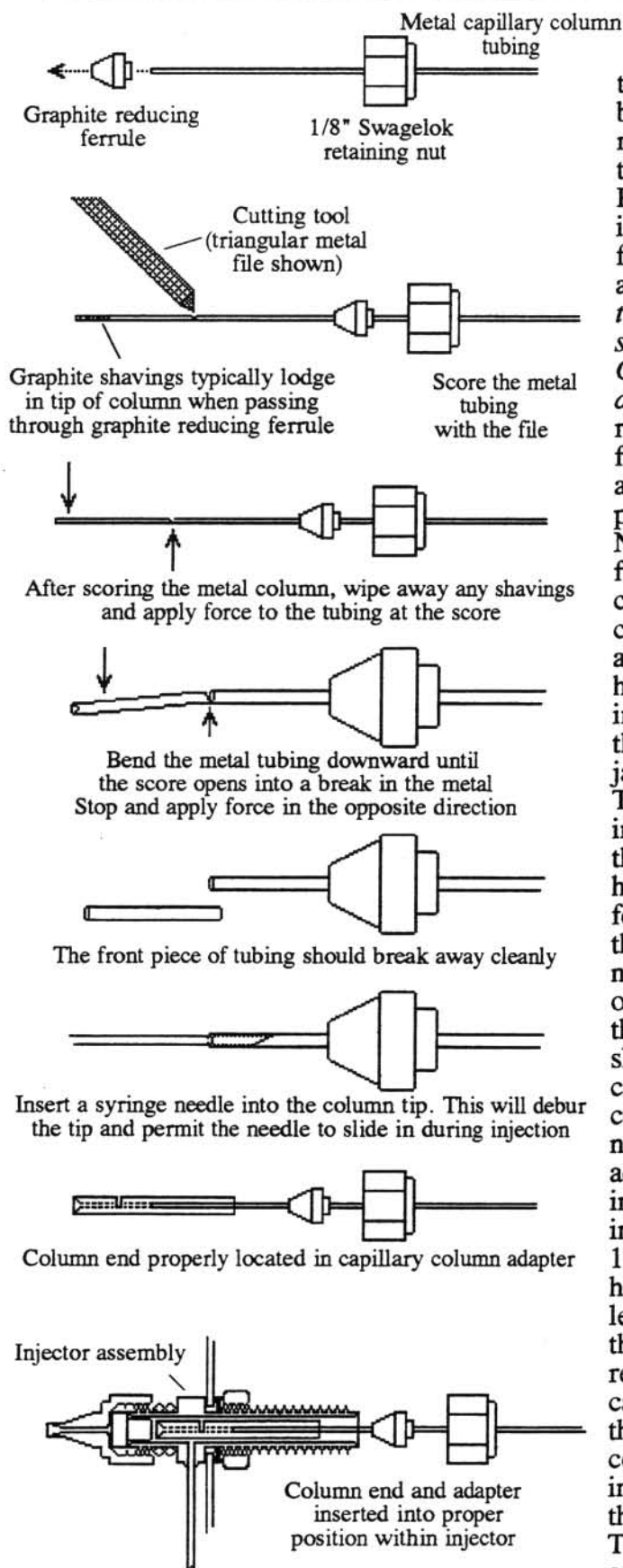


When installing a capillary column in the chromatograph, a graphite reducing ferrule must be used to secure the capillary tubing in the 1/8" retaining nut (Alltech RF200/0.8-G for 0.53mm tubing, RF200/0.5-G for 0.32mm tubing and RF200/0.4-G for 0.25mm tubing). The column is inserted first through the nut, and then through the ferrule. Note the orientation of these parts in the accompanying illustrations. The insertion of the tubing through the ferrule will cause graphite shavings to accumulate in the column entrance. Graphite is adsorbent and may cause peak tailing or a flow restriction if left in the column. For this reason, an inch or so of column tubing should be cut from the column tip after it has been passed through a graphite ferrule. A sapphire tool, a diamond scribe or a razor blade may be used to cut the column, in that order of preference. When the polyimide coating of the tubing has been scored, the tubing snaps apart cleanly. Check the cut end prior to use; it should be flat-ended, not jagged or with the polyimide coating peeling. The capillary column may now be inserted half-way into the capillary column adapter for installation into the injector. Once that the adapter and column end have been located in the injector as shown, the ferrule and nut are connected and tightened to secure the column in the injector. Note that the adapter does not contact the septum. If the septum nut were overtightened, the septa would be forced deeper into the injection port, sealing against the adapter. The slot cut in the adapter permits carrier gas to reach the column even if the septum is overtightened, so that column flow is unaffected. When the column is properly installed, a head pressure reading of between 4 and 12 psi should be observed. If there is little or no head pressure, the system should be inspected for leaks. If the head pressure rises to a level equal to the carrier gas supply pressure, suspect a flow restriction or plug either in the column (typically caused by an accumulation of cored septum slices in the entrance to the column) or at the outlet of the column (at the detector inlet).

Installation of a packed column in the chromatograph is simpler. With 1/8" stainless steel columns, standard metal ferrules are used to secure the column at the retaining nut. The ferrules are placed onto the column end as shown, and then the column end is inserted into the injector. The capillary column adapter is not used with packed columns and should be stored in the adapter holder under the red protective oven cover for future use. Columns manufactured by SRI include a slot in the injector end for carrier gas flow assurance.

Chapter: INSTALLATION

Topic: Analytical Column Installation (continued)



When installing a metal capillary column in the chromatograph, a graphite reducing ferrule must be used to secure the capillary tubing in the 1/8" retaining nut (Alltech RF200/0.8-G for 0.53mm I.D. tubing, RF200/0.5-G for 0.32mm I.D. tubing and RF200/0.4-G for 0.25mm I.D. tubing). The column is inserted first through the nut, and then through the ferrule. Note the orientation of these parts in the accompanying illustrations. *The insertion of the tubing through the ferrule will cause graphite shavings to accumulate in the column entrance.*

Graphite is adsorbent and may cause peak tailing or a flow restriction if left in the column. For this reason, an inch or so of column tubing should be cut from the column tip after it has been passed through a graphite ferrule. A fine-cut triangular metal file is provided with all SRI metal capillary columns.

Normal column cutting tools designed for use on fused silica will not work with metal columns. Metal columns are coated inside with fused silica and column phase. They offer the same performance, and are practically immune to breakage or rough handling damage. Score and cut the column tubing as indicated and the tubing snaps apart cleanly. Check the cut end prior to use; it should be flat-ended, not jagged or with metal covering the column orifice.

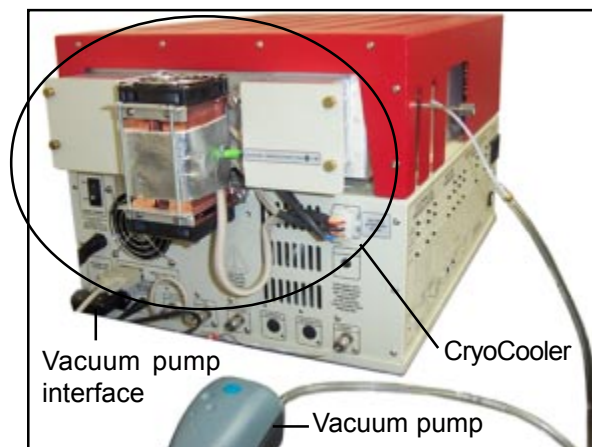
The capillary column may now be inserted half-way into the capillary column adapter for installation into the injector. Once that the adapter and column end have been located in the injector as shown, the ferrule and nut are connected and tightened to secure the column in the injector. Note that the adapter does not contact the septum. If the septum nut were overtightened, the septa would be forced deeper into the injection port, sealing against the adapter. The slot cut in the adapter permits carrier gas to reach the column even if the septum is overtightened, so that column flow is unaffected. Of course, septa should never be overtightened. A finger-tight septum nut is adequate for proper sealing of the silicone against the injection port. When the column is properly installed, a head pressure reading of between 4 and 12 psi should be observed. If there is little or no head pressure, the system should be inspected for leaks. If the head pressure rises to a level equal to the carrier gas supply pressure, suspect a flow restriction or plug either in the column (typically caused by an accumulation of cored septum slices in the entrance to the column) or at the outlet of the column (at the detector inlet). When plugged column inlets are encountered, cut off another inch or two of the column and reinstall the column in the injector. The capillary column adapter is not used with packed columns and should be stored in the adapter holder under the red protective oven cover for future use.

POPULAR CONFIGURATION GCs

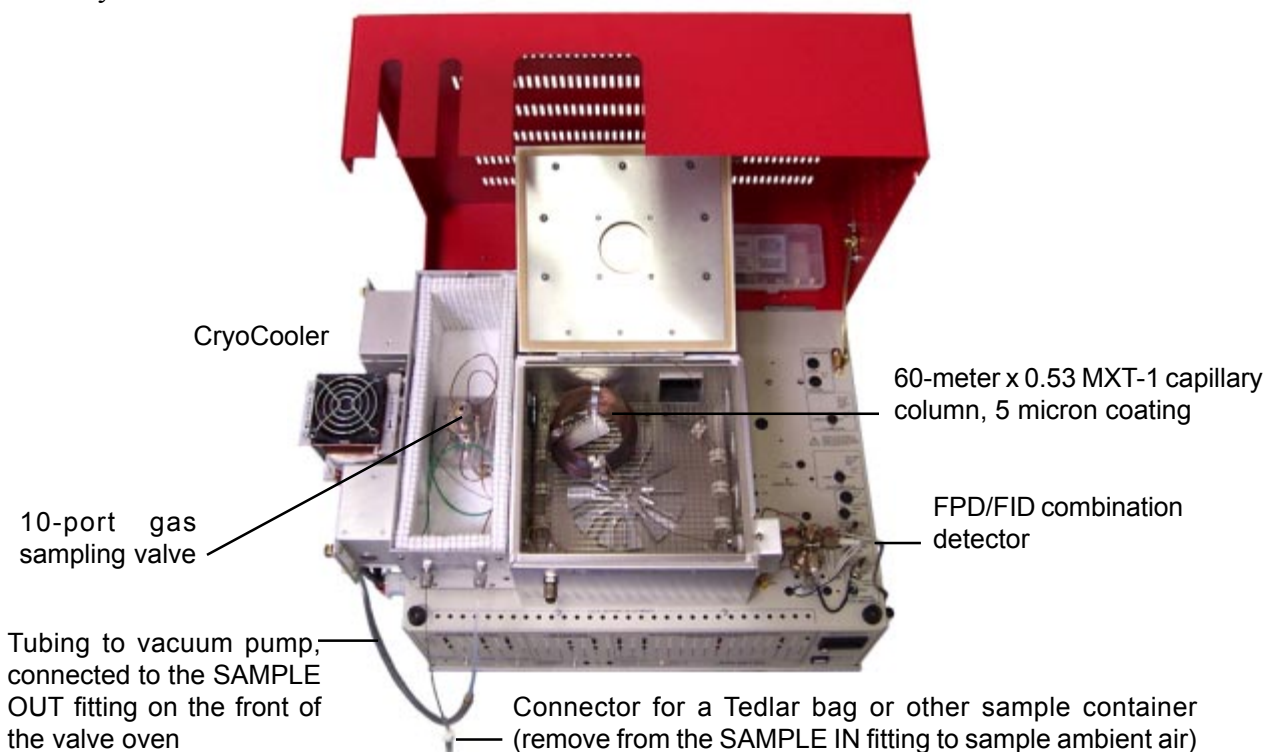
Cryosulfur GC

System Overview

The SRI CryoSulfur GC comes with everything you need to detect low-level sulfur compounds in gas samples. Since some sulfur compounds do not trap well, the CryoSulfur GC uses the CryoCooler Peltier Trap Accessory to enrich the sample, providing lower detection limits. The CryoSulfur GC uses a vacuum pump (provided) to draw gas or air samples into the CryoCooler. You can sample ambient air, or use the provided adaptor to connect a Tedlar bag. The vacuum pump interface, which is an electrical outlet on the left-hand side of the GC, allows the vacuum pump to be turned ON/OFF by the PeakSimple data system to provide consistent sampling times.



Like all SRI traps, the CryoCooler Peltier Trap Accessory is plumbed as the loop of a 10-port gas sampling valve. It has its own power cord that must be plugged into a wall outlet, and an interface cord that plugs into the left-hand side of the GC. After enrichment, the valve injects the sample onto the 60-meter capillary column. Once the sample components are separated by the column, they will be detected by the Flame Photometric and Flame Ionization detectors.



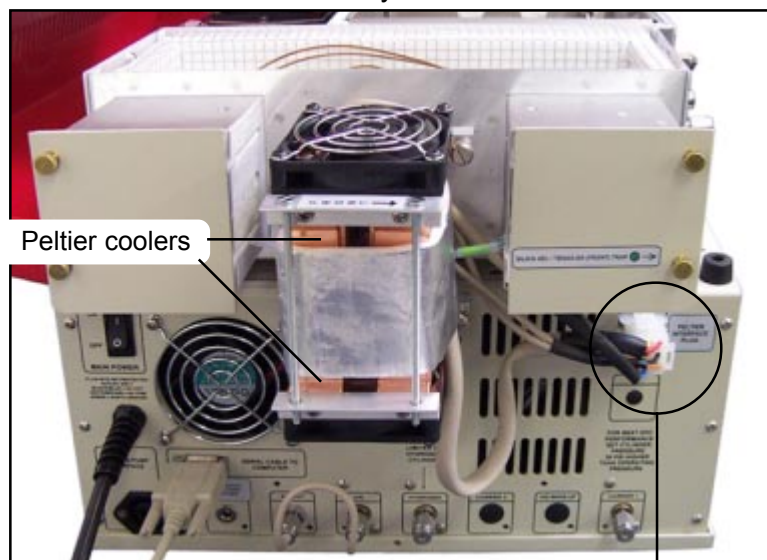
POPULAR CONFIGURATION GCs

Cryosulfur GC

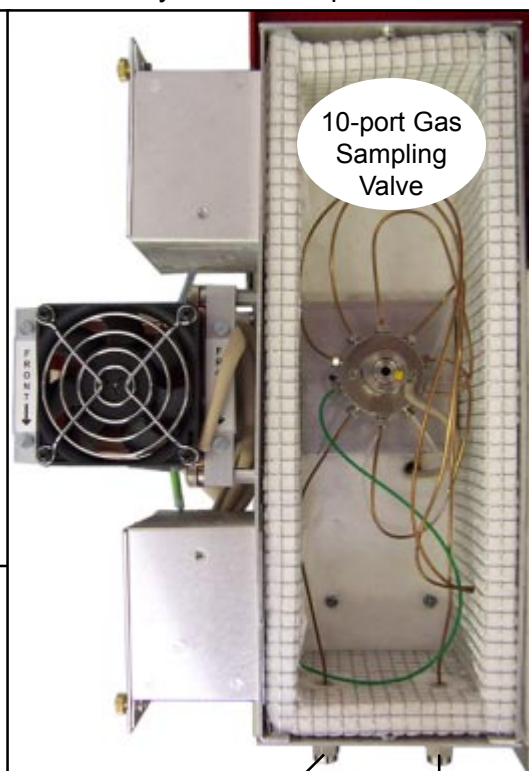
Theory of Operation

Some sulfur compounds do not trap well, but can be enriched for lower detection limits. The CryoCooler Peltier Trap Accessory is basically a heated trap sandwiched between two peltier coolers. The trap is filled with Tenax-GR (about 30%) and Silica Gel (about 70%) adsorbents. The vacuum pump draws sample through the CryoCooler from ambient air, or from a Tedlar bag or other sample container. The peltier coolers can cool the trap down to -15°C to enrich the sample.

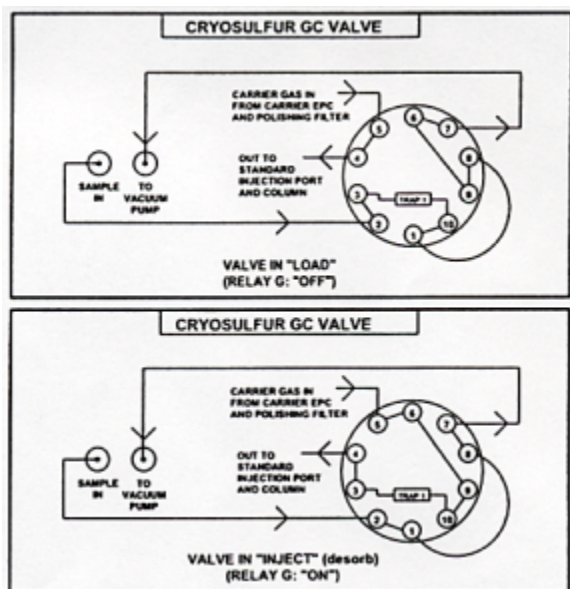
The CryoCooler



CryoCooler - Top View



Valve Diagram Label on Valve Oven Lid:



While the CryoCooler is enriching the sample, the 10-port gas sampling valve is in the LOAD position. At the conclusion of the sampling period, the heater heats the trap to $150\text{--}200^{\circ}\text{C}$ and the valve is actuated to the INJECT position; this places the trap in the carrier gas stream and sweeps the enriched analytes onto the column.

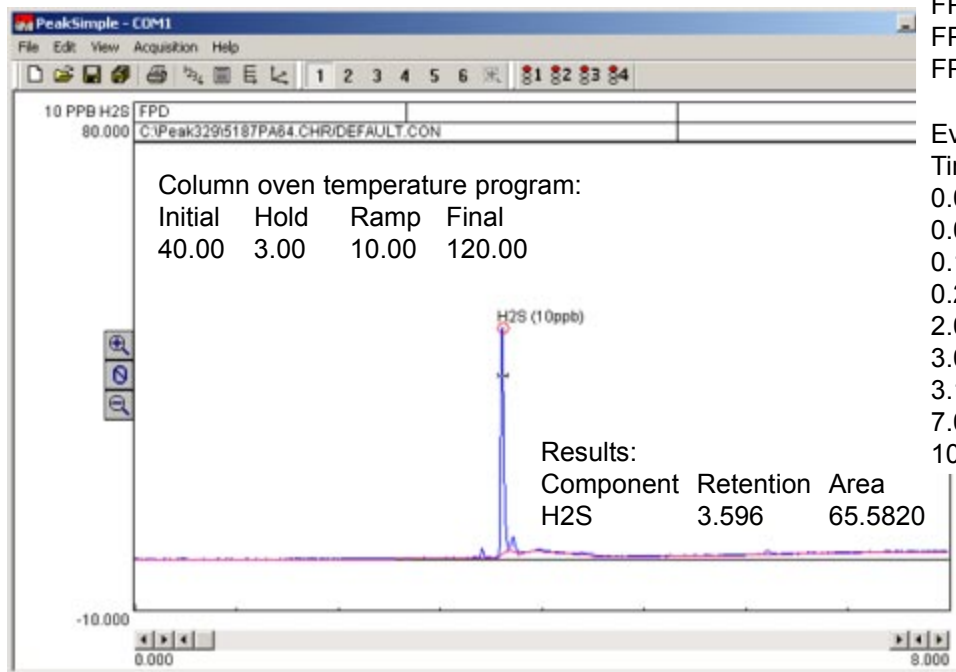
POPULAR CONFIGURATION GCs

Cryosulfur GC

Expected Performance

The following chromatograms were generated by an SRI CryoSulfur GC. The first chromatogram shows the FPD response to 10ppb hydrogen sulfide (H_2S), as enriched by the CryoCooler at $-10^{\circ}C$. The second chromatogram shows the FID response to 1000ppm C_1-C_6 hydrocarbons.

Sample: 10ppb H_2S
 Column: 60-meter MXT-1
 Carrier: helium at 10mLs/minute
 Vacuum pump: 20mLs/minute
 Trap: $-10^{\circ}C$
 FPD gain: HIGH
 FPD temperature: $150^{\circ}C$
 FPD volts: 500



Event Table:

Time	Event
0.000	Zero baseline
0.050	D OFF (vacuum pump)
0.100	A OFF (CryoCooler)
0.200	F ON (trap heat)
2.000	G ON (valve INJECT)
3.000	F OFF (trap heat)
3.100	G OFF (valve LOAD)
7.000	A ON (CryoCooler)
10.000	D ON (vacuum pump)

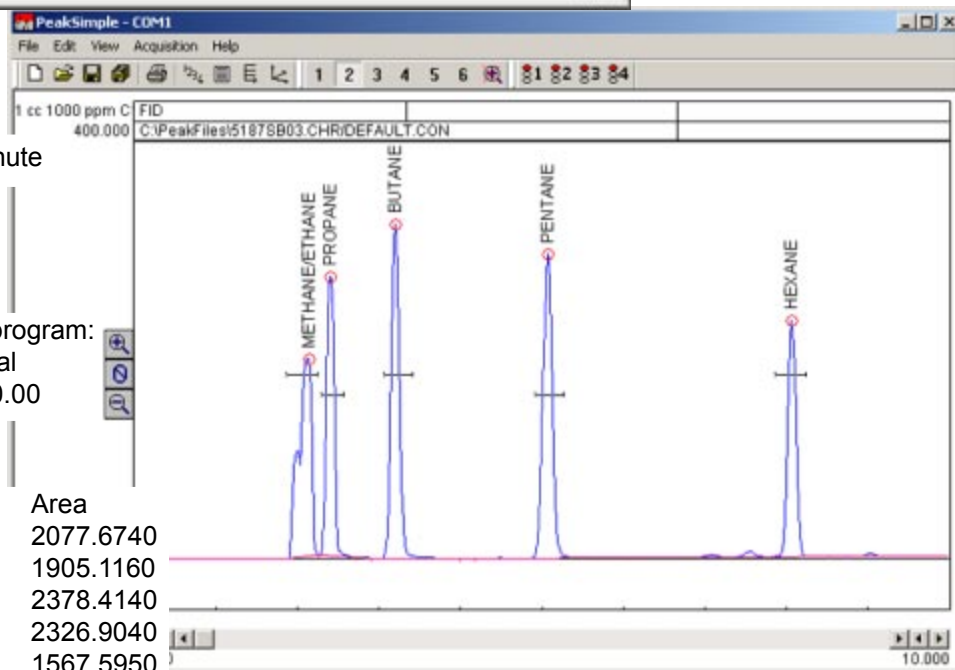
Sample: 1000ppm C_1-C_6
 Column: 60-meter MXT-1
 Carrier: helium at 10mLs/minute
 FID gain: HIGH
 FID temperature: $150^{\circ}C$
 FID ignitor: -400

Column oven temperature program:

Initial	Hold	Ramp	Final
40.00	3.00	10.00	140.00

Results:

Component	Retention	Area
Methane/ethane	2.116	2077.6740
Propane	2.383	1905.1160
Butane	3.183	2378.4140
Pentane	5.050	2326.9040
Hexane	8.050	1567.5950
Total		10255.7030

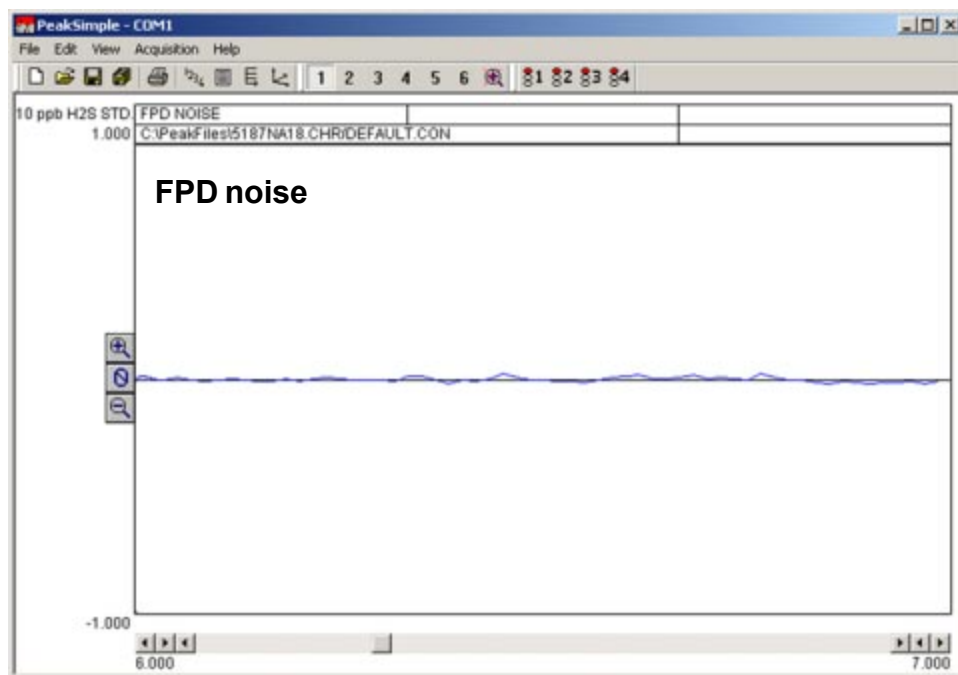


POPULAR CONFIGURATION GCs

Cryosulfur GC

Expected Performance continued

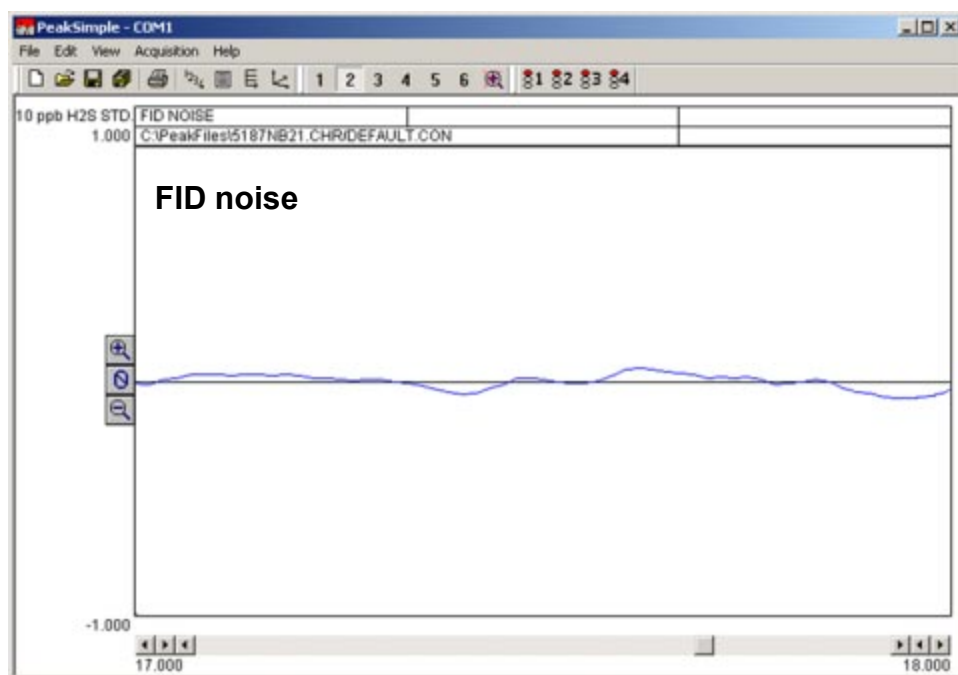
The following two noise runs were generated by a CryoSulfur GC. Both used the same isothermal column oven temperature program, 60-meter MXT-1 column, and helium carrier at 10 milliliters per minute.



FPD gain: HIGH
FPD temperature: 150°C
FPD volts: 500

Column oven temperature program:

Initial	Hold	Ramp	Final
80.00	24.00	0.00	80.00



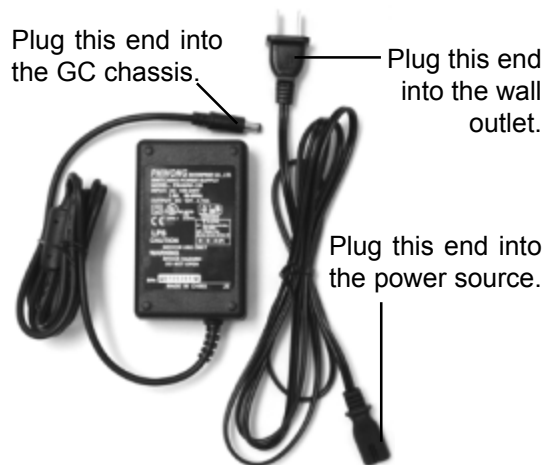
FID gain: HIGH
FID temperature: 150°C
FID ignitor: -400

POPULAR CONFIGURATION GCs

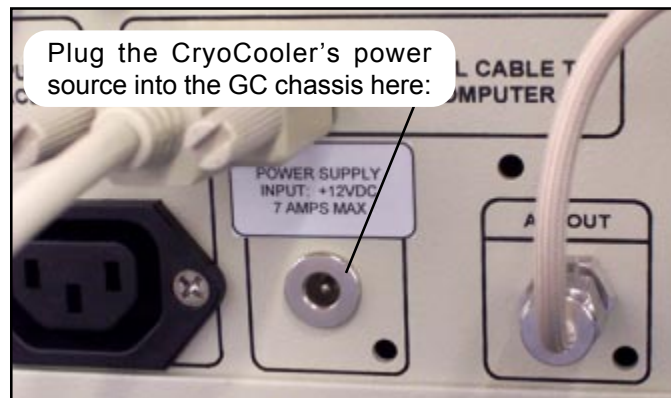
Cryosulfur GC

General Operating Procedure

CryoCooler power source:



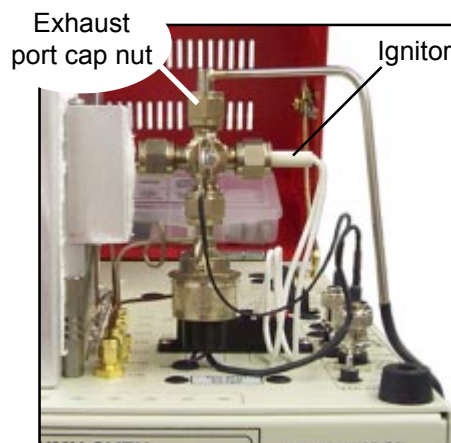
1. Plug the GC power cord into a wall outlet and turn the main power ON. Plug the CryoCooler into its power source, and plug the power source cord into the wall outlet. The CryoCooler power source plugs into the GC chassis between the vacuum pump interface and the Air Out fitting on the left-hand side of the GC as shown below.



2. Connect your helium source to the carrier gas inlet on the left-hand side of the GC. Connect your hydrogen source to the hydrogen inlet. Leave the jumper in place if you plan to use the built-in air compressor to supply air for the detectors. Otherwise, remove the jumper and connect your air source to the air inlet on the left control panel.

3. Set the hydrogen flow to 60-80mLs/minute; this correlates to a flow of 30-40mLs/minute each for the primary and secondary hydrogen used by the FPD/FID combination detector. Set the air flow to 100mLs/minute. The detector air supply tubing is T'd inside the GC so that 10-30mLs/minute of air flows across the face of the photomultiplier (PMT). The gas pressures required to achieve these specific flows are printed on the right-hand side of the GC.

4. Use the switch on the GC front control panel to light the detector flame (vertically labeled "FLAME IGNITE" under "DETECTOR PARAMETERS"). Often the flame can be difficult to light because of the hydrogen-rich atmosphere inside the detector body. Make sure that the PMT voltage is OFF (that switch is also on the GC front control panel, vertically labeled "PMT VOLTS" under "DETECTOR PARAMETERS"), then remove the cap nut on the detector exhaust port. **KEEP YOUR FACE AWAY FROM THE DETECTOR WHILE LIGHTING THE FLAME**, 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. The noise is accompanied by a flash of flame. Replace the exhaust cap nut after the flame is lit.



5. Switch ON the PMT voltage and set it at 400-500 by adjusting the appropriate trimpot ("PMT VOLTAGE" under "DETECTOR PARAMETERS"). The greater the voltage setting, the higher the FPD sensitivity. The PMT volts were set at 500 for the 10ppb H₂S analysis shown on the Expected Performance page.

POPULAR CONFIGURATION GCs

Cryosulfur GC

General Operating Procedure continued

6. Connect the provided vacuum pump to the SAMPLE OUT/VACUUM PUMP fitting on the front of the valve oven. Plug the vacuum pump power cord into the vacuum pump interface outlet on the left-hand side of the GC.

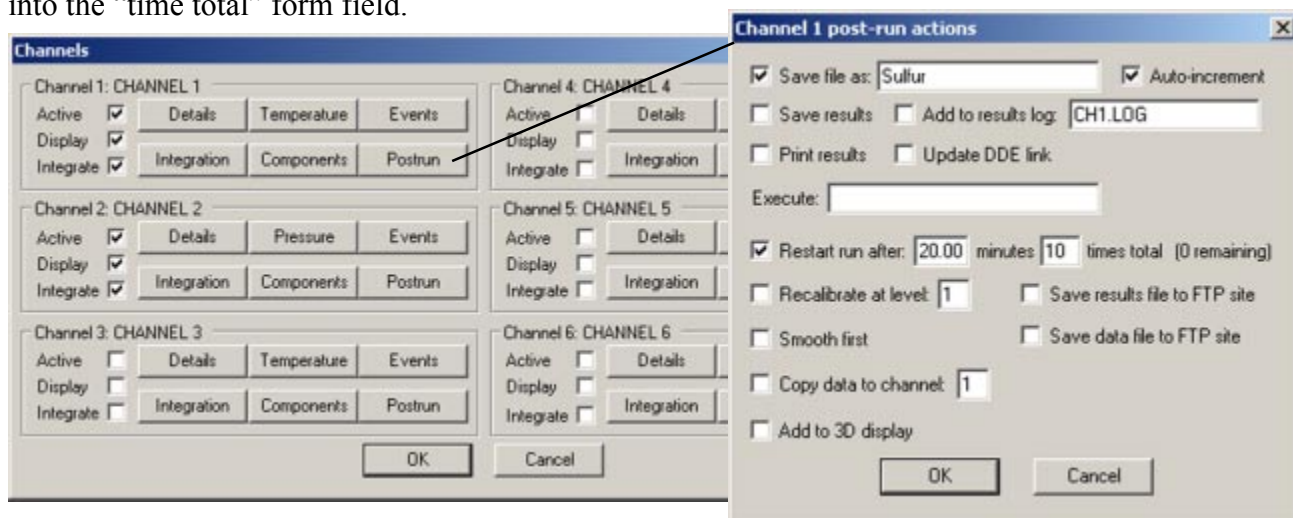
7. Connect your sample source to the SAMPLE IN fitting. It is critical to use as inert a sample pathway as possible to avoid absorption of sulfur compounds. You can also sample ambient air through the SAMPLE IN inlet, just remove the fitting.

8. In PeakSimple, type in the following event table:

Time	Event
0.000	Zero baseline
0.050	D OFF (vacuum pump)
0.100	A OFF (CryoCooler)
0.200	F ON (trap heat)
2.000	G ON (valve INJECT)
3.000	F OFF (trap heat)
3.100	G OFF (valve LOAD)
7.000	A ON (CryoCooler)
10.000	D ON (vacuum pump)

Then, in the Edit > Overall window, uncheck the box labeled “Reset relays at end of run.”

9. The system works best when operated automatically every 20 minutes. The CryoCooler and vacuum pump are left ON between runs for sampling. The first run's results are ignored, as it takes multiple runs to equilibrate the sulfur compounds inside the GC system and achieve reproducibility. In PeakSimple, open the Channels window by clicking Edit > Channels. Click the Postrun button for channel 1. Click the “Save file as” checkbox, type a name for the chromatograms in the form field, and click the “Auto-increment” checkbox. For example, if you type in Sulfur, the second chromatogram will be saved as Sulfur1, followed by Sulfur2, and so on. Click the checkbox labeled “Restart run after” and type 20 minutes in the form field. Type the number of times you want to repeat the analysis into the “time total” form field.



10. Press the START button on the front of the GC, or press your computer keyboard spacebar to start the run.

DETECTORS

Dry Electrolytic Conductivity Detector - DELCD

Overview

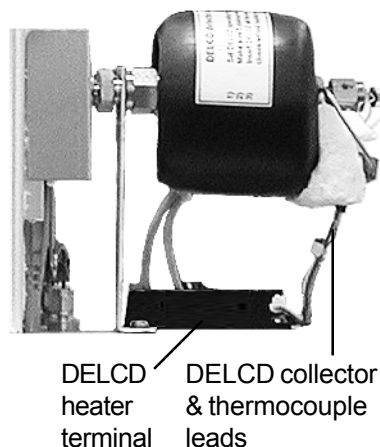
The Dry Electrolytic Conductivity detector, or DELCD, is selective to chlorinated and brominated molecules. It differs from the traditional wet ELCD in that it does not use a solvent electrolyte, and the reaction products are detected in the gaseous phase. The SRI DELCD is available alone or in combination 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 CO₂ and H₂O prior to reaching the DELCD, thus preventing contamination of the DELCD by large hydrocarbon peaks.



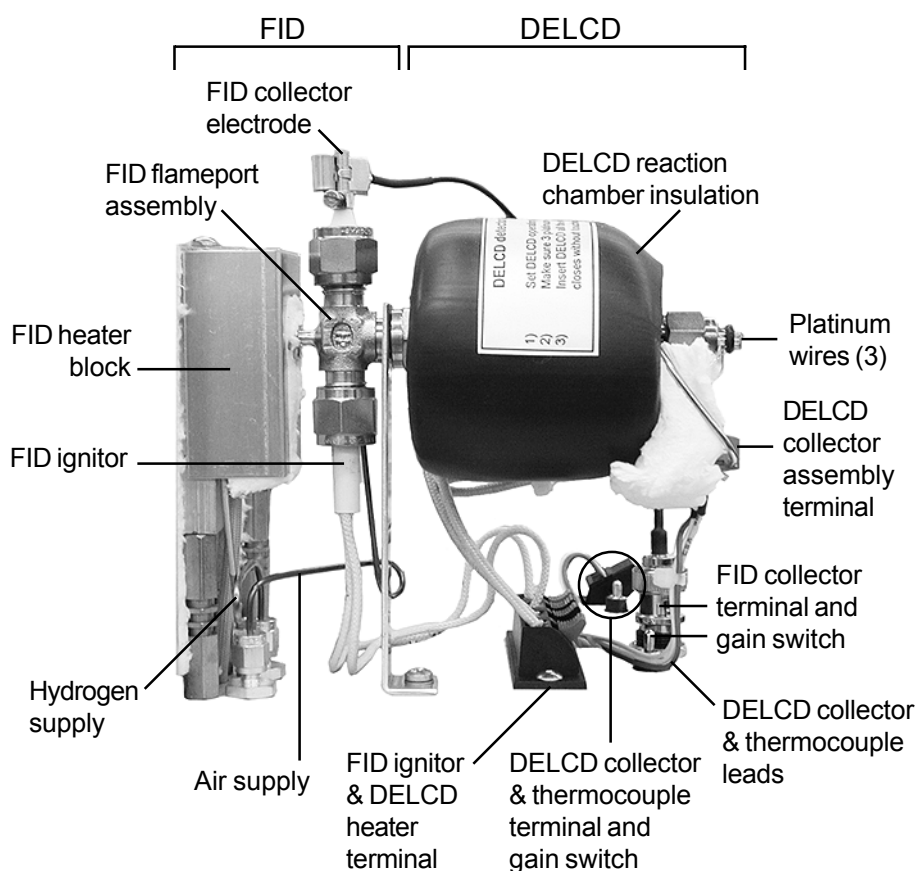
DELCD on an 8610C GC

DELCD - À la carte

Close-up of the same DELCD detector



FID / DELCD Combo Detector



DETECTORS

Dry Electrolytic Conductivity Detector - DELCD

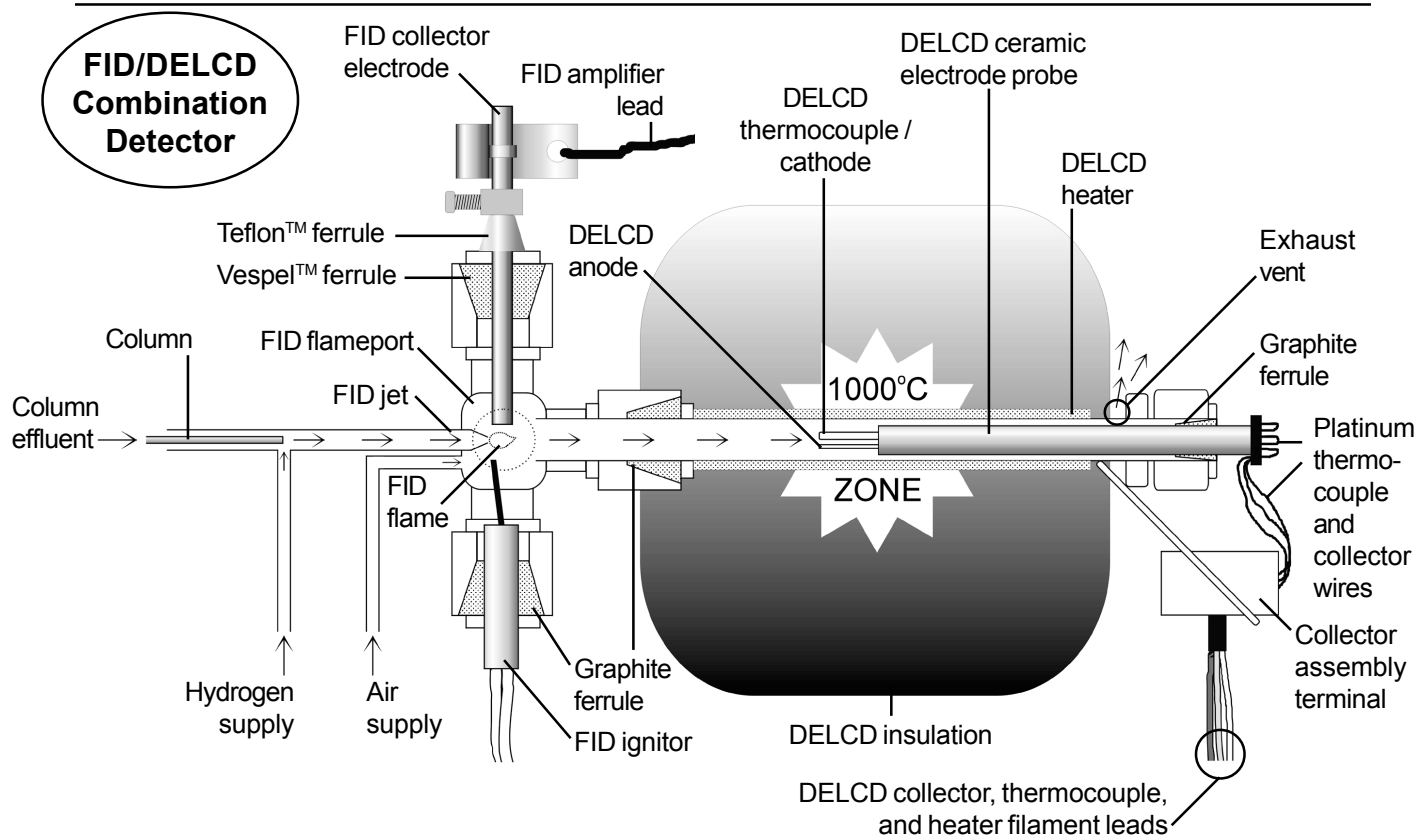
Theory of Operation

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 1 mm (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 ClO₂ and BrO₂. The DELCD detects the oxidized species of chlorine and bromine, such as ClO₂ and BrO₂. 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.

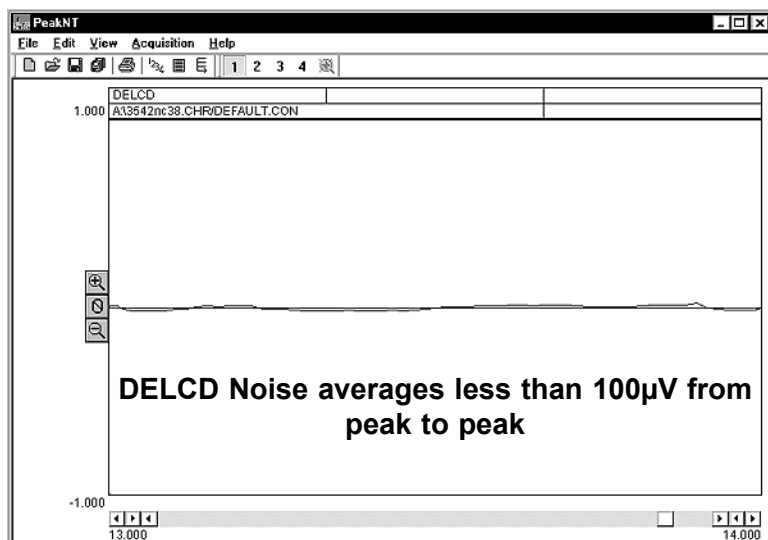
A DELCD only detector receives the sample laden carrier gas directly from the column or from a non-destructive detector outlet, like the PID. It is mounted on the heater block on the column oven wall so that the column effluent is maintained at a temperature consistent with the analysis. This type of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



DETECTORS

Dry Electrolytic Conductivity Detector - DELCD

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

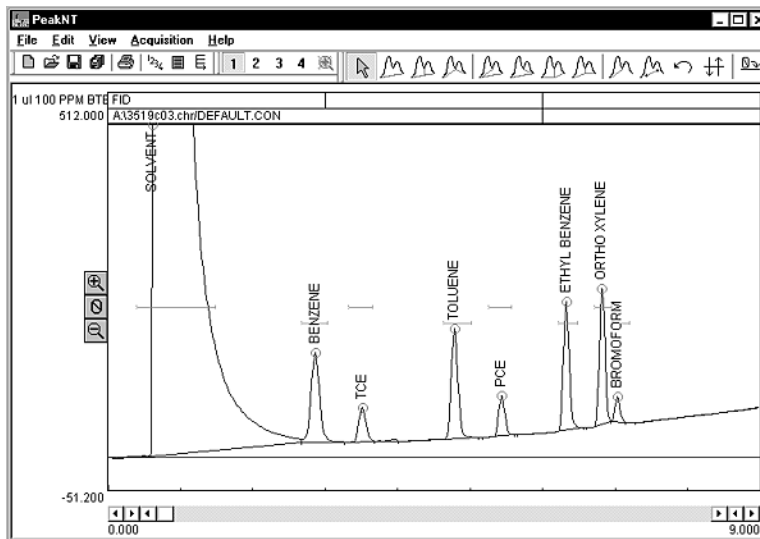
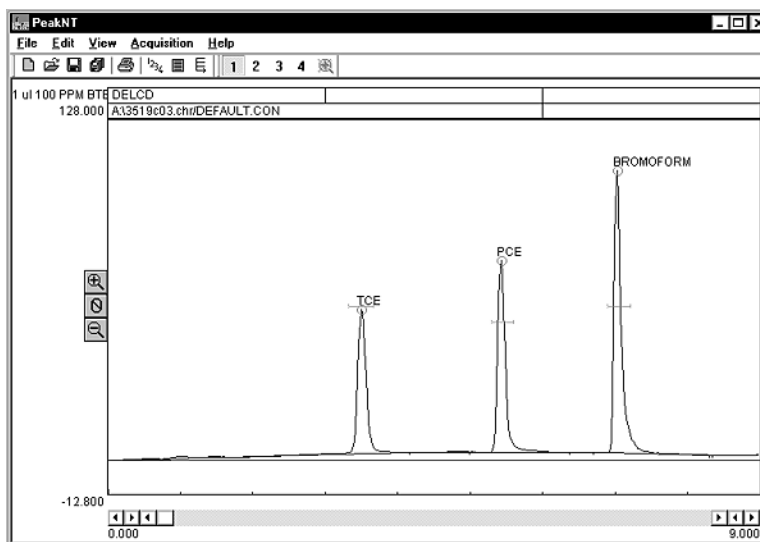
DELCD Results:

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 1 μL 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.

DETECTORS

Dry Electrolytic Conductivity Detector - DELCD

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 screw 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.

DETECTORS

Dry Electrolytic Conductivity Detector - DELCD

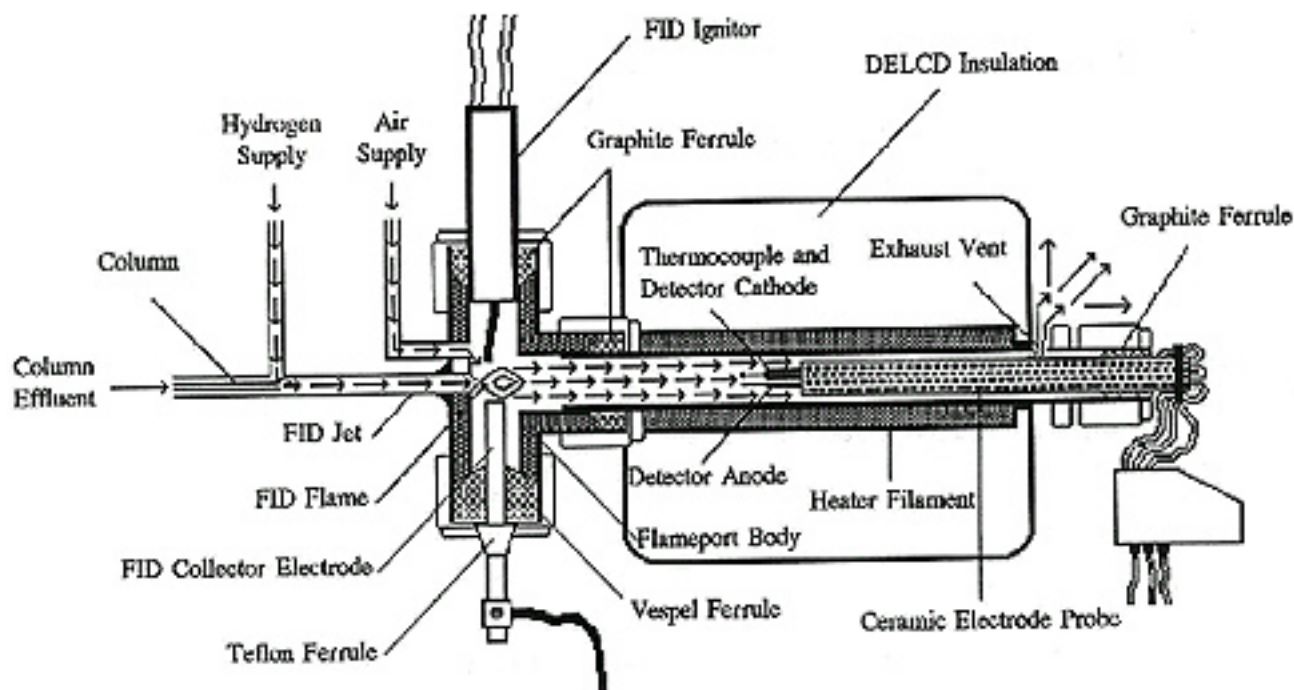
Troubleshooting and Maintenance

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.

DETECTORS FID/DELCD



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 1000°C. 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 1000°C. $\text{ClO}_2\text{-BrO}_2$ exhibits extremely high conductivity at 1000°C. So the DELCD actually responds to the $\text{ClO}_2\text{-BrO}_2$ 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.

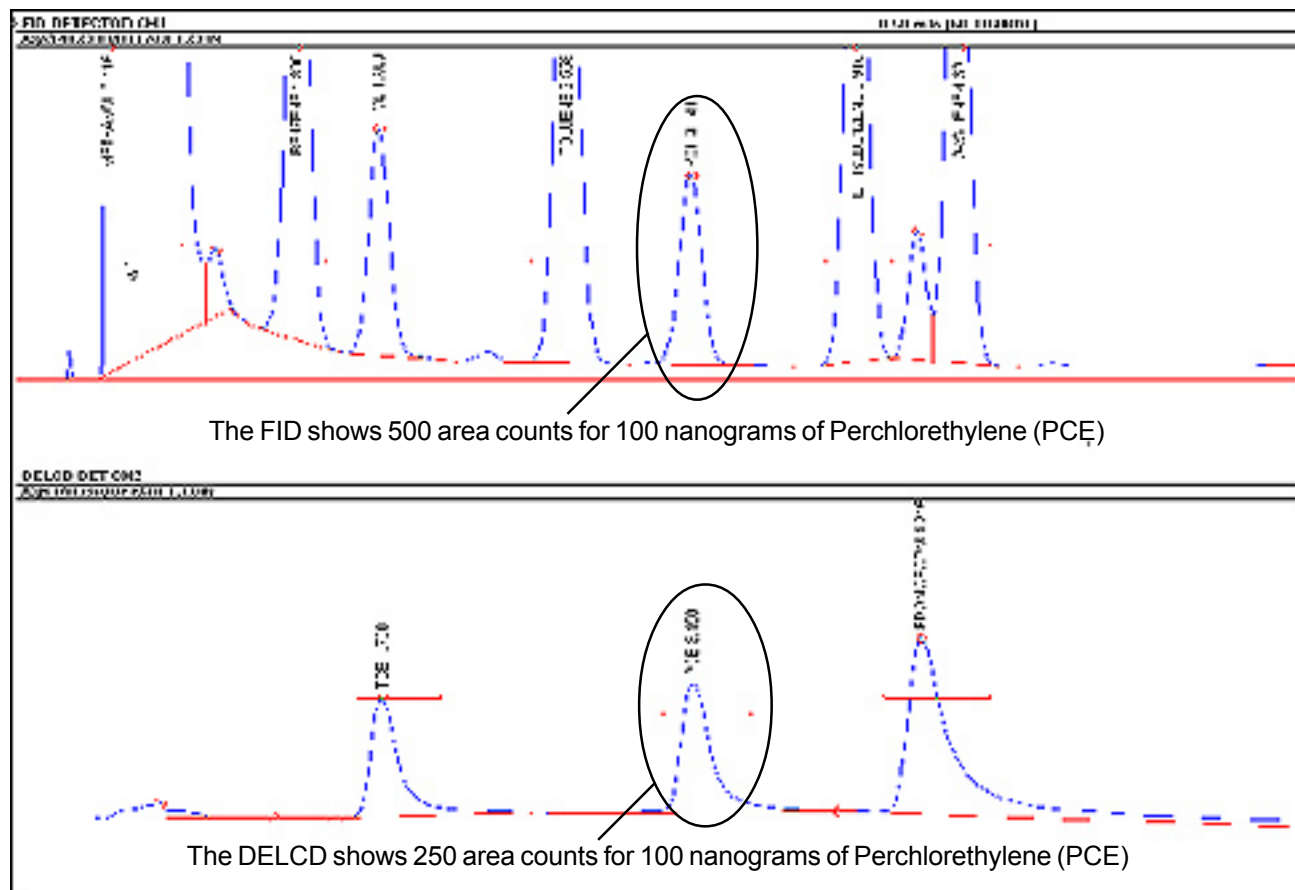
DETECTORS

FID/DELCD

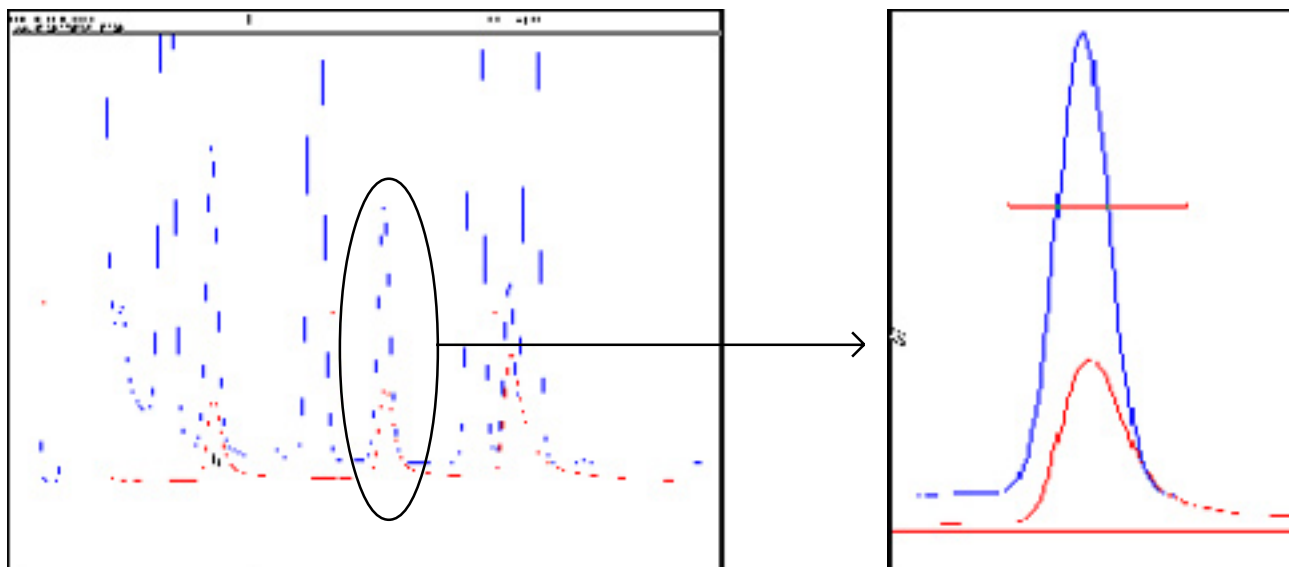
Operating the FID/DELCD in the Combo mode

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 1000°C. The DELCD will heat up to about 254 and stabilize. The quartz collector electrode will appear a bright red color due to the 1000C temperature.
- 3) In the FID/DELCD combo mode, the FID is normally operated on high gain or on hi-filtered gain if the peaks are more than 10 second wide at the base. The hi-filtered gain position is identical to the high gain except that extra noise filtering results in a quieter baseline. The DELCD amplifier is normally operated on low gain. In this configuration the FID and DELCD produce approximately the same response to chlorinated peaks such as TCE (same peak area counts). The FID will generate approximately 4 area counts per nanogram injected on column while the DELCD will generate 2-4 area counts per nanogram of chlorinated hydrocarbon. (see example chromatogram below).



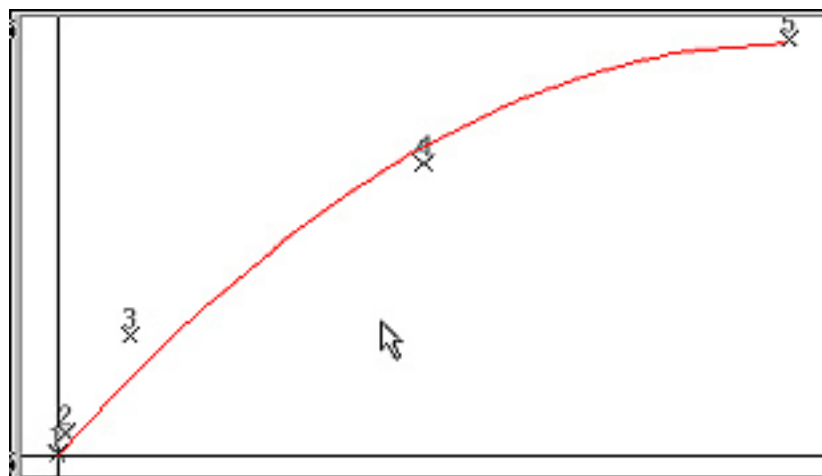
Operating the FID/DELCD in the Combo mode



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



DETECTORS

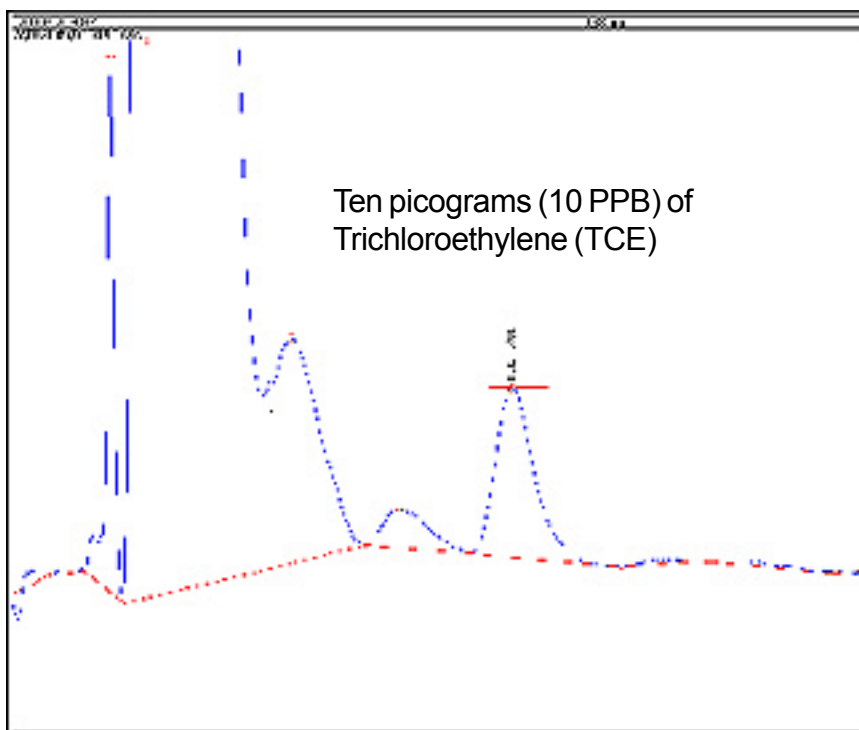
FID/DELCD

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 $\text{ClO}_2\text{-BrO}_2$. Because the chlorine/bromine atoms prefer to react with hydrogen to form non-detectable HCl-HBr , than with oxygen to form detectable $\text{ClO}_2\text{-BrO}_2$ 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 dissociated 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 sensitivity mode.

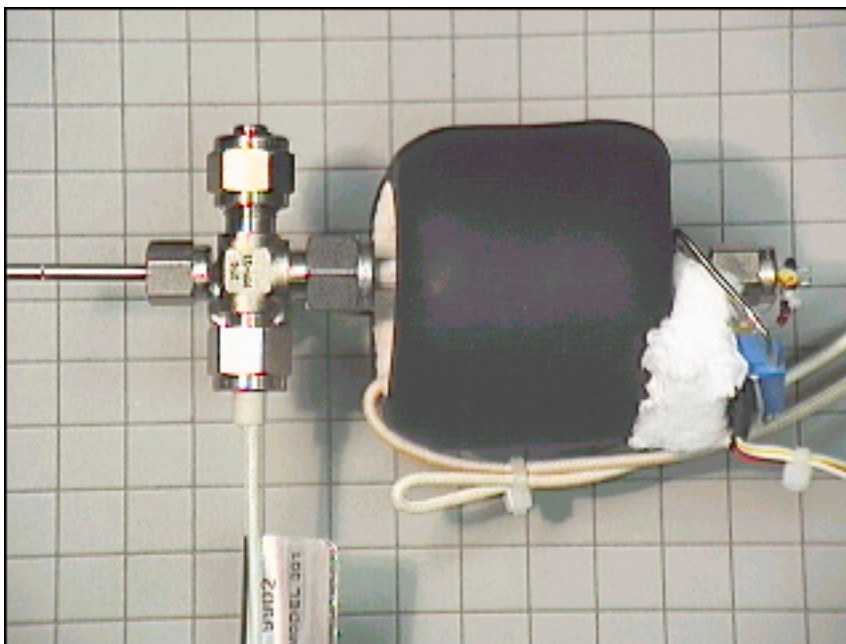
Note that in high sensitivity mode, there is some response to the methanol solvent.



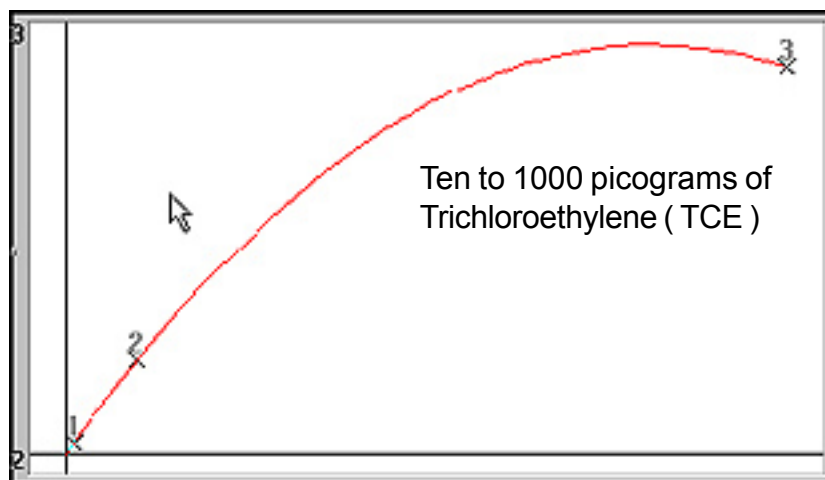
Operating the FID/DELCD in the high sensitivity DELCD only mode

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 μ l 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.

CHEMICAL DETECTION by DET

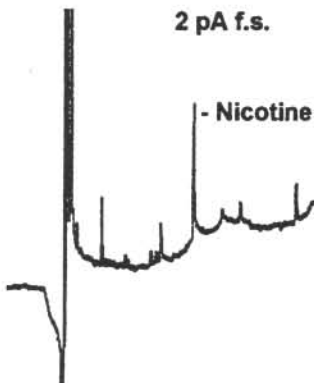
featuring novel applications of the principles of
THERMIONIC SURFACE IONIZATION and FLAME IONIZATION

Selective Detection for GC NPD - BEST N DETECTIVITY (less than 70 femtograms N/sec)

The combination of an Agilent 6890 NPD and a DET TID-4 ceramic ion source (bead) provides state-of-the-art N-selectivity for trace detection of drugs of abuse, pesticides, explosives, and pollutants.

The 6890 NPD hardware features a concentric cylinder ion source - collector electrode geometry for stream-lined gas flow and efficient ion collection. Similar DET equipment is available for HP5890, Varian 3400-3800, and SRI 8610 GC models.

600 femtograms Nicotine
TID-4 in 6890 NPD
2 pA f.s.



Selective Detection for GC PHOSPHORUS COMPOUNDS Very Big Signals with a New PTID

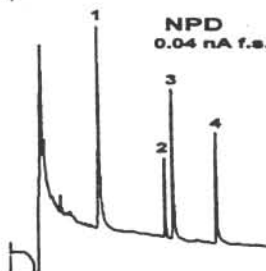
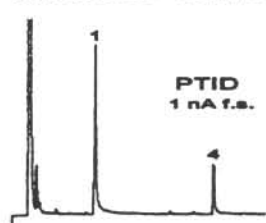
Pesticide Sample:

- 1-Mevinphos (P)
- 2-Trifluralin (N)
- 3-Simazine (N)
- 4-Methyl Parathion (NP)

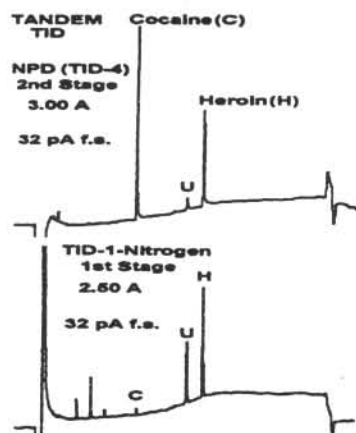
NPD detects both P and N. PTID detects only P with signals 10 times bigger than the NPD.

A Phosphorus Thermionic Ionization Detector (PTID) combines surface ionization principles with high flows of Hydrogen and Air for P/C selectivity of 100,000:1, P/N selectivity of 100:1, detectivity of 70 fg P/sec; and a dynamic response range more than 100,000.

NP pesticides 480 pg each



Tandem Thermionic Detection for GC COCAINE - HEROIN



NPD and TID-1 are two different modes of thermionic ionization.

Ceramic TID-1 surface operates at 400-600°C in a gas environment of Nitrogen or Air. TID-1 is non-destructive so it can be combined in series with another detector like the NPD.

Ceramic NPD surface operates at 600-800°C in an ignited, dilute mix of Hydrogen in Air.

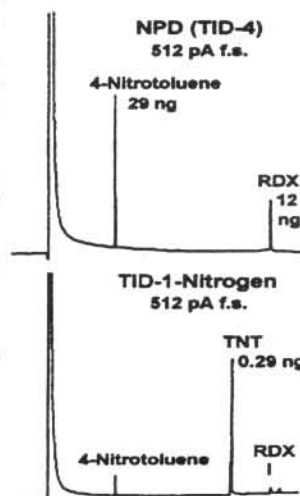
Sample analyzed: NPD detects both Cocaine (C) and Heroin (H). TID-1 detects Heroin and Heroin Impurity (U) Tandem combination gives simultaneous TID-1 and NPD signals for each sample injection.

- Femtogram GC Detection - NITRO-COMPOUNDS like TNT, 2,4-Dinitrotoluene, DNPH-Aldehydes, Methyl Parathion, 4-Nitrophenol, etc.

Unique TID-1 surface ionization provides better selectivity than ECD and NPD, and needs only Air or N₂ as the detector gas with no requirement for high purity.

TID-1 detection is an inexpensive modification of Agilent 6890 NPD equipment. DET NPD/TID-1 equipment is also available to fit HP 5890, Varian 3400-3800, and SRI 8610 GC models.

EXPLOSIVES Sample: NPD has a big response to RDX and 4-Nitrotoluene. TID-1 has a much larger response to TNT.

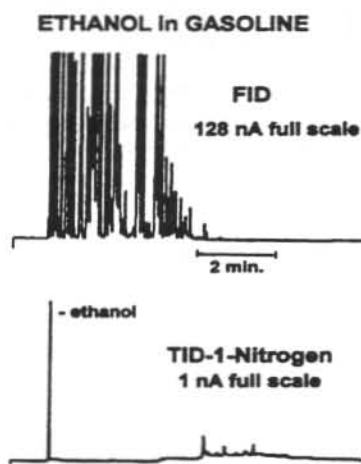


DETector Engineering & Technology, inc.

Oxygenate Selective Detection for GC ETHANOL in GASOLINE

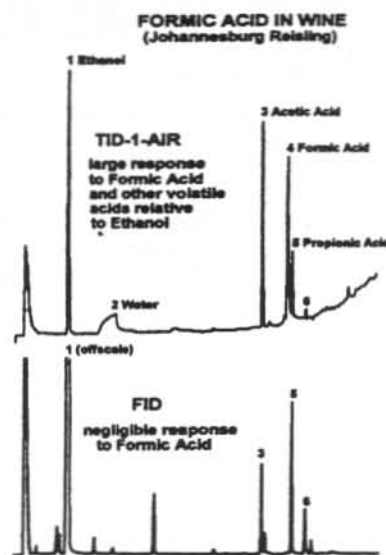
Negative ionization on a ceramic TID-1 surface detects Oxygenates with good selectivity vs. Hydrocarbons.

TID-1 detection provides a simple analysis for the Ethanol additive in gasoline. Only a single gas supply (Nitrogen) suffices for both GC carrier and detector gases. Short analysis times can be used because Ethanol is easily detected amidst many overlapping Hydrocarbon components. TID-1 also detects Phenols, Glycols, and other Oxygenated compounds.



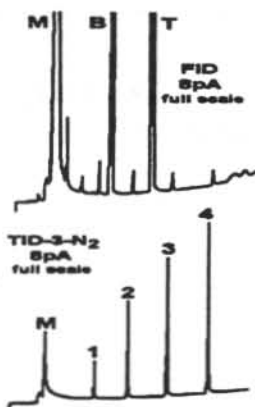
Oxygenate Selective Detection for GC CARBOXYLIC ACIDS

TID-1 surface ionization with an Air detector gas gives big signals for Carboxylic Acids relative to other Oxygenates like Alcohols. TID-1 detection includes Formic Acid which is not detected by an FID. TID-1 detection is also non-destructive so component aromas can be sensed at the detector exit. H_2O is also detectable to ppm levels.



Selective Detection for GC TRIHALOMETHANES

TID-3 surface catalyzed negative ionization process



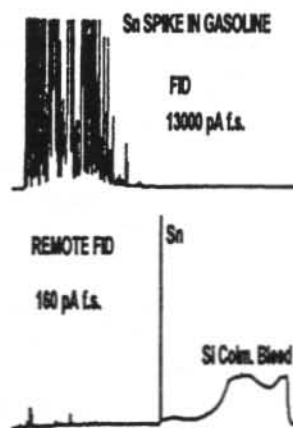
Volatile HALOGENATES detected with a sensitivity of 1 pg/sec, selectivity of 100,000:1 vs. hydrocarbons, and linear response exceeding a range of 10,000 in sample weight.

Unlike other halogen detectors, TID-3 response to Br is significantly more than Cl. Detector gas may be Nitrogen or Air with no requirement for ultra high purity. This detector is much easier and less costly to operate and maintain than an Electrolytic Conductivity Detector.

Sample analyzed:

640 pg each: 1=CHCl₃, 2=CHCl₂Br, 3=CHClBr₂, 4=CHBr₃,
47,000 pg each: B=benzene T=toluene
2,500,000 pg: M=methanol Solvent: water

Pb - Sn - P - Si (Lead, Tin, Phosphorus, Silicon) selective detection with a DET innovation Organically-Fueled Remote FID



A polarizer and ion collector located several centimeters downstream of a flame jet detect long-lived ion species that originate in a flame fueled by H_2 - CH_4 - Air. Ionization from Hydrocarbon combustion at the jet dissipates before reaching the downstream collector.

Detectivity of 1 pg/sec for Pb, Sn, P with a selectivity of 500,000:1 versus Carbon.

Sample:
12 ppm tetrabutyltin in gasoline

DETECTOR Engineering & Technology, inc.

486 North Wiget Lane, Walnut Creek, CA 94598 USA
ph: 925-937-4203, fx: 925-937-7581, www.det-gc.com

DETECTORS

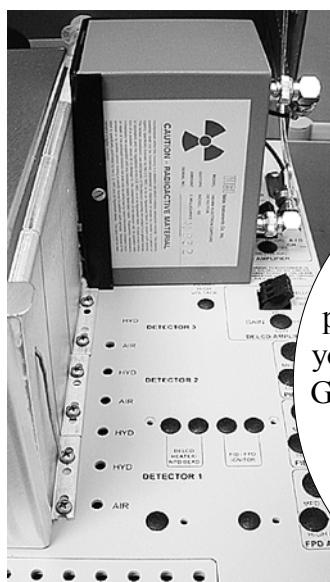
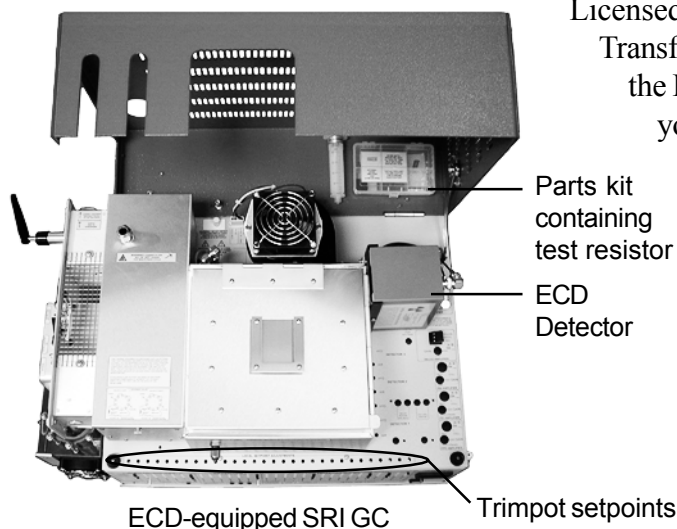
Electron Capture Detector - ECD

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 SRI 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 radioactive source is included in the ECD manual from Valco, the manufacturers. This documentation transfers possession of the ECD directly to you from Valco; 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.



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 Certification of Sealed Source and Test Specifications, which are proof that your ECD detector meets regulations.

1

VICI
Valco Instruments Co. Inc.

CERTIFICATION OF SEALED SOURCE

This instrument was tested for surface radiation and was found to be within acceptable legal limits. Sources from which 0.005 microcuries or more radioactivity is detected in wipe tests are considered to be leaking. Leak Tests are required at 6 month intervals on all NRC sources. These sources are tested initially for leakage before shipment in accordance with our Texas Department of Health Radioactive Materials License #10-1592.

Radionucleide: Ni-63 Source model number: 140BND
 Activity: 5.061 Instrument serial number: N 822
 Wipe date: 4-26-2000 File number: 15-8279
 Wiped by: [Signature]
RESULTS
 Date: 4-26-2000 Accepted by: [Signature]
 Activity assayed: -1.2 x 10⁻⁶ microcuries

This sealed detector source is distributed to persons generally licensed pursuant to TDCR 41.20(6).
 P.O. Box 55003 Houston, Texas 77255 Telephone (713) 688-5045 Fax (713) 688-5043

2

TEXAS DEPARTMENT OF HEALTH
BUREAU OF RADIATION CONTROL

3

VICI
Valco Instruments Co. Inc.

analytical devices/systems

Dear Valco Customer:

Model 140B (or Model 140BN) Electron Capture Detector is a sealed source detector. The detector is mounted on your carrier gas and system.

As described in the ECD Manual, the detector should be within 50 psi of the carrier gas pressure. If you are using a carrier gas pressure less than 50 psi, there is a problem and you should contact your nearest Valco representative. If you are using a carrier gas pressure of 50 psi or more, the detector is operating properly.

We adjust settings on the detector to match the manual.

For this instrument:
 Carrier gas: 50% UHI
 Flow rate: 80 mL/min
 Temperature: 240°C
 Carrier gas: Ar/CH₄
 Detector: N₂
 Job setting: Ar/CH₄
 Detector: N₂

I hereby certify to be fully operational. The unit was tested to maximum temperature specifications.

Model 140BN: 400°C ±

P.O. Box 55003 Houston, Texas 77255 Telephone (713) 688-5045

4

VICI
VALCO INSTRUMENTS CO. INC.

RECORD OF RADIOACTIVE SOURCE TRANSFER

Date: 6 June 2000

As required by our Radioactive Materials License # 10-1592 issued by the Texas Department of Health, the following generally licensed, sealed radioactive source:

File No. _____ Model _____ Source Material _____
 15-0279 Model 140BN Electron Capture Detector 5 mCi 63Ni
 (Serial Number 8022)

has been transferred to the following authorized individual:

Your name & address here

Acknowledged by: _____ Name _____
 _____ Title _____
 _____ Date _____
 Please sign and send one copy to: Your national agency in charge of radioactive source transfer
 Please sign and send one copy to: Valco Instruments Co., Inc.
 Attn: Radiation Safety Officer
 P.O. Box 55003
 Houston, TX 77255-5003

Please keep one copy for your information.

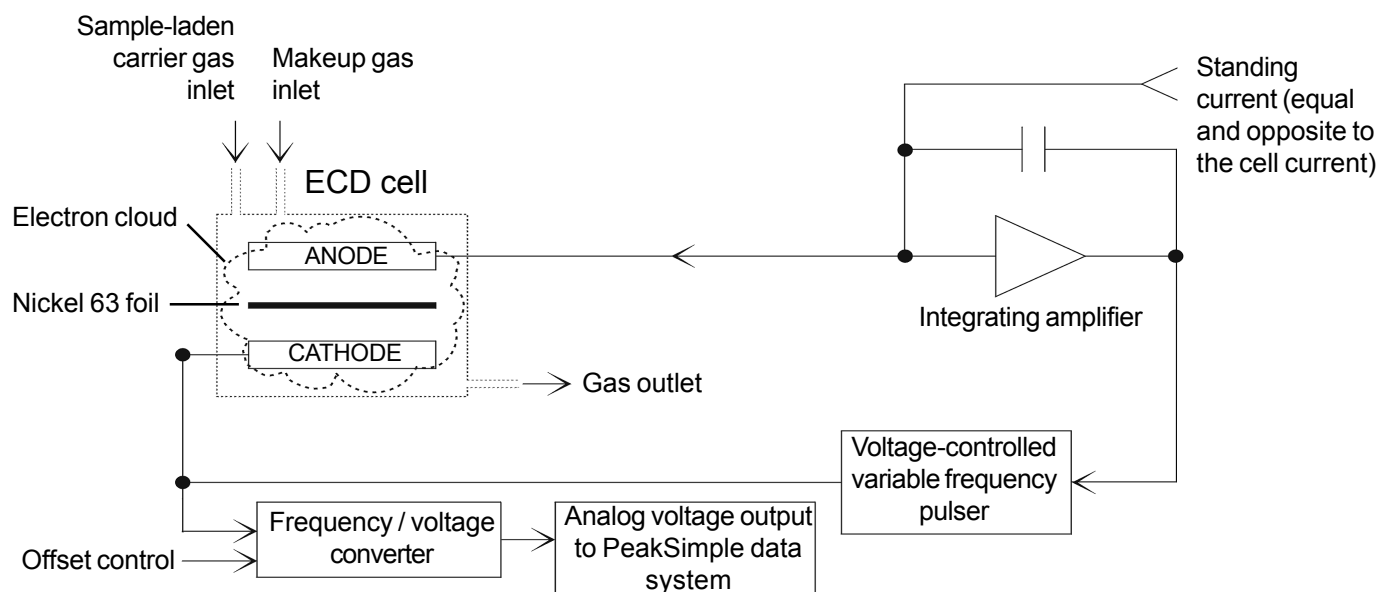
DETECTORS

Electron Capture Detector - ECD

Theory of Operation

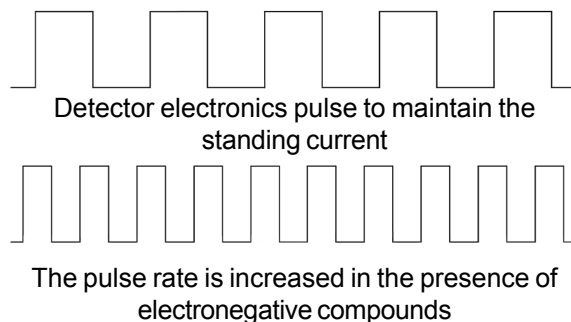
The radioactive Nickel 63 sealed inside the ECD detector emits electrons (beta particles) which collide with and ionize the make-up gas molecules (either nitrogen or P5). This reaction forms a stable cloud of free electrons in the ECD detector cell. The ECD electronics work to maintain a constant current 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



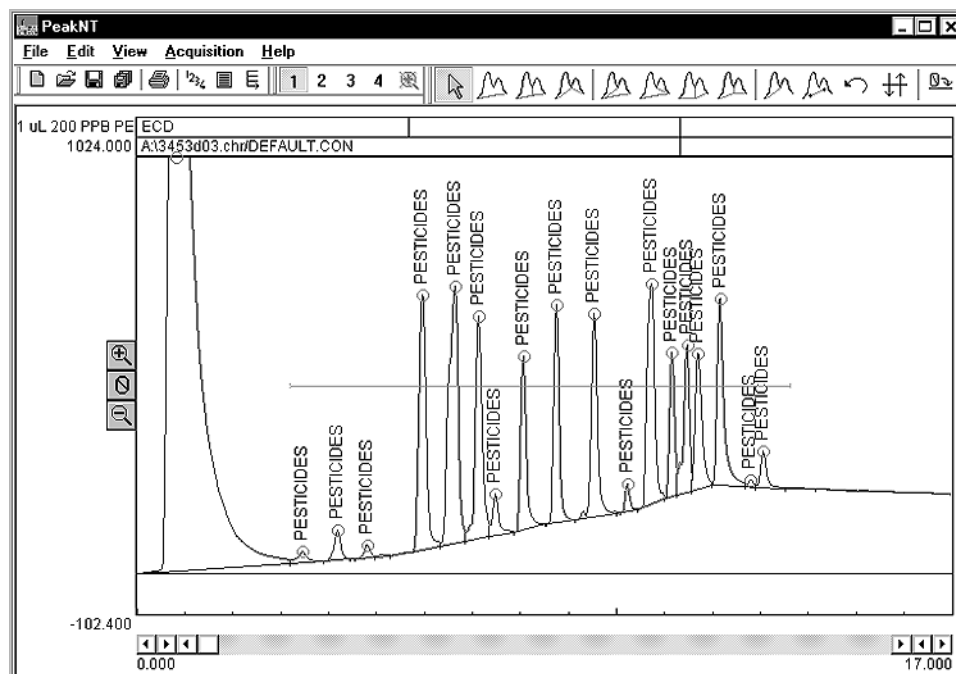
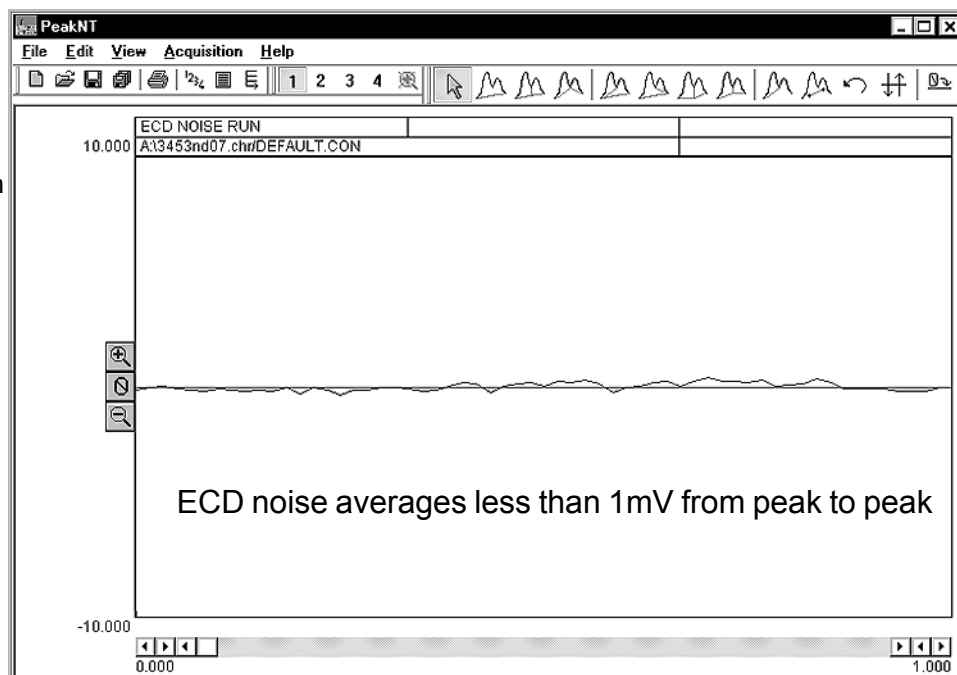
DETECTORS

Electron Capture Detector - ECD

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

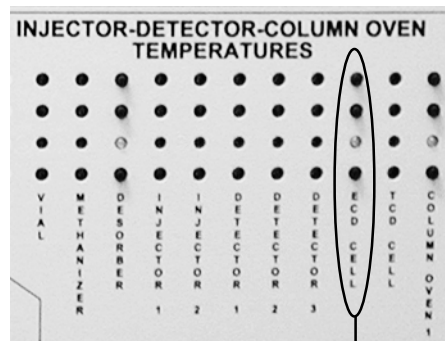
DETECTORS

Electron Capture Detector - ECD

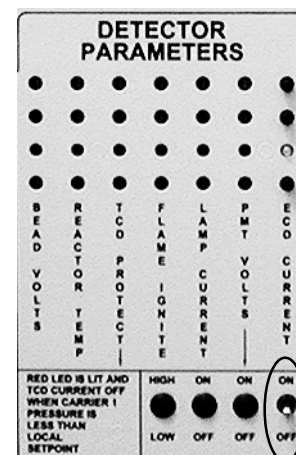
General Operating Procedure

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

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



ECD standing current ON/OFF switch

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.

DETECTORS

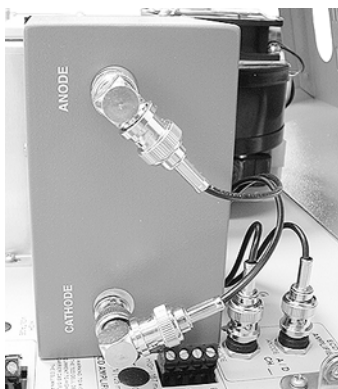
Electron Capture Detector - ECD

ECD Troubleshooting

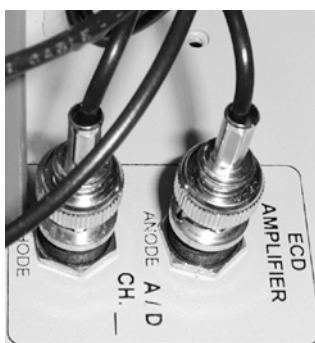
If you are experiencing baseline offset and noise problems with your ECD detector, try the following two diagnostic tests:

1. Verify that the ECD amplifier electronics are working properly by removing the detector from the circuit and inserting a 1000M Ω test resistor in its place. The parts kit in the tackle box included with your GC under the red lid contains a 1000M Ω 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 1000M Ω 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.

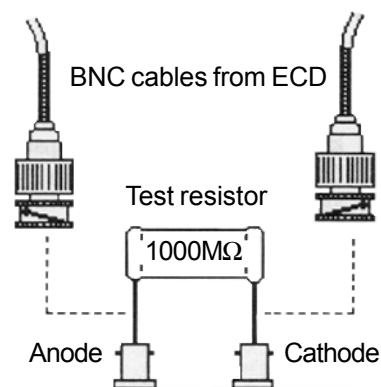
The ECD anode and cathode are connected to the ECD amplifier via BNC cables



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.

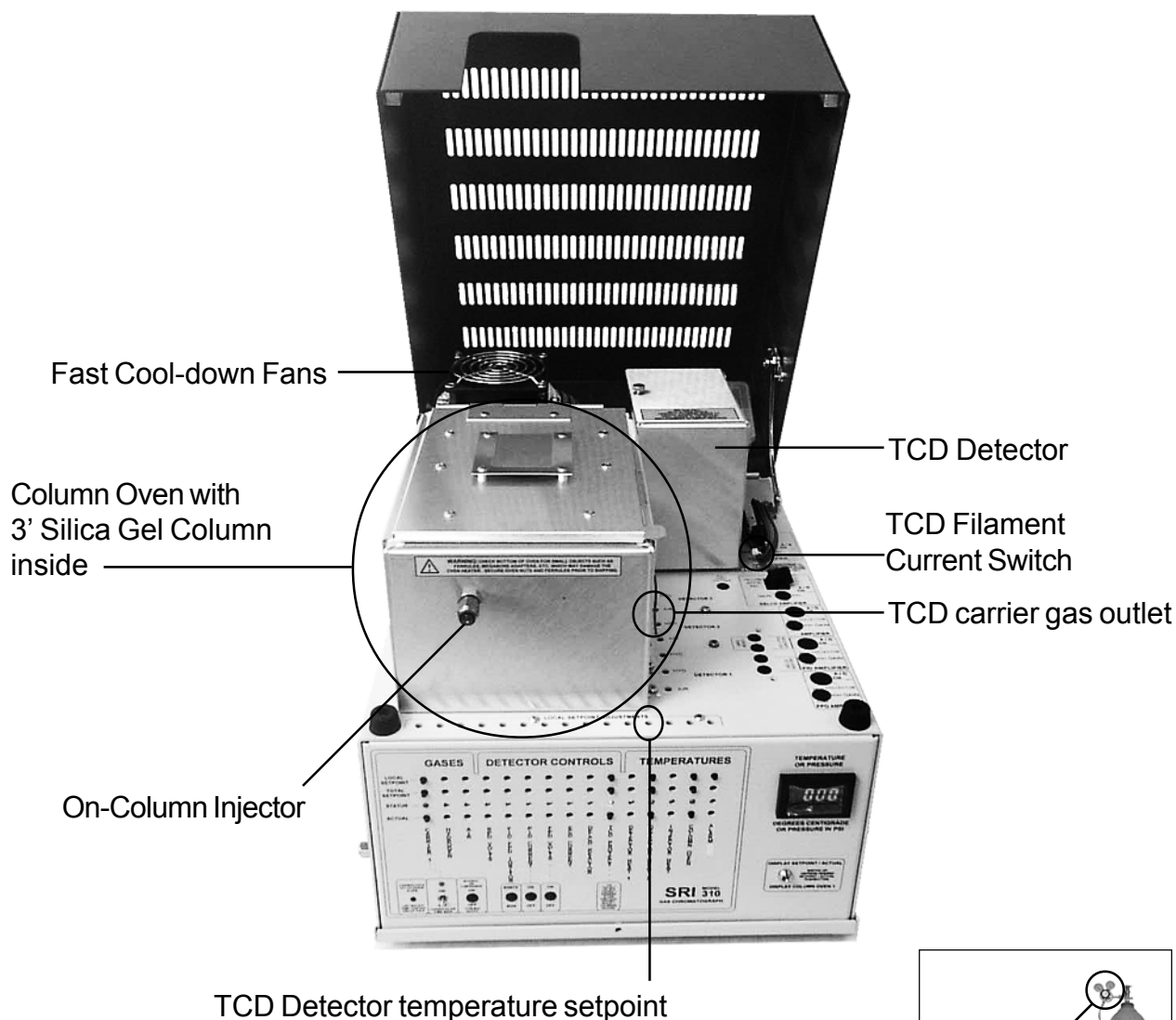
Tip: In most situations, the ECD will be used to detect sample components that are reactive with metal. Use glass, fused silica, or fused silica lined metal capillary columns to help avoid reactive sites and ghost peaks.

POPULAR CONFIGURATION GCs

Educational TCD

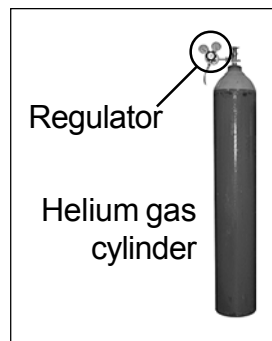
System Overview

Your educational TCD GC is configured on the compact 310 chassis. It is equipped with a TCD Detector, a temperature programmable Column Oven, a 3' Silica Gel packed column, Electronic Pressure Control (EPC) for carrier gas, On-column Injector, and a built-in, single channel PeakSimple Data System. The model shown below is equipped with optional Fast Cool-down fans.



The TCD Detector is located inside its own oven, mounted on the right rear of the Column Oven as shown above. Its temperature is factory preset at 100°C, but it may be heated up to 130°C by adjusting the trimpot with the small blade screwdriver attached to the front right corner of your GC. The trimpot looks like a small brass screw and is located inside the labeled hole on the top edge of the front control panel.

The TCD Detector requires helium to operate, which must be supplied by a gas cylinder and regulator. The helium cylinder pressure is normally set at 30psi, which is 10-20psi higher than the column head pressure.



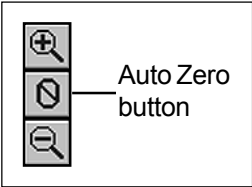
POPULAR CONFIGURATION GCs

Educational TCD

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.
2. The carrier gas head pressure is preset at the factory to 10mL/min for the Silica Gel 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. For this GC, carrier cylinder pressure is normally set at 30psi, which is 10-20mL higher than the column head pressure. The column head pressure is the pressure developed by the carrier gas as it flows through the analytical column.
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.
80.00	7.00	0.00	80.00
7. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Hit the RUN button on your GC or hit the spacebar on your computer keyboard to begin the run. You may also open the Acquisition pull-down menu and select Run, but this gets difficult unless you have a partner, since your hands are occupied with the sample syringe.


8. Using the 1mL syringe supplied with your GC, inject sample into column through the On-Column Injector.

POPULAR CONFIGURATION GCs

Educational TCD

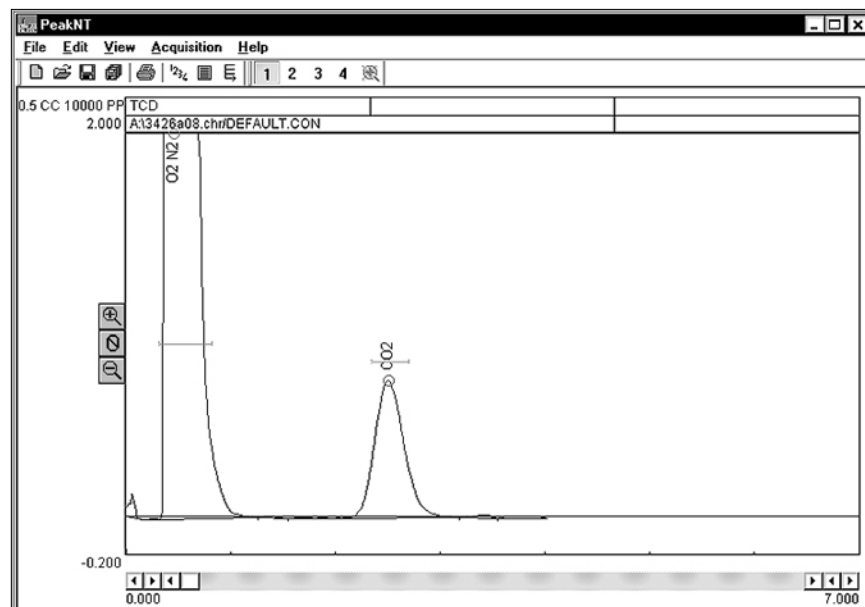
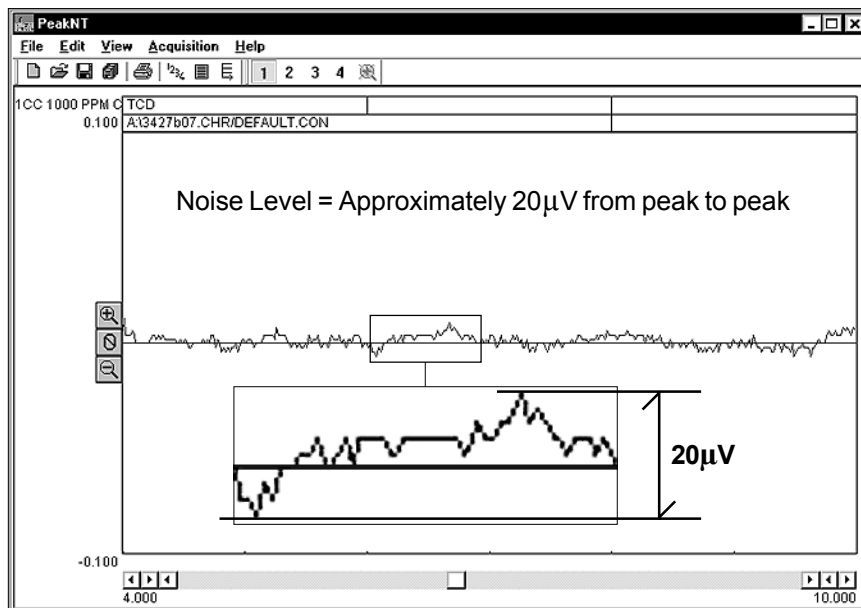
Expected Performance

Every compound possesses some degree of thermal conductivity and therefore may be measured with a TCD detector. TCD detectors are most often used with helium as a carrier gas because of helium's high thermal conductivity, but other gases such as nitrogen, argon, or hydrogen may also be used as a carrier gas. A TCD detects all molecules in concentrations from 100% down to around 100ppm, and is especially useful for measuring inorganic gases like O₂, N₂, CO & CO₂.

TCD Detector Noise

Column = 1m Silica Gel
Carrier = Helium at 10mL/min
TCD current = LOW
TCD Temp = 100°C

Temperature Program:
Initial Hold Ramp Final
80°C 10.00 0.00 80°C



Factory test run of an Educational TCD GC

Column = 1m Silica Gel
Carrier = Helium at 10mL/min
Sample = 0.5cc 10,000ppm CO₂
TCD current = LOW
TCD Temp = 100°C

Temperature Program:
Initial Hold Ramp Final
80°C 7.00 0.00 80°C

Component	Retention	Area
O2 N2	0.450	1252.9980
CO2	2.500	13.6460
Total		1266.6440

POPULAR CONFIGURATION GCs

Educational TCD

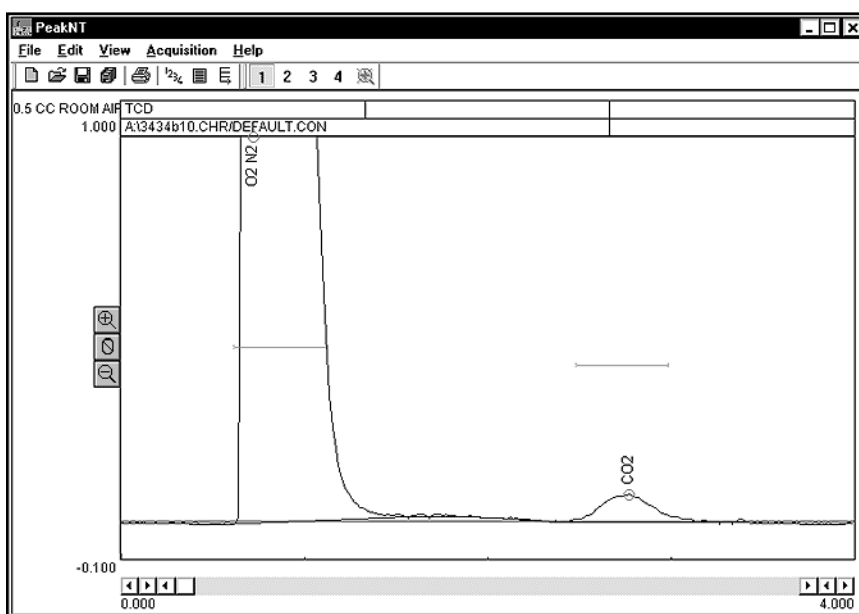
Expected Performance

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



Results:

Component	Retention	Area
O ₂ N ₂	0.716	1021.3830
CO ₂	2.766	1.5060
Total		1022.8890

The CO₂ content of the room air analyzed is approximately 350ppm.

POPULAR CONFIGURATION GCs Educational TCD

Suggested Class Experiment: "Waiting to Exhale"

CO₂ is a natural by-product of human respiration. Our lungs get oxygen when we inhale and release CO₂ when we exhale. When we hold our breath, the concentration of CO₂ increases. In this experimental gas chromatography analysis of human breath, the students will supply the samples. They will exhale into and trap their breath in the syringe, then it will be injected into the Educational TCD system and analyzed for CO₂ concentration. Have a contest for the highest CO₂ concentration: the student with the most CO₂ in his or her breath will win. Whomever passes out is disqualified!

1. Follow steps 1-4 of the **General Operating Procedure**.
2. In PeakSimple, set an isothermal Column Oven temperature ramp program as follows:

Initial Temp.	Hold	Ramp	Final Temp.
80.00	4.00	0.00	80.00
3. Locate the 3mL (3cc) syringe supplied with your GC, remove its needle, and give both parts to a student. Instruct the student to exhale into the tip of the syringe while pulling back on the plunger. Students need not touch the syringe with their mouths for it to work. Fill the syringe completely, then replace the needle. Depress the plunger until the syringe contains 0.5mL of breath.
NOTE: For sanitation concerns, it may be prudent to have one new, sterile syringe for each participating student. Sterile 3mL syringes complete with needles may be acquired for about \$0.18 each from:
VWR (800-932-5000):
BD-309587 Syringe-Needle, 3mL Sub-Q 26G 5/8 Luer-lok™
4. Click the Auto Zero button in PeakSimple, then press the RUN button on your GC or the spacebar on your PC keyboard to begin the run.
5. Inject sample into the On-Column injector.
6. Save and print the resulting PeakSimple chromatogram with the student's name for the sample identification. Typical results are about 12-14 area counts per 1% of CO₂.
7. Repeat steps 2-5 for each student. Compare chromatograms to find the winner.

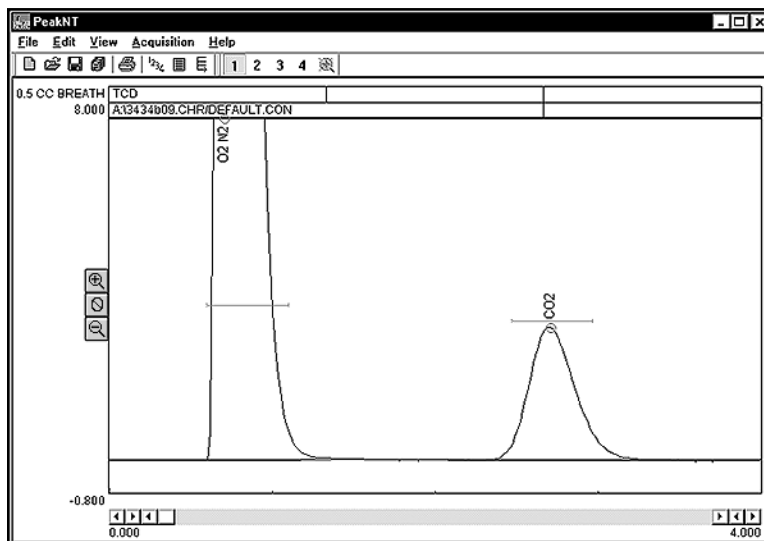
Example TCD Breath Analysis

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

Temperature Program:
Initial Hold Ramp Final
80°C 24.00 0.00 80°C

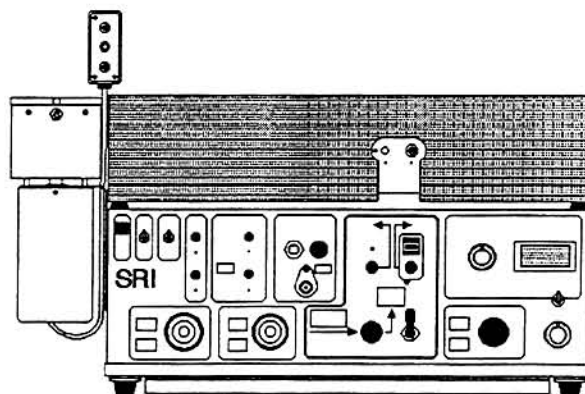
Results:

Component	Retention	Area
O ₂ N ₂	0.700	1379.4740
CO ₂	2.700	61.9540
Total		1441.4280



If the gas chromatograph in use is equipped with an electrically actuated multiport gas sampling valve and, after discussing the trouble experienced with the technical support staff at SRI Instruments, a valve actuator replacement is deemed necessary, then a replacement valve actuator may be purchased (or ordered under warranty) and the replacement may be performed in the field by the user.

By following the steps outlined below, the user may effect the replacement of the actuator in a relatively short period of time without much difficulty.



SRI gas chromatograph equipped with electrically actuated gas sampling valve

STEP 1: Remove power from the unit and allow it to cool to ambient temperature. Disconnect the power cord from the AC supply (wall outlet). Unplug the 5-wire modular plug on the cable that exits the base of the actuator housing.

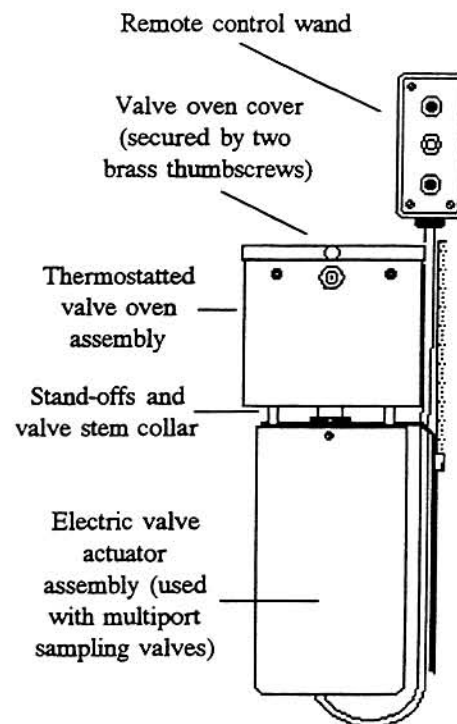
STEP 2: Remove the two brass thumbscrews securing the valve oven cover. Then remove the valve oven cover. Remove the top insulating blanket directly beneath the valve oven cover.

STEP 3: Note that there are four holes in the insulating blanket at the base of the valve stem. These holes permit access to four Phillips-head screws that secure the valve oven to the stand-offs mounted on the valve actuator through the bracket. Remove the four screws (a magnetic device may be needed to retrieve the screws through the insulation).

STEP 4: Using a 9/64" Allen wrench, loosen the set screw on the side of the collar securing the valve stem shaft. This will release the valve assembly. Then lift the valve oven slightly away from the actuator assembly in order to insert the Allen wrench into the two set screws present in the top surface of the collar ring. These screws go through the bracket must be removed.

STEP 5: Remove the four hexagonal stand-off posts mounted through the bracket into the valve actuator assembly. This will free the valve actuator assembly from the bracket and permit replacement. Remove defective actuator assembly and substitute with replacement valve actuator. Reassemble in the reverse order, from step 5 to step 1.

STEP 6: Verify proper valve operation after reinstallation. Verify that the valve position matches the position indicated on the remote control wand. Listen to the valve when rotating to hear for actuator jamming or other unusual noises.



VIEW OF VALVE OVEN AND ACTUATOR ASSEMBLIES AS SEEN FROM FRONT OF UNIT

If any difficulties are encountered during or after the valve actuator replacement process, contact SRI Instruments technical support for assistance at (310) 214-5092. If an actuator is suspected to be defective, consult with SRI before attempting removal. The problem may be located elsewhere in the system and diagnosis may be possible over the telephone.

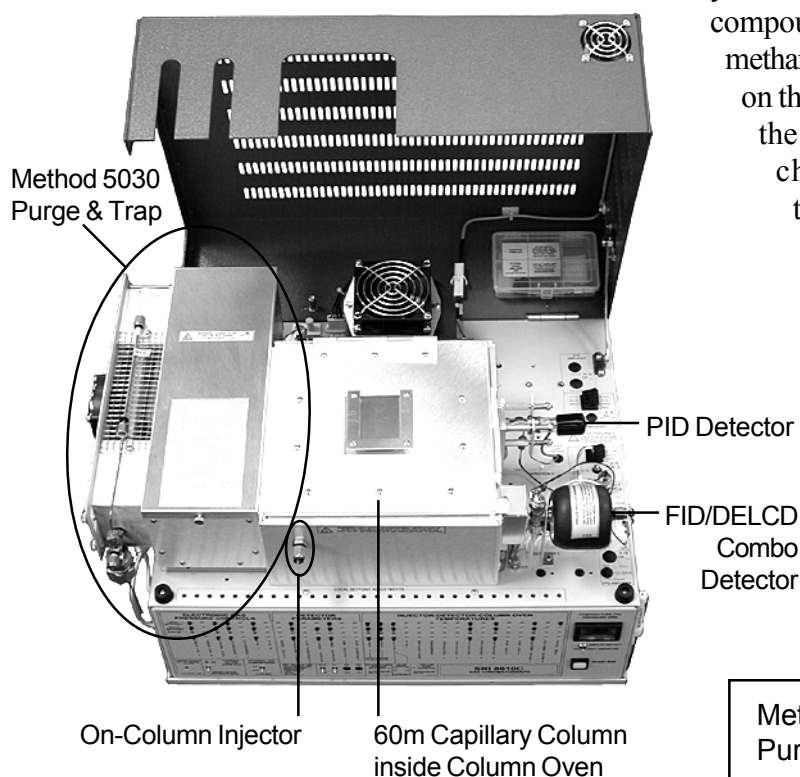
POPULAR CONFIGURATION GCs BTEX & Environmental

System Overview

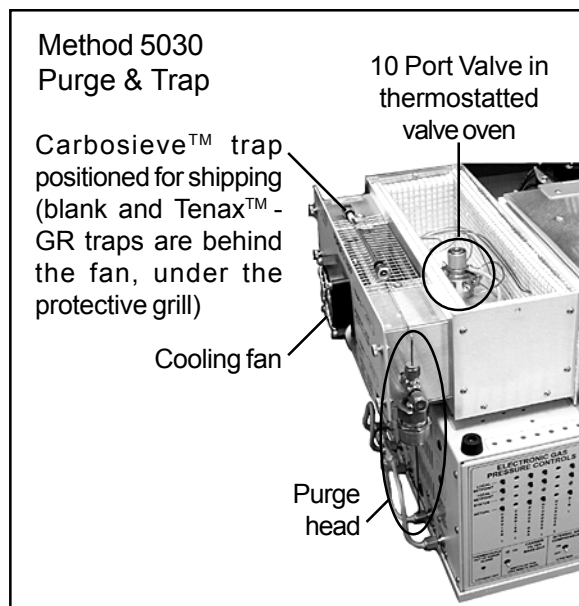
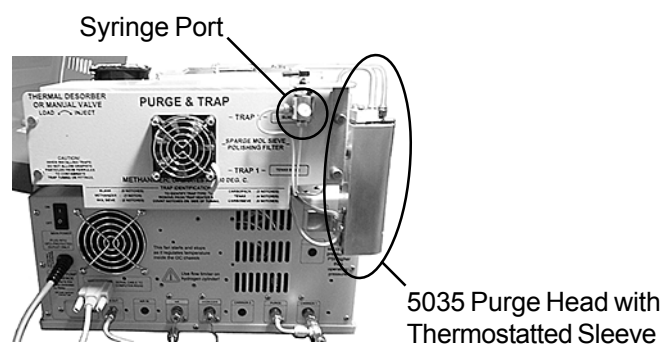
Your SRI Environmental GC is equipped with everything you need to generate certification quality data for EPA Methods 8010, 8015, 8021, and others. It is configured on the 8610C chassis, and includes a built-in Method 5030 or 5030/5035 compliant Purge & Trap for concentration of liquid and/or soil samples. Also included is an on-column injector for direct liquid injections. To detect commonly targeted pollutants, the Environmental GC uses a sensitive, non-destructive PID detector in series with a combination FID/DELCD detector. The PID detector responds to compounds whose ionization potential is below 10.6eV, including aromatics and chlorinated molecules with double carbon bonds. The FID detector responds to the hydrocarbons in the sample. The DELCD selectively detects the chlorinated and brominated compounds in the FID exhaust. Since the sample is pre-combusted in the FID flame, the DELCD is protected from contamination due to

hydrocarbon overload. The PID is blind to certain compounds which can cause interference, such as methanol, and is recommended by the EPA. Peaks on the FID chromatogram that are obscured by the methanol peak are visible on the PID chromatogram. Benzene and carbon tetrachloride are common target analytes which co-elute. The FID responds to both. The PID responds only to benzene, while the DELCD responds only to carbon tetrachloride.

The BTEX GC is the same as the Environmental GC without the DELCD detector. Both systems have a “whisper quiet” internal air compressor and can be used with an H₂-50 hydrogen generator for tankless field operation.

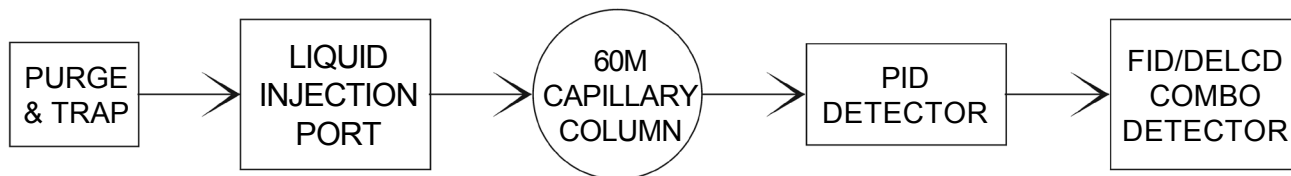


Method 5030/5035 Purge & Trap on an Environmental GC



POPULAR CONFIGURATION GCs BTEX & Environmental

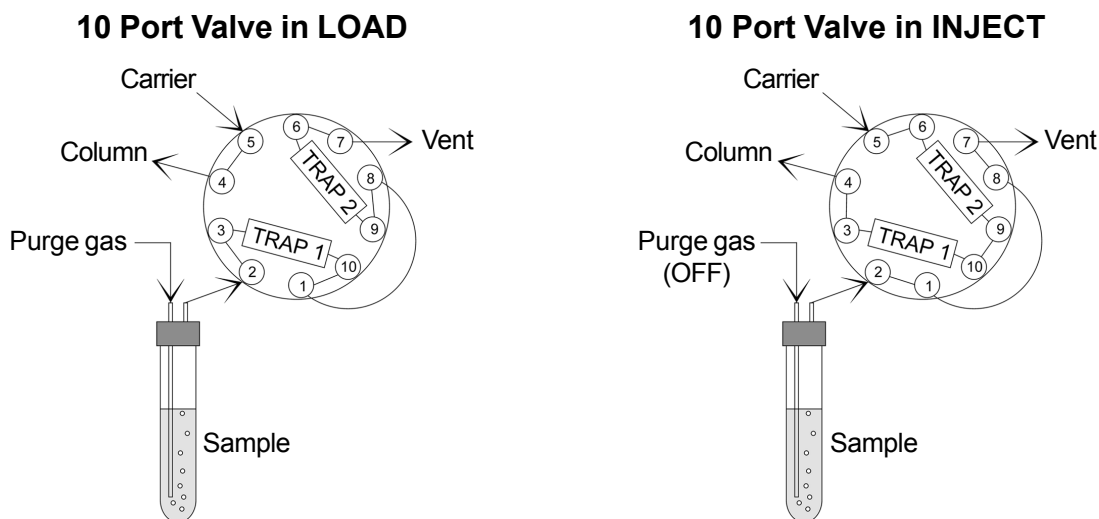
Theory of Operation



The versatile BTEX/Environmental GC systems can analyze gas, water, and soil samples. Four types of injection techniques can be used: purge and trap, direct liquid injection, TO-14 type gas sample concentration, and manual headspace injection. The Purge & Trap concentrator may be used for gas, liquid, and solid samples. For liquid samples up to 5µL and gas samples up to 1mL, direct injections can be made through the on-column liquid injection port. Larger gas samples can be injected through the syringe port on the 5030/5035 Purge & Trap concentrator or the septum port on the 5030 model.

Purge & Trap Injection

Designed for compliance with EPA Methods 5030 and/or 5035, the Purge & Trap system extracts volatile organic compounds from the sample solution in the test tube or VOA vial. Using a dual trap design plumbed with a 10 port gas sampling valve, the Purge & Trap system enables the use of two separate adsorbents with different desorption temperatures for a wide range of target analytes. Each trap is heated independently.



When the valve is in the LOAD position, the sample-laden purge gas from the test tube or VOA vial is directed through the two traps, then out to vent, loading the traps with sample at the adsorption temperature. The traps are heated to their respective desorption temperatures shortly after purging is stopped. When the traps reach desorption temperature, the valve is actuated to the INJECT position. In this position, the carrier gas backflushes through the traps in the direction opposite to the sample-laden purge gas flow with which the traps were loaded. The carrier gas flow sweeps desorbed analytes into the column, while flow from the purge vessel is stopped by the PeakSimple data system.

Theory of Operation continued

Direct Injection

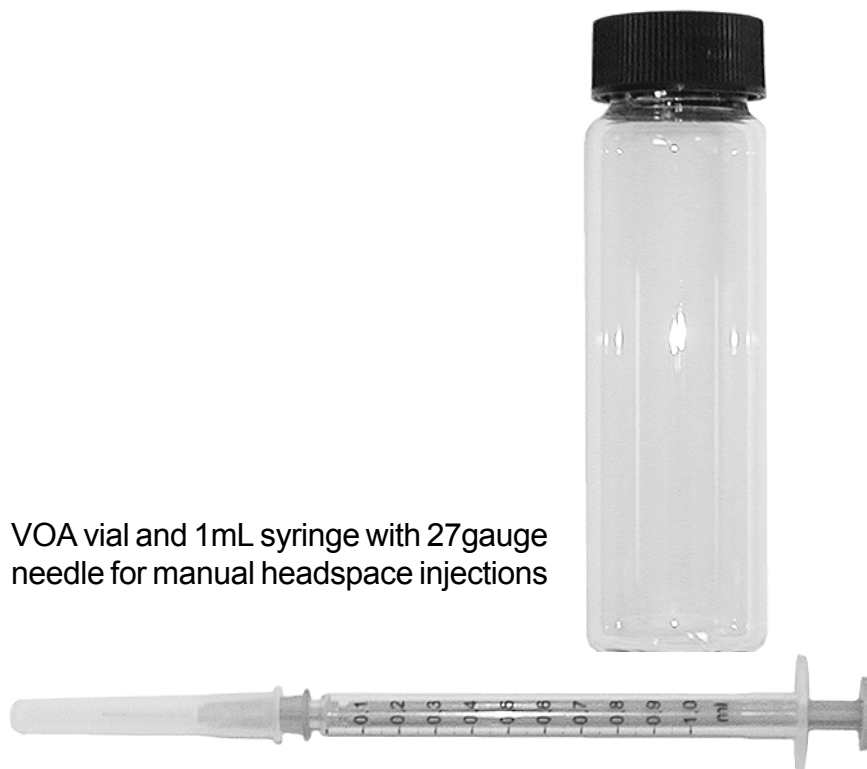
Direct injection with the BTEX or Environmental GC systems is simple and straightforward. This method uses the on-column injector to inject the sample directly into the column, bypassing the entire purge and trap injection system. Sample size for this technique is 1mL or less for gas, and 5 μ L or less for liquid. No event table is necessary, just a temperature program for the column oven.

Gas Sample Concentration

In this TO-14 type technique, a large volume of gas is pushed by syringe or pulled by vacuum pump through the dual traps. The trapped analytes are then desorbed and swept into the column. If the GC has the optional vacuum pump interface, the pump is plugged into it and may be controlled by the PeakSimple data system using an event table.

Room Temperature Manual Headspace Injection

When making headspace injections with the BTEX or Environmental GC systems, the sample is equilibrated offline at room temperature. It is then injected by syringe into the on-column injector. This technique is basically the direct injection of small gas samples.



VOA vial and 1mL syringe with 27gauge
needle for manual headspace injections

POPULAR CONFIGURATION GCs BTEX & Environmental

General Operating Procedures

EPA Style Purge & Trap Injection

This technique is limited to volatile organic compounds that purge efficiently from water at ambient temperature and VOC's that are purgeable from soil at 40°C. Sample preparation depends on the sample type, concentration, amount, etc. The third edition of SW-846 from the EPA is accessible on the Internet. Go to <http://www.epa.gov/epaoswer/hazwaste/test/main.htm> and click on the **5000 Series** link to download Methods 5030 and 5035. Also, please see the "**Sample Preparation**" page in the SRI Purge & Trap manual section (available online at www.srigc.com).

1. The purge gas flow is controlled with an Electronic Pressure Controller (EPC). Set the purge flow (measurable at the trap vent at the rear of the purge and trap system); 40mL/min is a typical purge flow. The pressure required for 40mL/min through a single Tenax trap is printed on the right panel of the GC. **NEVER use hydrogen as a purge gas.** SRI recommends helium purge gas.
2. TRAP 1 is in the lower position in the Purge & Trap, and TRAP 2 is in the upper position. The trap temperatures are factory set at 200°C for desorption. For adsorption temperatures, trap 1 is set at 30°C and trap 2 is set at 35°C. Trap heating will be controlled by the timed Event Table during the run. NOTE: the actual temperatures typically run 5°C over the setpoint. See the instructions in the Purge & Trap section of the manual for adjusting the trap adsorption temperature settings.
3. Load or create an Event Table that is appropriate to the sample to be analyzed, or that is designed for compliance with a particular EPA Method (such as **Epap&t1c.evt** for a single trap or **Epap&t2c.evt** for dual traps included in version 2.66 or higher of the PeakSimple software).
4. Load or create an appropriate Temperature Program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software for your convenience. As a basic rule for good separation using the purge and trap injection technique, the column oven should be kept at 40°C for 10-12 minutes: 6 minutes while the sample is purging, plus 4-6 more minutes while the traps heat and the gas sampling valve (in the INJECT position) transfers the sample to the column.

Epap&t1c.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.100	E "ON"	Purge "ON"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.100	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
13.000	G "OFF"	Valve in "LOAD"
13.100	B "ON"	Trap set "ON" (+50°C)
14.900	F "OFF"	Trap 1 "OFF"
15.050	E "OFF"	Purge "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.200	B "OFF"	Trap set "OFF" (+0)

Epap&t1c.evt is designed for one trap, while **Epap&t2c.evt** is for two traps.

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

General Operating Procedures continued

Direct Injection

This technique is useful for volatile and semi-volatile compounds, but is typically used for diesel and other compounds that don't purge well from aqueous or soil samples.

1. Perform **Detector Steps 1-4**, then proceed with step two below.

2. Load or create a Temperature Program for the column oven. You can create an isothermal or ramped temperature program; deciding which to use depends on the sample being analyzed, and the goals of the analysis. There are several preset **.tem** files included with version 2.66 and higher of the PeakSimple software. If the analysis requires the column to be hotter than 150°C, it is best to disconnect the column from the PID detector. The PID represents a cold spot in which higher boiling analytes will become trapped, never making it to the much hotter (300°C) FID for detection. Also, when the column is heated over 150°C, stationary phase bleed will

adhere to the PID lamp window. The higher boiling analytes and the column bleed will create a coating on the PID lamp window that will interfere with the analysis. The PID lamp window may be cleaned in the event of contaminant condensation, but the resulting change in the PID response usually requires detector recalibration. To bypass the PID, turn its lamp current OFF, then disconnect the column from the detector by loosening the swagelok-type nut from the bulkhead fitting in the column oven wall. Remove the tubing that connects the PID exit to the FID/DELCD by loosening that nut. Place the end of the column into the FID/DELCD bulkhead fitting instead and tighten it in place.

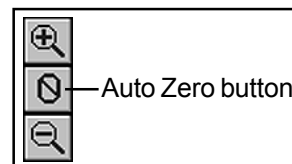
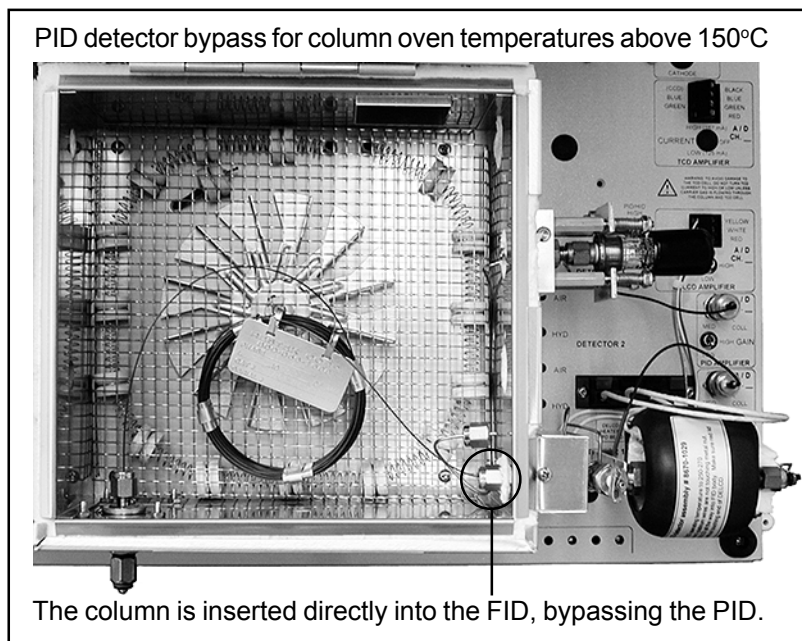
3. While the detectors are heating and stabilizing, prepare a diesel sample by shaking a known weight of the sample with a measured volume of methylene chloride for 1-3 minutes. Allow any particulates to settle before drawing the sample into the syringe.

4. Use a clean, standard glass 10µL GC syringe with a 26 gauge needle. Fill the syringe with sample, and work out any air bubbles. Depress the plunger until 1µL of sample remains in the syringe.

5. Zero the data system signal by clicking on the Auto Zero button on the left side of the chromatogram window. Or, make the first event ZERO (at time 0.00) in your event table.

6. Begin the analysis by pressing the RUN button on the GC or the computer keyboard spacebar.

7. Quickly and smoothly insert the syringe needle into the on-column injection port, and immediately depress the plunger.



POPULAR CONFIGURATION GCs BTEX & Environmental

General Operating Procedures continued

Gas Sample Concentration

This TO-14 type technique injects a gas or air sample using either a large syringe (60mL) or a Tedlar bag (1L). A vacuum pump may be used to pull the sample through the sorbent traps. The amount of sample that may be loaded onto the trap(s) is limited only by the capacity of the trap's adsorbent packings. The more gas that is loaded onto the traps, the lower the detection limit will be.

The volume and flow of sample and carrier gas that can be fed through the traps without adversely affecting the resulting chromatogram is known as the breakthrough volume. Different adsorbents have different breakthrough volumes. A breakthrough volume value is determined by the sample and target analytes, the adsorbent packing (pore size, natural affinities for certain compounds, etc.), the diameter of the trap, and the temperature at which the traps are loaded. Therefore, a given trap will have different breakthrough volumes in different analytical conditions.

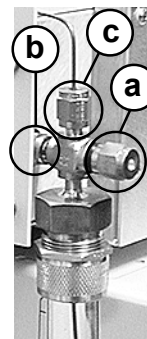
The SRI Purge & Trap concentrator is shipped with a blank trap and a Tenax™-GR trap installed, and a Carbosieve™ S-III packed trap for optional user installation. The Tenax-GR trap has a low affinity for water, making it a good adsorbent for the purge and trap technique. The Carbosieve has a high affinity for water, and is generally highly retentive; SRI recommends using it only when vinyl chloride is among the target analytes. The blank trap is provided for the user to pack with the adsorbent of choice.

Using a syringe:

1. Perform **Detector Steps 1-4**. While the detectors are heating and stabilizing, load or create an event table. The valve (Relay G) must be in the LOAD (G OFF) position while analytes are being adsorbed onto the traps. The valve is rotated to the INJECT (G ON) position during desorption. See the valve diagrams on the **EPA Style Purge & Trap Injection Theory of Operation** page. Relays C (trap 2) and F (trap 1) activate the traps' heat. The relays may also be activated by the operator during an analysis: open the Relay/pump window and click on the letter corresponding with the relay you want to turn ON or OFF.
2. Inject the sample into the 5030 septum nut or the 5030/5035 syringe port. Alternatively, the 5030 purge head may be removed by unscrewing nut **b**, allowing the sample to be injected directly into the bulkhead fitting on the front of the valve oven duct (see the photo, below right). Depending on the syringe you're using, you may have to make an adaptor for injection into the purge head.
3. Load or create a temperature program for the column oven. Once the detectors are activated and stabilized, begin the analysis.

Using a vacuum pump:

1. Connect the vacuum pump to the trap vent on the backside of the valve oven.
2. If your GC has the optional vacuum pump interface installed, plug the vacuum pump into that power socket on the left panel of the GC chassis. Enter events in the event table to turn the vacuum pump power ON and OFF as desired during the analysis. If your GC doesn't have the vacuum pump interface, plug the vacuum pump into a wall outlet instead, and control it's ON/OFF switch manually during the analysis.
3. Once the detectors are activated and stabilized, connect the Tedlar bag to the purge head septum nut (**a**), or remove the purge head and secure the Tedlar bag to the bulkhead fitting in the front valve oven duct. [To remove the purge head: loosen the nut (**b**) that secures the purge head to the bulkhead fitting in the valve oven duct wall. Loosen the nut (**c**) that secures the purge head to the purge gas tubing. Leave the second fitting (**c**) on the purge gas tubing and slide the purge head off of the tubing. See the photo, above right.] Load or create a temperature program. Begin the analysis.



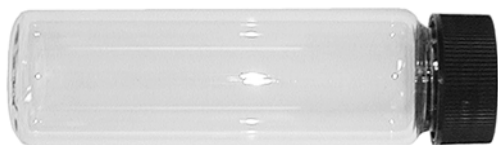
POPULAR CONFIGURATION GCs BTEX & Environmental

General Operating Procedures continued

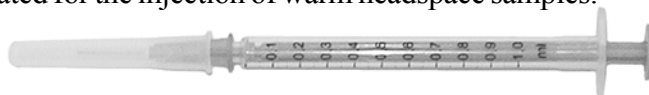
Room Temperature Manual Headspace Injection

1. In this technique, the sample is equilibrated offline. Transfer sample into a clean VOA vial until the vial is half full. Let it set at room temperature for 30 minutes to an hour to equilibrate.
2. Load or create a temperature program for the column oven.
3. Perform **Detector Steps** 1-4, then proceed with the following steps.
4. Fill a plastic medical syringe with the vial headspace. Inject the sample into the GC injection port, bypassing the Purge & Trap concentrator.
5. Begin the analysis by pressing the RUN button on the GC or the computer keyboard spacebar.

Note: both the sample vial and the syringe may be heated for the injection of warm headspace samples.



40mL VOA vials are available from Eagle Picher under part number 140-40C/EP/ES.
1-800-331-7425



Disposable, sterile 1mL syringes are available in packages of 100 from Aldrich under catalog number Z23072-3. 27 gauge precision glide needles in packages of 100 are available under catalog number Z19237-6.
1-800-558-9260

Detector Steps

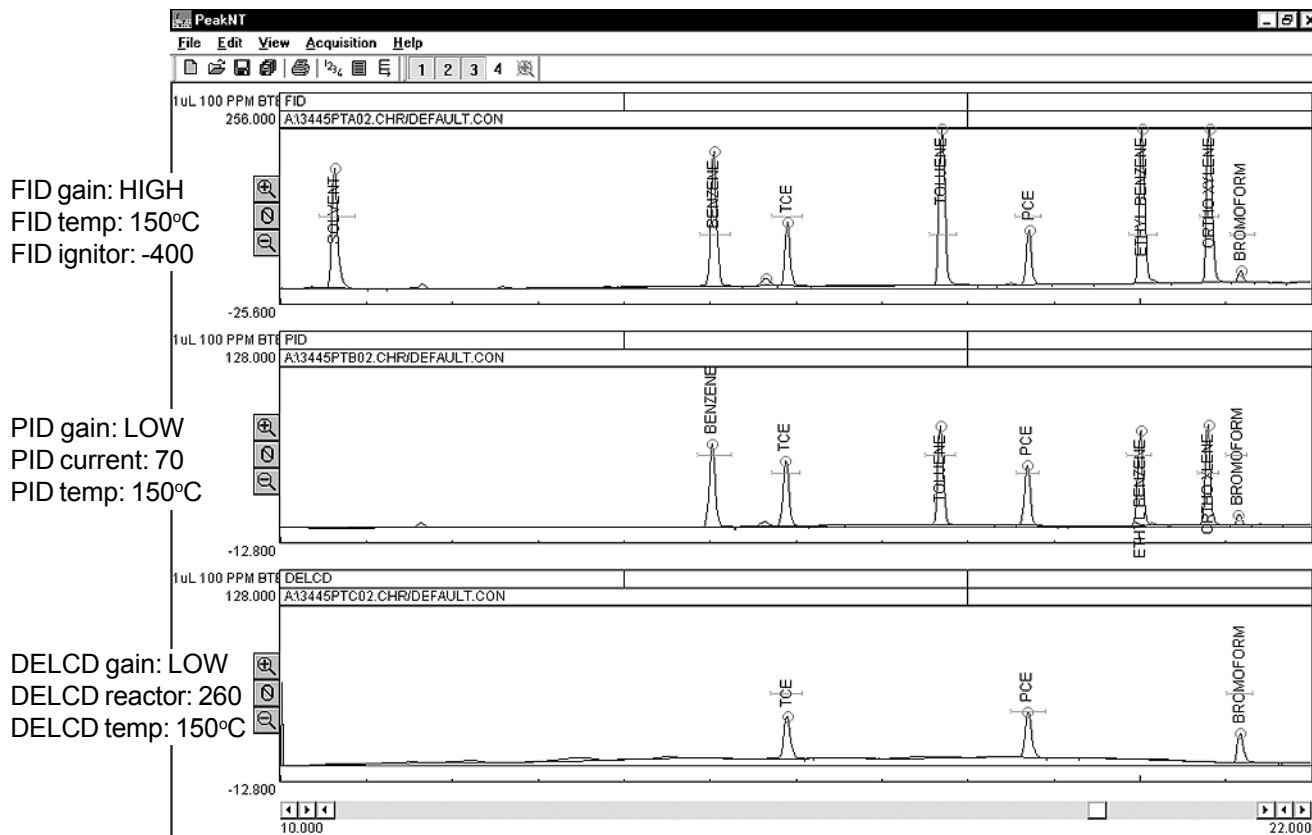
1. With the black plastic lamp hood in place on the PID lamp, turn ON the PID lamp current with the flip switch on the GC's front control panel. Set the PID current to 70 (= 0.70ma) by adjusting the appropriate trimpot setpoint on the top edge of the GC's front control panel. (Each detector zone is labeled on the front control panel under DETECTOR PARAMETERS, with the corresponding trimpot setpoint directly above it.) The lamp should emit a violet-colored light visible down the center of the tube. Set the PID temperature to 150°C. Set the PID gain to LOW.
2. Turn on the air compressor using the switch on the GC's front control panel. NOTE: since most ambient air will not cause interference with the DELCD, the built-in air compressor is appropriate for most analytical situations. However, if you are doing analyses in a lab environment with low levels of halogenated compounds in the ambient air, they 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.
3. Set the FID hydrogen flow to 25mL/min, and the FID air flow to 250mL/min. The pressure required for each flow is printed on the right hand side of the GC chassis. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. Ensure that the flame is lit by holding the shiny surface of a chromed wrench to the tip of the collector electrode; when the flame is lit, you should be able to see condensation on the wrench. Set the FID gain to HIGH. If the peaks are more than 20 seconds wide at the base, use the HIGH FILTERED gain setting. 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.
4. If a DELCD detector is installed, set the DELCD reactor temperature setpoint to 260 (=1000°C) by adjusting the appropriate trimpot. The DELCD will heat to around 254 and stabilize; the protruding end of the ceramic tube will glow bright red in the heat. Set the DELCD gain to LOW.
5. When the system has reached temperature and each detector is displaying a stable signal, begin the analysis by pressing the RUN button on the front of the GC or the spacebar on the computer keyboard.

POPULAR CONFIGURATION GCs

BTEX & Environmental

Expected Performance - Purge & Trap Concentrator

These chromatograms were produced from a 10ppb BTEX Plus standard analyzed in an Environmental GC equipped with a Method 5030 Purge & Trap injection system. The simultaneous display of all three detector channels illustrates their relative selectivity. The chromatogram on the next page shows the carry-over from the Purge & Trap concentrator on the subsequent analysis.



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

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

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

Time	Events
0.000	ZERO
0.100	E ON (PURGE GAS)
5.100	E OFF
6.000	C ON (TRAP 2 HEAT)
6.050	F ON (TRAP 1 HEAT)
8.000	G ON (VALVE INJECT)
12.000	E ON
12.900	B ON (BAKE)
13.000	G OFF (VALVE LOAD)
14.900	F OFF
15.100	C OFF
15.300	E OFF
15.500	B OFF

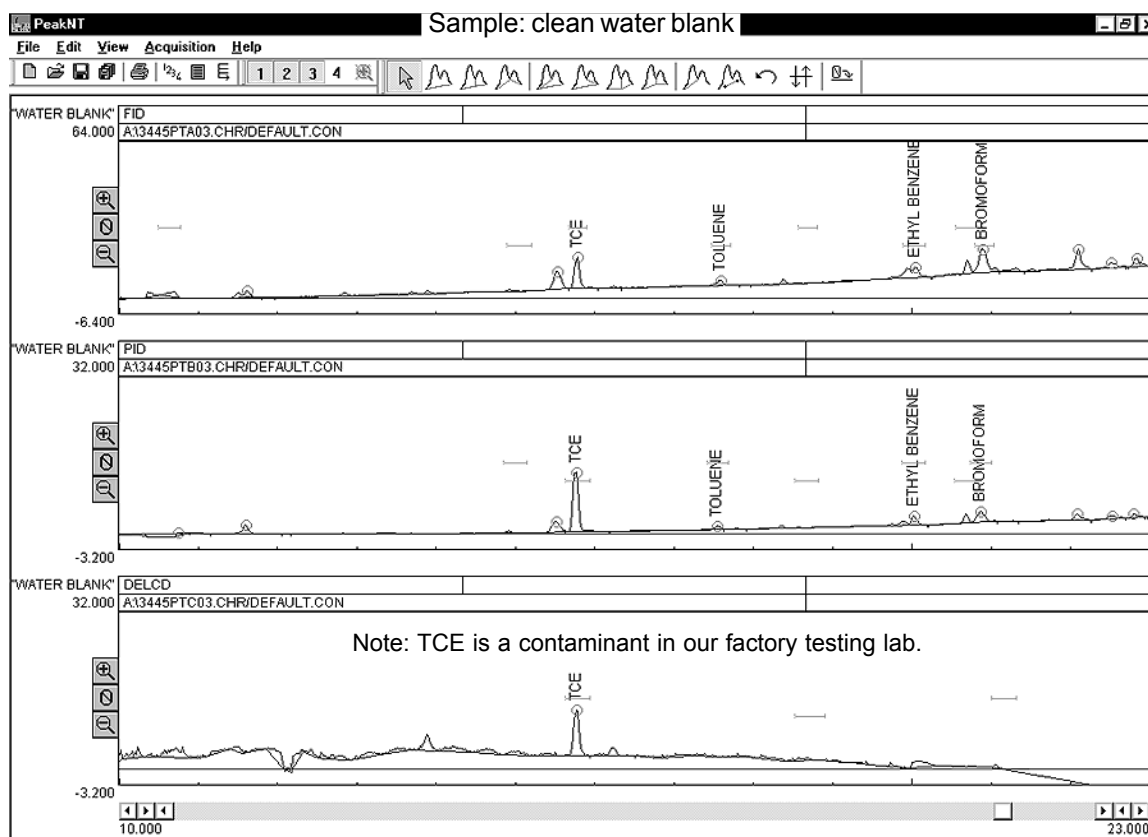
Sample: 1µL 100ppm BTEX Plus standard dissolved in 10mL of water to yield 10ppb of each analyte
 Method: 5030 P&T injection
 Column: 60m MXT-VOL
 Carrier: Helium @ 10mL/min

Temperature Program:
 (Etap&t.tem)
 Initial Hold Ramp Final
 40°C 10.00 10.00 180°C

POPULAR CONFIGURATION GCs BTEX & Environmental

Expected Performance - Purge & Trap Concentrator

This chromatogram was produced from analyzing a water blank immediately after the analysis of the BTEX Plus standard to show the Purge & Trap carry-over. The blank was run under the same conditions (event table, temperature program, detector settings) as the sample. Acceptable carry-over is a contamination level of 1% or 0.5ppb—whichever is lower—of an analyte (especially high boiling components), and is a normal condition of operation. This 1% of contamination from preceding analyses should not be significant enough to affect quantitation unless a very high concentration sample is followed by a very low concentration sample. It is standard laboratory practice to run a blank after a high concentration sample. Toluene is used as a representative of the carryover in the Purge & Trap system; if the carryover level of Toluene is below 1% or 0.5ppb on the PID chromatogram, then it will not affect subsequent analyses. (Note: the chromatograms are magnified for carryover visibility).



FID Results:

Component	Retention	Area
TCE	15.766	58.9100
Toluene	17.566	17.4000
Ethyl Benzene	20.033	51.9080
Ortho Xylene	20.833	91.5290
Total		219.7470

PID Results:

Component	Retention	Area
TCE	15.750	58.1920
Toluene	17.533	4.3400
Ortho Xylene	20.850	20.8720
Total		609.1300

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340

Determine the carryover level by comparing the areas of the two PID Toluene peaks resulting from the sample and blank runs:

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

$$353x = 40\text{ppb}$$

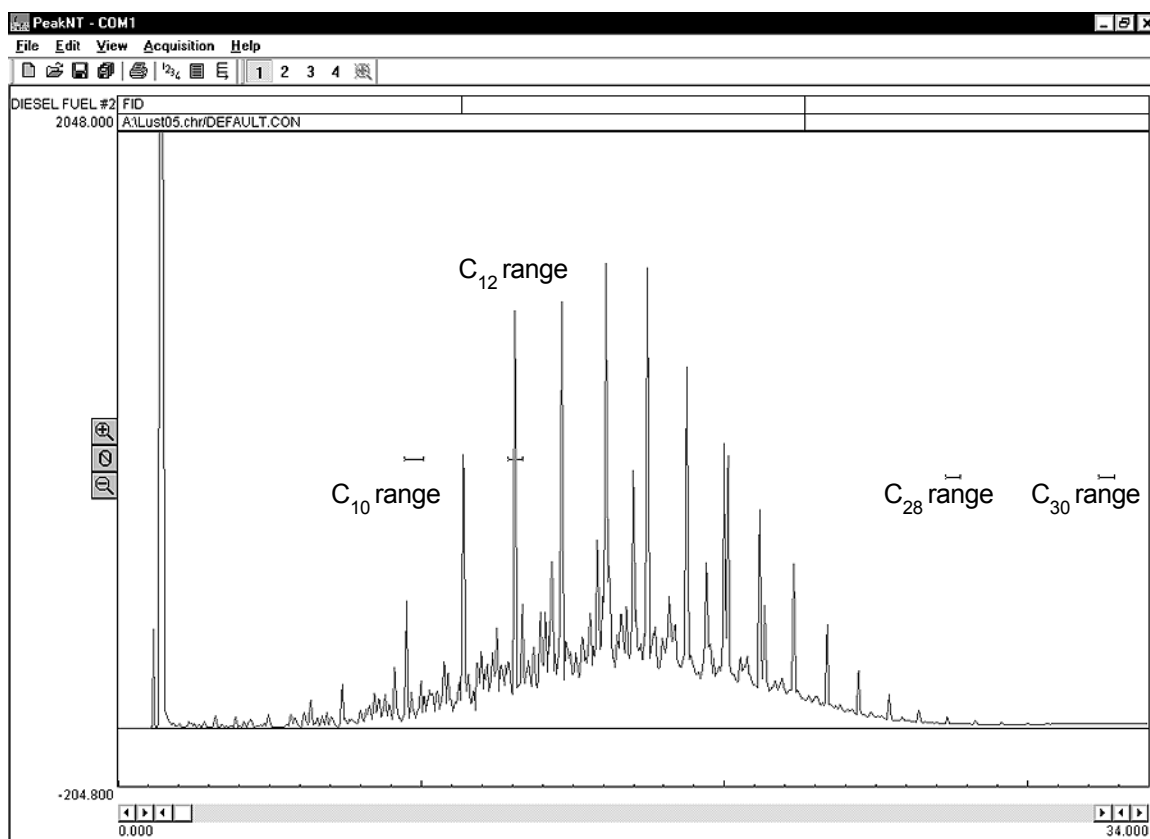
$$x = 0.1133\text{ppb}$$

(x represents the ppb concentration of the carryover)

POPULAR CONFIGURATION GCs BTEX & Environmental

Expected Performance - Direct Injection

This chromatogram is from an analysis of a diesel sample. The PID detector was bypassed, and the column was connected directly to the FID detector inlet. The results are identifiable as diesel because it shows the range of hydrocarbons that compose this fuel. A few retention windows are placed in the chromatogram to show the approximate ranges of C_{10} , C_{12} , C_{28} , and C_{30} .



Sample: diesel fuel #2
Method: direct injection
Column: 60m MXT-VOL
Carrier: helium @ 10mL/min

FID gain: HIGH
FID temp: 325°C
FID ignitor: -400

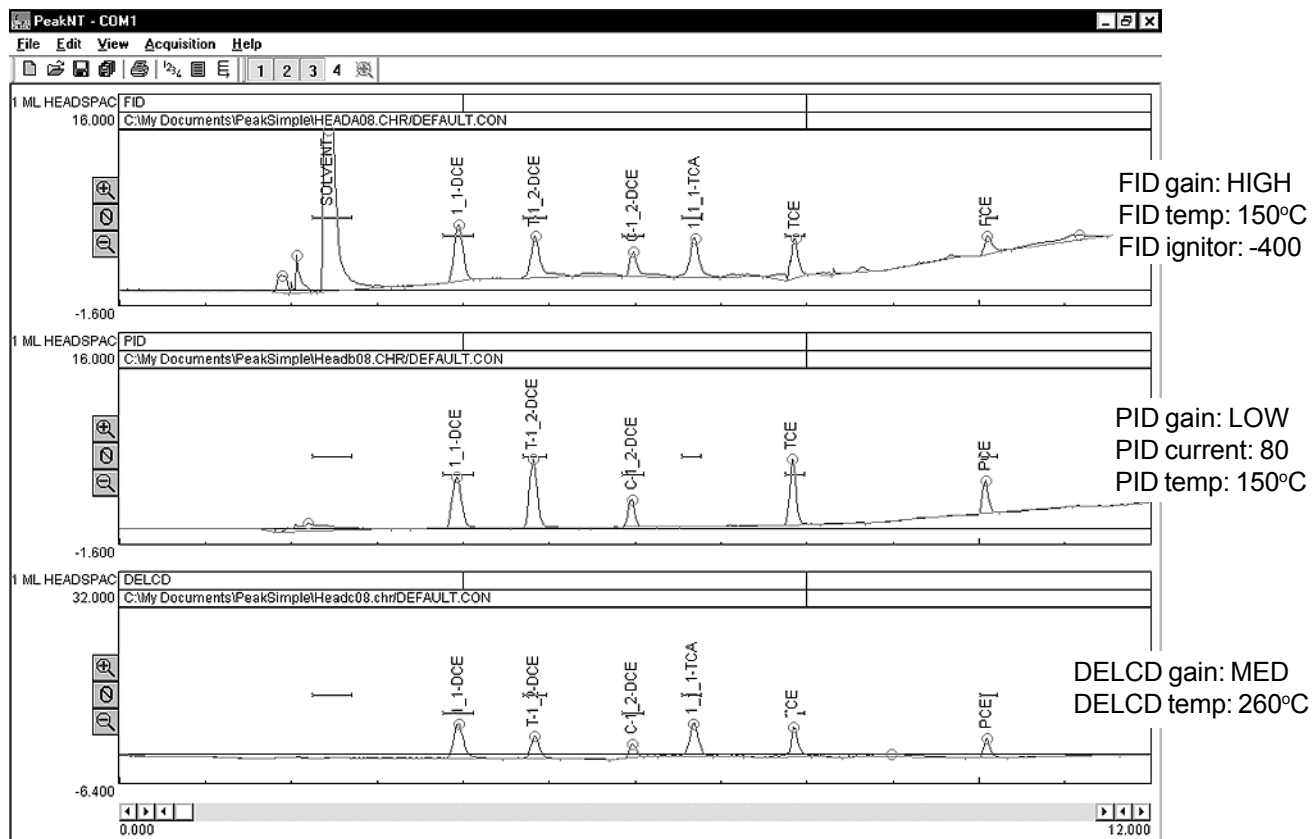
Temperature program:

Initial	Hold	Ramp	Final
50°C	3.000	10.000	320°C
320°C	30.00	0.000	320°C

POPULAR CONFIGURATION GCs BTEX & Environmental

Expected Performance - Manual Headspace Injection

To obtain the chromatograms below, 50ppb Japanese standard was placed into a VOA vial with water, and allowed to equilibrate at room temperature for 45 minutes. The FID (top) chromatogram shows all the components and the solvent. The PID (middle) does not detect the 1_1_1-TCA, while the DELCD (bottom) does not respond to the solvent.



Sample: 1mL headspace from 50ppb Japanese standard in water
Method: manual headspace injection
Column: 60m MXT-VOL
Carrier: helium @ 10mL/min

Temperature program:
Initial Hold Ramp Final
40°C 2.000 15.000 220°C
220°C 10.00 0.000 220°C

FID Results:

Component	Retention	Area
Solvent	2.416	290.1100
1_1-DCE	3.933	39.6100
T-1_2-DCE	4.833	34.3780
C-1_2-DCE	5.966	18.6020
1_1_1-TCA	6.683	29.6320
TCE	7.850	23.4490
PCE	10.083	10.7560
Total		446.5370

PID Results:

Component	Retention	Area
Solvent	2.183	22.7450
1_1-DCE	3.916	39.4070
T-1_2-DCE	4.800	45.0050
C-1_2-DCE	5.950	15.7380
TCE	7.816	33.7270
PCE	10.066	16.2780
Total		172.9000

DELCD Results:

Component	Retention	Area
1_1-DCE	3.933	63.1790
T-1_2-DCE	4.816	38.0780
C-1_2-DCE	5.950	18.0560
1_1_1-TCA	6.666	53.2210
TCE	7.833	39.6900
PCE	10.083	20.8340
Total		233.0580

Chapter: Injectors

Topic: EPC (electronic pressure control) operation

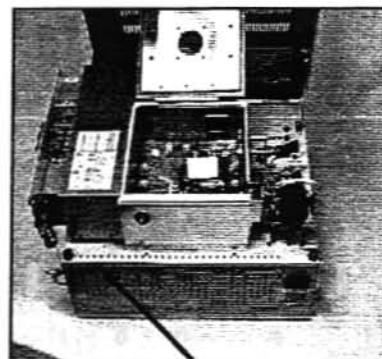
SRI GCs are equipped with electronic pressure control of all system gases. Detector support gases such as hydrogen and air are controlled by the screwdriver adjustable local setpoint on the GC, and once set are seldom altered. The carrier gas pressure may be controlled by either the local setpoint screwdriver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

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

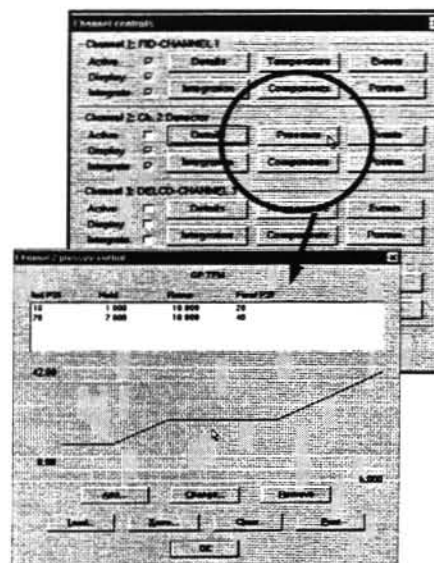
- 1) The screwdriver adjustment is simpler, and once set is not likely to be altered unintentionally.
- 2) The benefits of ramping the carrier gas pressure are often not worth the extra operational complexity.

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

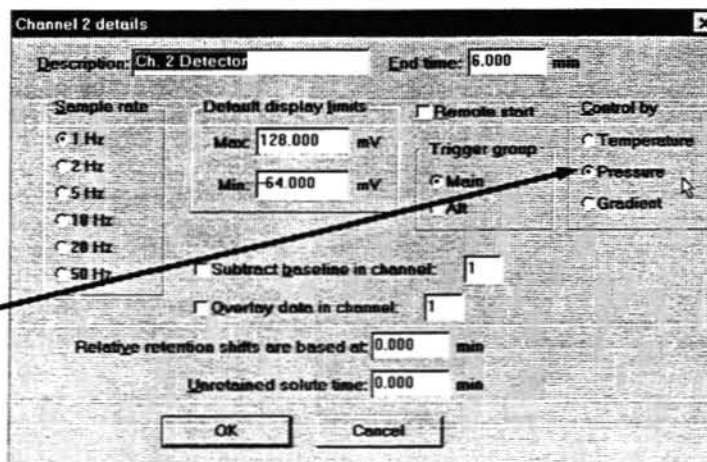
Once you make the changes, don't forget to save your control file (default.con) so PeakSimple will remember your changes the next time you boot up.



Local setpoint adjustments for temperatures and pressures using small screwdriver



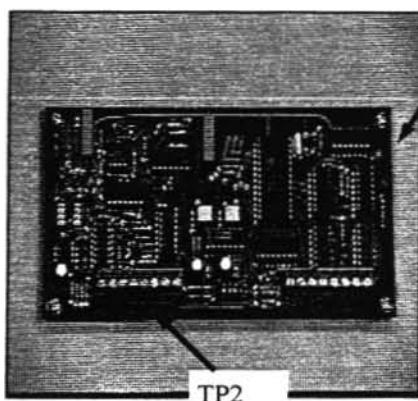
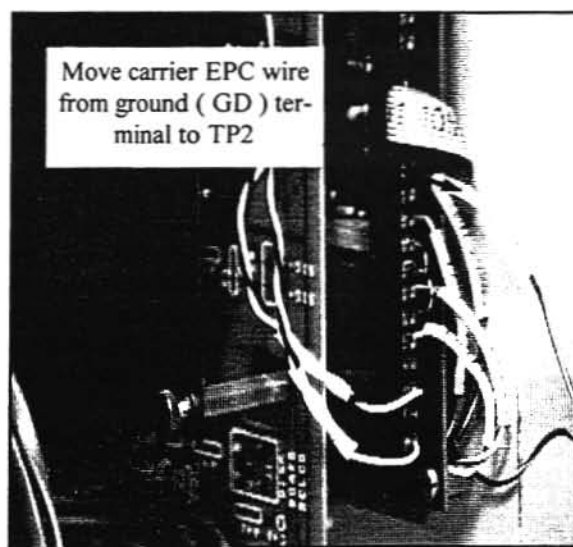
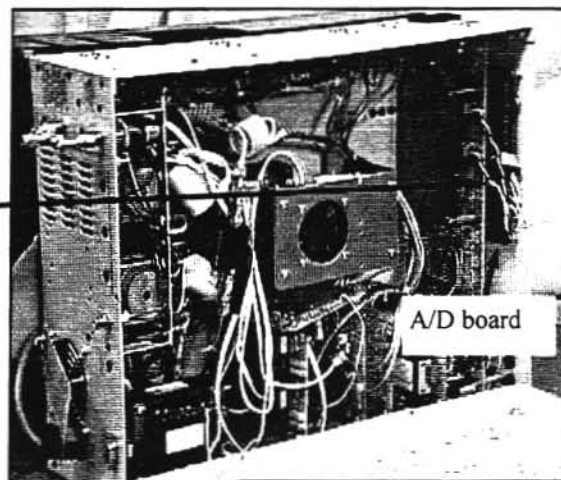
Set the "Control By" radio button in the Channel 2 Details screen to Pressure. Then enter the desired pressure program into Channel 2 by selecting the Pressure screen from the Edit/Channel menu



Chapter: Injectors

Topic: Enabling the carrier gas EPC

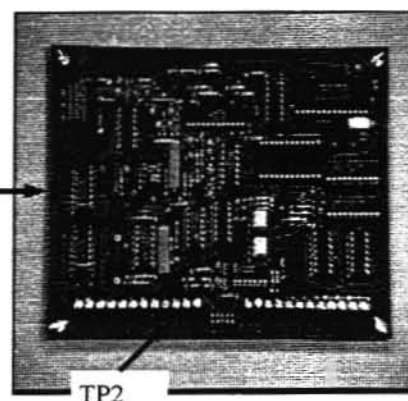
- 1) Un-plug the GC power cord.
- 2) Remove the six screw holding the bottom cover to the GC chassis.
- 3) Tilt the GC on its back and expose the interior.
- 4) Locate the A/D board which is mounted on the right hand interior wall.
- 5) Locate the carrier gas EPC wire (green with white stripe and labelled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



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

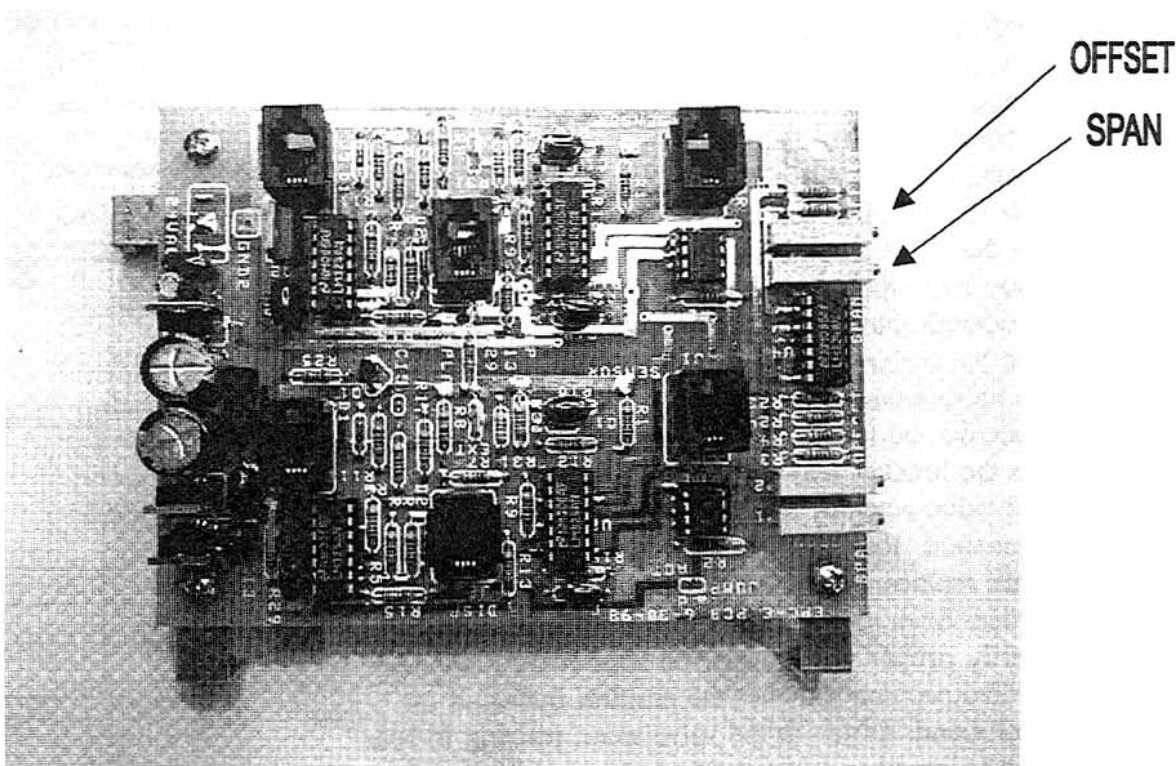
Other GCs will be equipped with the 4 channel Model 202 A/D board.

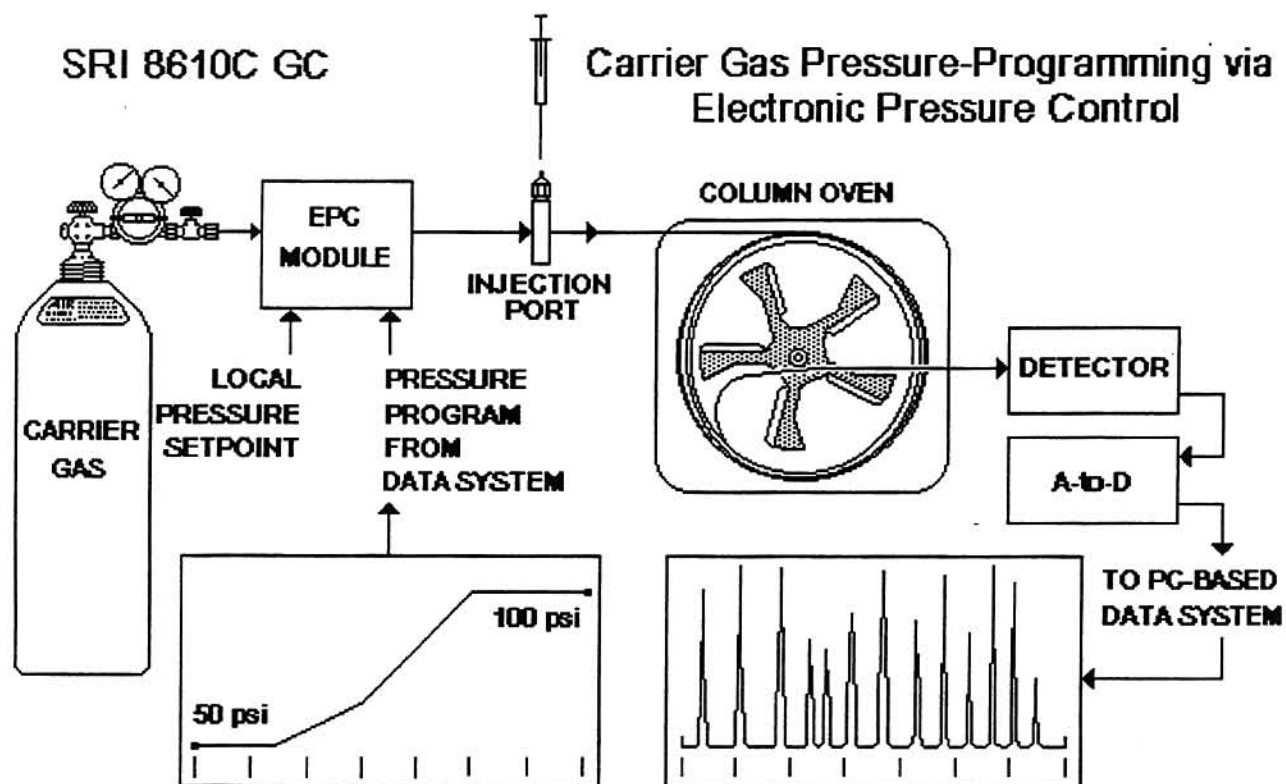
The procedure is identical on either board.



EPC Calibration:

1. With the gas off connect a 0 – 30 psi gauge to the union on the output of the EPC. (Inside the instrument, the EPC's are on the left)
2. Adjust the EPC SET POINT on the top of the front panel to 20 psi. Verify the SET POINT is at 20 psi with the pushbuttons.
3. With the supply gas off, Zero the display to a setting of -0.0 using the OFFSET POT adjustment on the EPC board shown below.
4. With a supply pressure of 30 psi turn on the supply gas.
5. Adjust the SPAN POT on the EPC board to make the gauge pressure equal to the SET POINT pressure.
6. Repeat steps 3-5, until the ZERO is -0.0 and the gauge and display both read 20 psi.





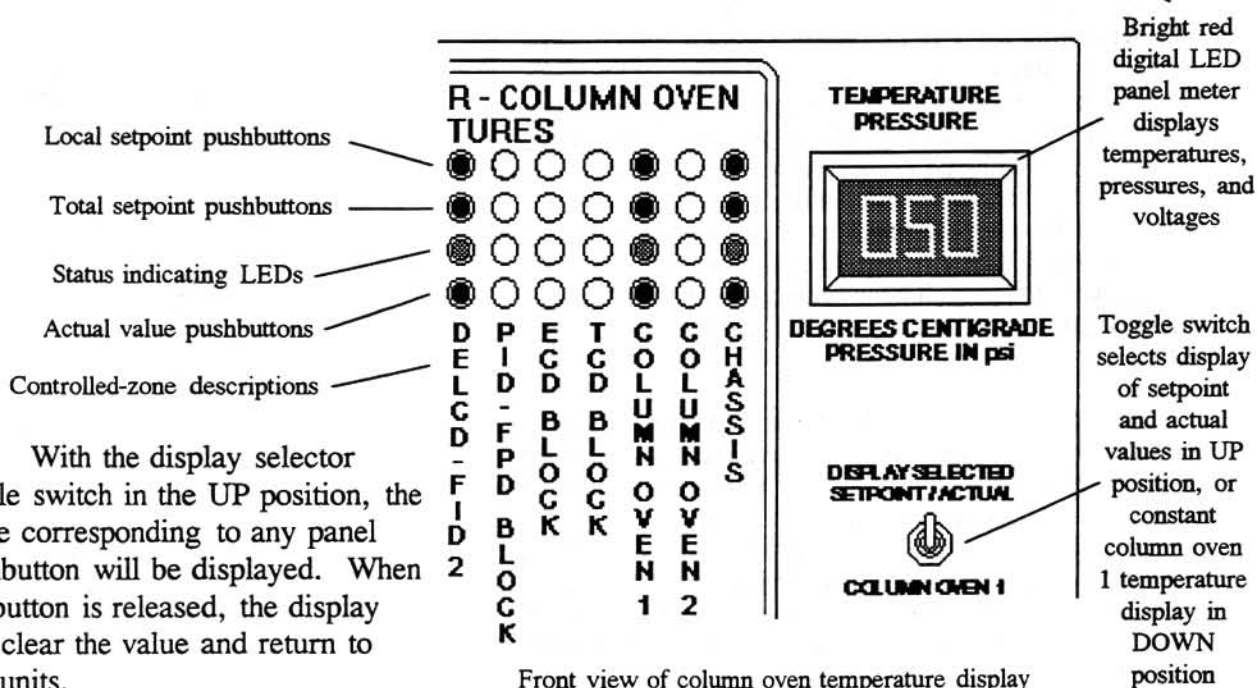
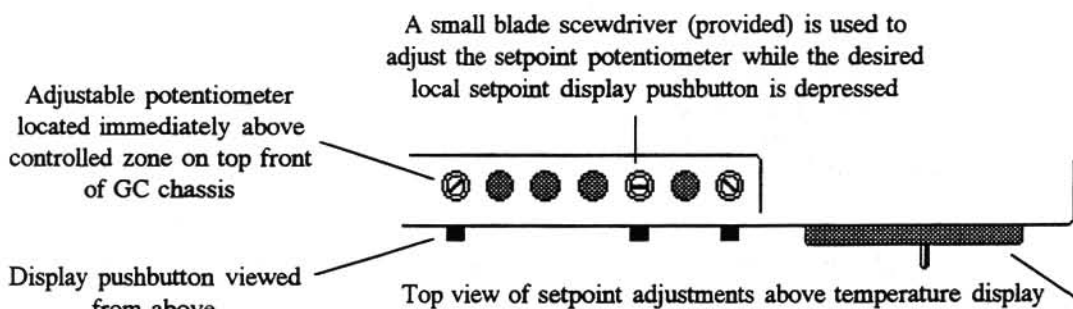
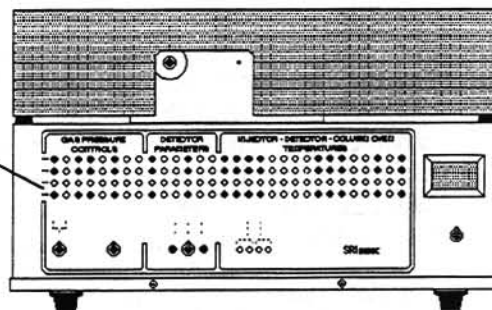
FLOW CHART ILLUSTRATING CARRIER GAS PRESSURE PROGRAMMING ON THE 8610C GC

All SRI 8610C gas chromatographs are equipped with Electronic (or Pneumatic) Pressure Control (EPC) of all system gases. Each gas, from the carrier gas, to the specific detector gases, such as FID hydrogen and FID compressed air, in the case of an FID detector, are controlled by a dedicated solid-state EPC module that electronically monitors and instantaneously adjusts the pressure being supplied to the particular feature. This electronic control facilitates extreme precision of gas flows to the various functions. Each EPC module features a local, user-adjustable setpoint accessed by a trimpot (variable potentiometer) located just above the particular function on the "at-a-glance" panel display. The carrier gas is among these adjustable setpoints. The term "local" refers to the fact that the "local" setpoint is set manually at the trimpot on the GC chassis. As in the case of the column oven temperature setpoint, the carrier gas pressure setpoint may be set "locally" (manually on the GC chassis), or from the computer via a pressure program. Created in the same format as a PeakSimple temperature program, the program signal is sent to the data system interface and converted to a control voltage that can increase, maintain, or decrease the carrier gas pressure automatically at the user's command.

The PeakSimple serial data system interface offers two rampable voltage outputs - one to program the column oven, and the other to program carrier gas pressure. Outputting a 0 to 5VDC variable signal, the EPC module will permit an output pressure of from 0 to 100psi (the carrier pressure shown is actually the column head pressure). Please note that any local setpoint value will be summed to this signal, resulting in the "total" setpoint value on the panel display. The carrier gas pressure regulator at the gas cylinder should be set 10psi higher than the highest programmed carrier gas head pressure desired for proper control. Ramping permits the head pressure to be varied, to speed or slow the elution of peaks from the analytical column as needed by the application or user.

The 8610C gas chromatograph permits easy display and adjustment of all controlled zone setpoints. To view a controlled zone, simply place the display selector switch in the UP position, and depress the desired feature pushbutton. Depending on the zone, the following values may be displayed: the actual value that the zone is being measured at, such as the current temperature of column oven 1; the local setpoint, which reflects the adjustable setpoint you currently have set, which, in the case of column oven 1, would be an offset value that could be summed with the temperature signal being sent from the data system; and the total setpoint, which is the sum of any signal being sent from the data system to the controlled zone, in addition to any local setpoint value you have set (for example, if column oven 1 has a local setpoint of 50 degrees, and the data system is instructing the GC to heat the column oven to 100 degrees, the total setpoint should display 150 degrees). Most zones will only display the local setpoint and actual value. Each zone also displays its status via a light-emitting diode (LED) that glows when the zone is active.

"At-a-glance" display panel also permits viewing of actual and setpoint values



With the display selector toggle switch in the UP position, the value corresponding to any panel pushbutton will be displayed. When the button is released, the display will clear the value and return to 000 units.

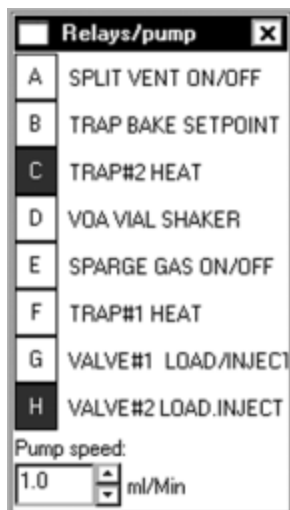
Events

PeakSimple gives you control of up to eight independent external events, or hardware events. A hardware event is the operation of a device external to the data system but integral to the analytical run. A hardware event can be a valve rotation, opening or closing a split vent, activation of an autosampler sequence, or operation of an electrical switch at a precise moment during the run. PeakSimple also gives you control over a comprehensive list of non-hardware events, such as integration events, data system signal control events (zero, reverse), and DOS command events. Events are controlled automatically with **Event tables** that use the system clock, which starts at 0:00 with each run. Using PeakSimple **Event tables** enhances the reproducibility of the resulting chromatograms by ensuring repeatable actuation of devices from run to run.

The eight timed event output signals are called relays, and are named A-H. For example, when an SRI GC is equipped with a 10-port gas sampling valve, its rotation/actuation is controlled by a relay: relay OFF = valve in the LOAD position; relay ON = valve in the INJECT position. The relay assignments for any given instrument are printed on the side panel (right hand side for GCs, left-hand side for HPLCs).

RELAY FUNCTIONS		
(DEFAULT / ACTIVE)		
A	SAMPLE SOLENOID #1	(CLOSED / OPEN)
B	SAMPLE SOLENOID #2	(CLOSED / OPEN)
C	SAMPLE SOLENOID #3	(CLOSED / OPEN)
D	SAMPLE SOLENOID #4	(CLOSED / OPEN)
E	TRAP #1 HEAT	(OFF / ON)
F	TRAP #2 HEAT	(OFF / ON)
G	VACUUM PUMP	(OFF / ON)
H	VALVE #1 POSITION	(LOAD / INJECT)

This list of assigned relay functions is printed on the side of a TO-14 GC customized with 4 sample solenoid valves.

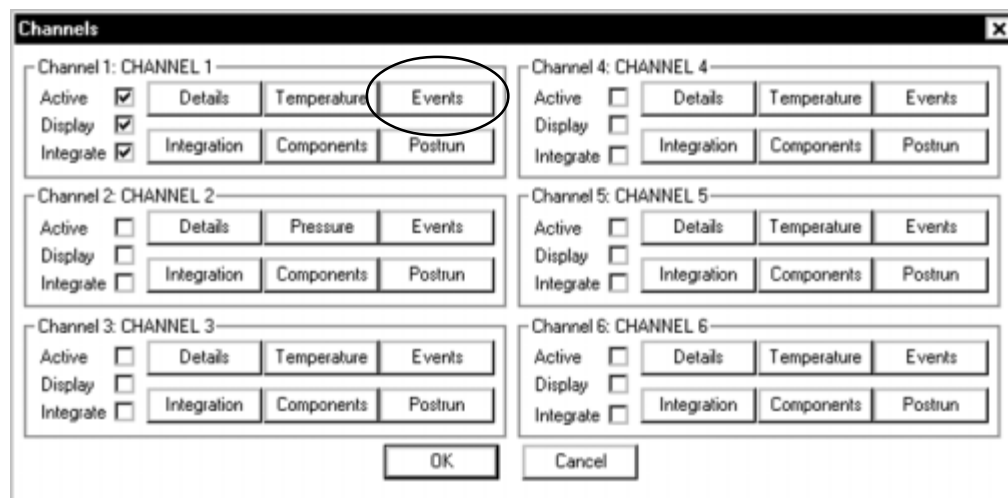


Users may manually control any relay event, either during the run or while in stand-by mode, by using the **Relays/pump** window. Click on View and choose **Relay/pump window**. In this window are eight buttons representing the relays with the appropriate letter. Activate a relay by clicking on its letter; it becomes highlighted to show its ON status. You can also toggle the relays from the keyboard by holding the control key (Ctrl) while pressing the letter of the relay: Ctrl+C, Ctrl+H, etc.

PEAKSIMPLE SOFTWARE

Events

Event Tables



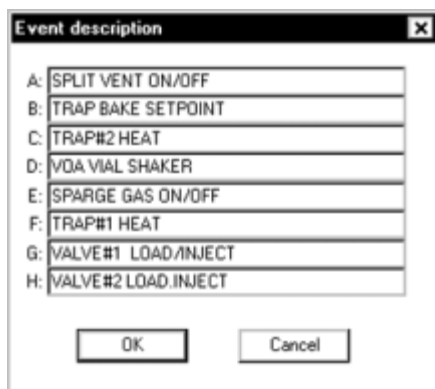
You can open the events table window from the Edit> Channels screen. Click on the Events button for channel 1. Or, right-click in the chromatogram window and select Events from the pop-up menu.



The event table window will open, either empty, or with whatever .EVT file was saved with the current control file (DEFAULT.CON, unless you have specifically opened another). At the bottom of the events table window there are several buttons for you to access event features.

Each channel has its own event table because of the signal processing type events that are available, such as Zero, Reverse, and Integration events. Hardware events may also be activated from any channel. SRI recommends entering hardware events only in the channel 1 event table to avoid confusion.

Click on the **Add...** button to add an event to the event table. The **Event details** screen will open, where you select the event and enter the time at which you want it to occur.



Type in your custom event descriptions.

Click on an existing event in the event table to select and highlight it, then click on the **Change...** button to edit the selected event. You can also simply double-click an event to open the **Event details** screen, from which you can edit any selected event.

Click on the **Remove** button to delete a selected event from the events table.

Click on the **Describe...** button to customize any or all of your eight relay (hardware) event descriptions.

PEAKSIMPLE SOFTWARE

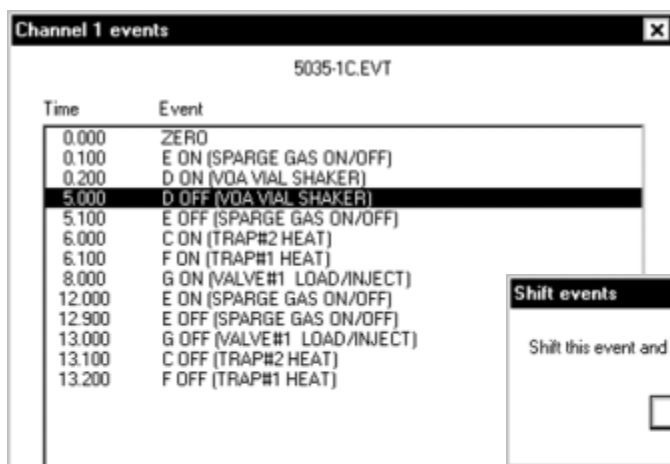
Events

Click the **Load...** button to open an existing event table. PeakSimple will open the program directory and display all .EVT files.

Click **Save...** to save the current event table shown in the window.

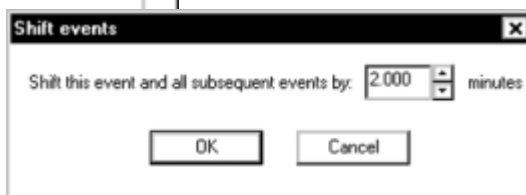
Click **Clear** to remove any and all events and .EVT files from the event table window (but not from the hard drive). PeakSimple will prompt you for confirmation before proceeding to clear the event table.

Click the **Print** button to send the current event table to the printer through the Windows print manager.



Time	Event
0.000	ZERO
0.100	E ON (SPARGE GAS ON/OFF)
0.200	D ON (VDA VIAL SHAKER)
5.000	D OFF (VDA VIAL SHAKER)
5.100	E OFF (SPARGE GAS ON/OFF)
6.000	C ON (TRAP#2 HEAT)
6.100	F ON (TRAP#1 HEAT)
8.000	G ON (VALVE#1 LOAD/INJECT)
12.000	E ON (SPARGE GAS ON/OFF)
12.900	E OFF (SPARGE GAS ON/OFF)
13.000	G OFF (VALVE#1 LOAD/INJECT)
13.100	C OFF (TRAP#2 HEAT)
13.200	F OFF (TRAP#1 HEAT)

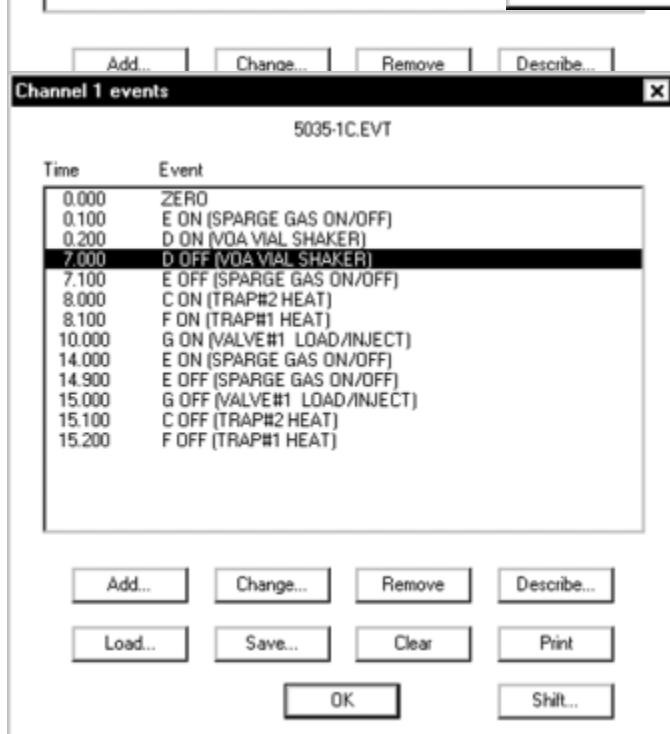
Click the **Shift...** button to shift the sequence of events following the selected event forward or backward in time.



Shift this event and all subsequent events by: 2.000 minutes

OK Cancel

In this example, we are shifting the highlighted event forward 2 minutes.



Time	Event
0.000	ZERO
0.100	E ON (SPARGE GAS ON/OFF)
0.200	D ON (VDA VIAL SHAKER)
7.000	D OFF (VDA VIAL SHAKER)
7.100	E OFF (SPARGE GAS ON/OFF)
8.000	C ON (TRAP#2 HEAT)
8.100	F ON (TRAP#1 HEAT)
10.000	G ON (VALVE#1 LOAD/INJECT)
14.000	E ON (SPARGE GAS ON/OFF)
14.900	E OFF (SPARGE GAS ON/OFF)
15.000	G OFF (VALVE#1 LOAD/INJECT)
15.100	C OFF (TRAP#2 HEAT)
15.200	F OFF (TRAP#1 HEAT)

Buttons: Add... Change... Remove Describe... Load... Save... Clear Print OK Shift...

Now the selected event takes place at 7.00 instead of 5.000; all subsequent events have also been shifted forward two minutes.

The **Shift...** button saves you having to re-type the entire event sequence.

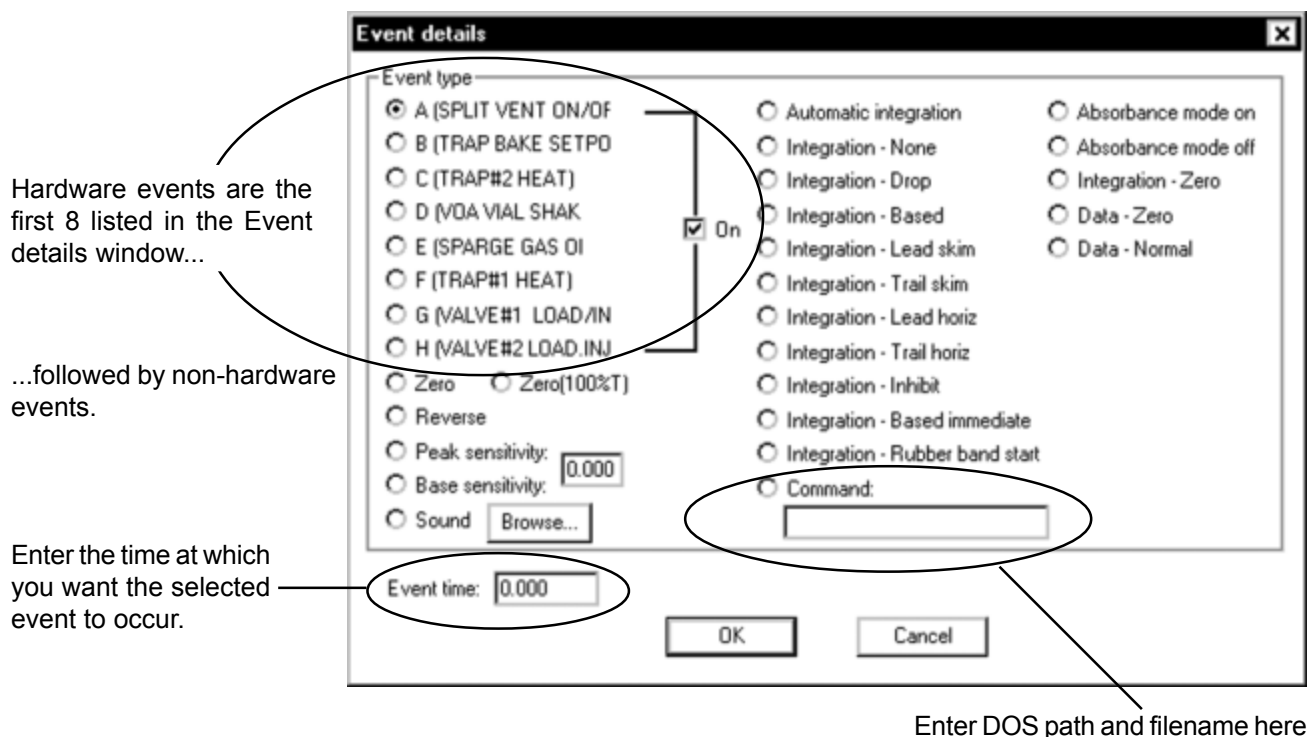
Click the **OK** button to exit the events table window.

PEAKSIMPLE SOFTWARE

Events

The **Event details** screen opens when you click the **Add...** or **Change...** buttons, or when you double-click any single event in the list. In the **Event details** screen are listed all the events you can enter into an event table. Starting on the left, the eight relays are listed, followed by non-hardware events. Hold your mouse cursor over any event to read its ToolTips description.

There is a checkbox labeled “On” to the right of the hardware events list. When the event is turning ON a relay, make sure this box is checked. Uncheck the box when the event is turning OFF a relay. Even though they control the same relay, ON and OFF are separate and distinct events in the timetable. On the bottom left of the Event details window is the Event time field. Enter the time at which you want the event to occur here. Typically, PeakSimple has a lot of tasks to perform at the beginning of an analytical run. Therefore, SRI recommends that you enter the first hardware event no earlier than 0.100 minutes (6 seconds).



PeakSimple also permits you to automatically execute a DOS command during the analytical run using an event table. A DOS command is the same as running an executable file. You may use this function to launch a macro to copy, rename the preceding file so the next file may be updated into a spreadsheet, or to copy the chromatogram data and results file onto a floppy or hard disk drive other than the destination to which it was originally saved. A DOS command may be executed at any time during the analytical run by typing in the DOS path and the filename of an .EXE, .COM, or .BAT file, and the time the event is to occur. To add a DOS command event: click the radio button next to “Command,” and type in your DOS path and executable filename. Example: C:\Excel\Macro1.bat Next, type in the time during the run at which you want the command to be activated in the form field labeled **Event time**. DOS events that require prolonged disk access should be executed after the run, using **Post-run actions** for channel 1.

You can choose to reset all eight relays at the end of the run by clicking Edit>Overall and checking the box in the lower left corner.

This option will return the relays to their default position—OFF.

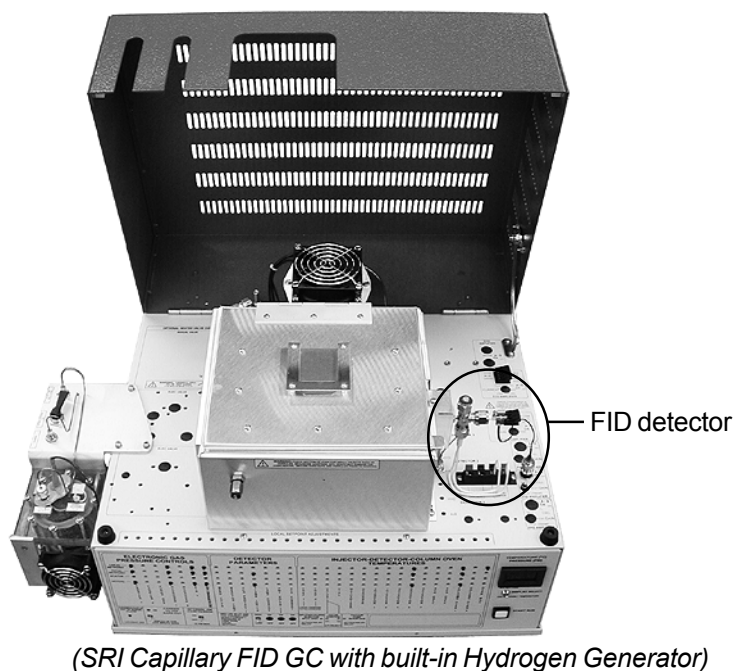
In some cases, a user may not want to reset the relays at the end of the run. For instance, when using our TO-14 Air Concentrator, users leave the gas sampling valve in the INJECT position at the run's end. This sweeps clean the trap and column, preparing the system for the next sample. In this case, the user would have an event table to turn OFF the valve relay and return it to the LOAD position sometime after the run has started. Therefore, such users would deselect the "Reset relays at end of run" option.

DETECTORS

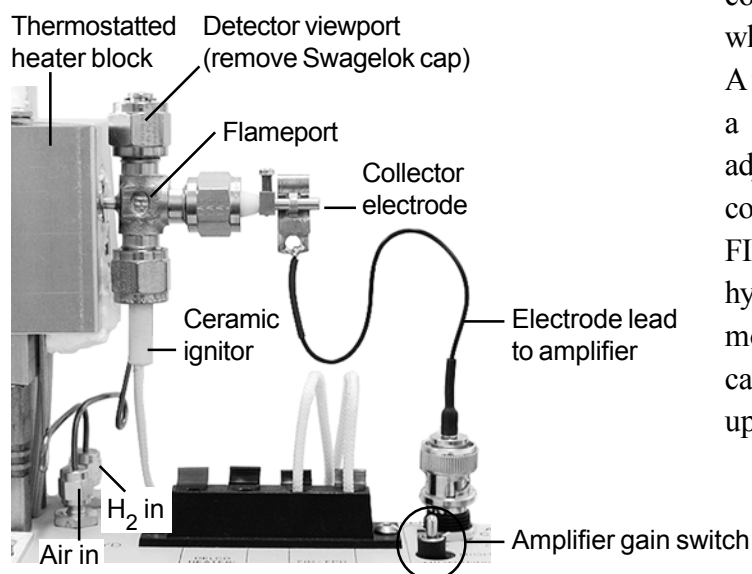
Flame Ionization Detector - FID

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 0.1 ppm to almost 100%. The FID's response is stable from day to day, and is not susceptible to contamination from dirty samples or column bleed. It is generally robust and easy to operate, but because it uses a hydrogen diffusion flame to ionize compounds for analysis, it destroys the sample in the process.



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



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

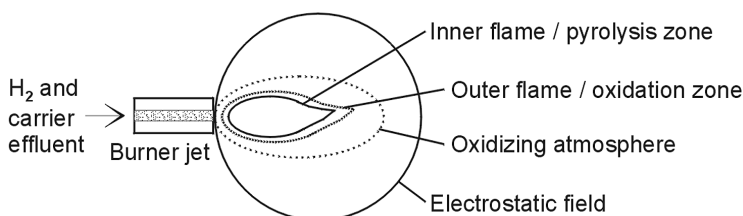
DETECTORS

FID - Flame Ionization Detector

Theory of Operation

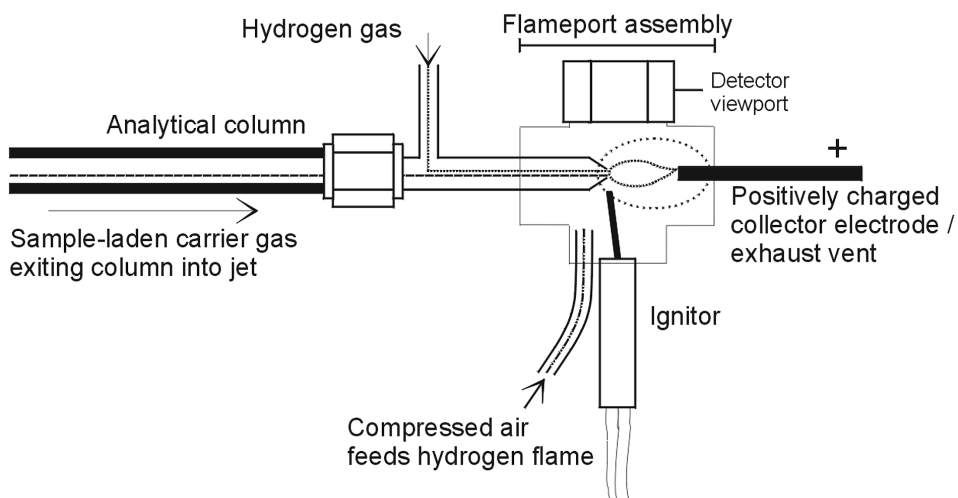
In the SRI FID, the carrier gas effluent from the GC column is mixed with hydrogen, then routed through an unbreakable stainless steel jet. The hydrogen mix supports a diffusion flame at the jet's tip which ionizes the analyte molecules. Positive and negative ions are produced as each sample component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

The FID hydrogen diffusion flame



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

FID detector schematic



DETECTORS

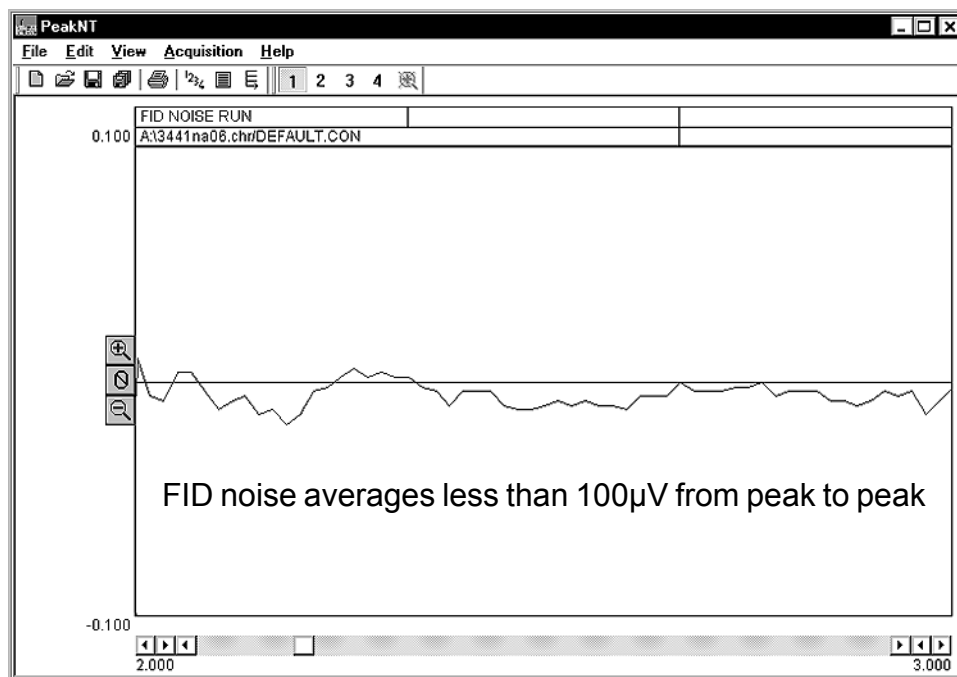
Flame Ionization Detector - FID

Expected Performance

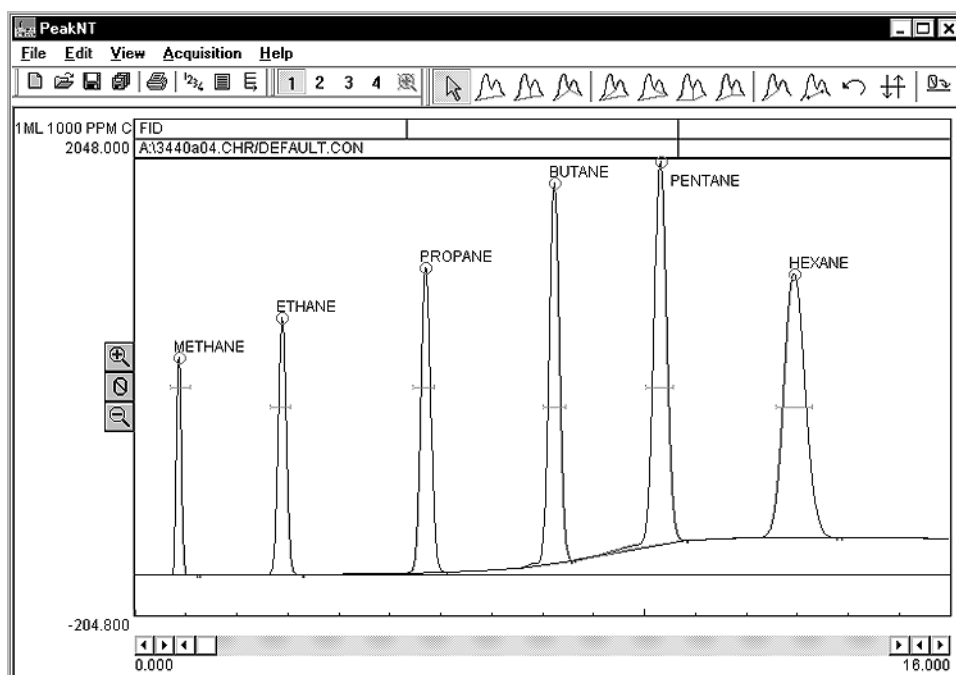
FID noise run

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



C₁-C₆ Hydrocarbon Test Analysis



Sample: 1mL of 1000ppm C₁-C₆
 Carrier: Helium @ 10mL/min
 FID H₂ 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

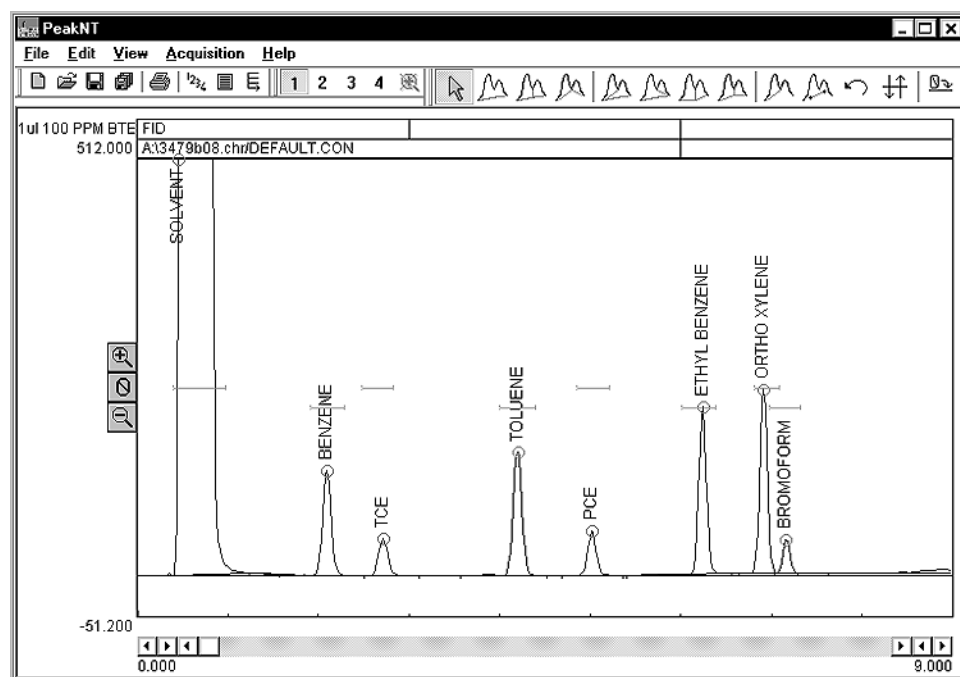
DETECTORS

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 SRI GC.



1 µL 100ppm BTEX sample

15m MXT-VOL capillary column

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

Results:

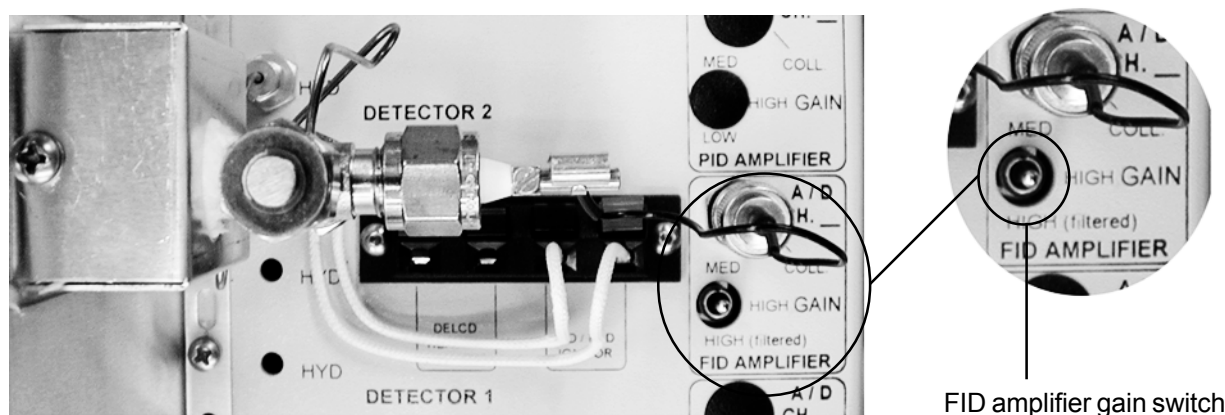
Component	Retention	Area
Solvent	0.433	95879.7560
Benzene	2.083	837.1000
TCE	2.700	319.2450
Toluene	4.183	1070.1060
PCE	5.000	344.8640
Ethyl Benzene	6.233	1200.3320
Ortho Xylene	6.900	1312.3070
Bromoform	7.150	225.2360
total		101188.9460

DETECTORS

FID - Flame Ionization Detector

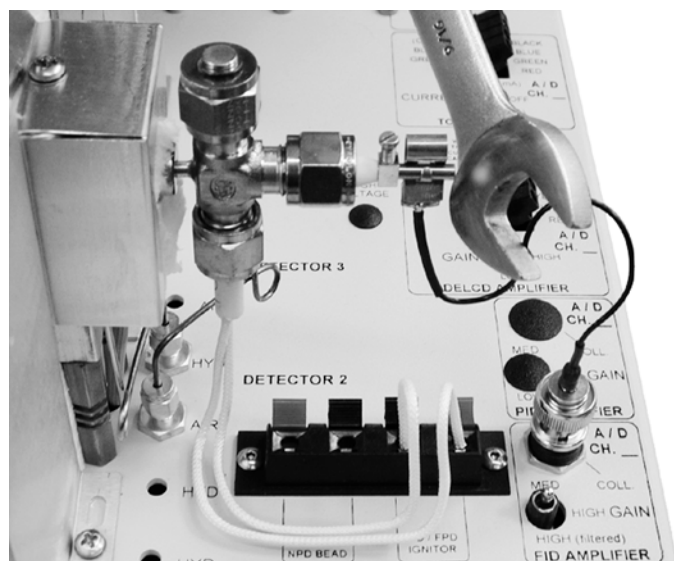
General Operating Procedure

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



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

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

DETECTORS

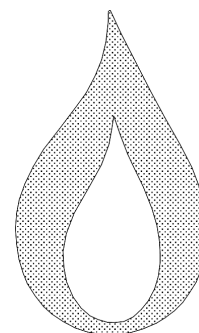
Flame Ionization Detector - FID

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.

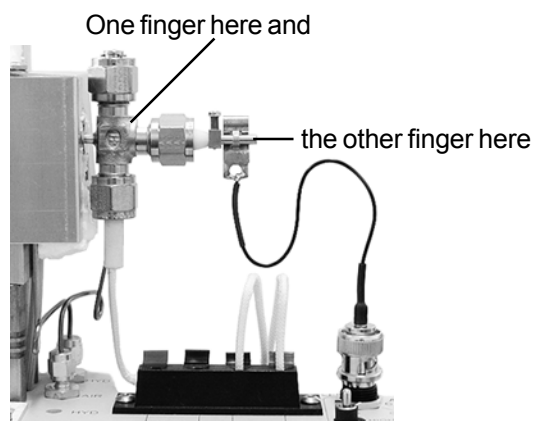
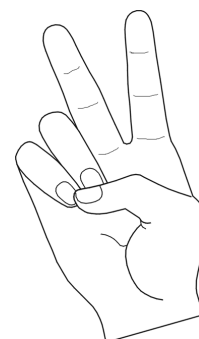
A. Flame ON/OFF Test

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.

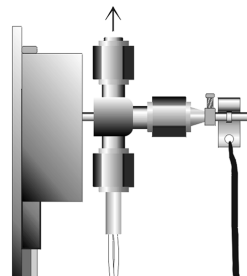
DETECTORS

FID - Flame Ionization Detector

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.

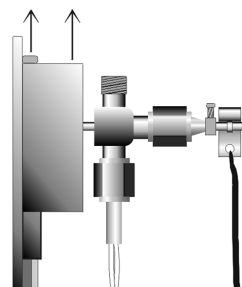
1. Unscrew the viewport cap nut and examine the flameport interior for coatings or films. If residue is found, the collector electrode and the flameport will need cleaning.



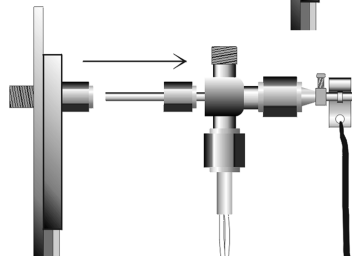
2. Remove flameport assembly from the heater block

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.

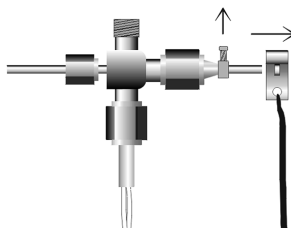


c. Gently pull off the white insulation to reveal the detector's bulkhead fitting on the column oven wall. Loosen this fitting to disconnect the flameport.

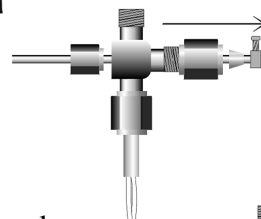


3. Remove the collector electrode

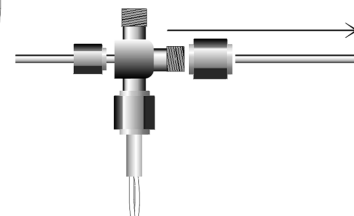
a. Unclip the electrode lead terminal and slide it off the electrode.



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.



DETECTORS

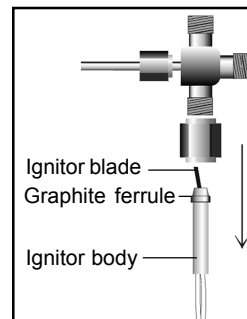
Flame Ionization Detector - FID

Cleaning the FID continued

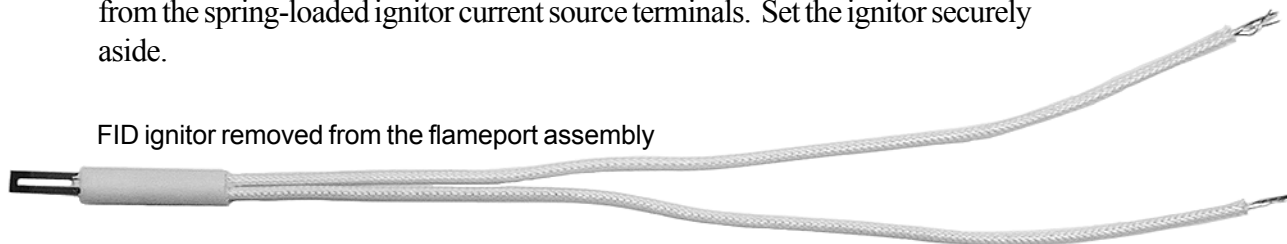
4. Remove the FID ignitor element

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

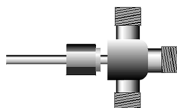
b. Carefully pull the ignitor down out of the flameport. Disconnect the ignitor from the spring-loaded ignitor current source terminals. Set the ignitor securely aside.



FID ignitor removed from the flameport assembly



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



Scrape, rinse, and bake out the FID flameport interior

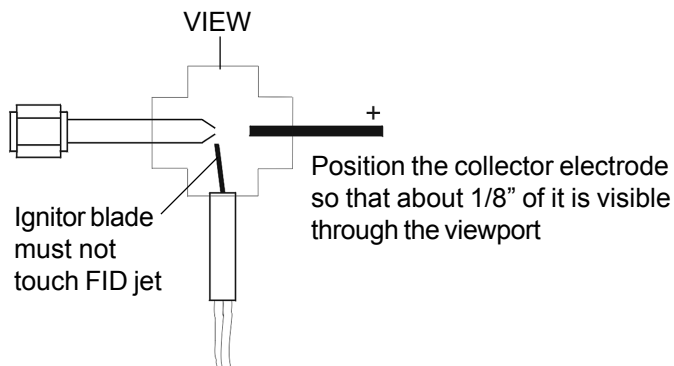
6. Re-assembly

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

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



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

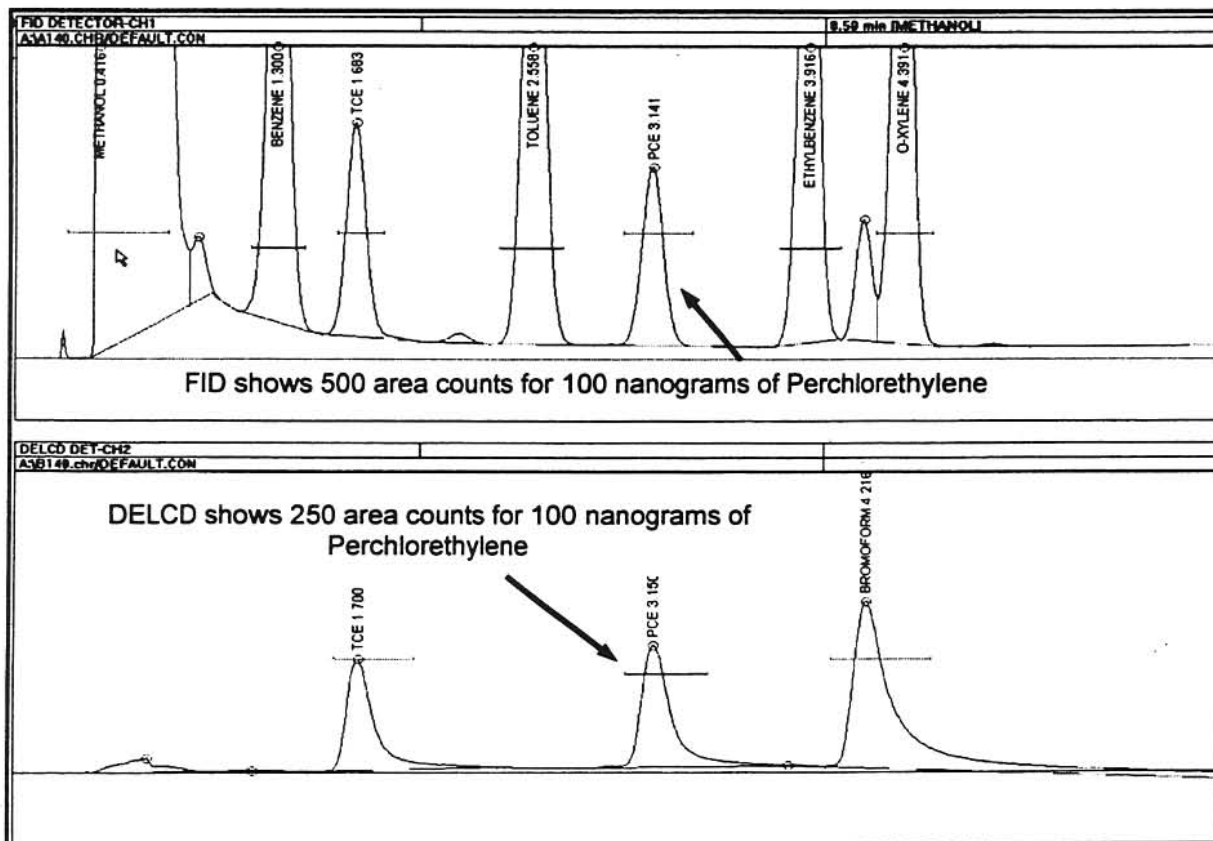


Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

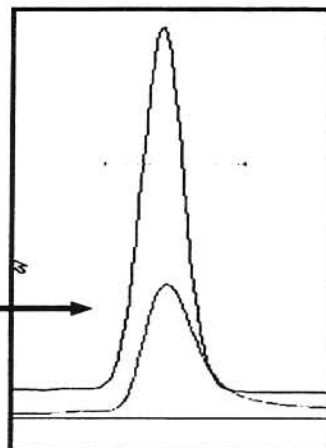
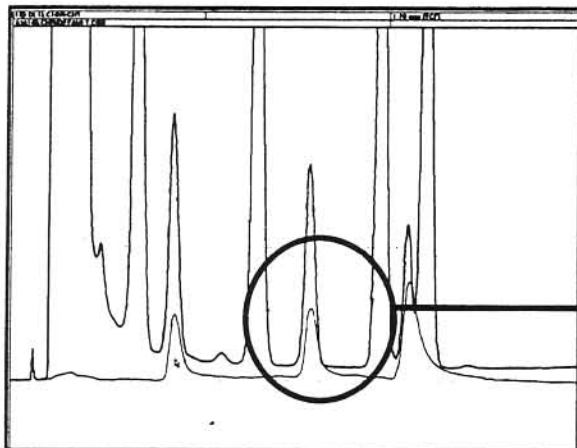
In the combo mode, the DELCD is operated after the FID. The FID signal is usually connected to Channel 1 on the PeakSimple data system. The DELCD signal may be on Channel 2 or 3. Each detector amplifier is labeled at the factory with the data channel to which it has been connected. Detector signals may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

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



Chapter: Detectors

Topic: Operating the FID/DELCD in the Combo mode

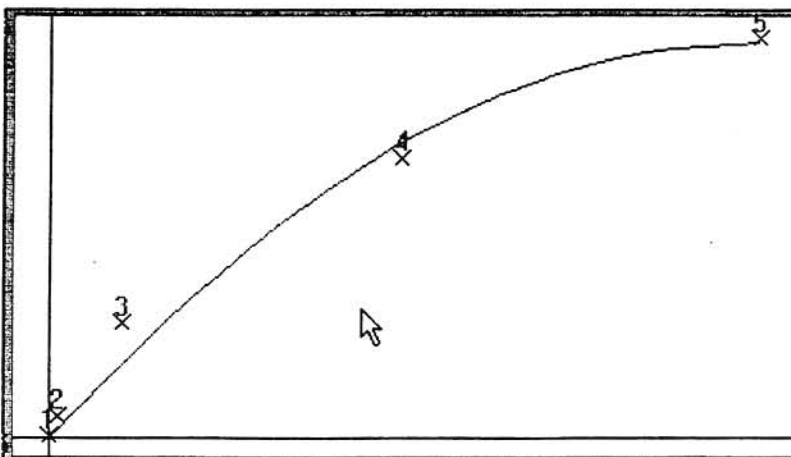


DELCD peak overlaid on FID peak for PCE, then expanded for clarity.

The smaller peak is the DELCD response.

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

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



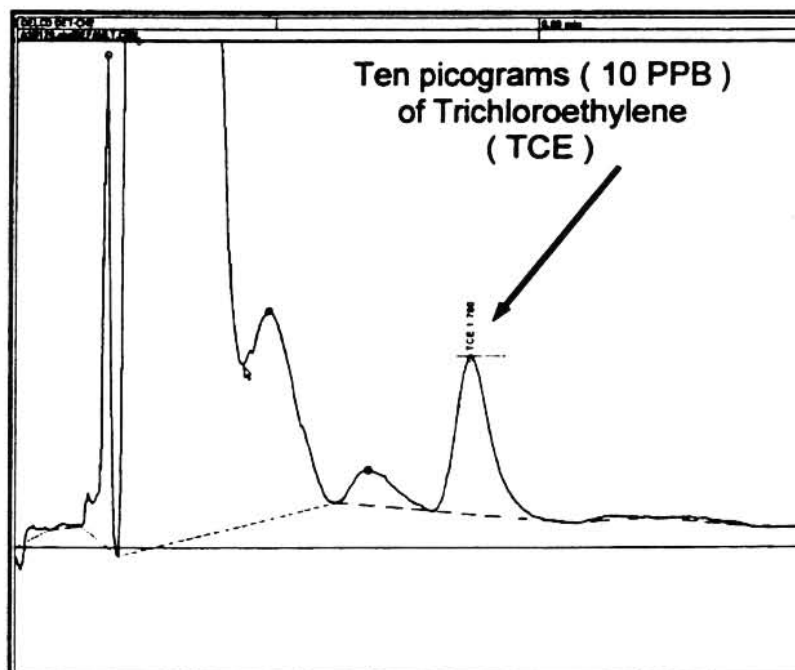
Chapter: Detectors

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

- 1) The DELCD can be operated in a high sensitivity mode by eliminating the hydrogen from the reactions which lead up to the detection of the $\text{ClO}_2\text{-BrO}_2$. Because the chlorine/bromine atoms prefer to react with hydrogen to form non-detectable HCl-HBr , than with oxygen to form detectable $\text{ClO}_2\text{-BrO}_2$ 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 dissociated from the H_2O molecule and available as a reactant. In practice, this means turning off the hydrogen and using clean dry tank air (not the built-in air compressor).
- 2) Remove the hydrogen supply from the GC by disconnecting the hydrogen supply at the GC's inlet bulkhead on the left hand side of the instrument. Reduce the air flow to the DELCD to 50 ml/min by turning the air pressure setpoint down to 1-2 psi. An additional air flow restrictor-consisting of 12" of .067 tubing (1/16", 1.58mm) with an internal diameter of .010 (.25mm) can easily be added to the air supply immediately below the detector to enable the flow to be controlled more precisely at higher pressures. With the extra restrictor installed a pressure setpoint of 10 psi will deliver an air flow of approximately 50 ml/min.
- 3) If using a capillary column, push the column through the FID jet until it just enters the ceramic tubing of the DELCD. This will improve the peak shape somewhat because the column effluent will be discharged into the flowing airstream and will be immediately swept into the DELCD detector volume. When switching back to FID/DELCD combo mode remember to pull the column back into the FID jet.
- 4) Remove the FID collector electrode and replace it with a 1/4" cap fitting. The FID collector electrode allows some gas to escape from the FID combustion area, and this is not desirable when operating in the high sensitivity mode.

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

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

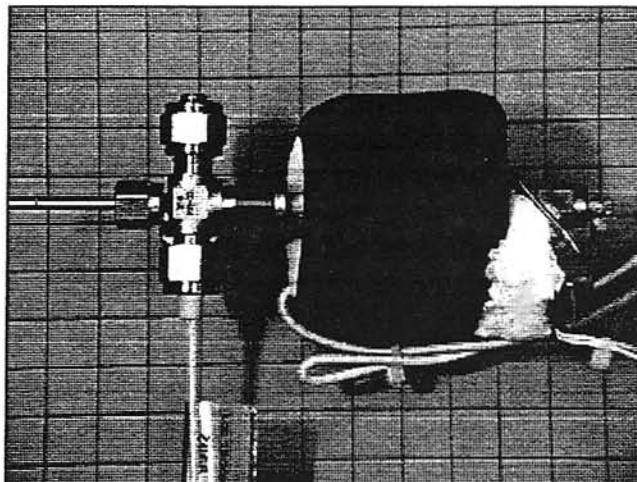


Chapter: Detectors

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

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

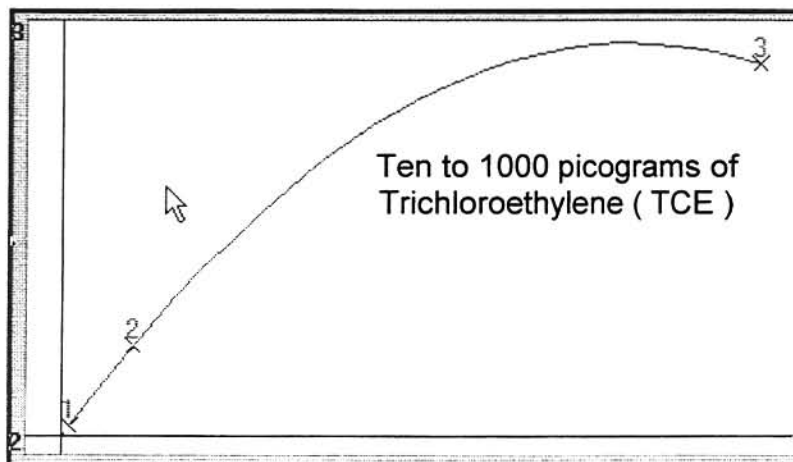
The collector electrode is removed and a 1/4" cap installed instead.



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

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

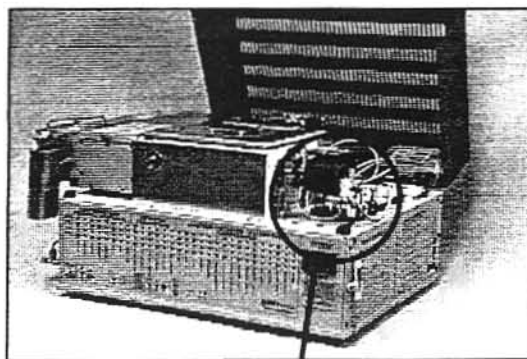
At levels above 10 nanograms the detector is saturated.



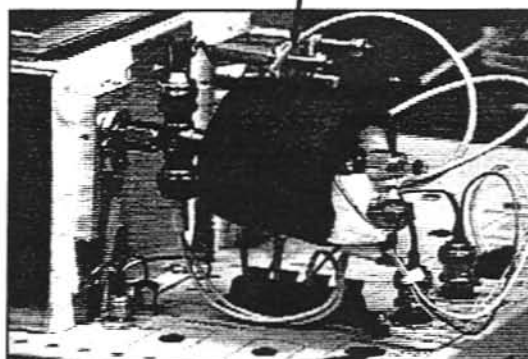
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

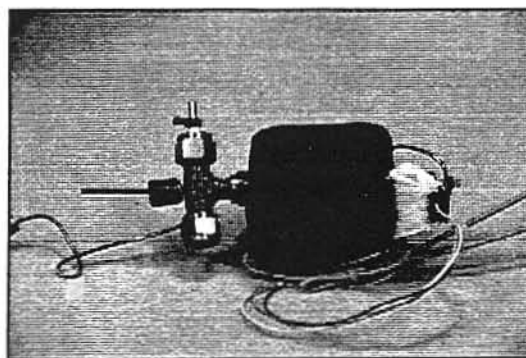
The DELCD detector is only available in combination with the FID detector.



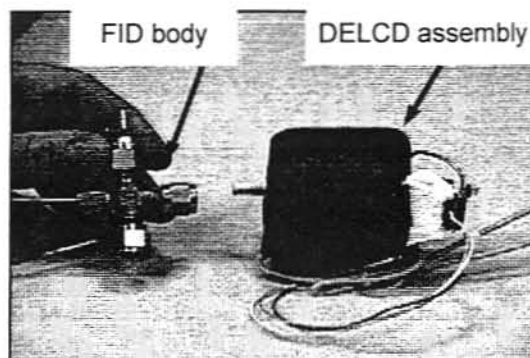
The FID/DELCD combo detector is mounted to a thermostatted aluminum heater block on the right hand side of the column oven.



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



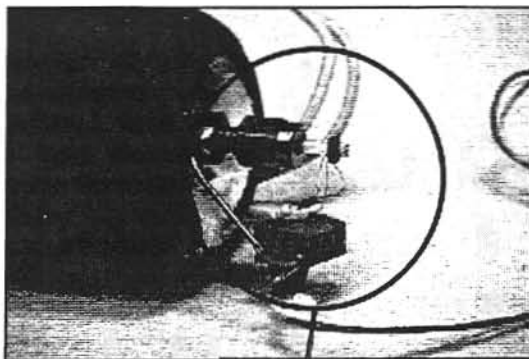
The DELCD part of the detector is the large black cylinder which mounts into the right hand port of the FID detector body. It can be separated from the FID body by loosening the 1/4" Swagelok nut and graphite ferrule which secures it in place.



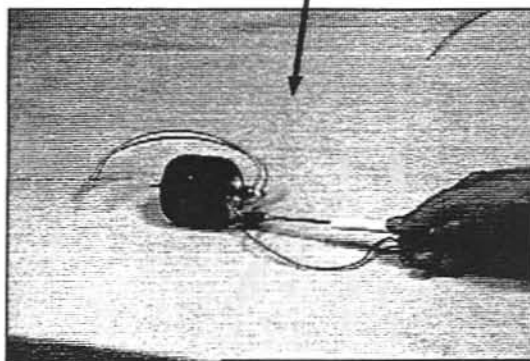
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

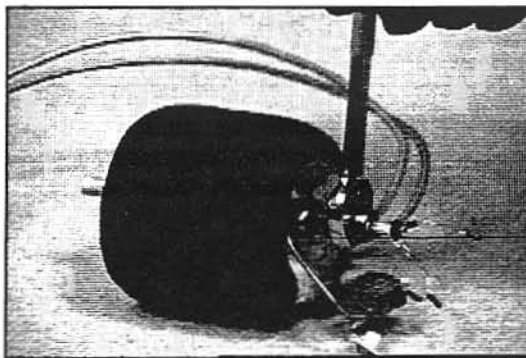
The DELCD collector electrode (part# 8670-1028) can be removed from the heater. Because the heater operates at close to 1000°C, it will fail eventually. A new heater (part # 8670-1027) is less expensive than the complete heater/collector assembly (part# 8670-1029), so it may make sense to remove the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



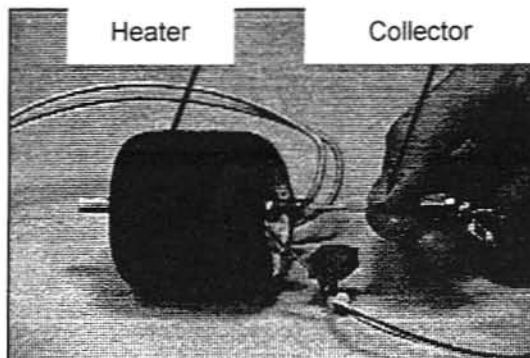
Dis-connect the three small platinum wires from the screw terminals and gently move them aside.



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



The collector can then be withdrawn from the heater.



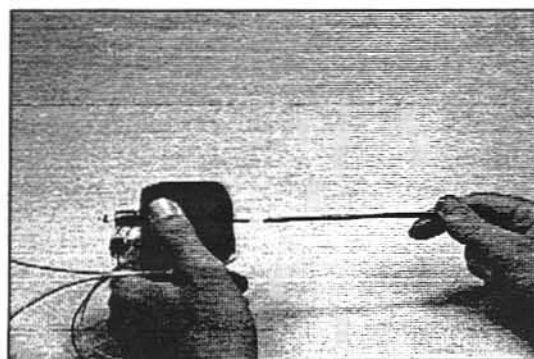
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

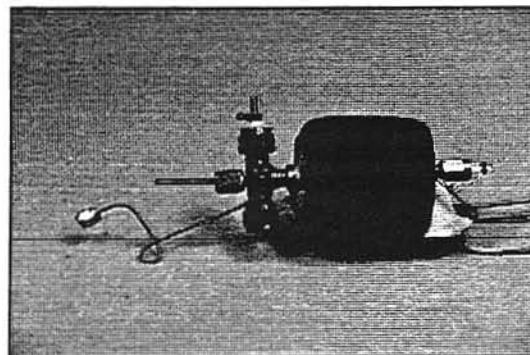
When the collector electrode is re-installed in the new heater, it is important that the tip of the electrode is positioned in the center of the heater.



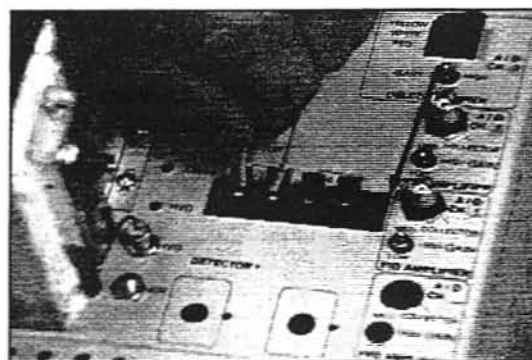
Use a file, rod, screwdriver or other long thin object as a gauge to verify that the electrode tip is centered in the new heater body. Gently re-position the electrode by sliding it through the graphite ferrule to get the proper adjustment. Finally, look down the bore of the heater and check to make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



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



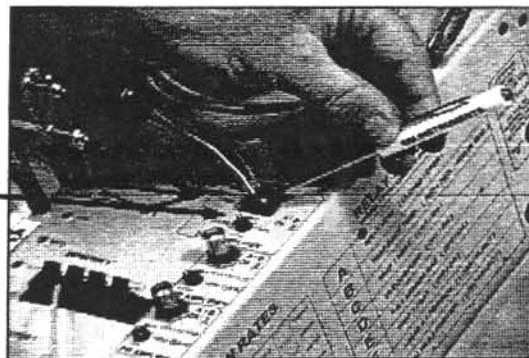
The two DELCD heater wires are connected to the push terminals on the deck of the GC which are labeled DELCD heater.



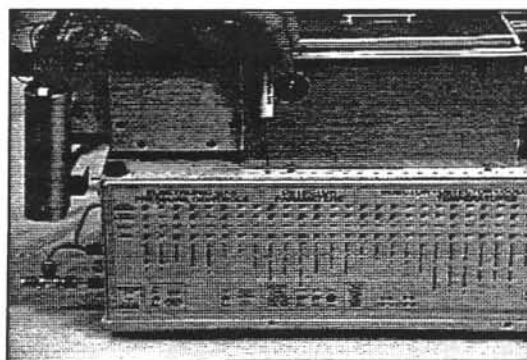
Chapter: Detectors

Topic: FID/Dry Electrolytic Conductivity Detector (DELCD)

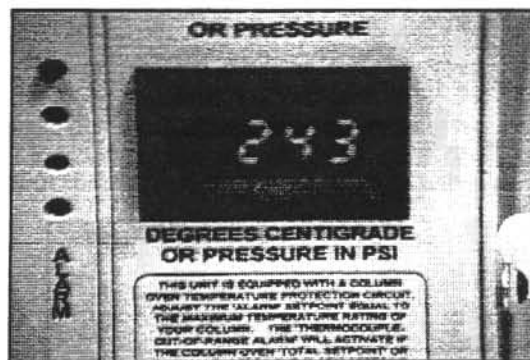
The red, white and yellow wires are inserted into the labelled screw terminals on the deck of the GC.



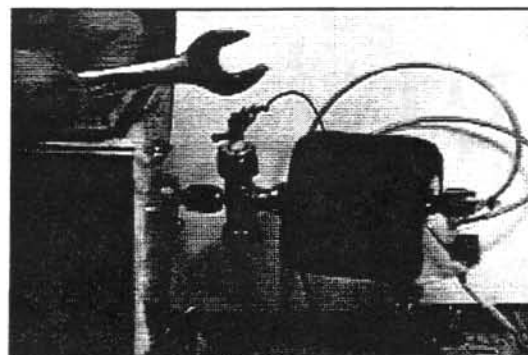
Set the DELCD heater setpoint temperature to 250. This is an arbitrary number which actually corresponds to a temperature of 1000°C. Better sensitivity can be obtained by raising the setpoint to 260 or 270, but at the cost of reduced heater lifetimes.



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



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



Chapter: FID DETECTOR

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

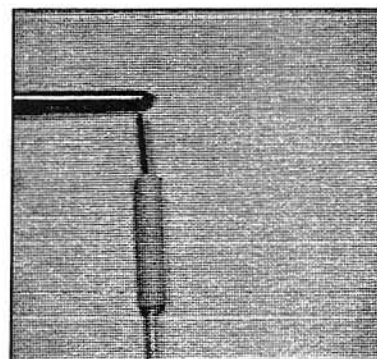
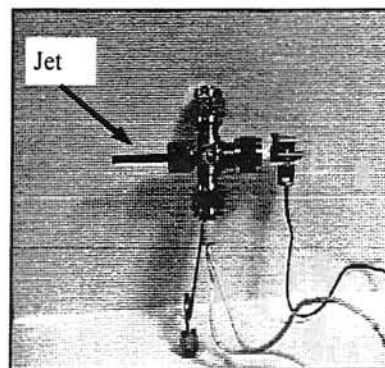
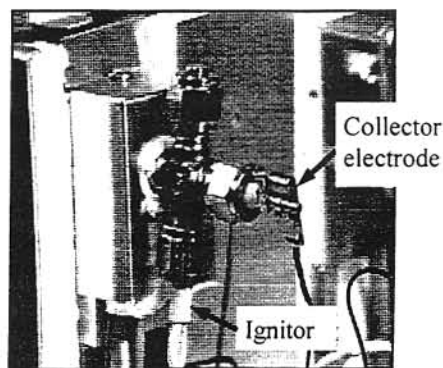
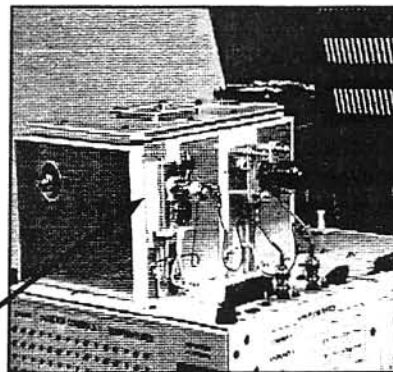
There are situations where it would be helpful to operate the FID detector using just the built-in air compressor for carrier gas and no other gases. SRI distributors demonstrating the GC and software may find it useful to run live chromatograms without the inconvenience of providing hydrogen and helium.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

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

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

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

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



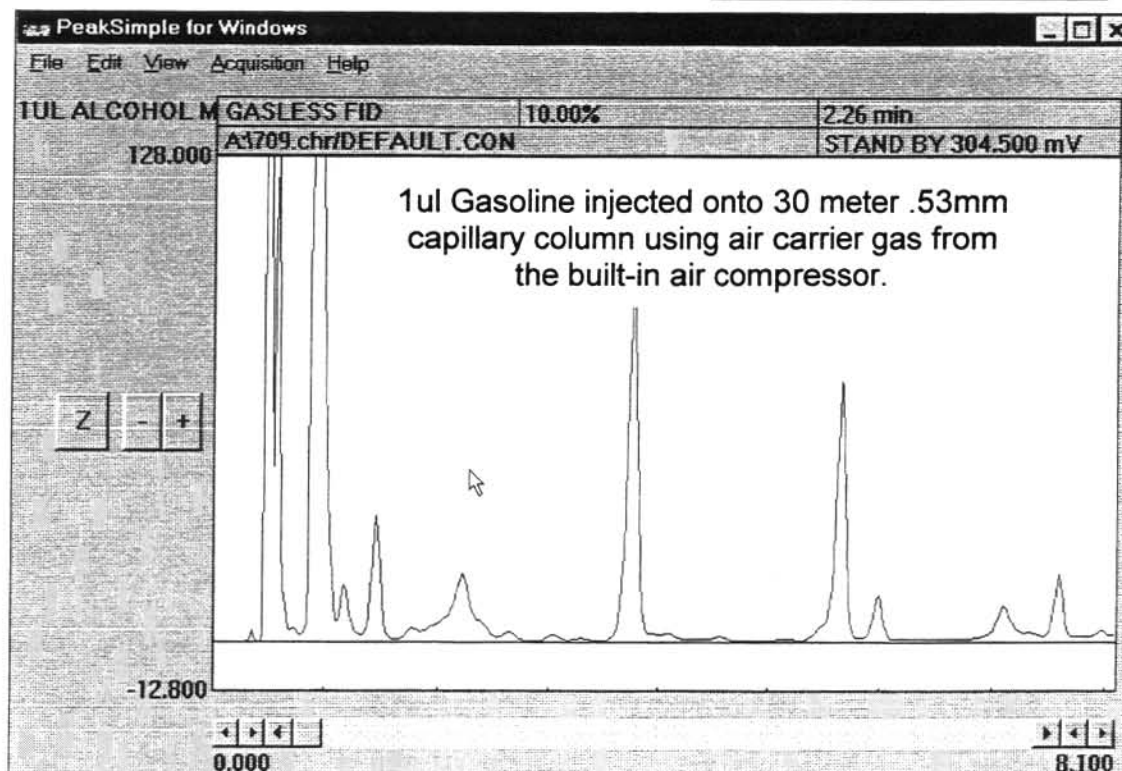
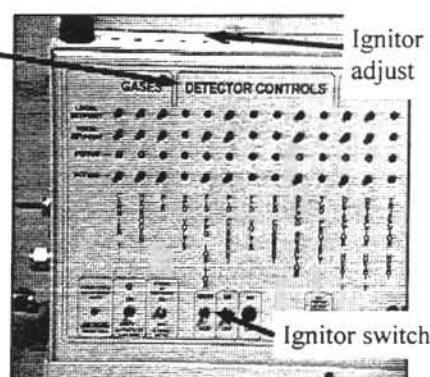
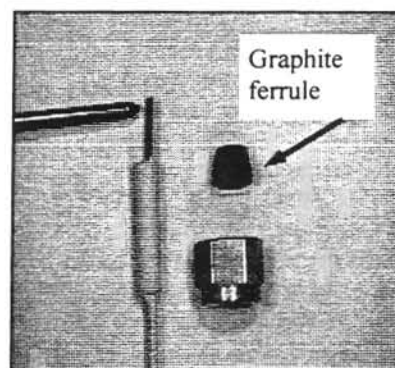
Chapter: FID DETECTOR

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

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

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

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



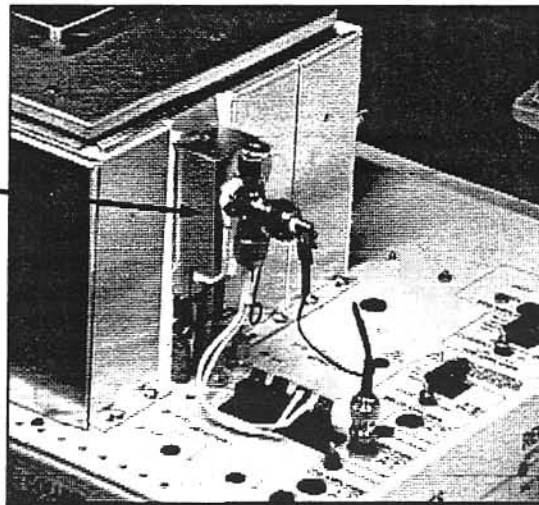
Chapter: Detectors

Topic: Converting from FID to NPD mode

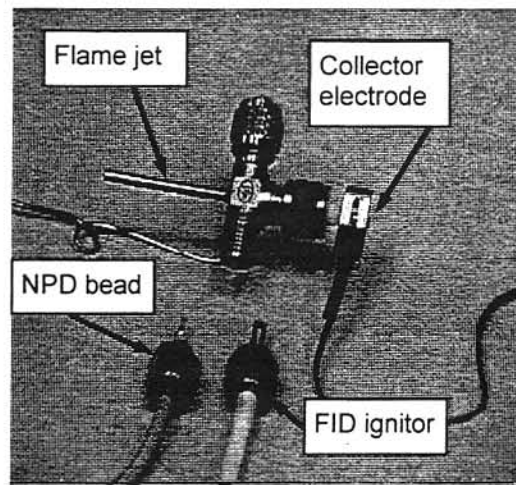
To convert the FID detector to NPD detector:

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

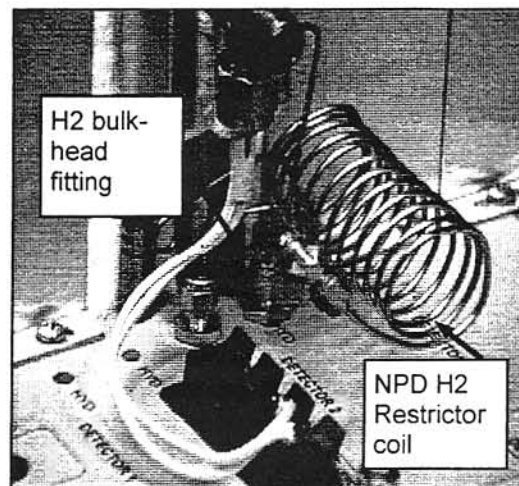
The NPD body is slightly different from the FID in that the NPD flame jet does not protrude as far into the detector body as it does on the FID. This allows the NPD thermionic bead to be positioned directly in front of the jet. Remove the FID body from the heated aluminum block and replace it with the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



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



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

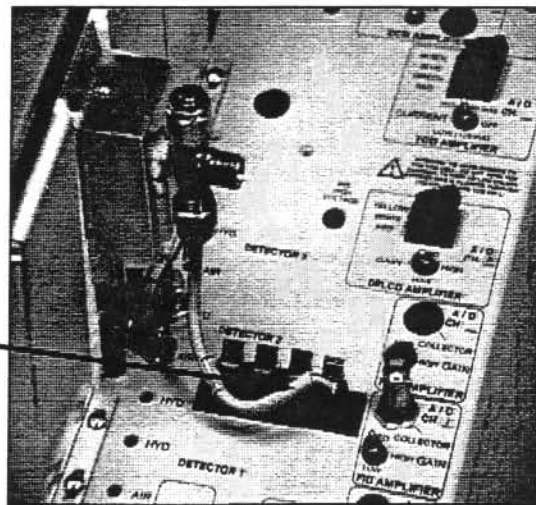


Chapter: Detectors

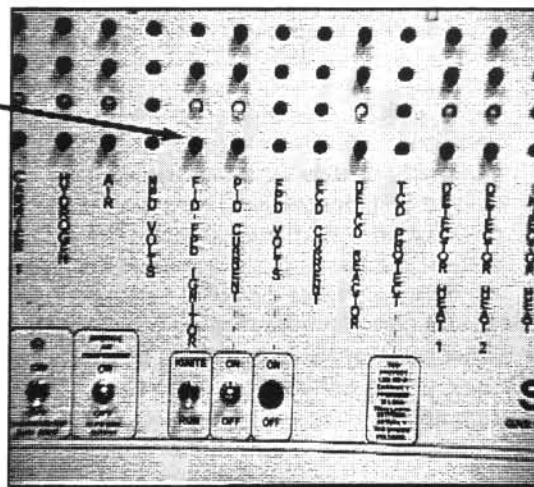
Topic: Converting from FID to NPD mode

To convert the FID detector to NPD detector:

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



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

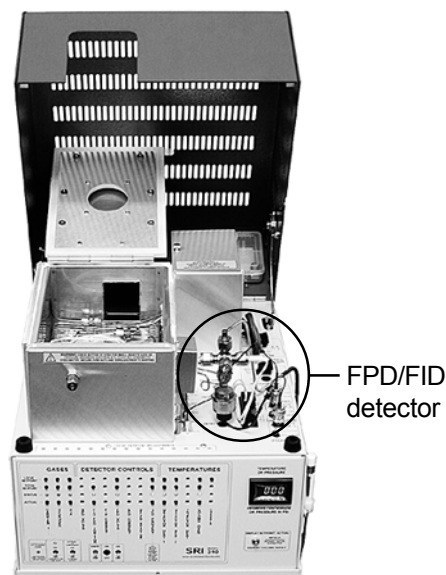


DETECTORS

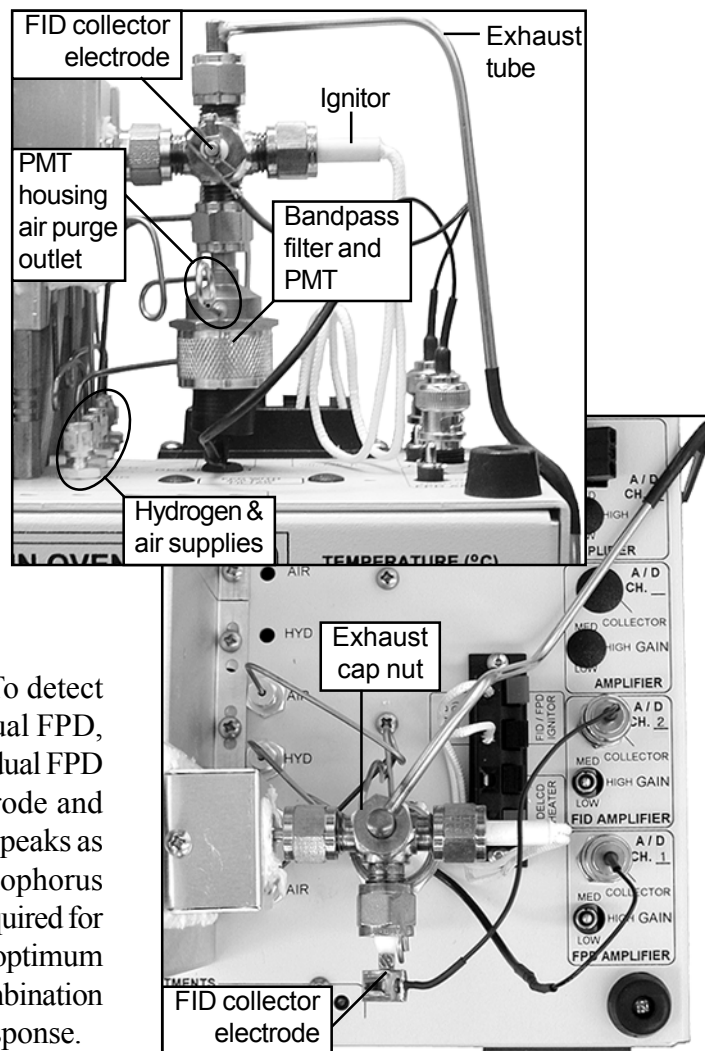
Flame Photometric Detector - FPD

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 phosphorus 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



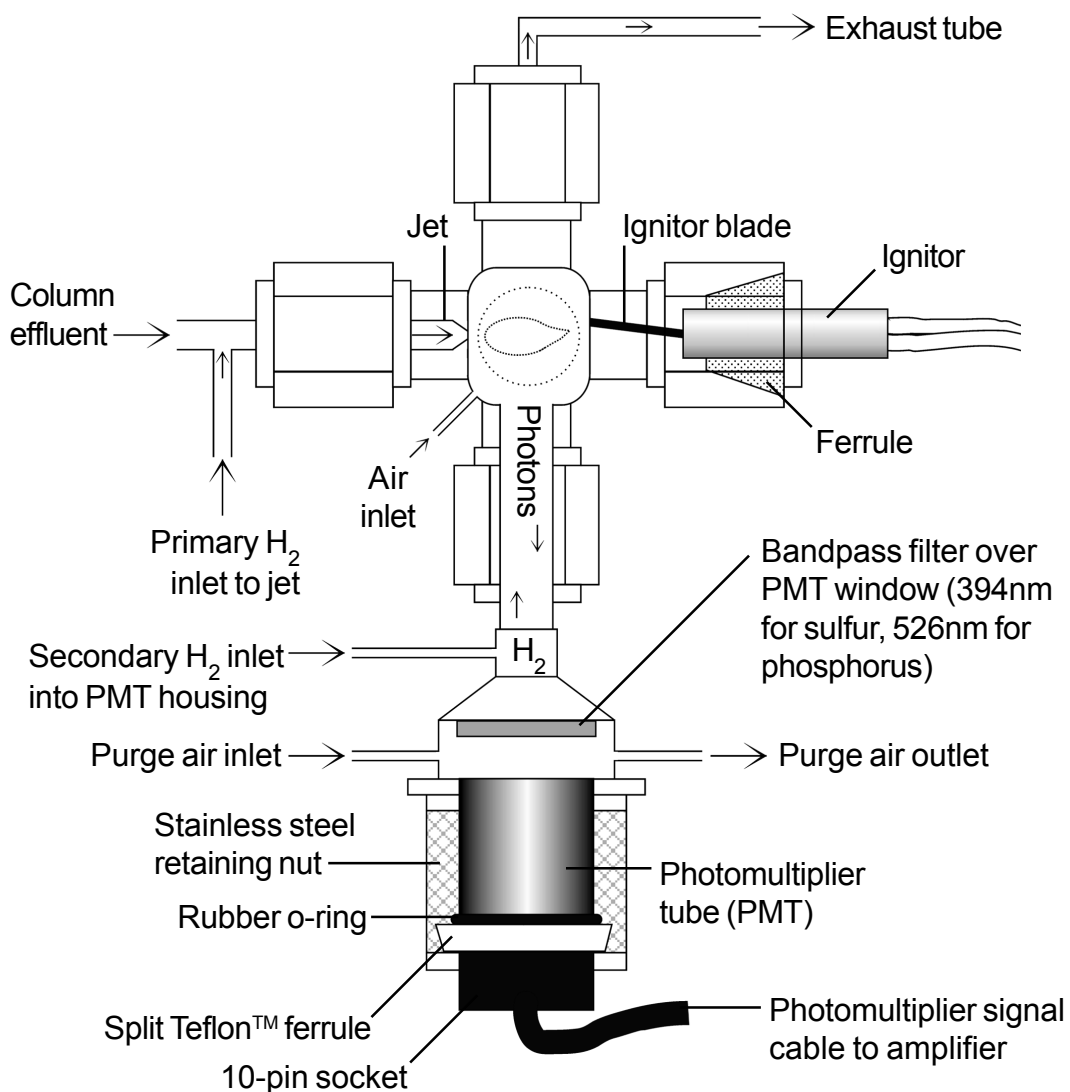
DETECTORS

Flame Photometric Detector - FPD

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



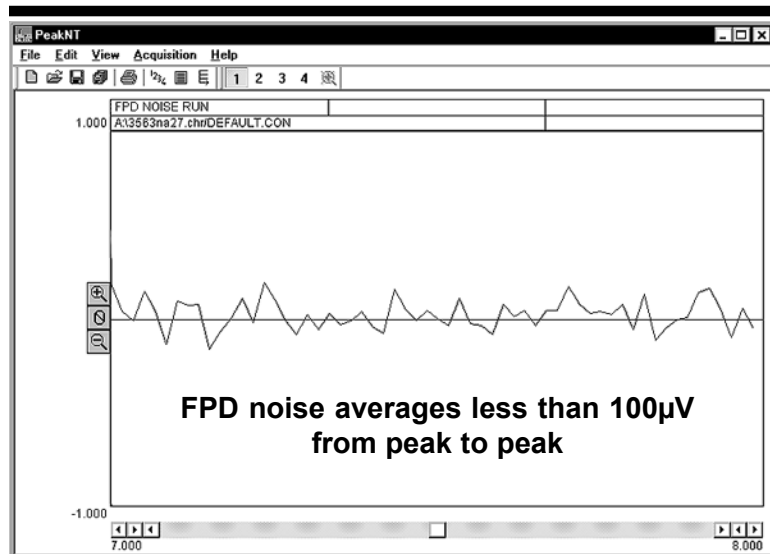
DETECTORS

Flame Photometric Detector - FPD

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 H₂: 60mL/min (30mL/min for each of the two hydrogen flows)
 FPD air: 100mL/min



FPD Sulfur (FID/FPD Combo)

Sample: 1cc 10ppm H₂S 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 H₂: 60mL/min (30mL/min for each of the two hydrogen flows)

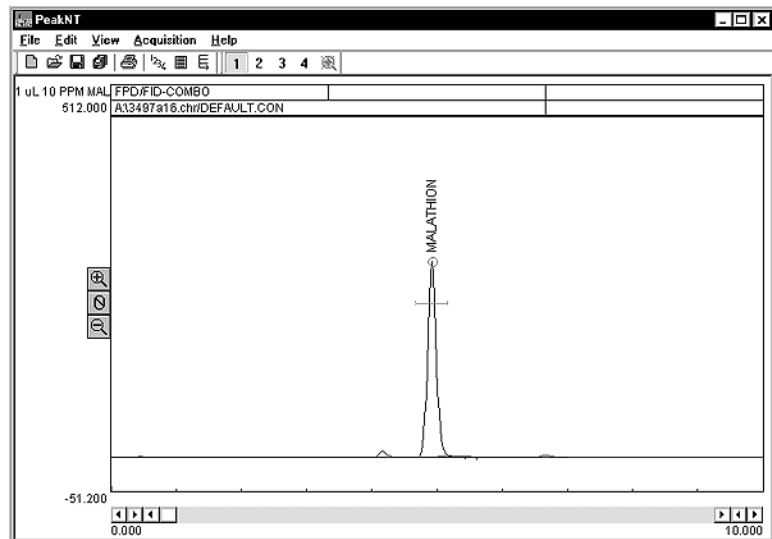
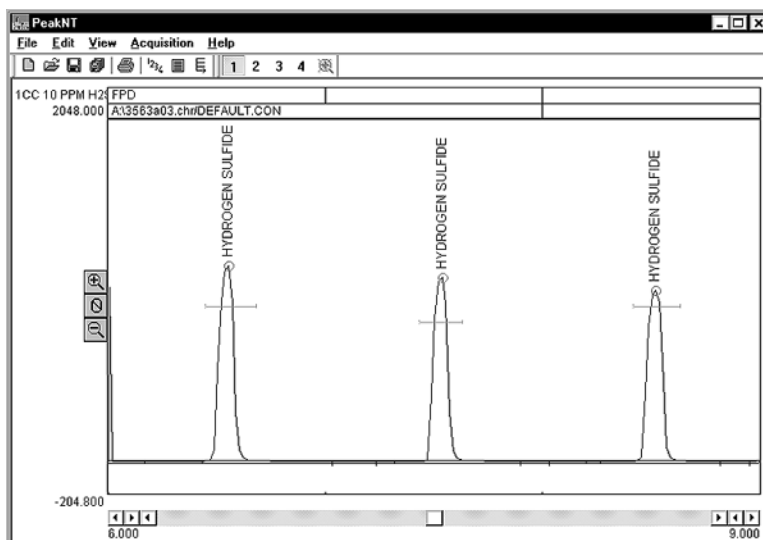
FPD air: 100mL/min

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 H₂: 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, SRI recommends 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.

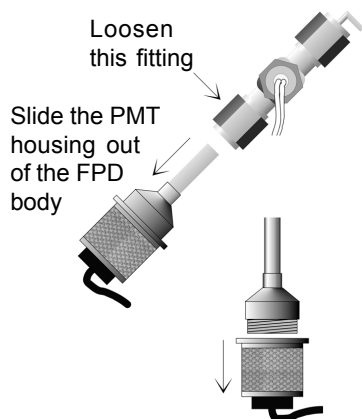
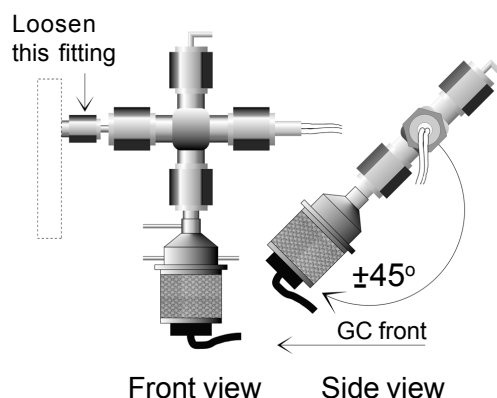
DETECTORS

Flame Photometric Detector - FPD

Switching Between Sulfur and Phosphorus Modes

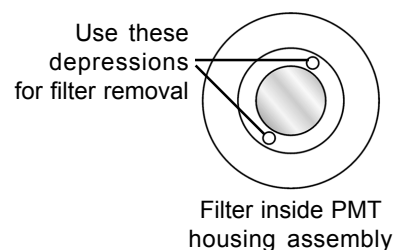
The bandpass filter specified when the instrument was ordered comes installed in the FPD detector assembly. There are two options for switching between sulfur and phosphorus modes. You can purchase either an additional filter or an additional PMT housing and switch them as necessary. Phosphorus wavelength filters are available under SRI part number 8670-0083; sulfur filters are available under SRI part number 8670-0082. A PMT housing with a bandpass filter of the other optional wavelength specifications is available under SRI part number 8670-0084 (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.

DETECTORS

Flame Photometric Detector - FPD

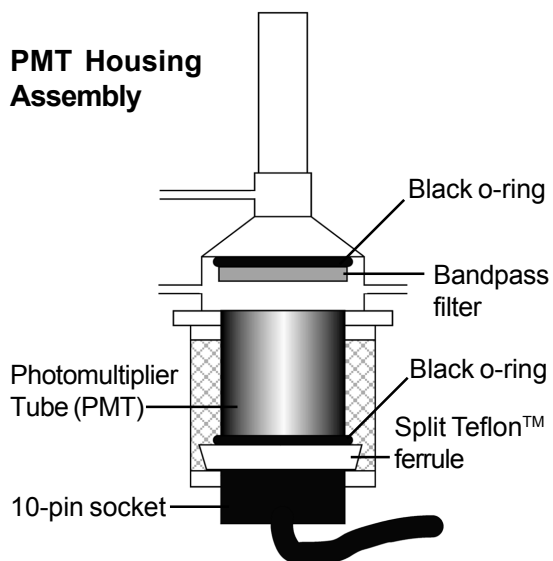
Troubleshooting and Maintenance

Changing the Photomultiplier Tube (PMT)

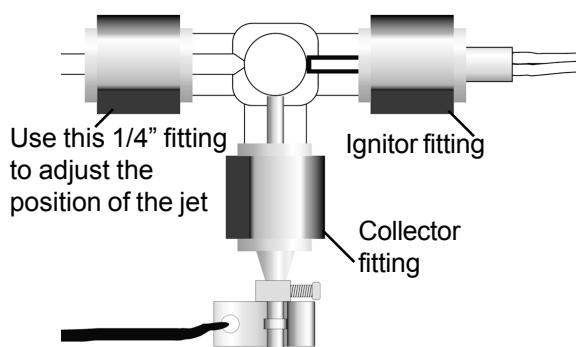
The Photomultiplier Tube (PMT) is a consumable part, and will eventually need replacement. Additional PMTs are available under SRI part number 8670-0080.

1. Follow steps 1-4 on the **“Switching Between Sulfur and Phosphorus 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.

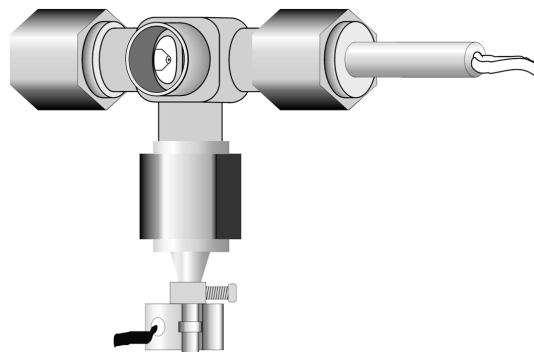
Note: When your FPD detector was assembled at the factory, the ignitor and jet (and collector electrode, if present) were all properly positioned within the FPD body. It is advisable to familiarize yourself with their proper positioning in case they require adjustment. Remove the 1/4” Swagelok cap nut 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.



Top view of FPD with exhaust cap nut removed



An angled view reveals the tip of the jet

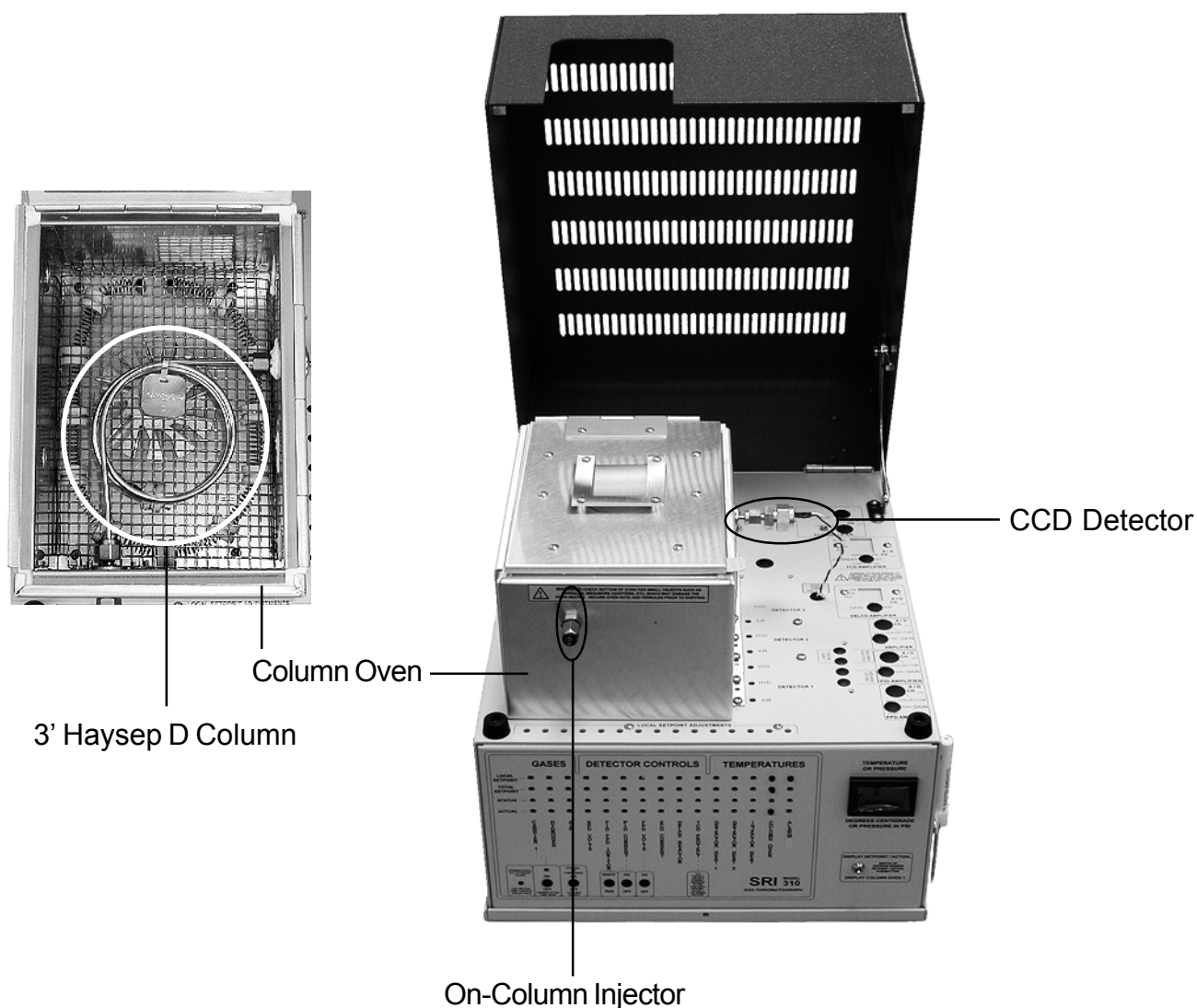


POPULAR CONFIGURATION GCs

Gas-less™ Educational

System Overview

Your SRI Gas-less™ Educational GC is equipped with a Catalytic Combustion Detector (CCD), built-in Air Compressor, temperature programmable Column Oven, Haysep D packed column, On-Column Injector and built-in, single channel PeakSimple Data System, and optionally, Fast Cool-down fans. It is designed to teach the principles of Gas Chromatography without the expense and safety hazards of compressed gas cylinders.



The CCD is about as sensitive as a TCD, but has the hydrocarbon selectivity of an FID. It operates on air alone, which is supplied by the built-in Air Compressor at around 12psi. If you chose optional fast cool-down fans, they will automatically reduce the Column Oven temperature at the end of an analysis to the initial temperature in less than five minutes. Most isothermal applications don't require fast cool-down fans; in these cases, the oven lid is simply manually raised for cooling.

POPULAR CONFIGURATION GCs

Gas-less™ Educational CCD

General Operating Procedure

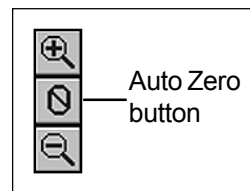
1. Connect your GC to your Windows PC with PeakSimple installed. Plug in your GC and turn its power on.
2. Set the Column Oven temperature to 130°C in PeakSimple as follows:

Initial Temp	Hold	Ramp	Final Temp
130.00	10.00	0.00	130.00

In an isothermal operation like this, the Hold period determines the length of the analytical run.

NOTE: The Haysep D packed column is standard for this GC system because of its separation qualities and durability. To avoid possible damage to the packing, do not program your Column Oven to heat above 150°C.

3. Let the system stabilize for at least 10 minutes, allowing the CCD detector to adjust to the increase in temperature.
4. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Otherwise, the signal starts out at 1000 millivolts. Press the RUN button on your GC or the spacebar on your computer keyboard to begin the run.



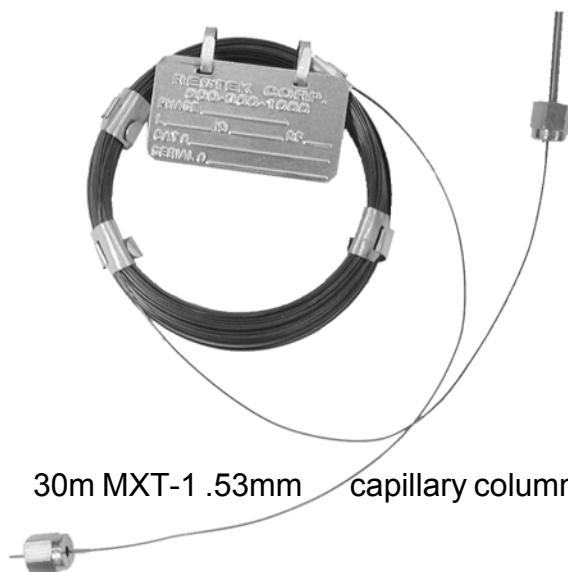
5. Inject sample into the On-Column Injector. A 1µL 1000ppm methanol/acetone sample is the factory test standard for this configuration.

Column Notes



1m Haysep D packed column

Haysep D packed columns are useful for analyzing gases and low molecular weight compounds such as alcohols, aldehydes, and ketones. For heavier molecular weight liquids, use a 30m or 60m MXT-1 capillary column.



30m MXT-1 .53mm capillary column

POPULAR CONFIGURATION GCs Gas-less™ Educational CCD

Expected Performance

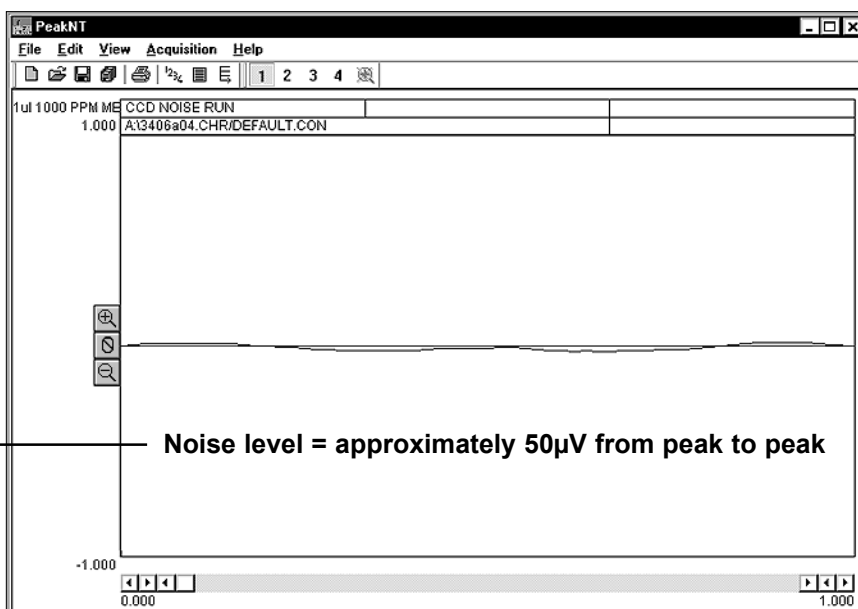
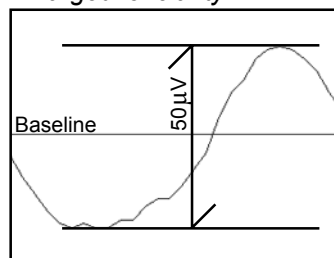
The CCD Detector in your Gas-less™ Educational GC is mounted on the wall of the Column Oven in a brass housing. It consists of a tiny coil of platinum wire embedded in a catalytic ceramic bead. This catalytic ceramic bead is housed in a plastic shell. A 150 milliamp current heats the bead to around 500°C. The CCD is maintained in an oxidative environment by the air being used as a carrier gas. When a hydrogen or hydrocarbon molecule impacts the hot bead, it combusts on the surface, raising the temperature and resistance of the platinum wire. This change in resistance causes the CCD Detector output to change, which produces a peak.

CCD Detector Noise Run

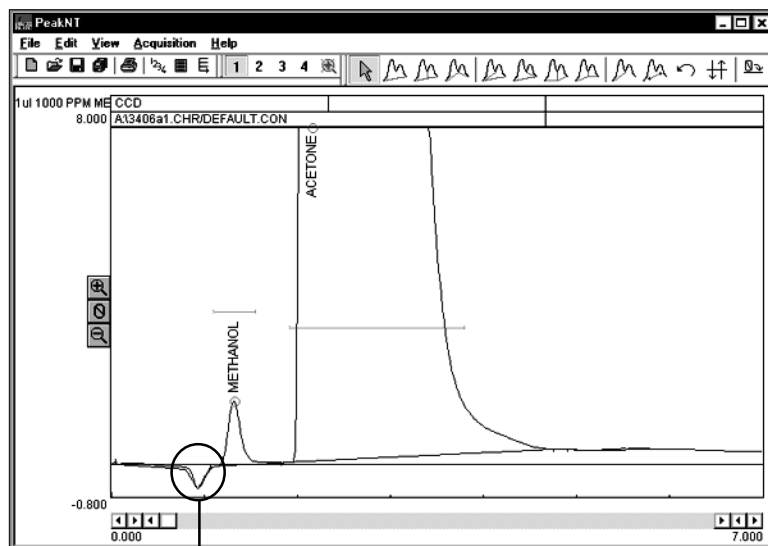
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:
Initial Hold Ramp Final
80°C 15.00 0.00 80°C

Enlarged for clarity



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



Column = 1m Hayesep D

Flow = 37mL/min

Sample = 1μL 1000ppm Methanol/Acetone
mix; direct injection

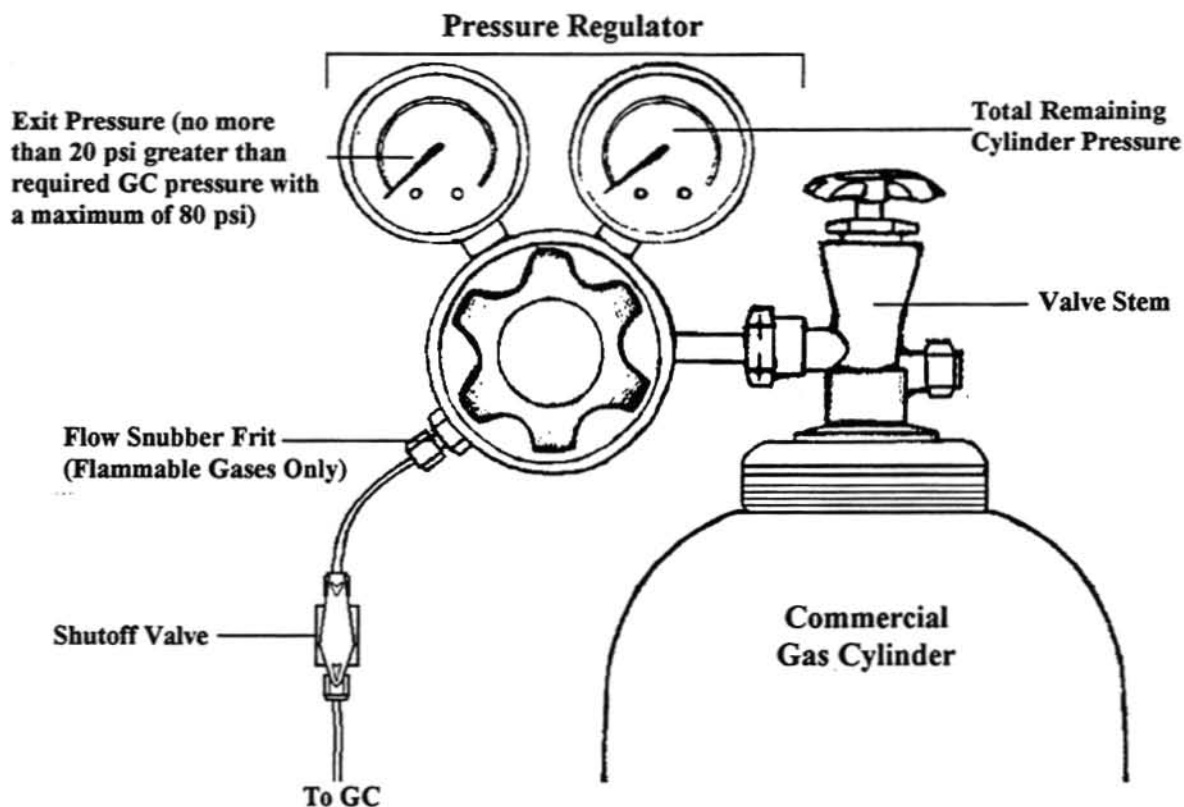
Isothermal Temperature Program:

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

RESULTS:

Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
Total		6958.5600

Negative water peak



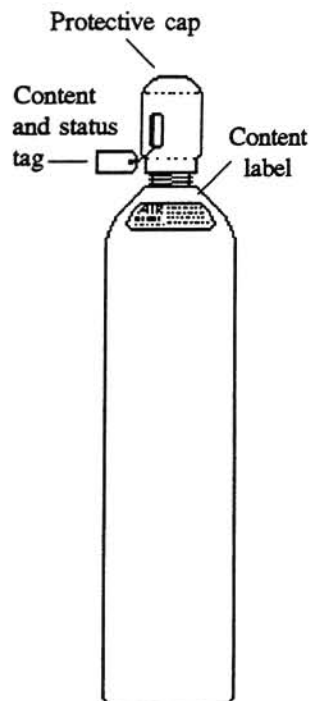
Once the necessary gas supply cylinders have been properly secured to a strong foundation (see previous page), related plumbing must be carefully installed and routed. Always use a pressure regulator on the cylinder to provide proper pressure regulation to the GC. A shutoff valve should be inserted on the output side of the regulator to permit line service when needed. A flow snubber on the output side of the regulator is also highly recommended for hydrogen and all other flammable gases. Unless you are utilizing an ECD, refrigeration grade 1/8" copper tubing is recommended for all of the gas lines from the cylinder pressure regulator to the GC. Due to the exceptionally high sensitivity of an ECD, GCs equipped with this detector require 1/16" stainless steel tubing to reduce the potential for gas line contamination. It is also advisable to flame the stainless steel tubing with a torch until it changes color while flushing with clean carrier gas. This will help to remove any potential preexisting contaminants from within the tubing. An oxygen filter is also a worthwhile option for ECD carrier gas supply lines due to the damaging effects of oxygen on the detector. Plastic tubing should never be used for the gas supply lines due to its permeability to contaminants such as oxygen which can cause damage to thermal conductivity detectors (TCDs) and capillary columns as well as ECDs. Proper supply line routing is also very important. **Avoid routing gas supply lines near electrical outlets** to eliminate any potential hazards associated with electrical shorts and/or flammable gases. Metal gas lines can very easily fall across the two prongs of any plugged in electrical device and start a fire if routed near an electrical outlet. After gas supply lines have been properly installed, pressurize the lines and check all associated fittings for potential leaks. In order for electronic pressure control units to operate properly, do not set gas pressure coming from the cylinders any more than 20 psi greater than GC requirements. For example, if carrier gas head pressure is set to 10 psi at the GC, then set carrier supply pressure from the cylinder above 10 psi but no greater than 30 psi.

Helium is the recommended carrier gas for all standard SRI installed detectors. These detectors include: TCD, FID, PID, ECD, DELCD, FPD, and NPD. If helium is unavailable, nitrogen is an acceptable carrier gas alternative. If nitrogen is used with a TCD, the filament current switch must be set to low to avoid filament damage. **Do not use hydrogen or any other flammable gas as a carrier gas for any SRI 8610C GC.** These units have electronic pressure control and a simple column or injection port leak could release dangerously high levels of flammable gases. Some detectors and accessories require additional gas supply types for proper operation. Argon/methane or nitrogen is required for ECDs as make-up gas. The recommended make-up gas is argon/methane which provides the best sensitivity and largest dynamic range for the ECD, but nitrogen is a readily available, cost effective alternative (see the manual section on the ECD for more details). FIDs, FPDs, and NPDs all require hydrogen and air in order to create the combustible fuel mixture for the detector flame. Hydrogen is an extremely flammable gas and must be handled appropriately. Always consult local safety regulatory agencies for proper procedures for handling compressed and/or flammable gases. An internal air compressor is an available SRI GC option as a source of air. GCs with a purge and trap accessory also require some type of sparge gas. Generally helium can be used as both a carrier and a sparge gas supply. Methanizer accessories require hydrogen gas as a reactant in the catalytic reduction of CO and CO₂ to CH₄.

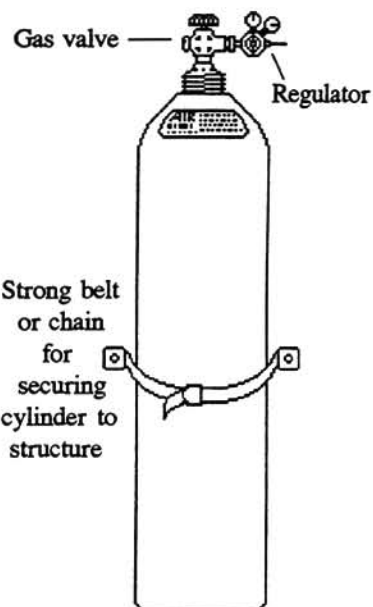
We recommend the use of medium to high quality gas sources for all required gases in order to prevent any operational problems associated with low quality gas. ECDs require an extremely pure carrier gas source of 99.9995% or higher. SRI GCs are equipped with small internal molecular sieve polishing filters on the carrier gas plumbing only to filter low levels of contaminants. If the quality of gas available is questionable, an larger external filter may be necessary to filter excess contaminants such as moisture. Please call SRI technical support with any additional questions on gas supplies or specialized applications.

IMPORTANT SAFETY NOTE

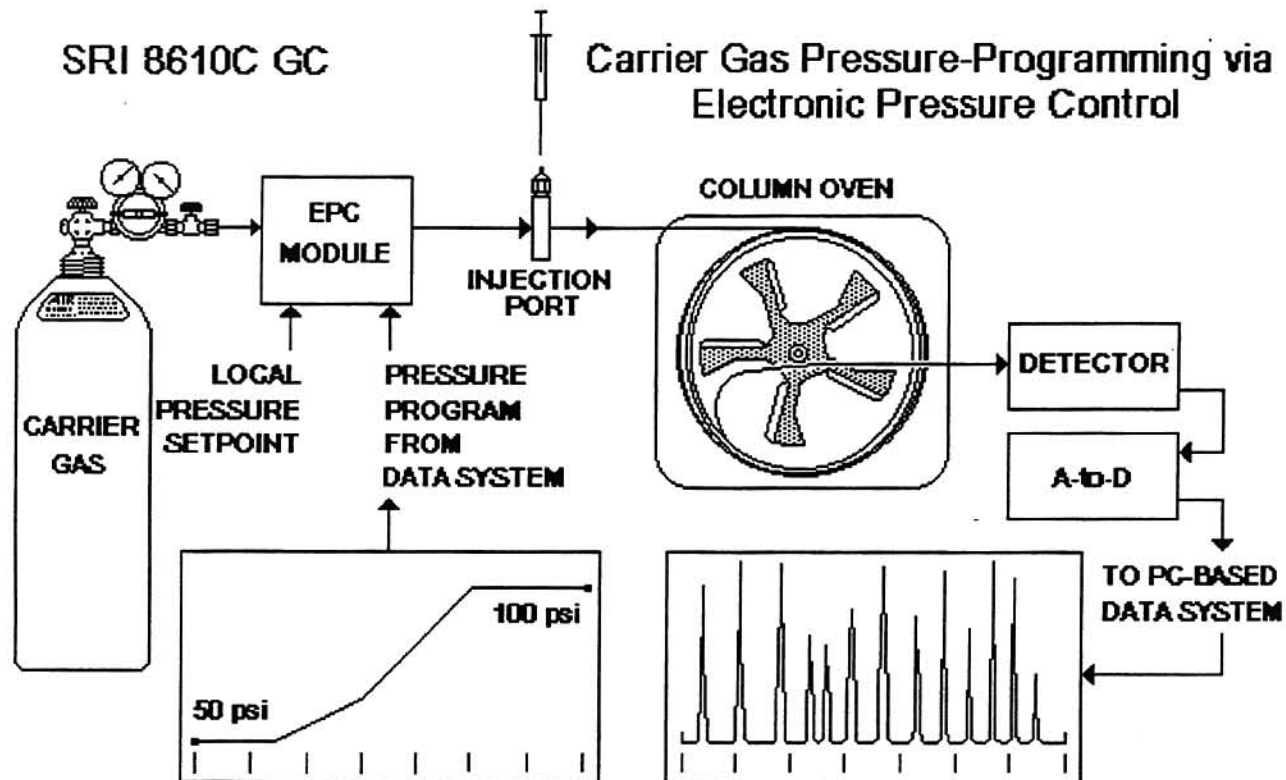
When handling gas cylinders, remember - never transport or move a gas cylinder without its protective cap securely in place. Gas cylinders can contain up to 2700 psi of compressed gas. If the cylinder were to suffer an accident causing the unprotected valve stem to be broken off, the force of the escaping gas could convert the cylinder into a lethal projectile capable of travelling hundreds of feet and penetrating structural walls. Once the gas cylinder has been placed in the location where it will be stored or utilized, it should be secured by means of a chain or belt securely fastened to the wall or other foundation. One strap may or may not be adequate depending on the installation - consult local safety regulations. Once the cylinder is in place and secured, the cap may be removed so that the gas pressure regulator may be attached for use.



Typical gas cylinder shown. Note that the protective cap is in place, protecting the valve from damage. Cylinders are clearly labelled and tagged when delivered for use. In some areas, cylinders are color-coded for handling safety



The protective cap is removed only after cylinder is in place and secured by at least one chain or belt



FLOW CHART ILLUSTRATING CARRIER GAS PRESSURE PROGRAMMING ON THE 8610C GC

All SRI 8610C gas chromatographs are equipped with Electronic (or Pneumatic) Pressure Control (EPC) of all system gases. Each gas, from the carrier gas, to the specific detector gases, such as FID hydrogen and FID compressed air, in the case of an FID detector, are controlled by a dedicated solid-state EPC module that electronically monitors and instantaneously adjusts the pressure being supplied to the particular feature. This electronic control facilitates extreme precision of gas flows to the various functions. Each EPC module features a local, user-adjustable setpoint accessed by a trimpot (variable potentiometer) located just above the particular function on the "at-a-glance" panel display. The carrier gas is among these adjustable setpoints. The term "local" refers to the fact that the "local" setpoint is set manually at the trimpot on the GC chassis. As in the case of the column oven temperature setpoint, the carrier gas pressure setpoint may be set "locally" (manually on the GC chassis), or from the computer via a pressure program. Created in the same format as a PeakSimple temperature program, the program signal is sent to the data system interface and converted to a control voltage that can increase, maintain, or decrease the carrier gas pressure automatically at the user's command.

The PeakSimple serial data system interface offers two rampable voltage outputs - one to program the column oven, and the other to program carrier gas pressure. Outputting a 0 to 5VDC variable signal, the EPC module will permit an output pressure of from 0 to 100psi (the carrier pressure shown is actually the column head pressure). Please note that any local setpoint value will be summed to this signal, resulting in the "total" setpoint value on the panel display. The carrier gas pressure regulator at the gas cylinder should be set 10psi higher than the highest programmed carrier gas head pressure desired for proper control. Ramping permits the head pressure to be varied, to speed or slow the elution of peaks from the analytical column as needed by the application or user.

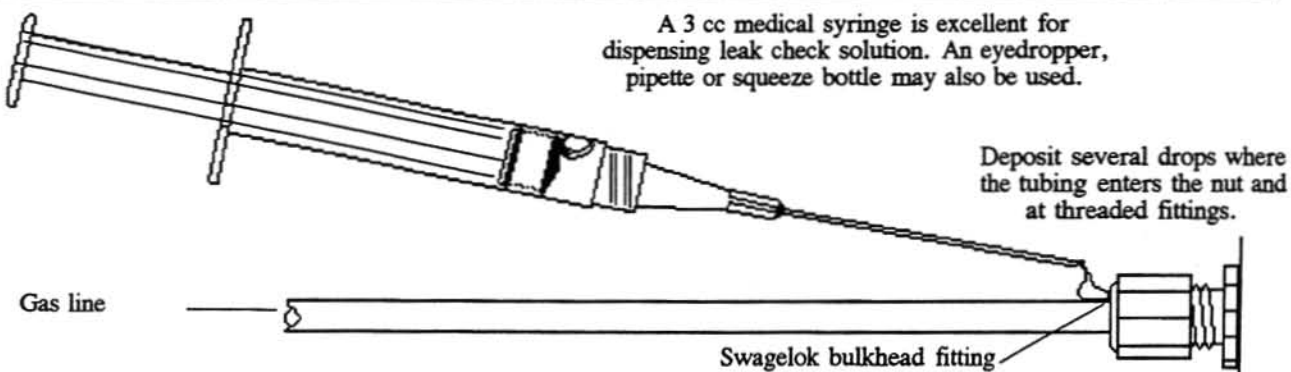
Once all of the appropriate gas supply sources and lines have been properly installed, along with all other GC columns and connections, the entire system should be systematically pressurized and checked for possible leaks. Begin by opening all of the compressed gas cylinder valves and setting exit pressures to the appropriate value for each cylinder regulator. Remember that cylinder exit pressures should never exceed the required GC pressure settings by more than 20 psi and 80 psi is the maximum pressure that the GC can safely handle.

First check for leaks in the lines and connections between the compressed gas cylinder and the GC flow control fluistors. With the system pressurized and the GC power turned off, close each of the compressed gas cylinder valves one at a time and closely watch the pressure indicator on the cylinder regulator to see if pressure decreases. If the system is leak free between these two points, the cylinder pressure indicator should not noticeably decrease for at least five minutes. If pressure does noticeably decrease over this time period, then it indicates a significant leak somewhere between the cylinder output and the GC fluistor. Any leak, especially with flammable gases, must be immediately located and repaired. The best way to check specific connections for leaks is with a leak check solution (see section below on Using Leak Check Solution). If pressure test indicates that the system is leak free from the cylinder to the fluistor, then proceed to check the rest of the carrier gas system for leaks. If the system does have a leak, locate and repair prior to proceeding.

Next check for leaks between the fluistor and injection port. Begin by disconnecting the column from the back side of the injection port. Next insert some type of pressure blocking fitting on the injection port where the column was attached. A standard Swagelok nut with an injection septum in place of the ferrule will work quite well. Turn the GC power and gas supply back on. Use the control panel to see what the **actual** carrier pressure value is and write it down. Now turn off the carrier gas supply at the cylinder once again. Wait 5 minutes and then use the GC control panel to view the **actual** carrier pressure once again. If this value has decreased in the 5 minute time frame and the previous test results were negative, it indicates that there is a significant leak somewhere in the internal GC carrier gas lines between the fluistor and the injection port. Once again immediately locate and repair any leaks using a leak check solution as described below.

After all of the leaks upstream from the column have been eliminated and confirmed by the two pressure tests described above, properly attach your column to the injection port. Use leak check solution to check all of the fittings within the column oven for leaks and repair any that you find.

Following all the instructions above will assure the operator that the system is leak free. Any time fittings are changed or the GC is relocated, the system should be rechecked for leaks. Failure to properly repair leaks can cause safety risks as well as operational malfunctions.



Leak Checking Solution

SRI recommends that a solution of 50% water and 50% alcohol (methanol, ethanol, or propanol) be used as a leak check solution. The water-alcohol mixture leaves no residue which could leak through the fittings and cause system contamination. Furthermore, water, when used alone and due to its high surface tension, tends to bead rather than flow into spaces between the tubing and the connectors where leaks may occur. A leak will show up as a stream or froth of tiny bubbles. Inspect any leaking fitting for damaged threads and reversed, missing, or damaged ferrules.

Quick Start

SRI GC Installation Guide

I. Gas Installation & Connection

1. To connect your GC to a gas supply, we recommend the following:

- A 50 foot length of copper tubing
- A stainless steel gas line filter
- At least 2 sets of stainless steel Swagelok nuts and brass ferrules (it is a good idea to keep a few extras on hand)
- A cylinder pressure regulator with 0-100psi output

NOTE: each type of cylinder has a different CGA connection to the regulator (CGA = Compressed Gas Association). Air is typically CGA 590 or 346. Helium and nitrogen are CGA 580. Hydrogen and argon-methane are CGA 350.

Gas line installation kits that include everything you need are available from SRI:

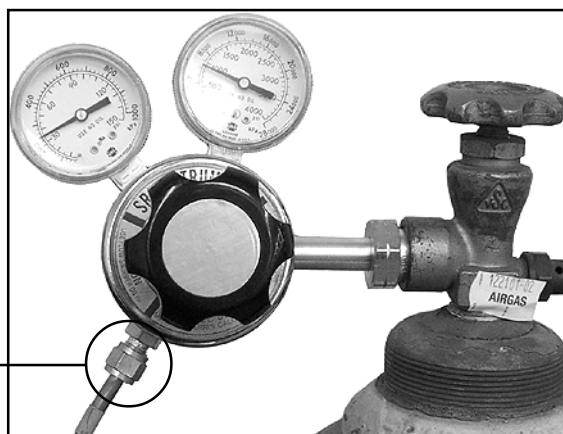
- | | |
|-----------|---|
| 8600-C590 | Air gas line kit (with both CGA 590 and 346 inlet fittings) |
| 8600-C580 | Helium/nitrogen gas line kit |
| 8600-C350 | Hydrogen/argon-methane gas line kit (the hydrogen CGA is equipped with a flow restrictor to limit the escape of gas in the event of a leak) |

These kits include everything in the list of recommended supplies above, plus a tubing cutter. Each regulator is supplied with a 1/8" Swagelok fitting for easy connection to the copper tubing.

2. Using the appropriate CGA connection as described above, attach the regulator securely to the gas cylinder.

3. Secure one end of the 1/8" copper tubing to the regulator with a Swagelok nut and ferrule. Cut the tubing to the desired length before connecting it to the GC. Make sure to leave it long enough to allow you to move your GC around your work area.

1/8"
Swagelok
fitting



4. If you don't already filter your gas, install gas line filter(s) in the gas line(s) where it is convenient to replace when needed.



cylinder air, a jumper tube is secured to the air inlet and outlet. If you ordered your GC with an air compressor, it is shipped with the jumper tube in place as shown.

5. Connect the gas or gases to the inlets on the left-hand side of the GC as labeled.

NOTE: the GC shown here is equipped with a built-in air compressor. When using the internal air compressor instead of

Quick Start GC Installation Guide

("Gas Installation & Connection" *continued*)

GAS FLOW RATES					
CARRIER 1:		:	7	PSI =	10 ml/min
CARRIER 2:		:		PSI =	ml/min
P&T PURGE:		:		PSI =	ml/min
HYDROGEN 1:	FID	:	21	PSI =	25 ml/min
HYDROGEN 2:		:		PSI =	ml/min
AIR 1:	FID	:	9	PSI =	250 ml/min

6. The pressure that correlates with the flow rate for the column, make-up gases, and detector supplies is labeled on the right-hand side of the GC. For best EPC performance, set the incoming gas pressure(s) 15-20psi higher than the operating pressure listed on the right-hand side of the GC.

II. Column Installation

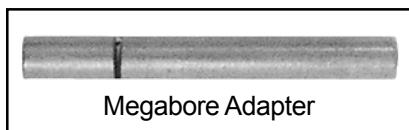
1. If you ordered a column with your GC, it is shipped installed in the column oven and you can skip this section. Otherwise, open the GC lid and the column oven lid.

2. These instructions will cover the installation of a 0.53mm capillary column into an on-column injector. The SRI on-column injector is designed for a 26 gauge syringe needle; a 10 μ L liquid injection syringe with a 26 gauge needle is included in the Accessories Kit shipped with your GC.



Spare Parts Kit

A megabore adapter for syringe injection onto 0.53mm capillary columns is included in the Spare Parts Kit affixed to the inside of the GC lid on the right-hand rear corner.



Megabore Adapter



Accessories Kit contents:

- 6' Serial **or** USB cable
- Tubing cutter
- 10 μ L liquid injection syringe
- 1mL gas injection syringe & needle
- 3mL leak check syringe

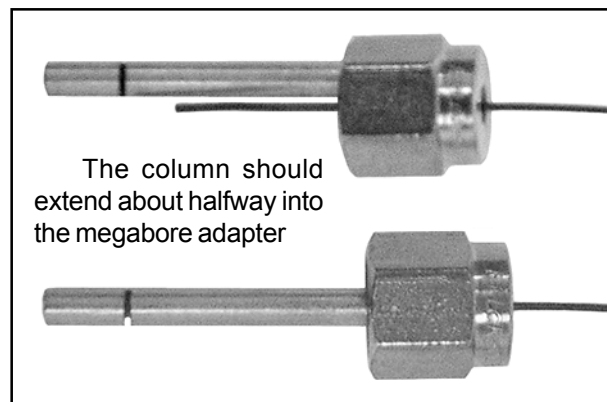
3. The megabore adapter is a 1" x 1/8"OD stainless steel tube with a perpendicular gash cut into it, and a conical entry to guide the syringe needle into the column. A 0.53mm capillary column connects to the SRI on-column injector with a graphite reducing ferrule and a 1/8" Swagelok

nut. Insert one end of the column through the nut, then through the graphite ferrule. It is a good idea to trim off about one inch of the column to avoid possible peak tailing from any graphite shavings left behind after inserting the column through the ferrule; make sure the cut is clean, with no jagged edges.

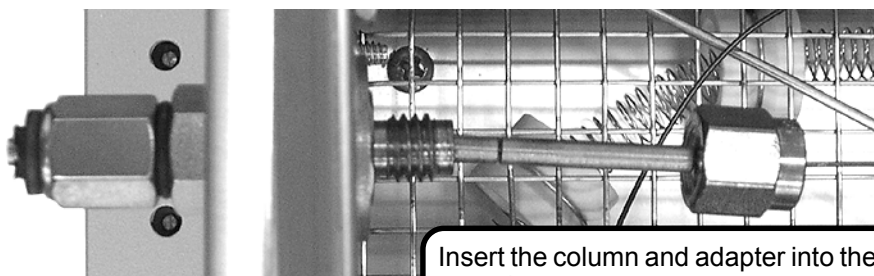
Quick Start GC Installation Guide

("Column Installation" *continued*)

4. Insert the column end with the graphite ferrule and Swagelok nut about halfway into the megabore adapter and tighten it with the nut and ferrule.



5. After inserting the column into the adapter, insert the column and adapter together into the injection port.



Tighten the Swagelok nut with a 7/16" wrench. You should feel a little give from the ferrule, but do not overtighten it. You want it tight enough to prevent leakage, but do not smash the ferrule.

Insert the column and adapter into the injection port

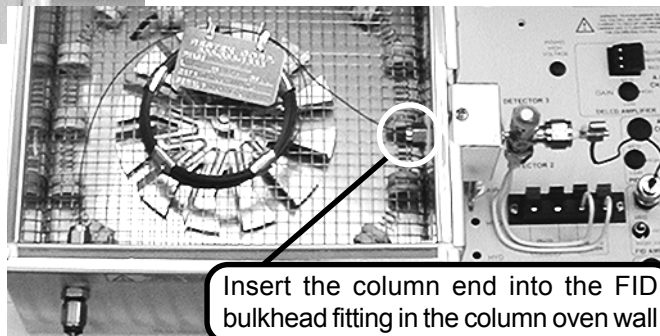
Connect the column to the TCD IN tubing



6. Slide another 1/8" Swagelok nut and graphite ferrule over the other end of the column. For a TCD detector, connect the nut to the fitting labeled "TCD IN" in the column oven.

For an FID detector, leave about 1" of the column protruding through the nut and ferrule. Insert the column into the FID bulkhead fitting in the column oven wall and tighten the Swagelok nut.

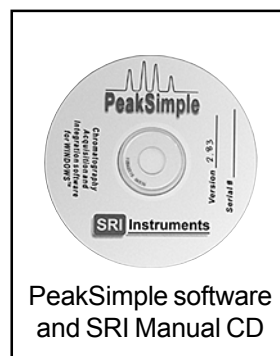
Please see "Analytical Column Installation" in the INSTALLATION section of your manual for more detailed information on column installation.



Quick Start GC Installation Guide

III. Software Installation

NOTE: There are tutorials in the manual and online at www.srigc.com (click on the “Download Our Documents” button) that will acquaint you with the basic functions of the PeakSimple chromatography software included with your GC. To view the manual, insert the CD into your computer’s CD drive, double click on “Manual Index” (an HTML document), then click on the manual title of your choice to view and print the PDF. You will need Adobe Acrobat Reader on your computer, a free download from adobe.com.



1. Connect the serial or USB cable to your computer and the GC. The serial port connection is on the left-hand side of the GC, and the USB connection is on the right-hand side.

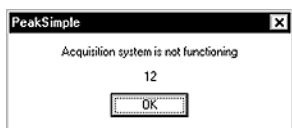


2. Locate your PeakSimple software and SRI Product Manual CD. Insert the CD into your computer’s appropriate drive.

3. Double click on “My Computer,” then double click on your computer’s CD drive to view the contents of the CD. To install PeakSimple, double click on the “setup***.exe” icon, and follow the instructions. (“***” represents the current version number of PeakSimple.)



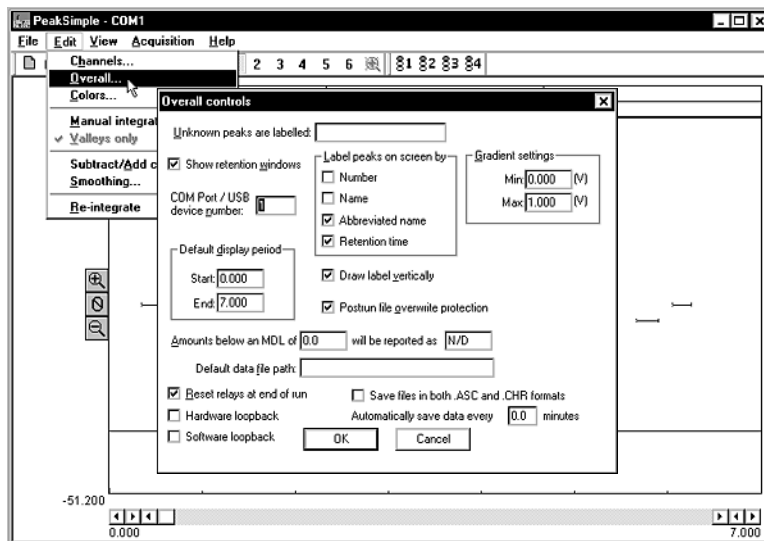
4. For USB, refer to “Installing the USB Drivers for Model 302 USB PeakSimple Data System” which you will find immediately behind these instructions in your manual, or online at www.srigc.com. Return to step #5 below when you are finished installing the USB drivers. For serial port, proceed to the next step.



5. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between the computer and the GC. An error message will appear if

communication is not established.

6. Open the Edit menu and choose Overall. In the dialog box that pops up, enter the number of the COM port to which you have connected the GC. For USB, enter the unique USB device number that is printed on your PeakSimple disk(s), and on the back of the GC. It is a 4-digit number that always begins with “5” (5093, 5276, etc.).



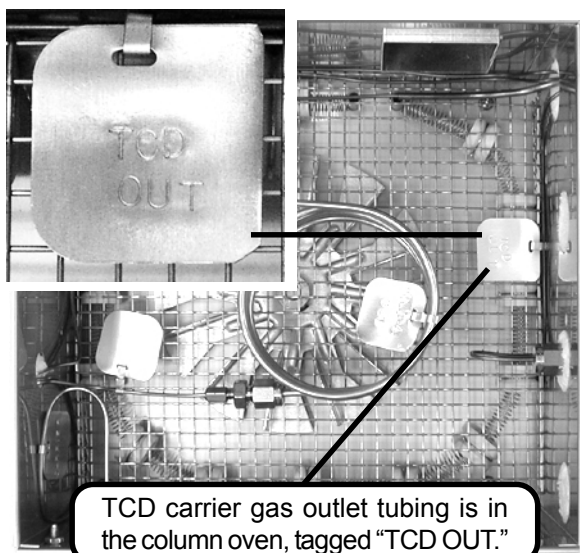
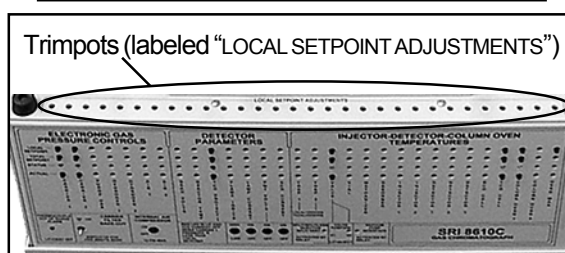
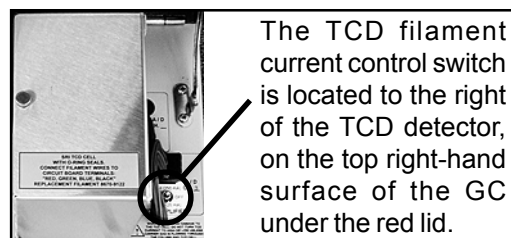
Quick Start GC Installation Guide

IV. Detector Activation

IMPORTANT: If you have a pre-configured GC system, please see the manual section for instructions on operating procedures. The manual is organized into sections with labeled tabs. In addition to preconfigured GCs, there are sections on detectors, injectors, autosamplers, valves, and more.

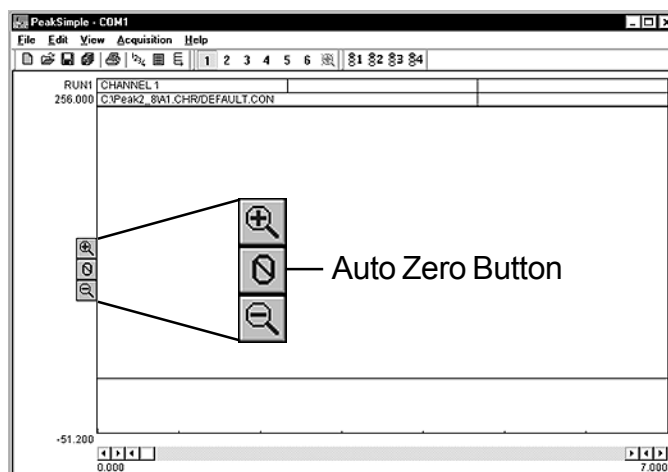
A. TCD Detector

1. Your GC power should still be ON, and the filaments should still be OFF. The TCD oven is set to 150°C at the factory. It is adjustable by turning the trimpot while observing the TCD CELL LOCAL SETPOINT temperature on the LED display. The trimpots are located on the top edge of the GC front control panel. Allow the TCD to reach desired operating temperature and stabilize.



2. TCD filaments will be damaged or destroyed if current is applied in the absence of flowing carrier gas. Therefore, always verify that carrier gas is exiting the TCD carrier gas outlet before energizing the TCD filaments. The TCD carrier gas outlet tubing is in the column oven, labeled "TCD OUT." Place the end of the tubing in some liquid; if no bubbles are exiting the tube, there is a flow problem. **DO NOT** turn the TCD current ON if you cannot detect carrier gas flow. 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. Correct any lack of carrier gas flow before proceeding.

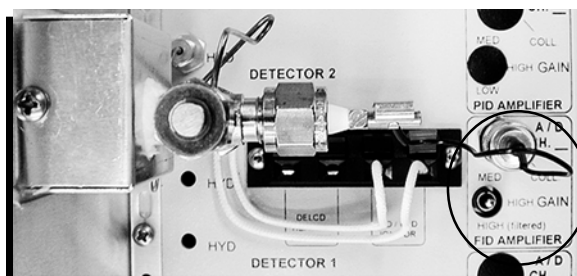
3. With the TCD filaments still OFF, zero the data system signal by clicking on the Auto Zero icon on the left side of the chromatogram. Switch the TCD current to LOW. The data system signal's deflection should not be more than 5-20mV for a brand-new TCD detector. There is also a HIGH current TCD filament setting, but to avoid filament damage, we recommend you use only the LOW setting until you are familiar with your GC and TCD detector.



Quick Start GC Installation Guide

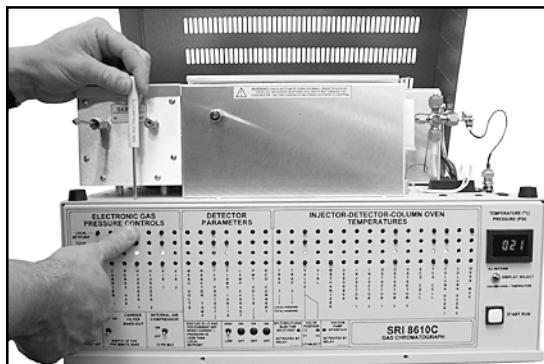
B. FID Detector

1. Set the FID amplifier gain switch to HIGH for most applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM.

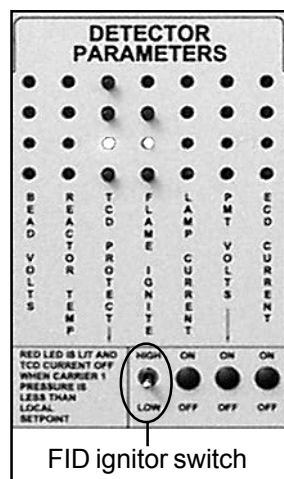
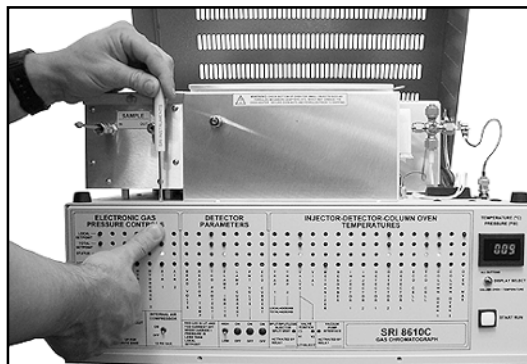


FID amplifier gain switch

2. Set the FID hydrogen flow to 25mL/minute, and the FID air to 250mL/minute. The approximate pressures required for this flow through your GC are labeled on the right-hand side of the GC chassis. In most cases, the pressure will have been set correctly at the factory. Check the hydrogen and air flow settings by pressing the LOCAL SETPOINT button while observing the LED display. The gas flow settings are adjusted using the trimpots on the top edge of the GC front control panel.

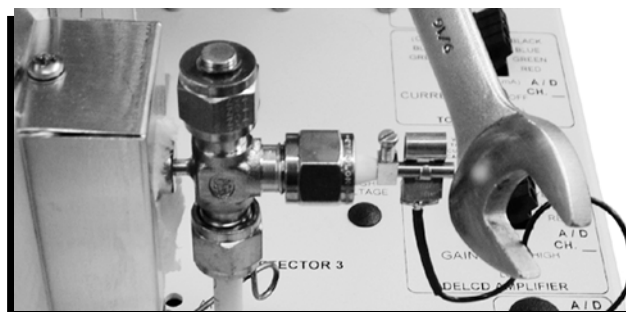


Turn the trimpot while holding down the "LOCAL SETPOINT" button until you read your desired setting in the LED display.



FID ignitor switch

3. Ignite the FID by holding the ignitor switch up 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, with a vertical label reading "FLAME IGNITE." Verify that the flame is lit by holding the shiny side of a wrench directly in front of the collector outlet/FID exhaust vent. If water condensation becomes visible on the wrench surface, the flame is lit.



C. For all other detectors, and for more information on the TCD and FID, please see the corresponding manual sections.

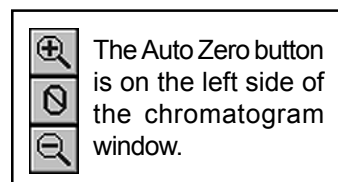
Quick Start GC Installation Guide

V. Inject Your Sample

NOTE: If you are injecting with a Purge & Trap, TO-14, or Headspace concentrator, a thermal desorber, an autosampler, or any of the heated on-column injectors (PTV, Split/Splitless, etc.), please see the corresponding manual section for operating procedures.

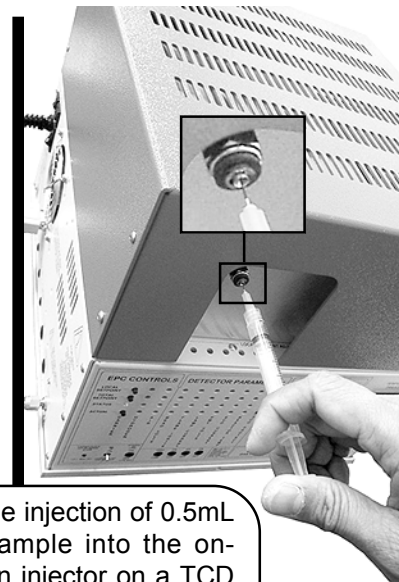
A. Syringe Injection

1. Enter a temperature program for the column oven. The temperature program is determined by the sample and the goals of the analysis.
2. For gas samples, fill the 1mL gas syringe with 0.5-1mL. For liquid samples, fill the 10 μ L liquid syringe with 1 μ L, removing the bubbles before injecting.



3. Click on the Auto Zero button to zero the data system signal. Hit the computer keyboard spacebar.

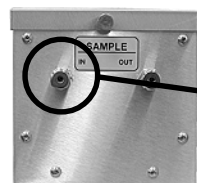
4. Pierce the septum in the on-column injector with the syringe needle. Insert the needle straight into the on-column injector port; avoid bending the needle. Depress the syringe plunger to inject the sample, then withdraw the syringe. For the best and most consistent results, use an easily reproducible injection technique with quick, smooth movements.



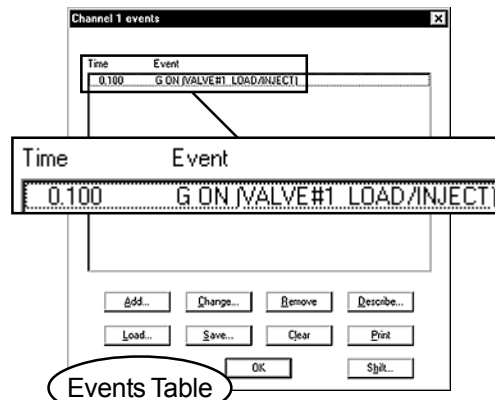
Syringe injection of 0.5mL gas sample into the on-column injector on a TCD equipped Model 310 GC

B. Valve Injection

1. Set the valve oven temperature between ambient and 175°C using the trimpot on the top edge of the front control panel. Enter a temperature program for the column oven.
2. Enter an event program to automatically inject the contents of the valve sample loop. The valve is usually in the LOAD position (default), during which Relay G is OFF. When relay G is activated, the valve is rotated to the INJECT position, in which the carrier gas stream sweeps the contents of the sample loop onto the column(s). Set the valve to INJECT (Relay G ON) 0.1 minutes into the run unless you have specific run parameters that require different timing.



3. Sample is injected into the bulkhead fitting labeled "SAMPLE IN" on the front of the valve oven. The fitting is equipped with a 1/8" Swagelok nut for easy connection of sample streams.

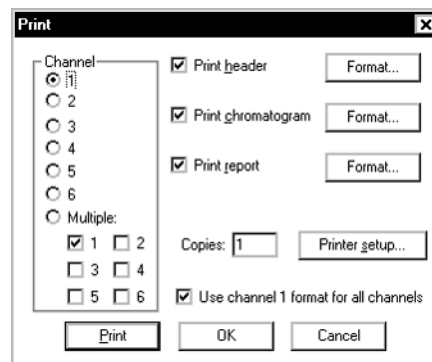


4. Press the computer keyboard spacebar to initiate the run. The valve will automatically rotate to the INJECT position at 0.1 minutes (or whatever time you entered in the Events Table).

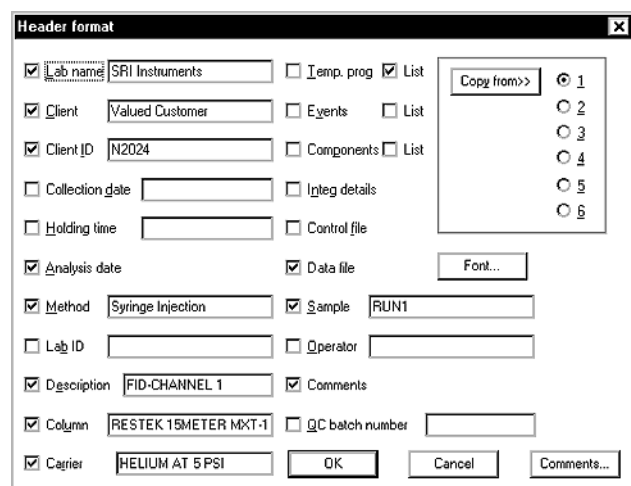
Quick Start GC Installation Guide

VI. Print Your Chromatogram

1. Choose File / Print from the main menu bar.
2. In the Print screen, designate which channel(s) you want printed. Use the radio buttons to pick a single channel, or select "Multiple:" and click the checkboxes to select the channels you want to print.



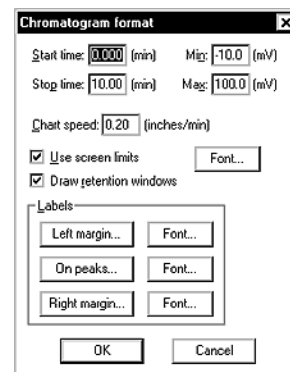
The Print dialog box contains a 'Channel' section with radio buttons for channels 1 through 6, and a 'Multiple:' section with checkboxes for channels 1 through 6. To the right, there are three checked checkboxes: 'Print header', 'Print chromatogram', and 'Print report', each with a 'Format...' button. Below these are 'Copies: 1' with a 'Printer setup...' button, and a checked checkbox 'Use channel 1 format for all channels'. At the bottom are 'Print', 'OK', and 'Cancel' buttons.



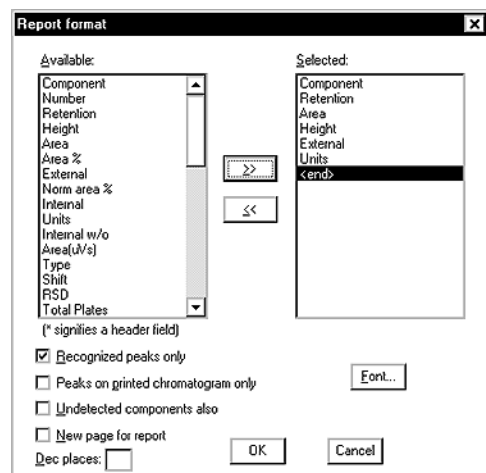
The Header format dialog box has a 'Copy from>>' list with channels 1 through 6. It contains various input fields and checkboxes for header information: Lab name (SRI Instruments), Client (Valued Customer), Client ID (N2024), Collection date, Holding time, Analysis date, Method (Syringe Injection), Lab ID, Description (FID-CHANNEL 1), Column (RESTEK 15METER MXT-1), Carrier (HELIUM AT 5 PSI), and others like Temp. prog, Events, Components, Integ details, Control file, Data file, Sample (RUN1), Operator, and Comments. It includes 'Font...' and 'Comments...' buttons, and 'OK', 'Cancel', and 'Comments...' buttons at the bottom.

3. Click the checkbox to select "Print header," then click on the "Format..." button to set up the Header. The Header is printed above the chromatogram on the page, and can contain such information as the analysis date, the sample and injection type, column and carrier gas used, client and lab names, and any special comments about the analysis that you want printed with the chromatogram. Click "OK" when finished formatting your header. The Print screen is still open.

4. In the Print screen, click the checkbox to select "Print chromatogram," then click on the "Format..." button. Choose "Use screen limits" to print the chromatogram as you see it onscreen. You can also choose the chart speed, which determines the number of inches per minute displayed in the chromatogram timeline. For example, if your chromatogram is 10 minutes long and you want it to occupy 5 inches on the paper, choose 0.5 inches/minute. Click "OK" when finished.



The Chromatogram format dialog box includes input fields for Start time (0.000 min), Stop time (10.00 min), Mix (-10.0 mV), Mag (100.0 mV), and Chart speed (0.20 inches/min). It has checked checkboxes for 'Use screen limits' and 'Draw retention windows', each with a 'Font...' button. A 'Labels' section contains 'Left margin...', 'On peaks...', and 'Right margin...' buttons, each with a 'Font...' button. 'OK' and 'Cancel' buttons are at the bottom.



The Report format dialog box features two lists: 'Available:' and 'Selected:'. The 'Available:' list includes fields like Component, Number, Retention, Height, Area, Area %, External, Norm area %, Internal, Units, Internal w/o, Area(uV/s), Type, Shift, FSD, and Total Plates. The 'Selected:' list contains Component, Retention, Area, Height, External, Units, and <end>. It has checkboxes for 'Recognized peaks only', 'Peaks on printed chromatogram only', 'Undetected components also', and 'New page for report'. It includes a 'Dec places:' field and 'Font...', 'OK', and 'Cancel' buttons.

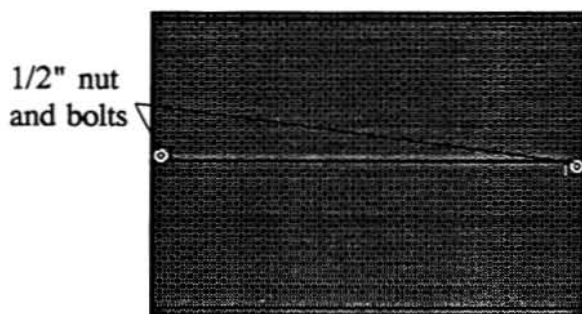
5. In the Print screen, click the checkbox to select "Print report," then click on the "Format..." button to choose the data that will be included in the report at the bottom, such as the component name, retention time, peak area and height, etc. Click "OK" when finished.

6. Now that your chromatogram is ready to print, click on the Print button in the Print screen.

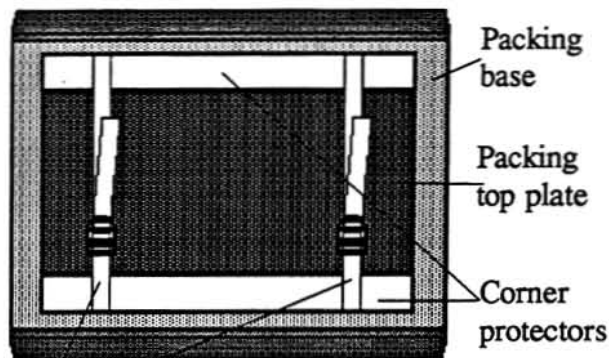
Chapter: INSTALLATION

Topic: Removing The Chromatograph From The Shipping Container

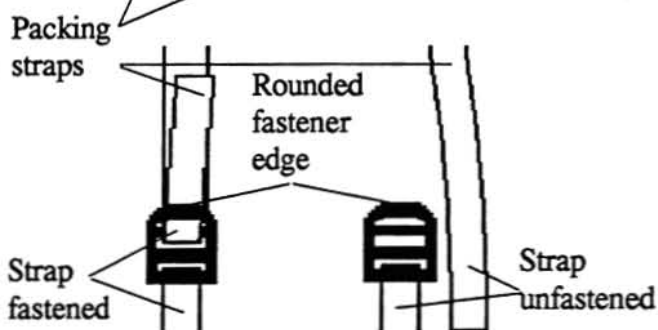
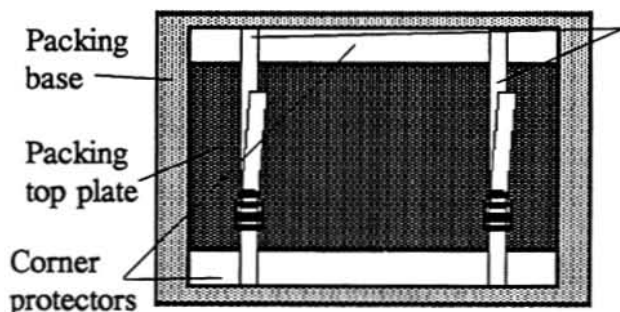
Top view of container, lid closed



Top view, lid open, GC in container



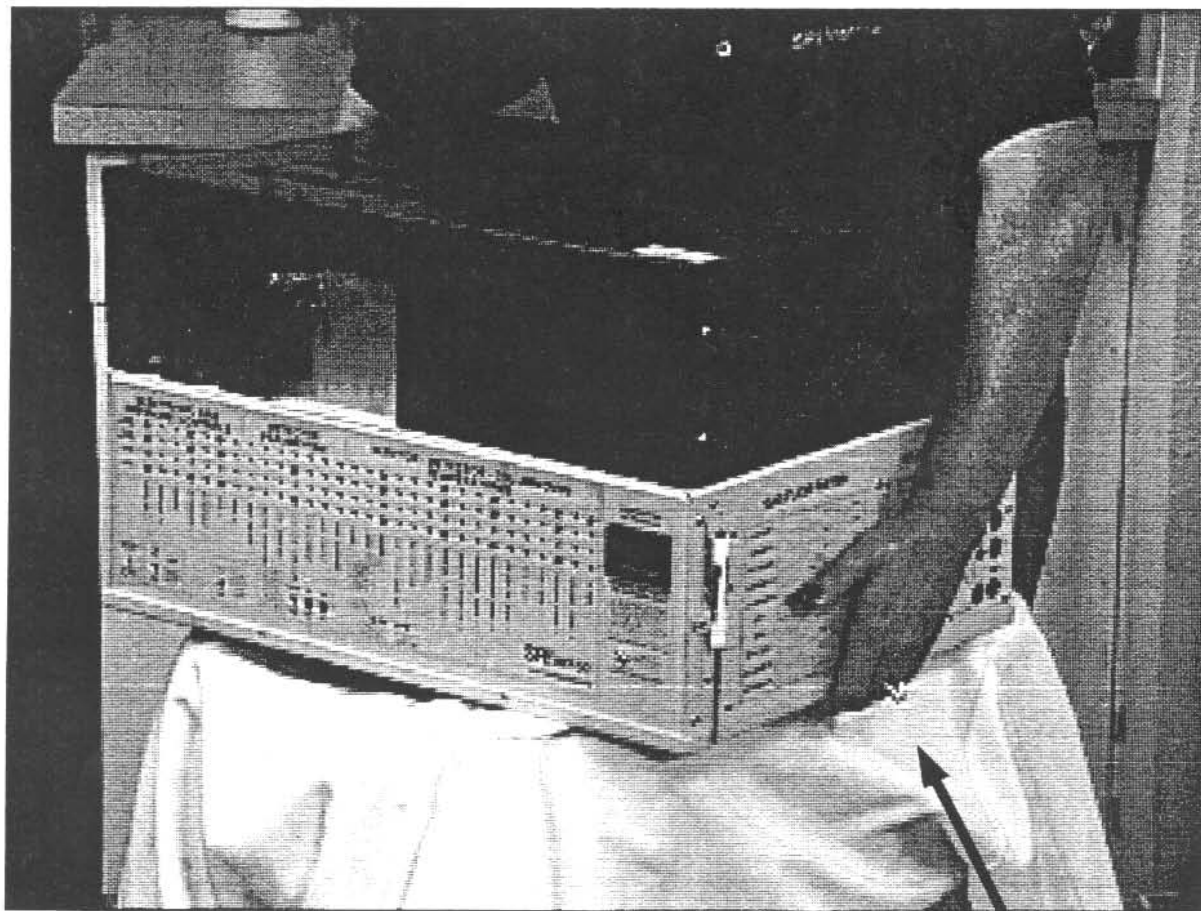
Top view, GC out of container



SRI gas chromatographs and stand-alone units are shipped in a sturdy, protective shipping container. The molded gray plastic container is reinforced and resistant to blows and crushing pressures typically encountered while en route to the customer or job site. Upon receipt, check to see that there is no obvious damage to the exterior of the shipping container. Notify the delivery person immediately of any such damage. The lid of the shipping container is secured closed by a 1/2" nut and bolt set each located on either side of the container. To open the lid of the shipping container, completely remove the two nut and bolt set and simply open lid. Screw the nuts back onto the bolts and place in shipping container for future use. The GC is held in place within the shipping container in custom packing material consisting of (1) rigid foam bottom packing base, (1) soft foam top plate, (2) cardboard corner protectors, and (2) straps with fasteners to bind GC within packing material. Some SRI GCs can weigh more than 70 pounds, and care must be taken to prevent injury when removing from shipping container. To properly remove the GC from within the shipping container, firmly grasp the two visible straps running across the soft foam top plate between the two cardboard corner protectors. Being careful to properly bend your knees, lift the entire GC, still contained within the packing material, straight up and out of the shipping container. To remove the packing material from around the GC, begin by removing the two straps holding it all in place. Place your fingers beneath the rounded strap fastener edge and pull up and back. When the strap loosens up, pull the free end of the strap completely through the fastener. Once both straps have been unfastened, remove the two cardboard corner protectors along with the soft foam top plate and place back in the empty shipping container for safe keeping. Next, slide your fingers between the metal GC base plate and the rigid foam bottom packing base, and firmly grasp the bottom of the GC with both hands. Once again being careful to properly bend your knees, lift the GC up and out of the packing base. Place the packing base, with straps still attached, in the shipping container with the other packing materials. Be sure to save all packing materials along with the shipping container for all future shipping needs.

Chapter: Installation:

Topic: Lifting the 8610C and 310 GCs

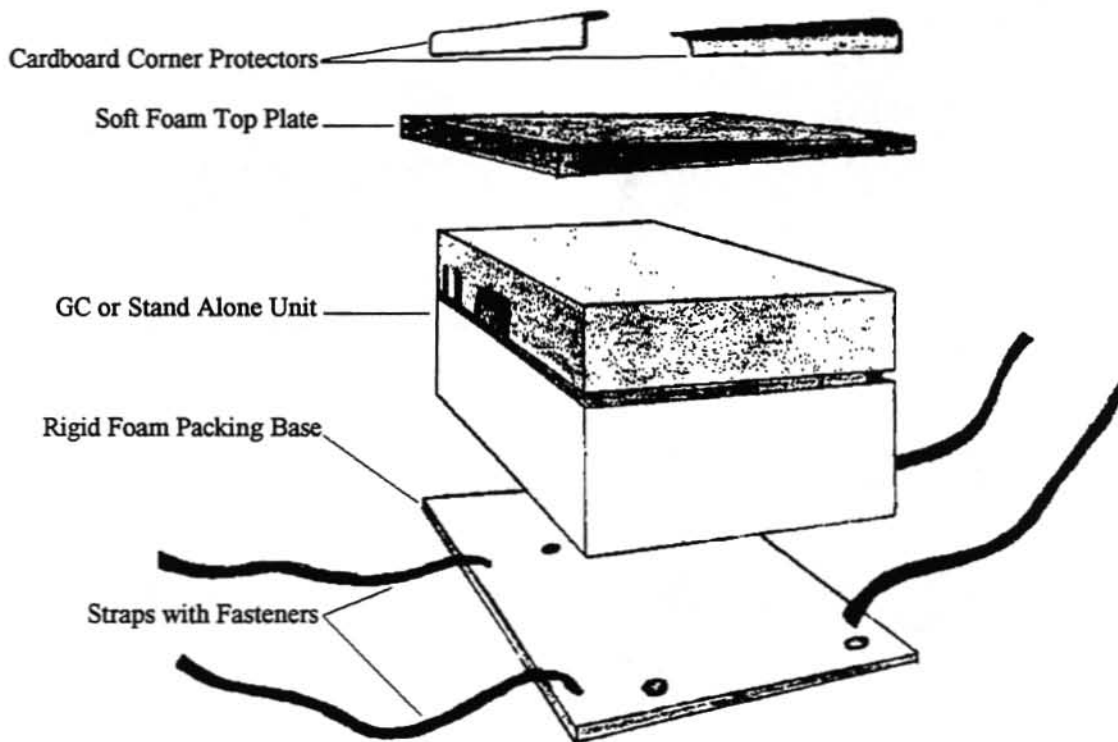


Lift here

As illustrated by the photo above, lift the 8610C and 310 GCs by grasping the GC under each side. Before lifting, check to make sure the bottom cover is securely attached to the chassis with six screws, and that the power cord, gas line connections and serial port cable are disconnected.

Chapter: Installation

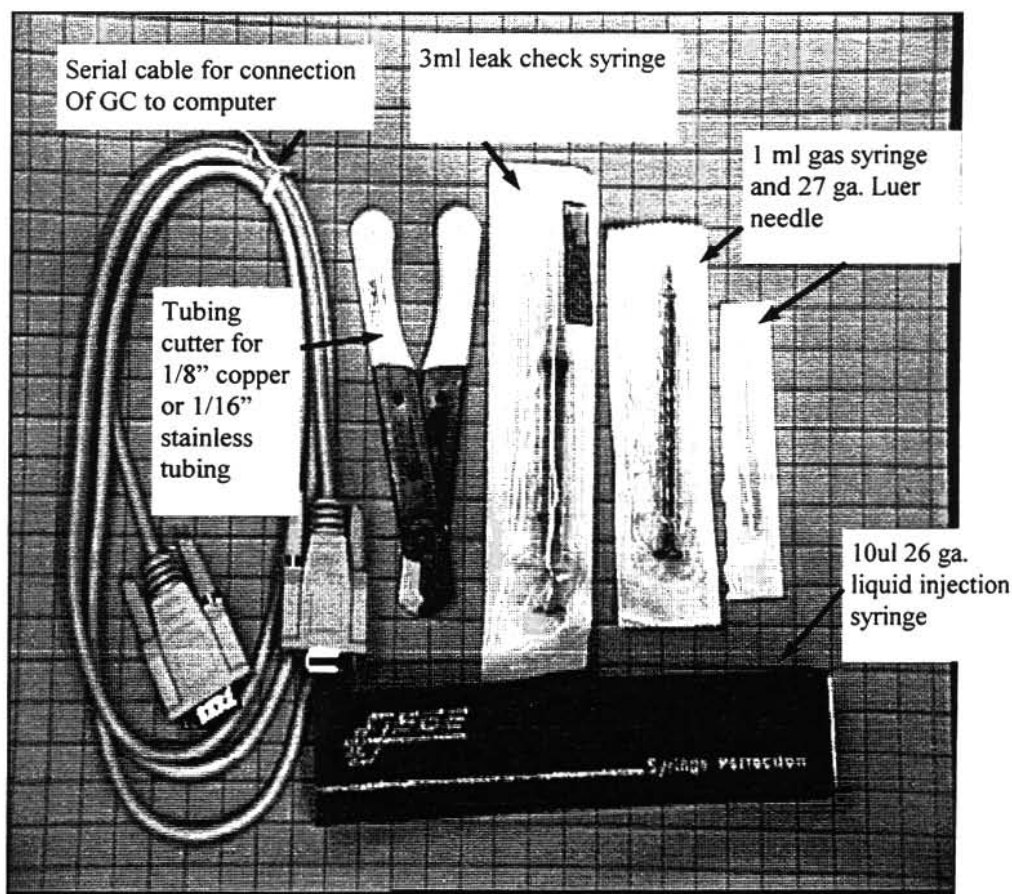
Topic: Repacking Your Gas Chromatograph or Stand Alone Unit For Safe Shipping



When reshipping an SRI GC or stand alone unit, be sure to use the original shipping container and all of the original packing material to minimize the potential for damage during shipment. First, make sure that you have all of the primary packing pieces: (1) molded gray shipping container, (1) rigid foam bottom packing base with (2) straps and fasteners, (1) soft foam top plate, and (2) cardboard corner protectors. To properly pack your GC or stand alone unit, begin by placing the bottom packing base flat on the floor with the straps coming up through the surface of the base as shown in the diagram. Place your GC on top of the base with the legs inserted in the appropriate cutouts. Next, place the soft foam top plate on top of the GC and place the cardboard corner protectors over the soft foam top plate. Pull the straps coming through the packing base up and around the GC, as well as all the other packing material and secure the two strap ends together. It may be helpful to straddle the GC and use your knees to squeeze all the packing material together as you firmly tighten the straps. Be sure the straps firmly secure the GC or stand alone unit in the packing material to properly protect your instrument. When you are sure the straps are firmly and securely fastened, grasp the two straps running across the soft foam top plate between the two corner protectors. Properly bend your knees and lift up the entire GC, contained within the packing material, and gently place into the molded gray shipping container. Place bubble-wrap in the remaining empty spaces within the container to prevent any potential shifting during shipment. Also, include a packing slip inside, as well as one on the outside of the container, and then close the lid. Lastly place the 1/2" bolts in the two holes each side of the top surface and properly secure the lid closed with the 1/2" nuts. It is also important to properly insure your GC with the shipping company due to its high value. Your GC is now ready for safe shipping.

Chapter: INSTALLATION

Topic: Contents of Accessories Kit included with GC



Contents of accessories kit shipped with new SRI GCs.

- 1) 6' DB-9 serial cable for connection of GC to computer (Student model without data system will not have this item).
- 2) Tubing cutter for easy installation of 1/8" copper or 1/16" stainless steel tubing
- 3) 3ml leak check syringe (fill with alcohol/water mix to check fitting for leaks)
- 4) 1ml gas syringe and needle for injection of gas samples into GC
- 5) 10ul liquid injection syringe

Chapter: INSTALLATION

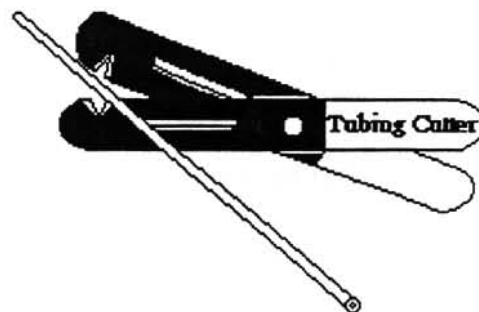
Topic: Tubing Cutter - For Facilitating Gas Connections

Included in the optional gas line installation kits that may be purchased with each SRI Instruments gas chromatograph is a disposable tubing cutter. This tool is capable of producing clean, fast cuts in chromatography tubing that rival more time-consuming tubing cutting methods. The hardened, beveled cutting surface of the tool enables the user to effect a through-and-through cut upon the tubing in one motion, cutting copper and stainless steel tubing with ease. The cut obtained allows both metal and graphite ferrules to slide onto the tubing without the normal filing or reaming necessary after cutting tubing using other methods. No smearing or burring is produced if this tool is used as directed.

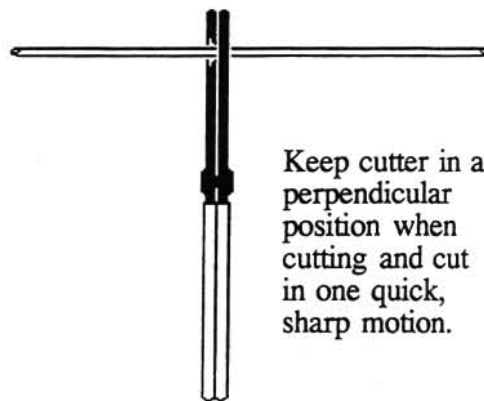
Users can make up to ten connections in the time that it took to cut, file, ream and connect one single tubing connection. Since the tubing is sheared and not twisted or stressed, the inside passage is not deformed or restricted, enabling the user to cut very small internal diameter stainless steel tubing (such as 1/16" O.D. x 0.005" I.D.) that would likely collapse or otherwise become restricted when cut by any other tool. Cuts on very small tubing is seldom attempted due to the difficulty encountered using ordinary methods. By using this tool, delicate tubing cuts become as easy and routine as larger tubing cuts.

Tubing cuts in tight or hard-to-reach locations can be performed without difficulty with the use of this tool. Since the cutting head is practically flat and requires relatively little clearance, it can be inserted into otherwise difficult spots to perform high precision cuts. As an example, if gas tubing routed through a hard-to-reach area inside the gas chromatograph required cutting for the insertion of an adapter or other special fitting, the cutter head could be inserted to the location and the cut achieved without having to disassemble and relocate or remove the adjacent hardware blocking access to the tubing. Once cut, the tubing ends could be reached with another tool, such as a needle-nosed plier, and pulled to gain accessibility for the installation of the fitting.

When making cuts, the tubing should be located between the two "jaws" of the cutter, making sure that the cutter grabs the tubing in the "V" notches located on the blades. The cutter should be held completely perpendicular to the tubing at the time the cut is made, to avoid obtaining a bad angle on the tubing end. Care should be exercised to avoid pinching the fingers or hand when operating this tool, as with any other hand-held cutting tool.



TO USE: Locate the tubing to be cut between the beveled cutting surfaces while maintaining the cutter at an angle completely perpendicular to the tubing. Holding the cutter steadily, cut the tubing in one quick, hard motion. Do not hesitate during the cut to prevent any possible twisting of the blades or the tubing. This cutter cuts 1/8" and 1/16" copper and stainless steel with ease. After extended use, especially when used to cut stainless steel tubing, the cutter blade will become dull. Discard and replace the tubing cutting tool when this occurs to prevent damage to any future tube cuts.



Keep cutter in a perpendicular position when cutting and cut in one quick, sharp motion.

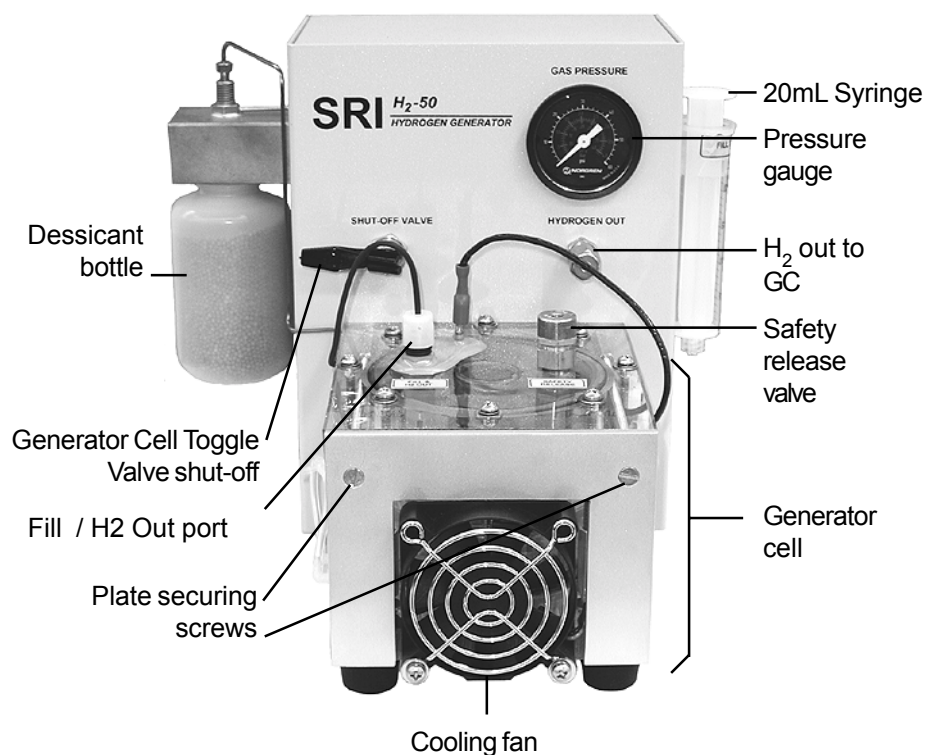
GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

Overview

Your SRI H₂-50 Stand-Alone Hydrogen Generator consists of a generator cell mounted on a metal chassis. The generator cell is attached to the chassis with two screws for easy disassembly—just unscrew them, unplug the power cord, and the entire cell comes off. On the metal chassis is a pressure gauge, an interior pressure switch, a dessicant bottle, and a toggle valve shut-off for isolating the generator cell. The dessicant bottle contains Indicating Molesieve dessicant beads which turn from blue to gray when they absorb water. Water vapor that is released from the generator cell with the hydrogen is removed by the dessicant before reaching the GC column, thus drying the hydrogen gas. The H₂-50 can supply enough gas for a detector or two as well as the GC carrier gas. During operation, there is about 40mL of hydrogen gas stored in the dessicant, which is enough to operate a split injector for short periods, in addition to the detector(s) and carrier. The toggle valve shut-off facilitates checking for leaks and allows the H₂-50 to reach operating pressure more quickly, while the interior pressure switch maintains the operating pressure. As a safety measure, a pressure release valve protects the generator cell from pressure overload. An external power supply/transformer that is provided enables the H₂-50 to operate on various voltages around the world. You may use any approved power supply rated 100-240VAC with 12VDC, 7amp output. Conveniently, the H₂-50 produces 50mL/min at 35psi (241316Pa, 2.4bar) using distilled water from the grocery store.

The SRI H₂-50



Approved external power supply



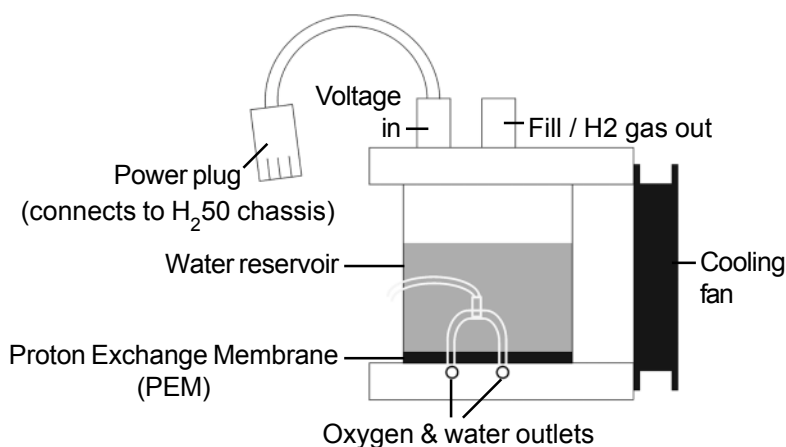
GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

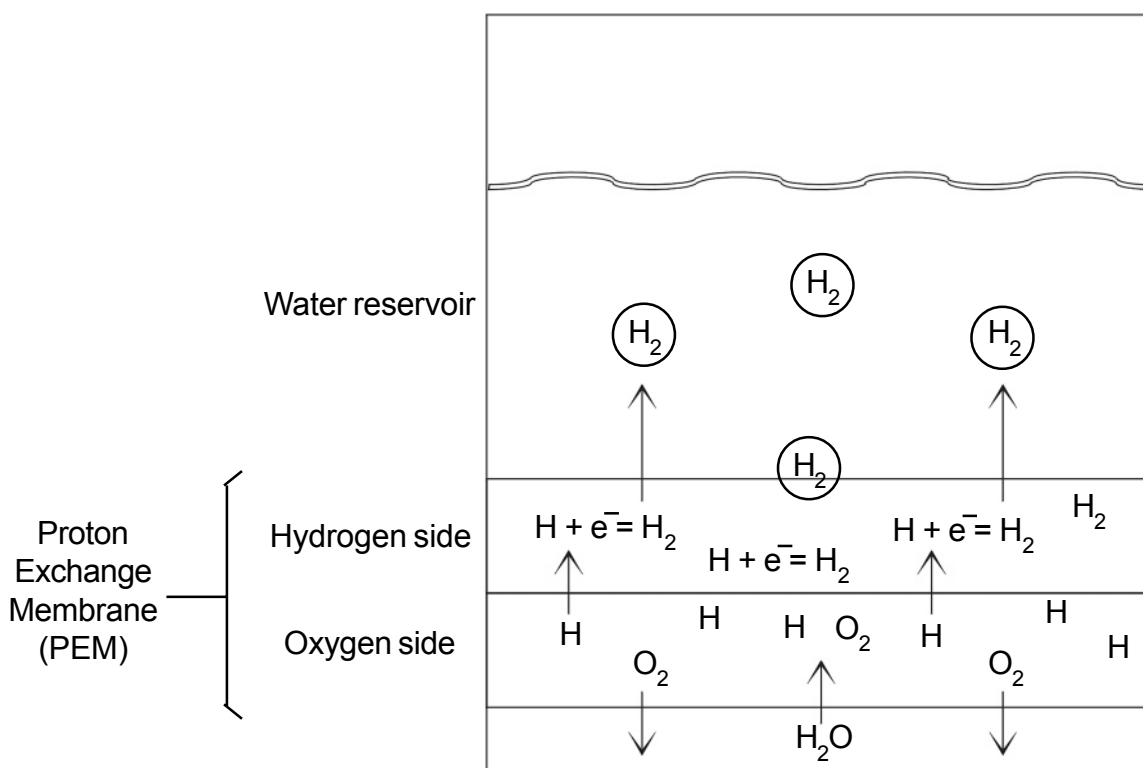
Theory of Operation

The SRI H₂-50 Hydrogen Generator separates water into hydrogen and oxygen using a Proton Exchange Membrane (PEM). The water on the oxygen side of the PEM is disassociated into O₂ and hydrogen protons. The hydrogen proton is transported through the PEM to the hydrogen side, where it recombines with an electron to make H₂, then bubbles up through the water reservoir.

The H₂50 Generator Cell



Operational Diagram of the H₂50 Generator Cell



GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

General Operating Procedure

Use the H₂-50 on a flat, level surface, away from open flame and any other ignition sources, including spark sources.

1. Remove the nut with the septum from the Fill / H₂ Out port on the top of the generator cell.
2. Use the 20mL syringe mounted on the right-hand side of the H₂-50 chassis to inject clean distilled water into the water reservoir. Although clean tap water will work in a pinch, use distilled water whenever possible. Fill only to the top fill line; do not overfill. Replace the nut and septum on the fill port and hand tighten until the nut contacts the black o-ring on the fitting.
3. Make sure the dessicant bottle contains dry beads. Dry dessicant beads are blue in color; they turn grey when wet. See below for instructions on recharging and replacing the dessicant beads.
4. Connect the H₂-50's "H2 OUT" fitting to the GC's hydrogen gas inlet. Output from the H₂-50 "HYDROGEN OUT" fitting is connected to the GC with 1/8" or 1/16" O.D. tubing. Make sure the red and black power cord is plugged into the H₂-50 chassis, and connect the external power supply cord to the generator and a wall outlet. Make sure you have the correct input cord for the voltage you are using. Properly used, the transformer is not a spark source and poses no ignition threats.
5. Close the H₂ shut-off valve. Always build up pressure initially with the toggle valve shut; it will take 5-15 minutes.
6. The H₂ gas pressure is preset to 35psi (241316Pa, 2.4bar). Once this pressure is attained, the interior pressure switch will shut off the current to the generator. The water in the generator cell reservoir should stop bubbling.
7. Wait 10 minutes to make sure that 35psi (241316Pa, 2.4bar) pressure is maintained. If pressure is not being maintained, there is probably a leak. Check the dessicant bottle; it should be snug against the o-ring. Make sure the Fill / H₂ Out port nut and septum are intact and snug. Check the bottom of the water reservoir around the PEM for moisture to ensure generator cell integrity; if you find any seepage, tighten each of the eight screws that hold the cell layers together.
8. If you find no indication of a leak after 10 minutes of stabilization at 35psi (241316Pa, 2.4bar), open the toggle valve to let the H₂ gas flow into the GC.
9. When the water in the generator cell water reservoir reaches the bottom fill line, it is time to refill it.
10. Close the toggle valve.
11. Unscrew the Fill / H₂ Out port nut and septum and use the syringe to refill the cell to the top fill line.
12. Replace the nut and septum, and tighten until snug.
13. Since you have the cell pressure vented, it is a good idea to check the dessicant for any grey coloring to see if the beads need recharging. If they do, follow the instructions on the next page (***General Operating Procedure continued***).

GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

General Operating Procedure continued

Recharging and Replacing the Dessicant Beads

Periodic recharging of the dessicant beads will be necessary as they absorb water during operation and turn grey. The blue color of the dry beads comes from cobalt chloride. Take care not to bake out the dessicant beads with any food item.

1. Before you loosen the dessicant bottle on the H₂-50 chassis, vent the hydrogen pressure in the generator cell by unscrewing the nut capping the fill port on the top of the cell. It will hiss audibly until it is released.
2. Dry the dessicant beads by pouring them onto a paper plate and cooking them in a microwave oven for 2-3 minutes. Or, pour them onto a glass or metal pan and bake them in the GC oven at 250°C. Do not microwave or bake the plastic dessicant bottle. The dessicant beads can be recharged over and over again; they last indefinitely. Should you need them, dry dessicant beads are available in kilogram quantities from Alltech (1-800-ALLTECH; part # 05553).
3. Let the beads cool, especially after microwaving them. Refill the dessicant bottle with the dry, blue beads.
4. Replace the bottle on the H₂-50 chassis and hand tighten it. There is an o-ring that engages with the bottle top; tighten the bottle until it is snug against the o-ring.
5. The dry dessicant contains some air which will purge out during the first few minutes of operation. You may notice your retention times change temporarily since the carrier gas may initially be a mixture of hydrogen and air for a few minutes after dessicant replacement. The FID flame may also be hard to light until pure hydrogen comes through. You can speed up this equilibration process by building up pressure in the generator cell then venting with the toggle valve 2-3 times before reconnecting the H₂-50 to the GC. Keep in mind that the internal pressure switch will cut the current when the cell reaches 35psi (241316Pa, 2.4bar), so you don't need to build up too much pressure before venting it. Experiment to learn what works best for your particular GC system.

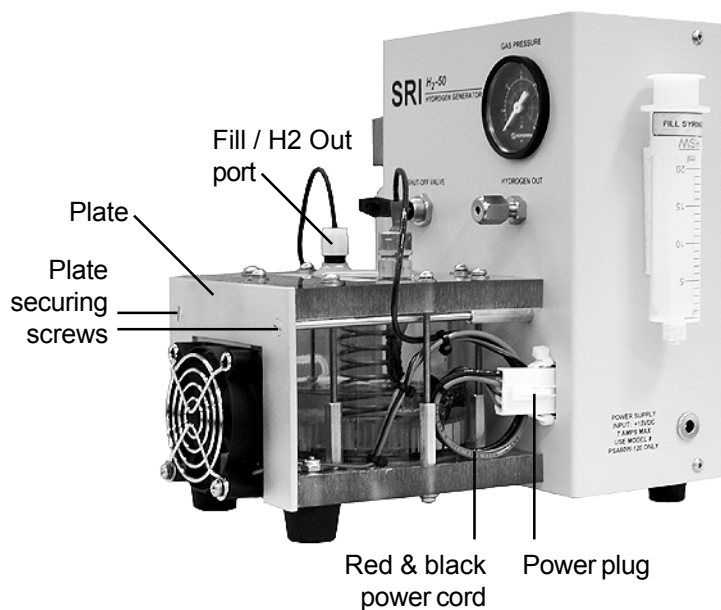
GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

Maintenance and Troubleshooting

If the water in the H₂-50 water reservoir looks cloudy, it needs to be replaced:

1. Remove the nut with the septum from the Fill / H₂ Out port on the top of the generator cell.
3. Turn the generator over and pour the water out. When the water is almost all out, shake the generator to help it drain.
4. Use the syringe to refill the water reservoir with clean, distilled water through the Fill / H₂ Out port.
5. Replace the nut and septum on the Fill / H₂ Out port.



See the following page for PEM replacement

For service, call 310-214-5092.

GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

Maintenance and Troubleshooting continued

If the Proton Exchange Membrane (PEM) changes color, it most likely needs to be replaced. New H₂-50 PEMs are available from SRI under part # 8690-0151.

1. Put the replacement PEM in clean distilled water to soak while you take apart the generator cell.
2. Remove the generator cell from the H₂-50 chassis by unplugging the red and black power cord from the chassis, and unscrewing the two screws that hold the clamping plate against the cell.

3. Loosen the eight screws that hold the water reservoir with a philips head screwdriver. Loosen each screw in increments; first one, then the one opposite, and so on in a star-like pattern. As you progress, be mindful of the spring in the water reservoir; don't loosen the screws too suddenly, or it may pop open the reservoir, presenting safety and damage risks. You can feel the pressure of the spring relax as you loosen the screws sufficiently; hold the top of the generator cell firmly with one hand while loosening the screws with the other.

4. Once the screws are removed, carefully take the water reservoir off the bottom of the cell and remove the old PEM. Be very careful handling and moving the graphite coil, as it can easily come apart.

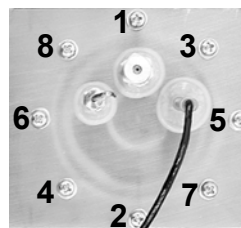
5. Take the new PEM out of its bath and position it centrally within the ring of screws. Place the water reservoir back on the bottom, over the PEM; the PEM should protrude slightly on all sides of the water reservoir.

6. Once the PEM is properly positioned, tighten the screws in increments until the water reservoir is snug against the bottom of the generator cell.

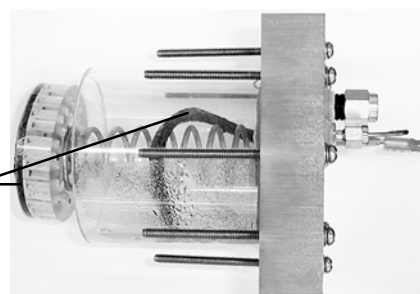
7. Put the generator cell back on the chassis and secure it with the plate and two screws. Plug the red and black power cord into the chassis.

8. Plug the H₂-50 into a wall outlet and pressurize the generator cell to 30psi. Check the bottom of the water reservoir around the PEM for moisture; if you see any seepage, tighten each of the eight screws a little more.

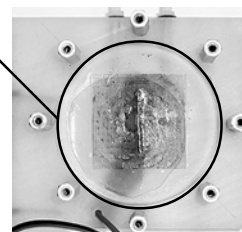
Incrementally
loosen the
screws in this
pattern:



Handle the
water
reservoir
with care;
the
graphite
crumbles
easily



Position the PEM
centrally within
the circle of
screws



GC ACCESSORIES

H₂-50 Stand-Alone Hydrogen Generator

WARNING!



Warnings and Safety Precautions:

The H₂-50 generates hydrogen, which is an extremely flammable gas. Under normal operation, the safety features of the H₂-50 protect the operator. However, operators must use common sense and take basic precautions. Hydrogen burns with a flame that is invisible to the naked eye. Do not use the H₂-50 near any flames, sparks, or sources thereof, including lab ovens, heater elements, bunsen burners, torches, etc. When venting the hydrogen from the generator cell, NEVER open the H₂-50 toggle valve near an ignition source!

Hydrogen is non-toxic, but it can cause asphyxiation in confined spaces by displacing oxygen. Use the H₂-50 in a ventilated room with an ambient temperature of 5-40°C (40-100°F). If the GC power is interrupted or cut off during hydrogen generation, flip the toggle valve to isolate the generator cell, then disconnect the external power source from the H₂-50 and the wall outlet. This is a good general response in any situation of uncertain risk; if you're not sure what's happening, isolate the cell and pull the power plug. That way, you can take the time to diagnose any problems without H₂ accumulation. Familiarize yourself with the safe operation of the GC and other equipment to which you intend to connect the H₂-50.

The H₂-50 is designed to be safe under the following Environmental Conditions:

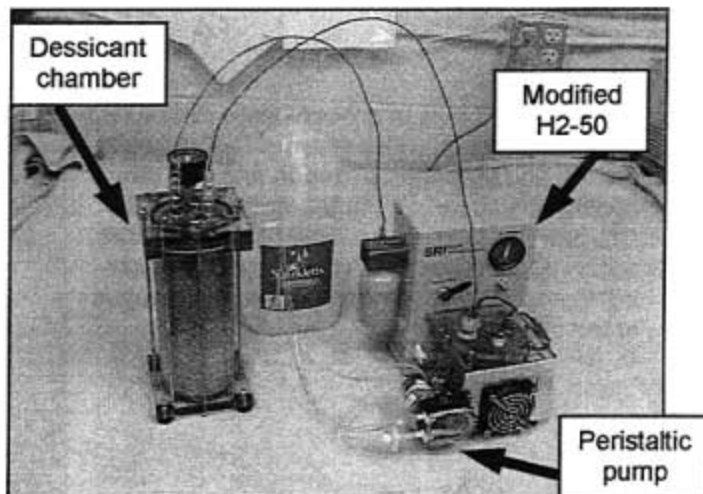
- indoor use;
- altitude up to 2000 meters;
- temperature 5°C-40°C;
- maximum relative humidity 80% for temperatures up to 31°C, decreasing linearly to 50% relative humidity at 40°C;
- POLLUTION DEGREE = 2 in accordance with IEC 664.

WARNING!

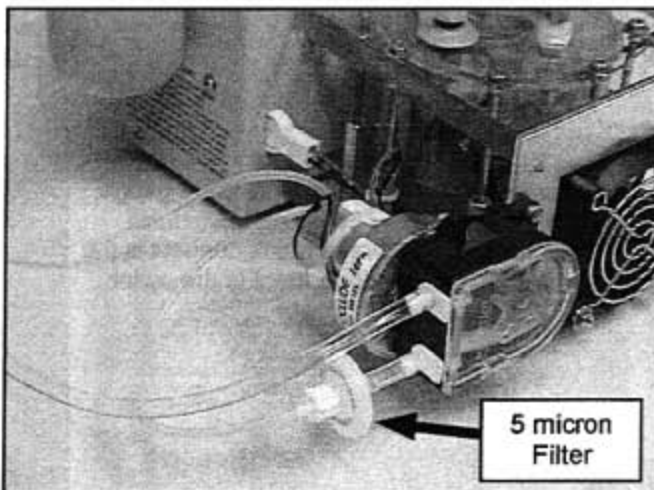
Chapter: Hydrogen Generator

Topic: Using the Extended Run H2 Generator kit

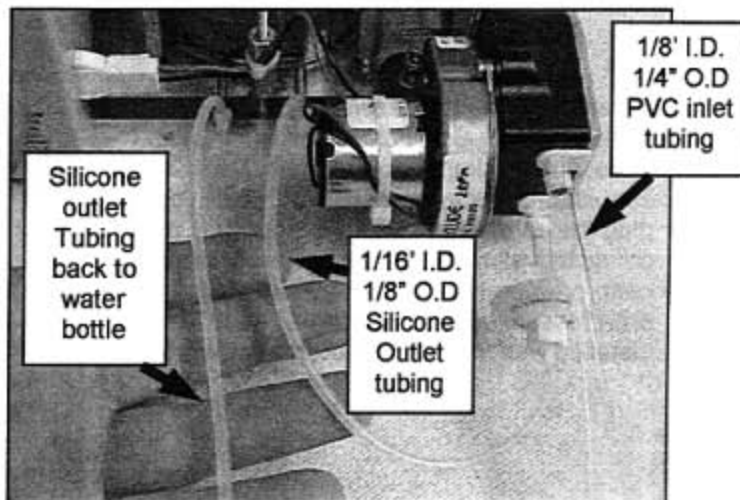
The SRI Extended Run kit for the H2-50 Hydrogen Generator consists of a modified Hydrogen generator cell and electronics, a peristaltic pump, and a large dessiccant chamber. The extended run cell comes equipped with a water level sensor which turns the peristaltic pump on and off automatically to maintain a constant water level inside the cell. A one gallon bottle of grocery store quality distilled water is sufficient for two months or more of operation. The large dessiccant chamber holds about two pounds of indicating mole sieve dessiccant. This quantity of dessiccant is also enough for two month continuous operation at 40ml/min or longer if the H2 flow requirement is lower. In the photo to the right, you can see the bottom third of the chamber has turned grey after one month of use.



The peristaltic pump re-circulates the distilled water from the bottle past the oxygen side of the PEM (proton exchange membrane). Electro-osmotic drag pulls the water through the membrane to the hydrogen side. When the water level rises to the tip of the water level sensor, the peristaltic pump shuts off. Excess water is returned to the distilled water bottle.



By pumping the water across the oxygen side of the PEM instead of directly into the water reservoir we can avoid pumping against the 30 psi of H2 pressure in the water reservoir, which is hard on any pump and prone to leaks. The oxygen side of the membrane is at ambient pressure, so a simple peristaltic pump can be expected to work reliably and for a long time. A disposable 5 micron 25mm syringe filter is used to prevent clogging of the passageways inside the H2 generator from dust and small fibers which seem to find their way into the water reservoir despite all precautions. This filter should be changed whenever the dessiccant is re-generated. Almost any brand of syringe filter is OK to use, but we supply a Millipore Millex-LS part# SLLS025NS.



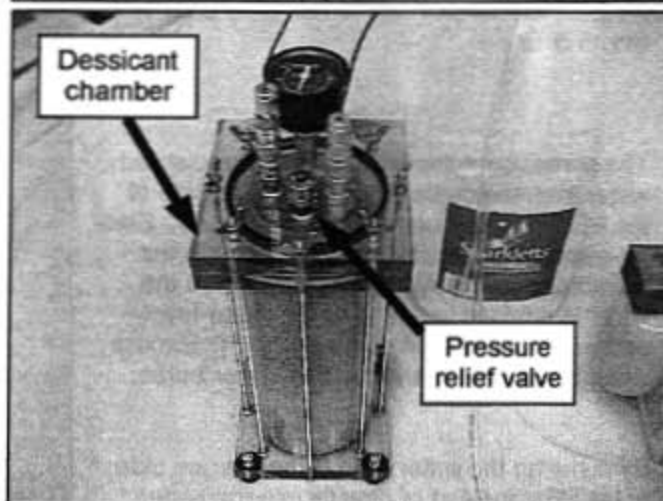
Chapter: Hydrogen Generator

Topic: Using the Extended Run H2 Generator kit

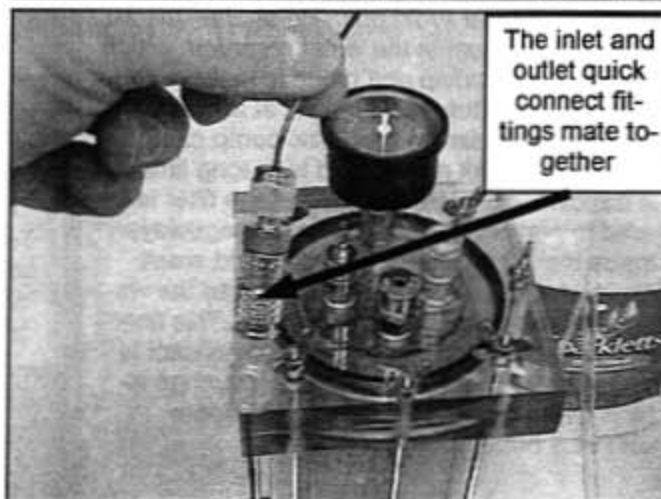
A one gallon bottle of grocery store distilled water makes a good water reservoir. Cut holes in the top and feed the 1/4" tygon and 1/8" silicone tubes all the way to the bottom. Put the cap back on to keep dust and fibers out of the water. Most pump problems result from clothing fibers clogging the internal water passages of the H2 cell.



The dessicant chamber has a pressure gauge, pressure relief valve (45psi) and two quick connect fittings. The brass quick connect is the inlet and the silver quick connect is the outlet.



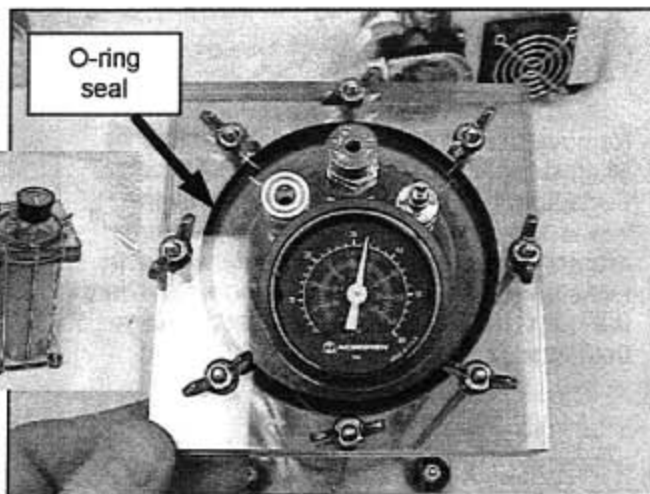
When changing the dessicant, or if you want to bypass the dessicant chamber, the inlet and outlet fittings simply plug together. The dessicant chamber stays pressurized when you unplug the connections. This is important because this allows you to re-generate the dessicant, pre-purge the air out and leave the unit pre-pressurized with hydrogen, ready to be re-installed with minimum system down-time.



Chapter: Hydrogen Generator

Topic: Using the Extended Run H₂ Generator kit

Many customers opt to purchase a spare dessicant chamber so that when they arrive on-site to perform the monthly or periodic maintenance, they have a pre-charged dessicant chamber which they can swap right into the system. This avoids the down-time which would otherwise result from the time it takes to purge air out of the chamber after re-generating the dessicant beads. Since the H₂-50 makes a maximum of 50 ml/minute, this can take hours. It makes more sense to swap dessicant chambers in the field and re-generate the old chamber back in the lab.

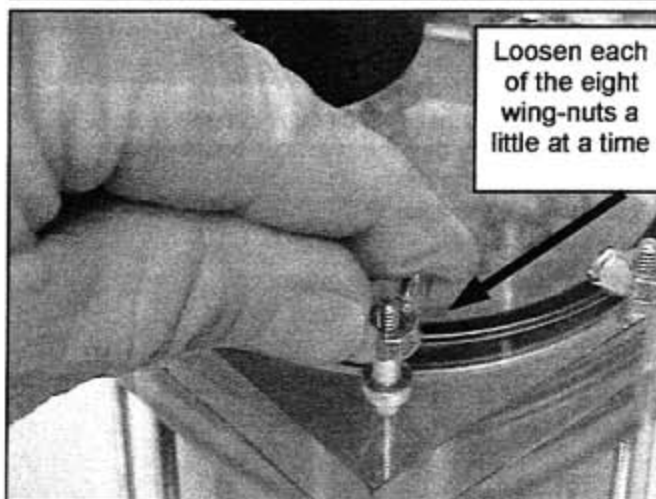


The dessicant chamber has eight wing-nuts which secure the top, compressing a rubber o-ring which seals in the pressure.

To change or re-generate the dessicant beads, release the pressure in the dessicant chamber by pushing the button on the top of the silver outlet quick connect fitting. Verify (using the pressure gauge) that the pressure has bled down to ambient before removing the nuts.



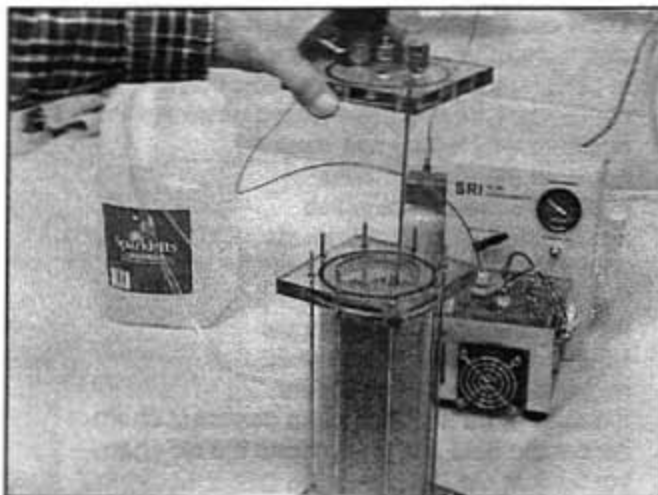
Loosen the wing-nuts evenly. Loosen each wing-nut a little bit at a time before removing any single wing-nut. This protects the plastic top from un-necessary stress.



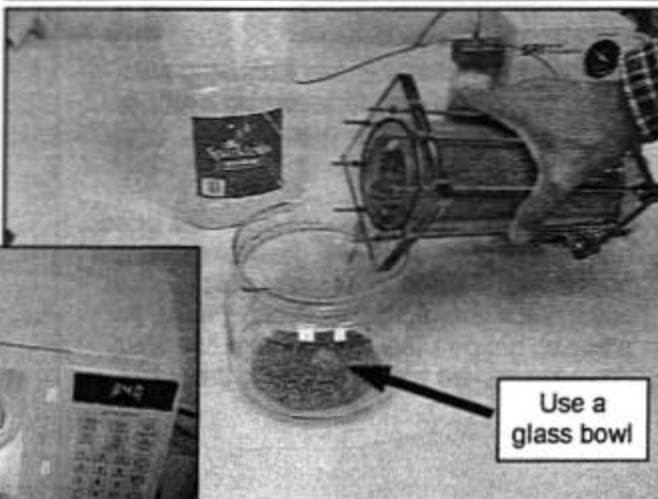
Chapter: Hydrogen Generator

Topic: Using the Extended Run H₂ Generator kit

Remove the top of the dessicant chamber by lifting straight up. The brass inlet quick connect fitting has a tube which extends all the way to the bottom of the dessicant chamber. Inspect the outlet at the bottom of the tube to make sure it is not plugged or blocked. (There is a metal frit in the tube to prevent blockage from dust).



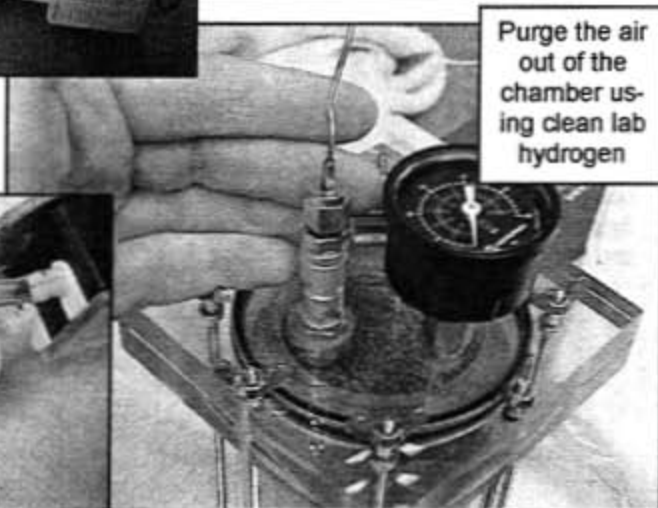
Pour the dessicant beads into a glass bowl. Don't use a plastic or metal bowl. Microwave the beads for 5-10 minutes until the blue color returns. **DO NOT USE A MICROWAVE WHICH IS ALSO USED FOR FOOD.**



The beads will be very hot when you remove them from the microwave oven, so allow them to cool, then pour them back into the dessicant chamber. **DO NOT PUT THE DESSICANT CHAMBER INTO THE MICROWAVE OVEN.**



Re-assemble the dessicant chamber and then purge the chamber with clean hydrogen from a cylinder. Verify that the chamber holds pressure by watching the pressure gauge after pressurizing the chamber to 30 psi.



Don't forget to change the filter.

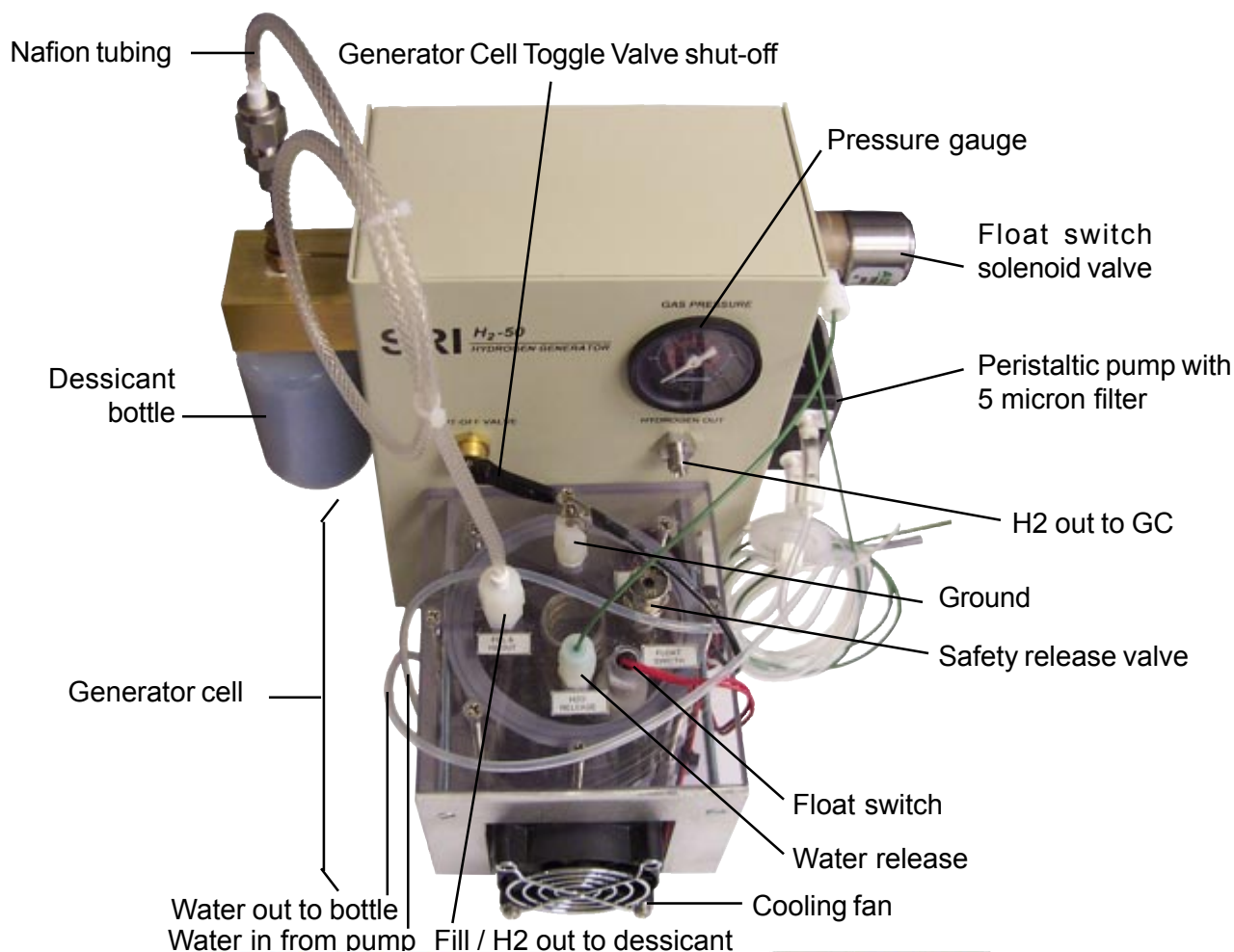


GC ACCESSORIES

H₂-50XR Hydrogen Generator

Overview

The SRI H₂-50XR Hydrogen Generator consists of a generator cell mounted on a metal chassis. The generator cell is attached to the chassis with two screws for easy disassembly. On the metal chassis is a solenoid valve, a pressure gauge, an interior pressure switch, a dessicant bottle, and a toggle valve shut-off for isolating the generator cell. The Nafion tube that connects the cell with the dessicant bottle removes most of the water vapor from the H₂ gas; the rest is removed by the dessicant before reaching the GC column. The dessicant bottle contains Indicating Molesieve dessicant beads which turn from blue to gray when they absorb water. Water is supplied to the generator cell by the peristaltic pump from the provided reservoir bottle. If the water level in the cell gets too high, the float switch opens up the solenoid to drain the excess water back into the reservoir bottle. The H₂-50XR can supply enough gas for a detector or two, plus the GC carrier gas. During operation, there is about 40mL of hydrogen gas stored in the dessicant, which is enough to operate a split injector for short periods, in addition to the detector(s) and carrier. The toggle valve shut-off facilitates checking for leaks and allows the H₂-50XR to reach operating pressure more quickly, while the interior pressure switch maintains the operating pressure. As a safety measure, a pressure release valve protects the generator cell from pressure overload. The external power supply with its own transformer enables the H₂-50XR to operate on various voltages around the world. The H₂-50XR produces 50mL/min at 30psi using distilled water from the grocery store, and can run unattended for up to 30 days.



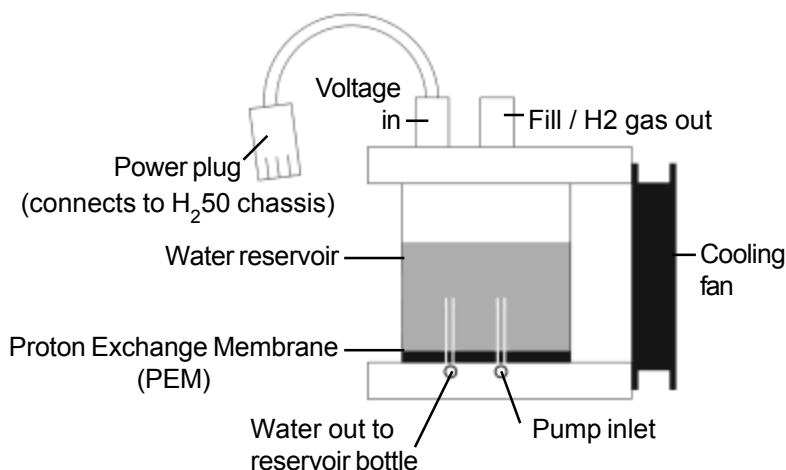
GC ACCESSORIES

H₂-50XR Hydrogen Generator

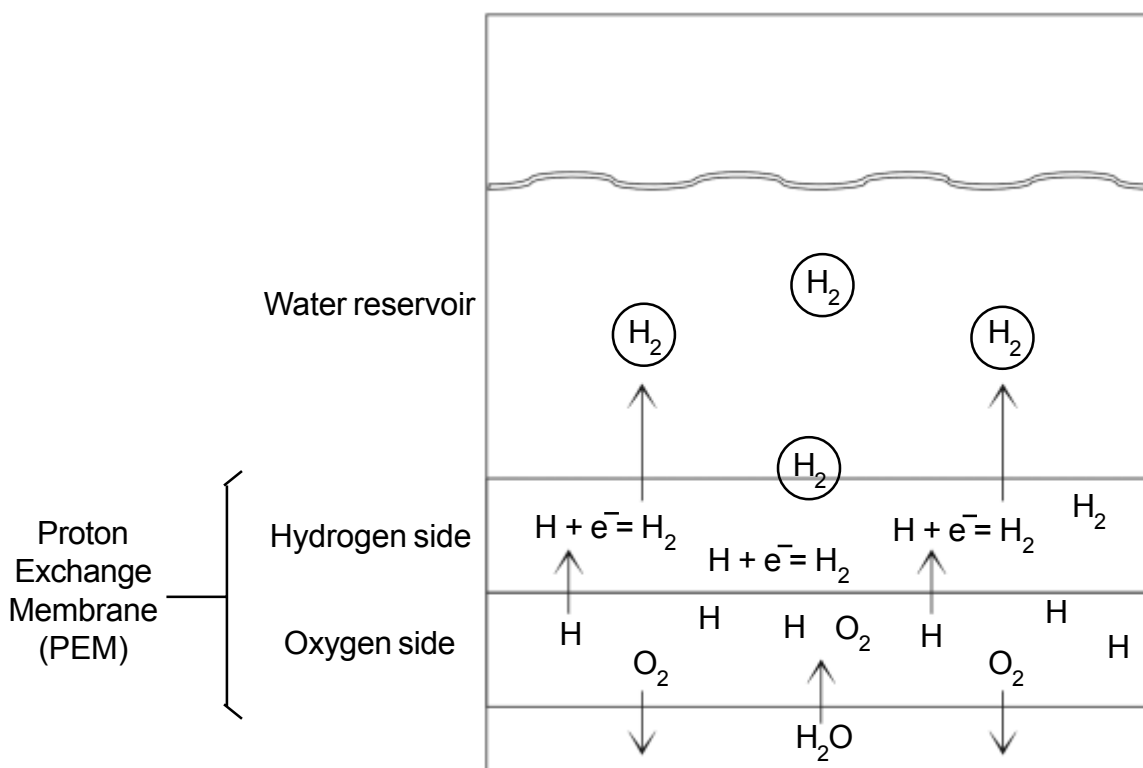
Theory of Operation

The SRI H₂-50XR Hydrogen Generator separates water into hydrogen and oxygen using a Proton Exchange Membrane (PEM). The water on the oxygen side of the PEM is disassociated into O₂ and hydrogen protons. The hydrogen proton is transported through the PEM to the hydrogen side, where it recombines with an electron to make H₂, then bubbles up through the water reservoir.

The H₂50 Generator Cell



Operational Diagram of the H₂50 Generator Cell



GC ACCESSORIES

H₂-50XR Hydrogen Generator

General Operating Procedure

NOTE: Use the H₂-50XR on a flat, level surface, away from open flame and any other ignition or spark sources.

1. Remove the nut and septum with the Nafion tubing from the “Fill/H₂ OUT” port on the top of the generator cell. Use the provided 20mL plastic syringe to inject clean water into the cell through this port. Fill only to cover the plastic disk in the bottom of the cell. The cell will automatically fill the rest of the way when you power up the H₂-50XR. Replace the nut and septum on the “Fill/H₂ OUT” port and 1/4 turn past finger tight. Fill the plastic bottle provided by SRI with the same distilled water used in the cell.
2. There are three tubing lines attached to the H₂-50XR: two clear, one green. Place the ends of all three tubes into the water bottle, through the holes drilled in the lid. The ends of the two clear tubes should be submerged in the water, and the green one should hang just above the water level.
3. Make sure the black ground wire is clipped onto the electrode protruding through the top of the generator cell, and that the red and black power cord is plugged into the H₂-50XR chassis. Then, connect the external power supply cord to the generator and a wall outlet. Make sure you have the correct input cord for the voltage you are using; SRI provides a 110VAC cord. The power supply can run on 100-240VAC. Properly used, the transformer is not a spark source and poses no ignition threats.
4. Make sure the H₂ shut-off valve is closed; the back toggle should be switched back toward the chassis. Always build up pressure initially with the toggle valve shut; it should take 5-15 minutes. The hydrogen gas pressure is preset to 30psi. Once this pressure is attained, the interior pressure switch will shut off the current to the generator. The peristaltic pump will shut off, and water in the generator cell should then stop bubbling.
5. Wait about 10 minutes to ensure that the 30psi pressure is maintained. If pressure is not being maintained, there is probably a leak. Check the dessicant bottle: it should be snug against the o-ring. Make sure the “Fill/H₂ OUT” port nut and septum are snug and intact. Check the bottom of the water reservoir around the PEM for moisture to ensure cell integrity. If you find any seepage, carefully tighten each of the eight screws that hold the cell layers together; don’t force them. If you find no indication of a leak after 10 minutes of stabilization at 30psi, open the H₂ shut-off valve by pulling the black toggle switch out away from the chassis. Pressurize and release 2-3 times to get rid of oxygen and nitrogen left in the cell after filling.
6. Connect the “H₂ OUT” fitting on the H₂-50XR to the GC’s hydrogen gas inlet with 1/8” or 1/16” O.D. tubing. Now the hydrogen gas will flow into the connected GC.

GC ACCESSORIES

H₂-50XR Hydrogen Generator

General Operating Procedure continued

Recharging and Replacing the Dessicant Beads

Periodic recharging of the dessicant beads will be necessary as they absorb water during operation and turn grey.

1. Before you loosen the dessicant bottle on the H₂-50XR chassis, vent the hydrogen pressure in the generator cell by unscrewing the nut capping the "H₂O RELEASE" port on the top of the cell. It will hiss audibly until the pressure is released.
2. Dry the dessicant beads by pouring them onto a paper plate and cooking them in a microwave oven for 2-3 minutes. Or, pour them onto a glass or metal pan and bake them in the GC oven at 250°C. Do not microwave or bake the plastic dessicant bottle. The dessicant beads can be recharged over and over again; they last indefinitely. Should you need them, dry dessicant beads are available in kilogram quantities from Alltech (1-800-ALLTECH; part # 05553).
3. Let the beads cool, especially after microwaving them. Refill the dessicant bottle with the dry, blue beads.
4. Replace the bottle on the H₂-50XR chassis and hand tighten it. There is an o-ring that engages with the bottle top; tighten the bottle until it is snug against the o-ring.
5. The dry dessicant contains some air which will purge out during the first few minutes of operation. You may notice your retention times change temporarily since the carrier gas may initially be a mixture of hydrogen and air for a few minutes after dessicant replacement. The FID flame may also be hard to light until pure hydrogen comes through. You can speed up this equilibration process by building up pressure in the generator cell then venting with the toggle valve 2-3 times before reconnecting the H₂-50XR to the GC. Keep in mind that the internal pressure switch will cut the current when the cell reaches 30psi, so you don't need to build up too much pressure before venting it. Experiment to learn what works best for your particular GC system.

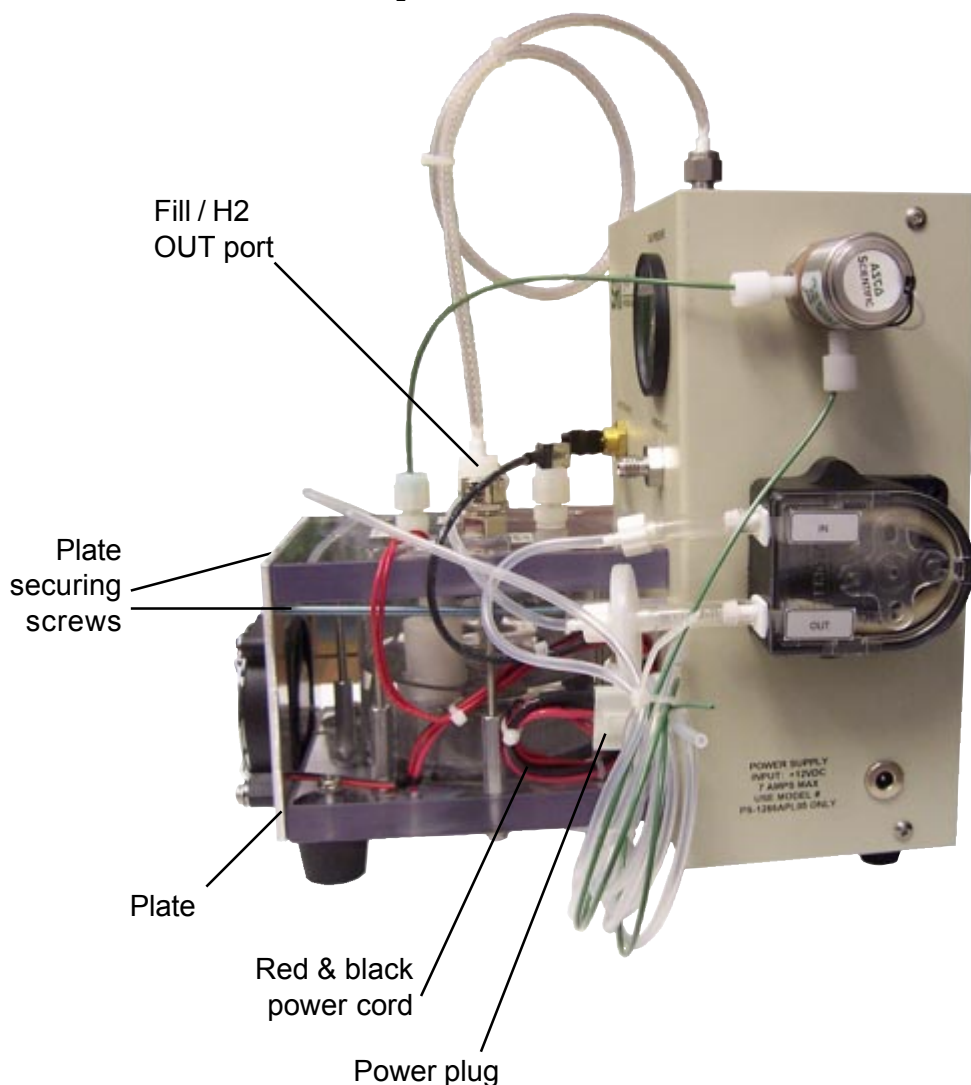
GC ACCESSORIES

H₂-50XR Hydrogen Generator

Maintenance and Troubleshooting

If the water in the H₂-50XR water reservoir looks cloudy, it needs to be replaced:

1. Remove the nut with the septum from the “Fill / H₂ OUT” port on the top of the generator cell.
3. Turn the generator over and pour the water out. When the water is almost all out, shake the generator to help it drain.
4. Use the syringe to refill the water reservoir with clean, distilled water through the “Fill / H₂ OUT”.
5. Replace the nut and septum on the “Fill / H₂ OUT”.



See the following page for PEM replacement

For service, call 310-214-5092.

GC ACCESSORIES

H₂-50XR Hydrogen Generator

Maintenance and Troubleshooting continued

If the Proton Exchange Membrane (PEM) changes color, it most likely needs to be replaced. New H₂-50XR PEMs are available from SRI under part # 8690-0151.

1. Put the replacement PEM in clean distilled water to soak while you take apart the generator cell.
2. Remove the generator cell from the H₂-50XR chassis: unplug the red and black power cord from the chassis, un-clip the black wire from the ground, and unscrew the two screws that hold the clamping plate against the cell.

3. Loosen the eight screws that hold the water reservoir with a philips head screwdriver. Loosen each screw in increments; first one, then the one opposite, and so on in a star-like pattern. As you progress, be mindful of the spring in the water reservoir; don't loosen the screws too suddenly, or it may pop open the reservoir, presenting safety and damage risks. You can feel the pressure of the spring relax as you loosen the screws sufficiently; hold the top of the generator cell firmly with one hand while loosening the screws with the other.

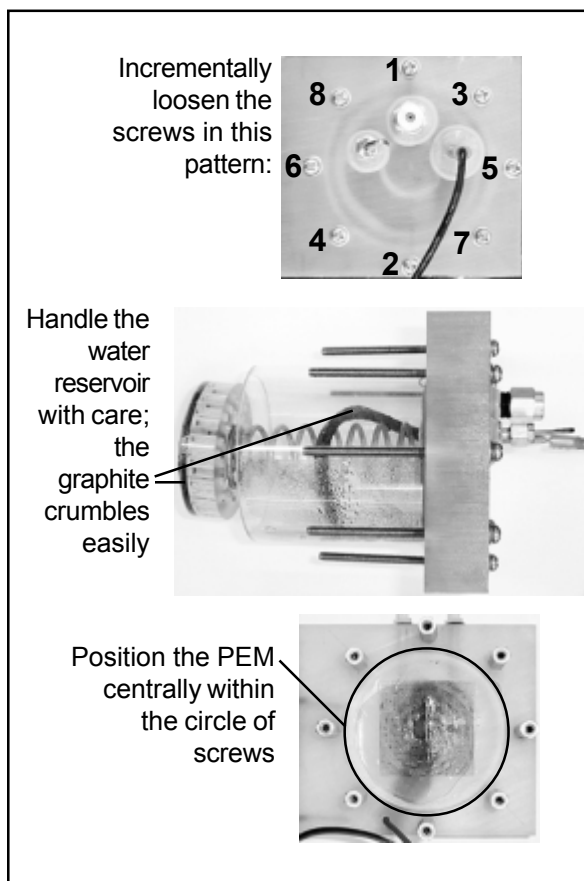
4. Once the screws are removed, carefully take the water reservoir off the bottom of the cell and remove the old PEM. Be very careful handling and moving the graphite coil, as it can easily come apart.

5. Take the new PEM out of its bath and position it centrally within the ring of screws. Place the water reservoir back on the bottom, over the PEM; the PEM should protrude slightly on all sides of the water reservoir.

6. Once the PEM is properly positioned, tighten the screws in increments until the water reservoir is snug against the bottom of the generator cell.

7. Put the generator cell back on the chassis and secure it with the plate and two screws. Plug the red and black power cord into the chassis. Clip the black wire onto the ground.

8. Plug the H₂-50XR into a wall outlet and pressurize the generator cell to 30psi. Check the bottom of the water reservoir around the PEM for moisture; if you see any seepage, tighten each of the eight screws a little more.



WARNING!



Warnings and Safety Precautions:

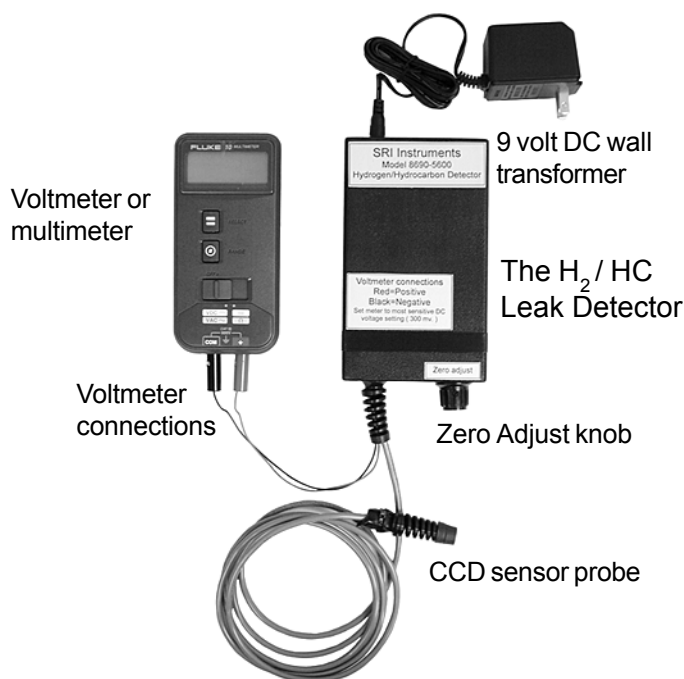
The H₂-50XR generates hydrogen, which is an extremely flammable gas. Under normal operation, the safety features of the H₂-50XR protect the operator. However, operators must use common sense and take basic precautions. Hydrogen burns with a flame that is invisible to the naked eye. Do not use the H₂-50XR near any flames, sparks, or sources thereof, including lab ovens, heater elements, bunsen burners, torches, etc. Hydrogen is non-toxic, but it can cause asphyxiation in confined spaces by displacing oxygen. Use the H₂-50XR in a ventilated room with an ambient temperature of 40-100°F. If the GC power is interrupted or cut off during hydrogen generation, flip the toggle valve to isolate the generator cell, then disconnect the external power source from the H₂-50XR and the wall outlet. This is a good general response in any situation of uncertain risk; if you're not sure what's happening, isolate the cell and pull the power plug. That way, you can take the time to diagnose any problems without H₂ accumulation. Familiarize yourself with the safe operation of the GC (or other equipment) to which you intend to connect the H₂-50XR.

WARNING!

GC ACCESSORIES

Hydrogen/Hydrocarbon Leak Detector/Monitor

Equipped with a CCD detector, the SRI Hydrogen/Hydrocarbon Leak Detector/monitor connects to your voltmeter, which provides the digital readout. As the hydrogen and/or hydrocarbon concentration increases, the voltmeter reading increases. The detection limit is approximately 500ppm. The 110 volt AC unit is shipped with a 9 volt DC wall transformer, but it may be run on any battery source with voltage between 8 and 15 volts. Power consumption is approximately 200 milliamps.



To use the Hydrogen/hydrocarbon Leak Detector:

1. Connect the leak detector to your voltmeter: plug the red and black wires into the corresponding sockets on the voltmeter (RED = positive, BLACK = negative).
2. Plug in the detector's power source.
3. Set the voltmeter to its most sensitive setting (typically 200 millivolts).
4. Use the Zero Adjust knob to zero the detector signal to the voltmeter.



To sniff the fittings on a GC for hydrogen or argon/methane leaks, simply hold the CCD sensor near each potential leak site while observing the voltmeter readout. Make sure to zero the signal first, using the Zero Adjust knob. Check all potential leak sites on the outside of the GC, then inspect the chassis interior: gas line fittings, T's, and restrictors.



Post Office Box 1674

Bandera, Texas 78003

(830) 796-4512

FAX (830) 796-4655

POROUS POLYMERS

Since the development of porous polymers for use in gas chromatography by Hollis and Hayes in the mid 1960's, very little has been done to improve their performance. Commercial polymers such as Porapak® and Chromasorb® have been available since this time for chromatographic use. However these commercially available polymers have been plagued with problems such as batch-to-batch variations, incomplete or inadequate cleanup and shrinkage. These variations and inconsistencies in production and handling have led to poor performance and reproducibility.

HayeSep® analytical polymers and packed columns are now available to chromatographers at a reasonable cost. Hayes Separations, Inc. takes pride in providing the necessary technical assistance to support our users. We guarantee that our polymers are better than any other on the market and we are continuing to develop and test new packings for specific separation problems.

HayeSep® polymers are thoroughly cleaned and preconditioned for twelve hours under oxygen-free nitrogen before packaging. These handling techniques produce polymers which are consistently the same, with no shrinkage and minimum bleed. Columns packed with HayeSep® require minimum conditioning.

HayeSep® Polymer	Maximum Operating Temp.	Surface Area m ² /gram	Tapped Bulk Density gram/cc	Polymer Composition*	Polarity (1=lowest 9=highest)
A	165°C	526	0.356	DVB (high purity) EGDM (high purity)	7
B	190°C	608	0.330	DVB/PEI	8
C	250°C	442	0.322	DVB/ACN	6
D	290°C	795	0.3311	DVB (high purity)	1
N	165°C	405	0.355	DVB/EGDM	9
P	250°C	165	0.420	DVB/Styrene	3
Q	275°C	582	0.351	DVB	2
R	250°C	344	0.324	DVB/NV2P	5
S	250°C	583	0.334	DVB/4VP	4
T	165°C	250	0.381	EGDM	10

*DVB	Divinylbenzene	ACN	Acrylonitrile
EGDM	Ethyleneglycoldimethacrylate	NV2P	N-vinyl-2-pyrrolidinone
PEI	Polyethyleneimine	4VP	4-vinyl-pyridine

HayeSep® A This polymer separates permanent gases (hydrogen, nitrogen, oxygen, argon, carbon monoxide, and nitric oxide) at ambient temperatures. It also exhibits good separation characteristics for the C2's, hydrogen sulphide and water at higher temperatures.

Figure 1

PERMANENT GASES

Column: 36' x 1/8" packed
with HayeSep® A
80/100 mesh
Column Temp: 25°
Detector: P.E. 900 T.C. 225 ma
Att. x 2 180°C
Flow: He 23 cc/min
Sample: 25 microliters Air
plus Hydrogen and
Carbon Monoxide

1. Hydrogen 5%
2. Nitrogen 48.5%
3. Oxygen 13%
4. Argon 0.5%
5. Carbon Monoxide 33%

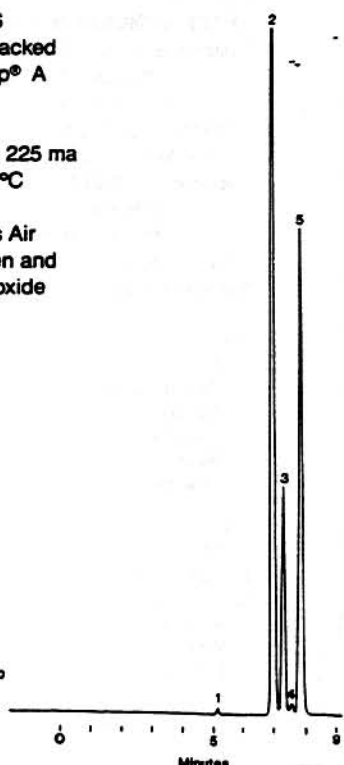
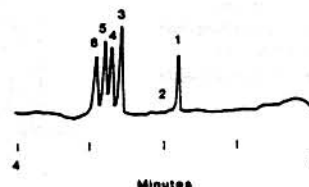


Figure 2

PERMANENT GAS

STANDARD 500 ppm
Column: 25' x 1mm packed
with HayeSep® A
170/200 mesh
Column Temp: 23°C
Flow: He 15 cc/min
Sample: 20 microliters

1. Neon
2. Hydrogen
3. Nitrogen
4. Oxygen
5. Argon
6. Carbon Monoxide



HayeSep® B Designed to separate the C1 and C2 amines as well as trace levels of ammonia and water, this polymer eliminates the need for caustic washing of material prior to packing.

Figure 3

AMINES #1

Column: 5' x 1/8"
SS packed with
HayeSep® B
80/100 mesh
Column Temp: 140° up to
190°C at
16°C/min
Injector Temp: 150°C
Detector: P.E. 900 T.C.
175 ma
Att. x 8 180°C
Flow: He 30 cc/min
Sample: 0.2 microliters
with on-
column
injection

1. Air
2. Water
3. Methylamine
4. Dimethylamine
5. Trimethylamine
6. Ethylene diamine

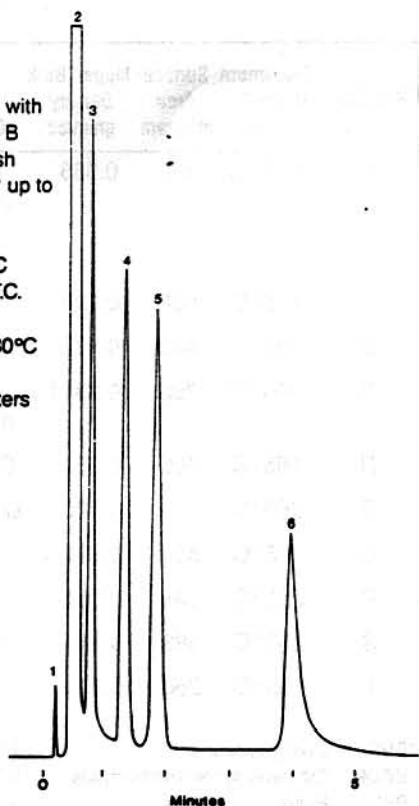
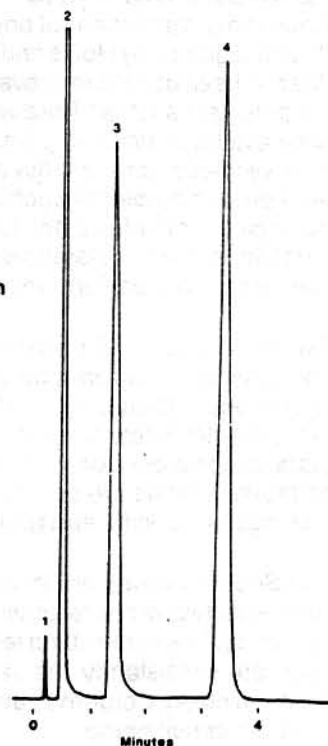


Figure 4

AMINES #2

Column: 5' x 1/8" packed
with HayeSep® B
80/100 mesh
Column Temp: 140°C up to
190°C at 16°C/min
Injector Temp: 150°C
Detector: P.E. 900 T.C.
175 ma
Att. x 8 180°C
Flow: He 30 cc/min
Sample: On-column injection

1. Air
2. Water
3. Ethylamine
4. Diethylamine



HayeSep® C This polymer is designed for polar hydrocarbons such as hydrogen cyanide, ammonia, hydrogen sulphide and water. HayeSep® C has similar separation characteristics to Chromosorb® 104.

Figure 5

**AMMONIA IN
HYDROGEN SULPHIDE**

Column: 5' x 1/8" packed
with HayeSep® C
80/100 mesh
Column Temp: 70°C
Injector Temp: 200°C
Detector: P.E. 900 T.C.
225 ma
Att. x 2 180°C
Flow: He 30 cc/min
Sample: 100 microliters

1. Air
2. Carbon Dioxide
3. Ammonia
- Trace 1: approx 15%
- Trace 2: approx 1.5%
4. Hydrogen Sulphide
5. Unknown in
Hydrogen Sulphide
6. Unknown in
Hydrogen Sulphide
7. Water

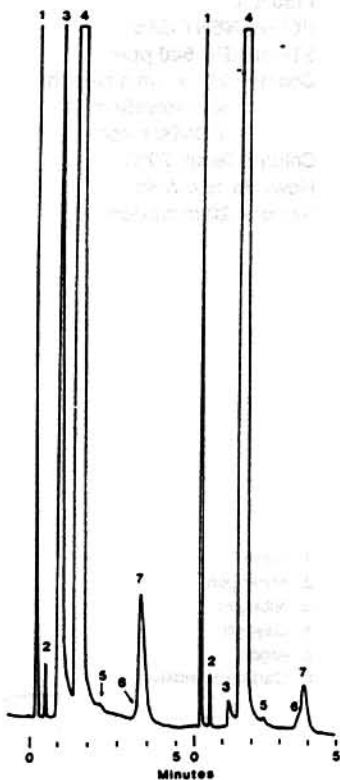


Figure 6

TRACE AMMONIA IN WATER

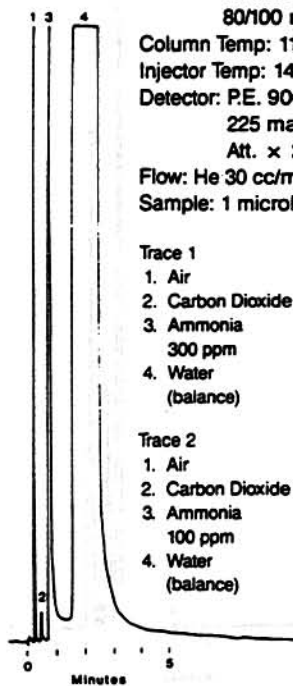
Column: 5' x 1/8" SS packed
with HayeSep® C
80/100 mesh
Column Temp: 115°C
Injector Temp: 140°C
Detector: P.E. 900 T.C.
225 ma
Att. x 2 180°C
Flow: He 30 cc/min
Sample: 1 microliter

Trace 1

1. Air
2. Carbon Dioxide
3. Ammonia
300 ppm
4. Water
(balance)

Trace 2

1. Air
2. Carbon Dioxide
3. Ammonia
100 ppm
4. Water
(balance)



HayeSep® N, P, Q, R, S, and T These polymers are interchangeable with the Porapak® series for separations of low molecular weight materials containing halogens, sulphurs, water, alcohols, glycols, free fatty acids, esters, ketones and aldehydes.

Figure 7

AMMONIA

Column: 8' x 1/8" SS packed
with HayeSep® P
60/80 mesh

Column Temp: 80°C
Injector Temp: 150°C
Manifold Temp: 180°C
Detector: T.C. 175 ma 200°C
Flow: He 30 cc/min
Sample: 0.1 microliters of
NH₄OH with on-
column injection

1. Air
2. Ammonia 35%
3. Water 65%

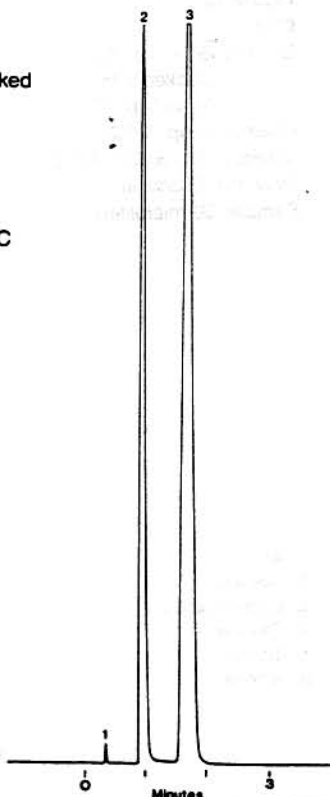


Figure 8

SOLVENTS

Column: 8' x 1/8" SS packed
with HayeSep® P
60/80 mesh

Column Temp: 80°C up to
180°C at
16°C/min
Injector Temp: 150°C
Manifold Temp: 180°C
Detector: 175 ma 200°C
Flow: He 30 cc/min
Sample: 0.2 microliters with
on-column injection

1. Air
2. Water
3. Methanol
4. Ethanol
5. Acetone
6. Chloroform

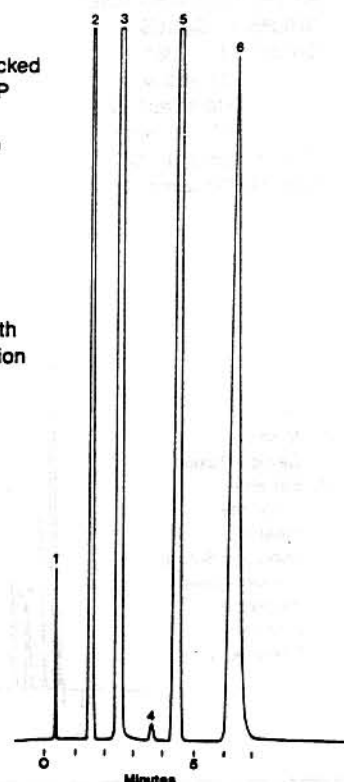


Figure 9

TRACE WATER ANALYSIS

Column: 9' x 1/8" Ni packed
with HayeSep® R
80/100 mesh

Column Temp: 118°C
Flow: He 30 cc/min
Detector: Varian T.C.
with Bendix On-Line
Process Analyzer
Sample: 10 microliters
Ethyl Chloride

1. Air
2. Water 12 ppm
3. Hydrogen Chloride
4. Ethyl Chloride

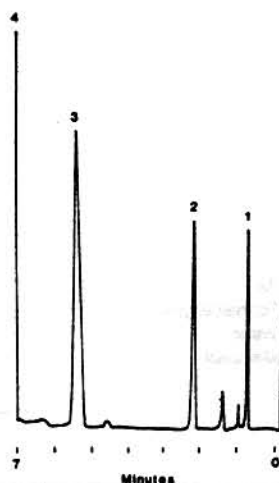


Figure 10

MAPP GAS

Column: 10' x 1/8" SS
packed with
HayeSep® R

Column Temp: 80°C
Flow: He 30 cc/min
Sample: 15 microliters

1. Air
2. Methane
3. Carbon Dioxide
4. Ethane
5. Propylene
6. Propane
7. Propadiene
8. Methyl Acetylene

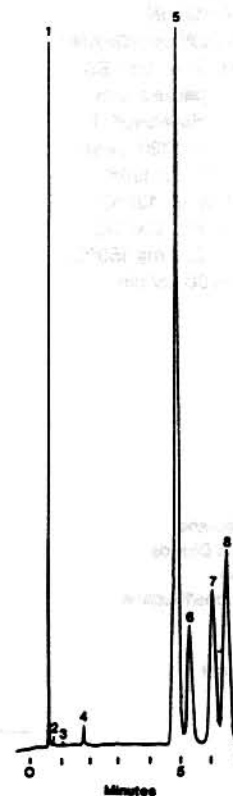


Figure 11
HYDROCARBONS AND
SULPHUR GASES

Column: 8' x 1/8"
packed with
HayeSep[®] Q
80/100 mesh
Column Temp: 90°C
Flow: He 30 cc/min

1. Air
2. Methane
3. Carbon Dioxide
4. Ethylene
5. Acetylene
6. Ethane
7. Hydrogen Sulfide
8. Carbonyl Sulfide
9. Propylene
10. Propane
11. Propadiene

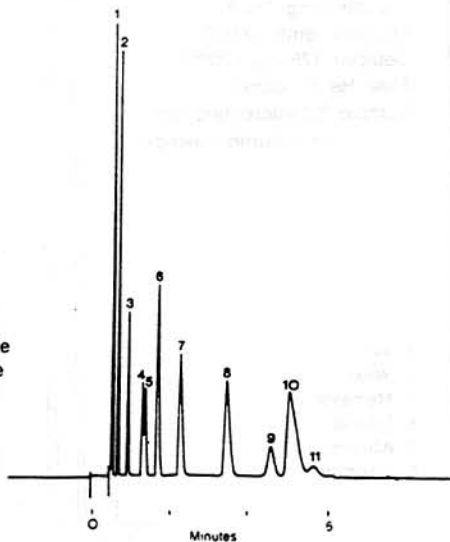


Figure 12
C2's

Column: 5' x 1/8" SS
packed with
HayeSep[®] T
Column Temp: 32°C
Detector: Att. x 216 180°C
Flow: He 30 cc/min
Sample: 50 microliters

1. Air
2. Methane
3. Carbon Dioxide
4. Ethylene
5. Ethane
6. Acetylene

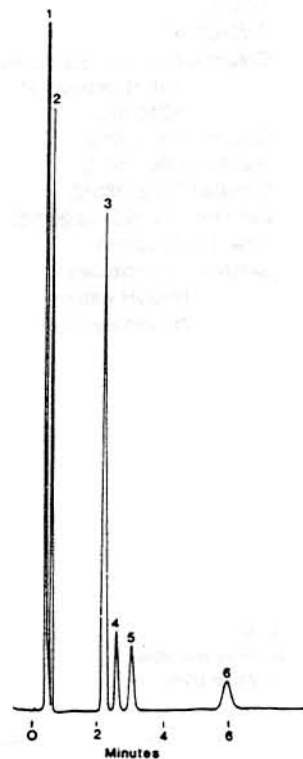


Figure 13
METHANOL IN
PROPYLENE/PROPANE

Column: 5' x 1/8" SS
packed with
HayeSep[®] T
100/120 mesh
Column Temp: 120°C
Injector Temp: 132°C
Detector: P.E. 900 T.C.
225 ma 150°C
Flow: He 30 cc/min

1. Air/Methane
2. Carbon Dioxide
3. Ethane
4. Propylene/Propane
5. C4
6. C4
7. Methanol

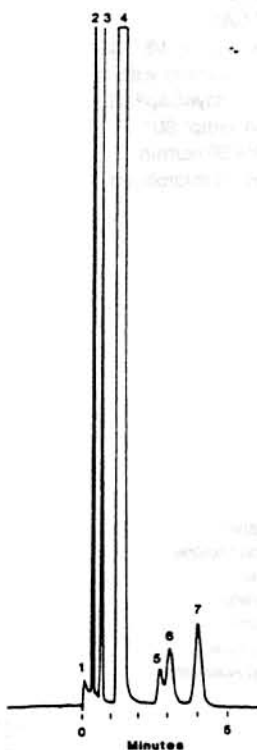
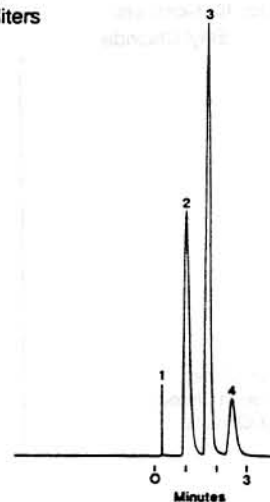


Figure 14
FORMALDEHYDE

Column: 5' x 1/8" SS packed
with HayeSep[®] T
100/120 mesh
Column Temp: 132°C
Injector Temp: 165°C
Detector: P.E. 900 T.C.
175 MA
Att. x 32 180°C
Flow: He 30 cc/min
Sample: 0.2 microliters

1. Air
2. Formaldehyde
3. Water
4. Methanol



INTRODUCING A UNIQUE NEW PRODUCT

HayeSep® D This new polymer made from high purity divinylbenzene is unavailable anywhere else. It has a high surface area and higher operating temperatures than competitive polymers. Available in four different porosities with surface areas from 790 to over 800 m²/gram, this range allows flexibility, since in water/ethane separations porosity determines the order of elution.

These D formulations exhibit superior separation characteristics for light gases. Significant separation abilities include the separation of CO and CO₂ from room air at ambient temperatures and the separation of acetylene prior to other C₂'s. HayeSep® D is particularly useful in the separation and analysis of water and hydrogen sulphide.

Comparisons of D Formulations

	Average Diameter (microns)	Bulk Density gram/cc	Porosity %	Surface Area m ² /gram
Dip	.0317	.3283	69.1	774
D	.0308	.3311	70.35	803
D _B	.0332	.3334	64.2	781

Figure 15

SCOTT MIX 237

Column: 20' x 1/8" Ni packed
with HayeSep® D
100/120 mesh

Column Temp: 25°C

Injector Temp: 100°C

Detector: P.E. 900 T.C.
225 ma 140°C

Flow: He 30 cc/min

Sample: Valco valve
50 microliters vapor
(ambient)

1. Nitrogen (balance)
2. Oxygen 7% Att. x8
3. Carbon Monoxide 7% Att. x8
4. Methane 4.5% Att. x4
5. Carbon Dioxide 15% Att. x4

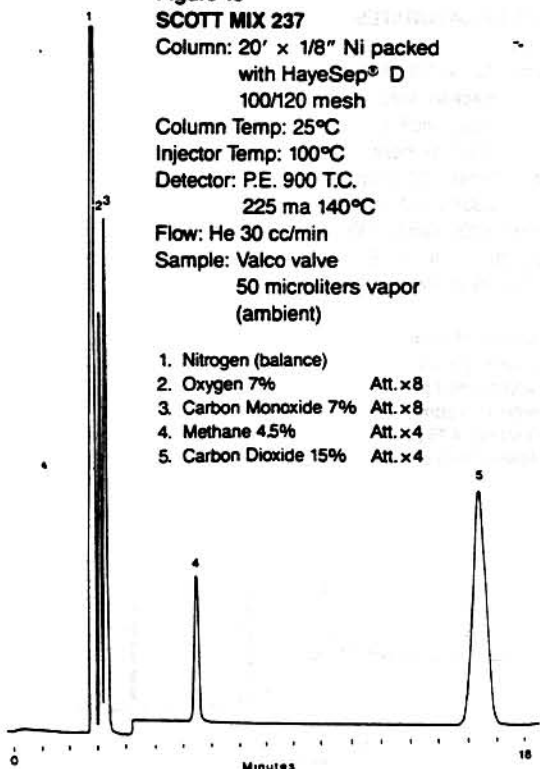


Figure 16

GAS MIXTURE

Column: 10' x 1/8" SS packed
with HayeSep® D
100/120 mesh

Column Temp: 80°C

Injector Temp: 140°C

Detector: P.E. 900 T.C.
225 ma

Att. x 4

Flow: He 30 cc/min

Sample: Valco valve
100 microliters

1. Nitrogen (balance)
2. Carbon Dioxide 2%
3. Nitrous Oxide 3%
4. Water 0.5%
5. Hydrogen Sulphide 3%

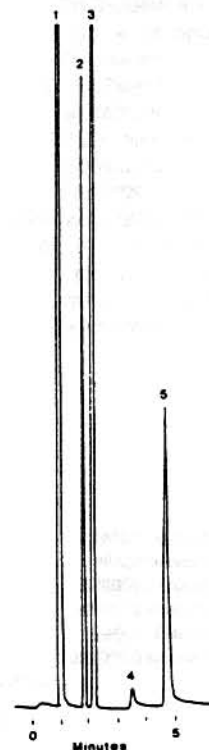


Figure 17

SCOTT MIX 216 (Programmed)

Column: 20' x 1/8" Ni packed
with HayeSep® D
100/120 mesh

Column Temp: 40°C/2 min
programmed
up to 110°C
at 24°C/min

Injector Temp: 100°C
Detector: P.E. 900 T.C.
225 ma 140°C

Flow: He 30 cc/min
Sample: Valco valve
100 microliters
(ambient)

1. Nitrogen (balance)
2. Carbon Monoxide 1% Att.x2
3. Methane 1% Att.x2
4. Carbon Dioxide 1% Att.x2
5. Acetylene 1% Att.x2
6. Ethylene 1% Att.x2
7. Ethane 1% Att.x2

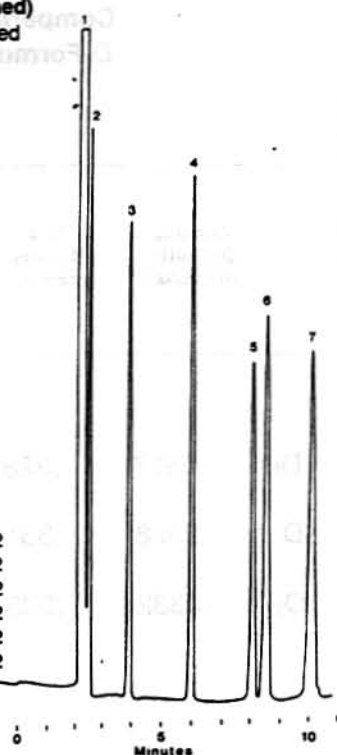


Figure 18

C1 - C2's

Column: 10' x 1/8" SS packed
with HayeSep® D
100/120 mesh

Column Temp: 80°C Isothermal

Detector: 3700 Varian FID

Range: 10⁻¹¹ Att. x 16

Flow: He 35 cc/min

1. Methane 1%
2. Acetylene 1%
3. Ethylene 1%
4. Ethane 1%

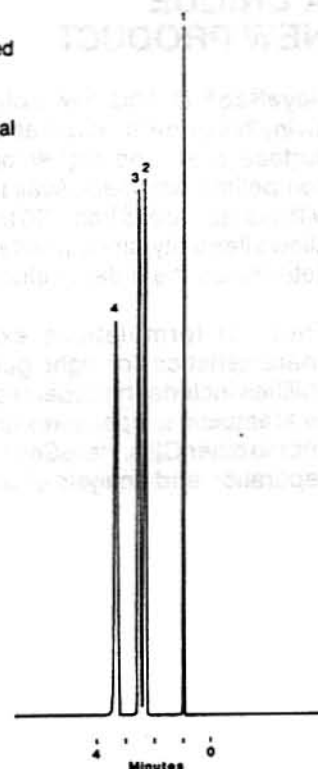


Figure 19

C1 - C5 PARAFFINS

Column: 10' x 1/8" SS
packed with
HayeSep® D
100/120 mesh

Column Temp: 120°C
up to 200°C
at 20°C/min

Detector: 3700 Varian FID
Range: 10⁻¹¹ Att. x 16

Flow: He 35 cc/min
Sample: Valco valve
50 microliters

1. Methane 0.1894%
2. Ethane 0.0965%
3. Propane 0.0989%
4. Isobutane 0.1019%
5. Butane 0.1019%
6. n-Pentane 0.2002%

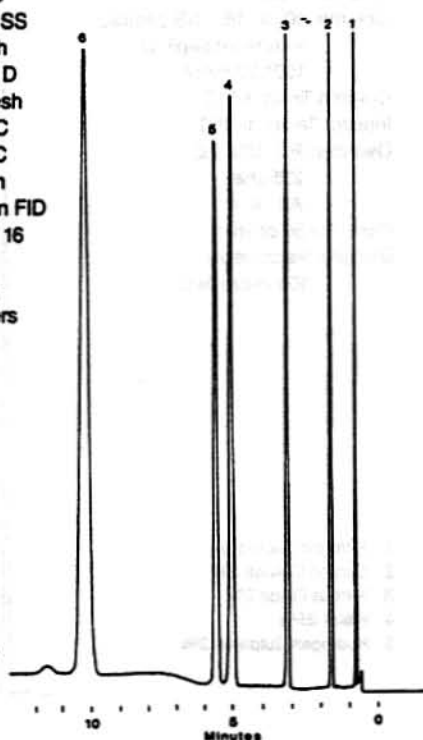


Figure 20

TRACE UNSATURATES

C2 - C6

Column: 10' x 1/8" SS
packed with
HayeSep® D
100/120 mesh

Column Temp: 120°C up to
200°C at 24°C/min

Detector: 3700 Varian FID

Range: 10⁻¹¹ Att. x 16

Flow: He 35 cc/min

1. Acetylene 16 ppm
2. Ethylene 15 ppm
3. Propylene 14.3 ppm
4. 1-Butene 15 ppm
5. 1-Pentene 14.75 ppm
6. 1-Hexene 16 ppm

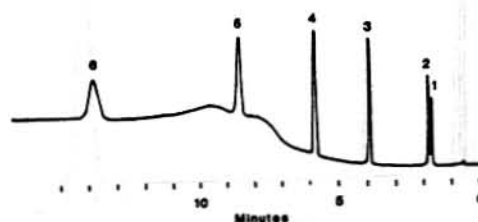


Figure 21

**TRACE ACETALDEHYDE
IN AIR 2500 ppm**

Column: 3' x 1/8" SS
packed with
HayeSep® D
100/120 mesh
Column Temp: 100°C
Injector Temp: 140°C
Detector: P.E. 900 T.C.
225 ma 140°C
Flow: He 30 cc/min
Sample: Valco valve
100 microliters

1. Air
2. Water
3. Acetaldehyde
2500 ppm (vol.)

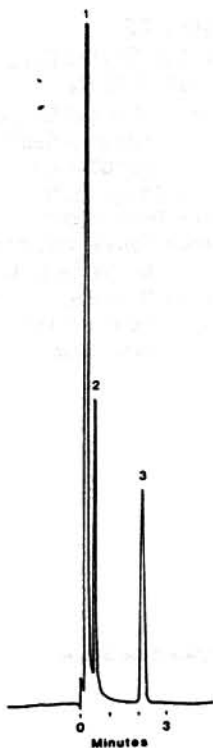


Figure 22

TEQUILA HEADSPACE

Column: 3' x 1/8" SS packed
with HayeSep® D
80/120 mesh
Column Temp: 100°
Injector Temp: 140°C
Detector: P.E. 900 T.C.
225 ma 140°C
Flow: He 30 cc/min
Sample: Valco valve
100 microliters

1. Air Att. x1
2. Carbon Dioxide Att. x1
3. Water Att. x1
4. Methanol Att. x1
5. Acetaldehyde Att. x1
6. Ethanol Att. x8

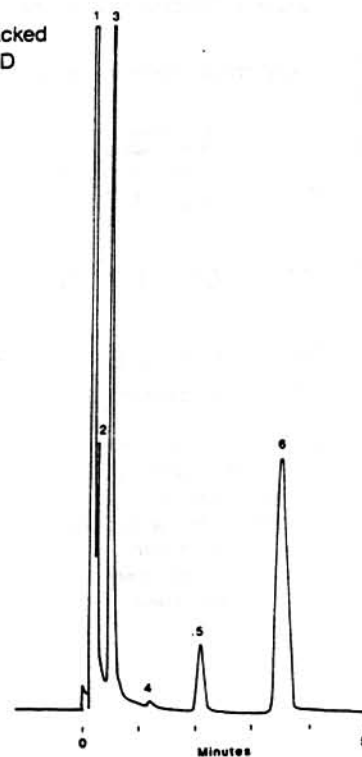


Figure 23

**TRACE ALCOHOLS
IN WATER**

Column: 10' x 1/8" packed
with HayeSep® D
80/100 mesh
Column Temp: 75°C up to
150°C at 16°C/min
Flow: He 33 cc/min
Injector Temp: 125°C
Detector: P.E. 900 T.C.
225 ma
Att. x 1 140°C
Sample: 3 microliters

1. Methanol
500 ppm
2. Ethanol
200 ppm

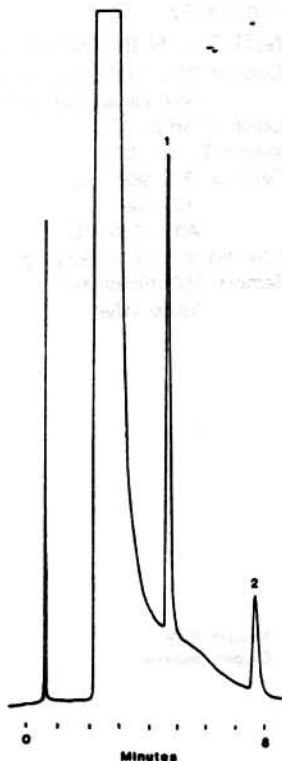
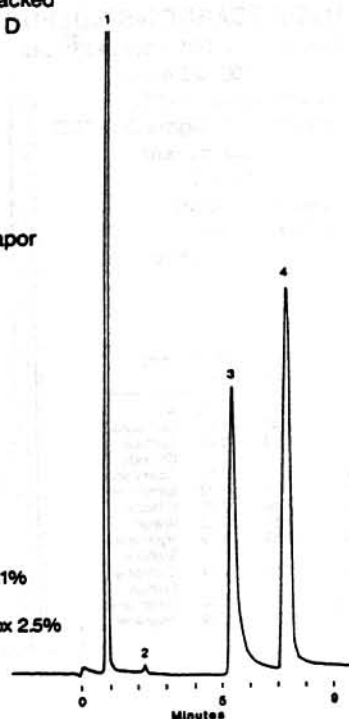


Figure 24

HYDROGEN SULPHIDE

Column: 10' x 1/8" Ni packed
with HayeSep® D
100/120 mesh
Column Temp: 60°C
Injector Temp: 100°C
Detector: P.E. 900 T.C.
225 ma 140°C
Flow: He 30 cc/min
Sample: Valco valve
50 microliters vapor
(ambient)

1. Air (balance)
2. Carbon Dioxide approx 0.1%
3. Water approx 2.5%
4. Hydrogen Sulphide approx 2.5%



COMPARISON OF "D" FORMULATIONS

Retention Time in Minutes

	AIR	CH ₄	CO ₂	C ₂ H ₂	C ₂ H ₄	C ₂ H ₆	H ₂ O
Dip	0.9	1.7	3.1	5.4	5.8	8.3	9.0
D*	0.9	1.7	3.1	5.8	6.1	8.4	8.6
DB	0.9	1.6	3.1	6.1	6.6	8.7	8.1

*This is our standard D.

Column: 10' x 1/8" SS
80/100 mesh

Column Temp: 45°C

Detector: 175 ma Att. x 2 150°

Flow: He 30 cc/min

Sample: 50 microliters

Valco valve

Figure 25 TRACE ETHYLENE OXIDE IN NITROGEN

Column: 10' x 1/8" SS packed
with HayeSep® D
80/100 mesh

Column Temp: 130°C

Injector Temp: 100°C

Detector: Varian 1400 F.I.D.

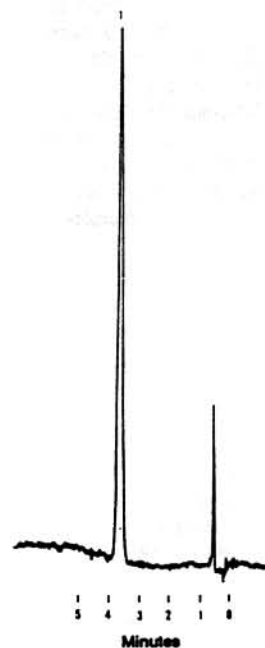
Att. 10⁻¹² x 2 140°C

Flow: He 30 cc/min

Sample: 250 microliters

Valco valve

1. Ethylene Oxide 23 ppm



Courtesy of John Booker Co.

Figure 26 HYDROCARBONS/SULFUR GASES

Column: 9' x 1/8" HayeSep® Dip
100/120 mesh

Column Temp: 100°C

Detector: P.E. Sigma 300 TCD

Low current

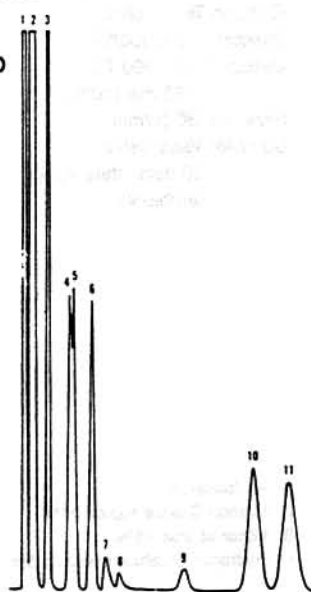
Att. x 1

Flow: He 30 cc/min

Sample: 0.5 cc

Valco valve

PEAK	RET. TIME	AREA %	CPD
1.	0.71	31.59	Air
2.	0.88	46.88	Methane
3.	1.23	5.48	Carbon Dioxide
4.	1.71	2.17	Acetylene
5.	1.79	2.72	Ethylene
6.	2.19	3.09	Ethane
7.	2.52	0.45	Water
8.	2.83	0.19	Hydrogen Sulfide
9.	4.30	0.44	Carbonyl Sulfide
10.	5.82	3.36	Propylene
11.	6.57	3.59	Propane



Courtesy of John Booker Co.

Figure 27 NITROGEN IN OXYGEN

Column: 30' x 1/8" SS packed
with HayeSep® DB

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C.

300 ma

Att. x 2 140°C

Flow: He 30 cc/min, 90 psig

Sample: 250 microliters

Valco valve

1. Nitrogen 0.4%
2. Oxygen - balance

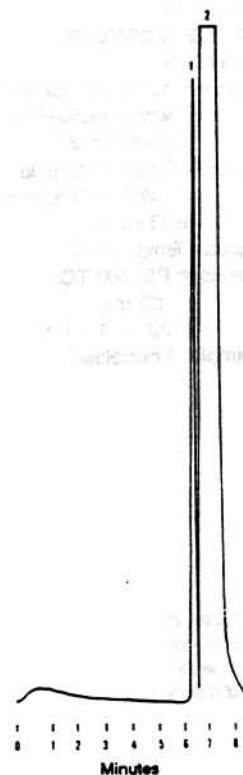


Figure 28
SCOTT MIX 234

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C.

300 ma 140°C

Flow: He 30 cc/min, 90 psig

Sample: 250 microliters

Valco valve

- | | |
|----------------|-----------|
| 1. Hydrogen 4% | Att. x 01 |
| 2. Nitrogen 5% | Att. x 32 |
| 3. Carbon | |
| Monoxide 5% | Att. x 32 |
| 4. Methane 4% | Att. x 32 |
| 5. Carbon | |
| Dioxide 5% | Att. x 08 |

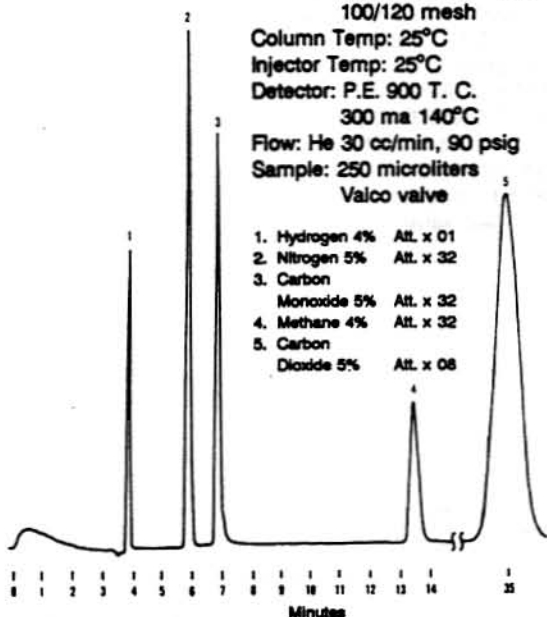


Figure 29
AIR

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C.

300 ma

Att. x 32 140°C

Flow: He 30 cc/min, 90 psig

Sample: 250 microliters

Valco valve

- | |
|-----------------|
| 1. Nitrogen 78% |
| 2. Oxygen 21% |
| 3. Argon 0.94% |

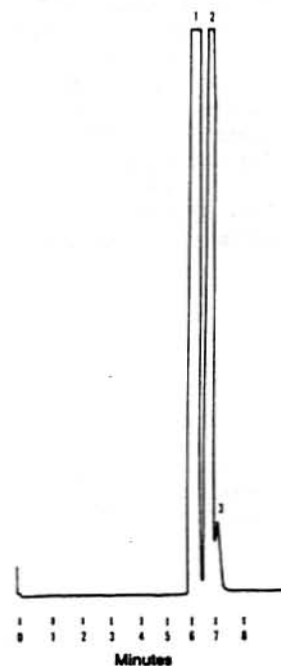


Figure 30
NITROGEN IN ARGON

Column: 30' x 1/8" SS packed
with HayeSep® D
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C.

300 ma

Att. x 1 140°C

Flow: He 30 cc/min, 90 psig

Sample: 250 microliters

Valco valve

- | |
|---------------------|
| 1. Nitrogen 373 ppm |
| 2. Argon - balance |

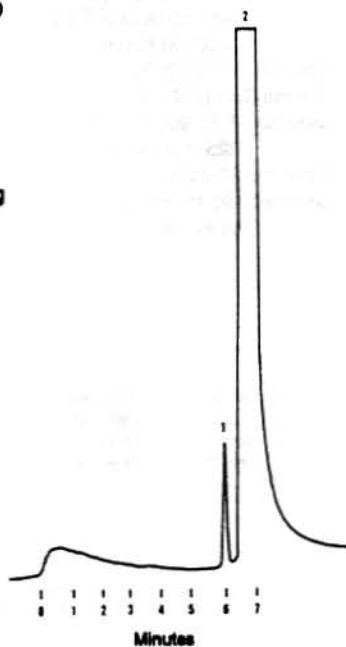


Figure 31
ETHYLENE AND SCOTT MIX 216

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 120°C

Injector Temp: 120°C

Detector: P.E. 900 T. C.

300 ma

Att. x 1 140°C

Flow: He 30 cc/min, 90 psig

Sample: 250 microliters

Valco valve

- | |
|----------------------|
| 1. Nitrogen |
| 2. Carbon |
| Monoxide |
| 3. Methane |
| 4. Carbon |
| Dioxide |
| 5. Acetylene 924 ppm |
| 6. Ethylene |
| 7. Water |
| 8. Ethane |

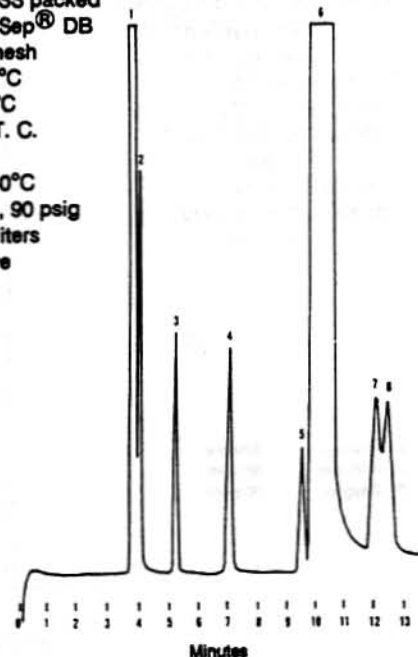


Figure 32**NITRIC OXIDE IN NITROGEN**

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

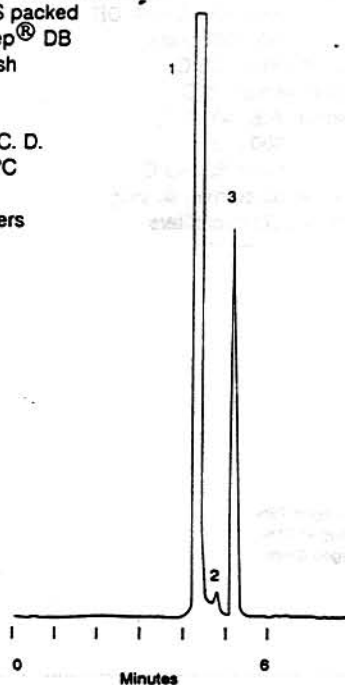
Injector Temp: 22°C

Detector: P.E. 900 T. C. D.
225 ma 140°C

Flow: 30 cc/min

Sample: 100 microliters

- | | |
|-----------------|---------|
| 1. Nitrogen | Balance |
| 2. Argon | |
| 3. Nitric Oxide | 0.58% |

**Figure 33****HYDROGEN IN HELIUM**

Column: 25' x 1/8" SS packed
with HayeSep® D
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

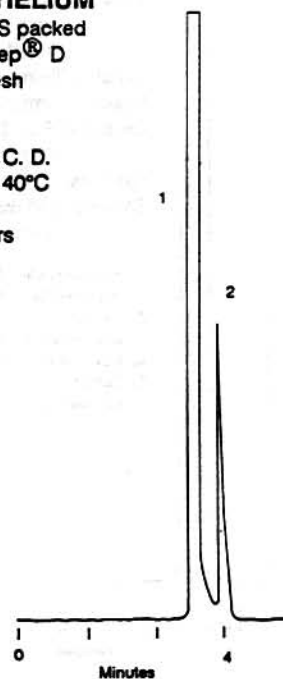
Detector: P.E. 900 T. C. D.
150 ma at 140°C

Flow: 24 cc/min N₂

Sample: 50 microliters

Valco valve

- | | |
|-------------|---------|
| 1. Helium | Balance |
| 2. Hydrogen | 1% |

**Figure 34****IMPURITIES IN HYDROGEN**

Column: 25' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

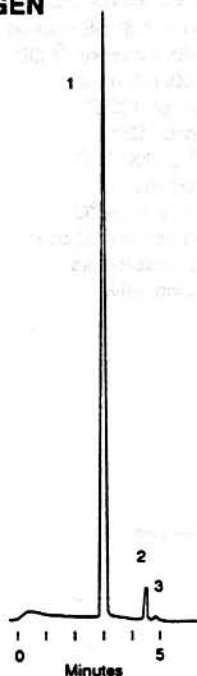
Detector: P.E. 900 T. C. D.
225 ma

Flow: He 25 cc/min

Sample: 250 microliters

Valco valve

- | | |
|-------------|---------|
| 1. Hydrogen | Balance |
| 2. Nitrogen | 80 ppm |
| 3. Oxygen | 20 ppm |

**Figure 35****AIR IN ARGON**

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

Injector Temp: 22°C

Detector: P.E. 900 T. C. D.
225 ma 140°C

Flow: He 30 cc/min

Sample: 100 microliters

Valco valve

- | | |
|-------------|----------|
| 1. Nitrogen | 8000 ppm |
| 2. Oxygen | 1800 ppm |
| 3. Argon | 25% |
| 4. Helium | Balance |

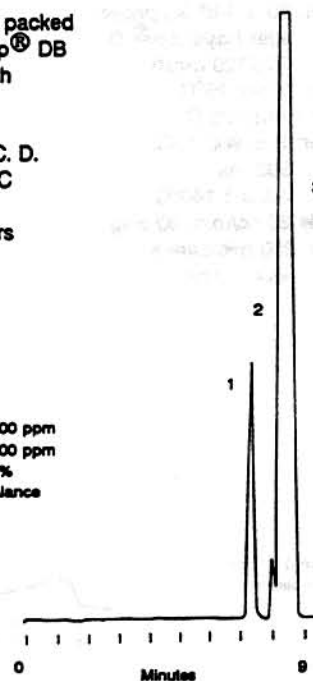


Figure 36

HYDROGEN/AIR 50/50

Column: 25' x 1/8" SS packed
with HayeSep® D
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C. D.

225 ma 140°C

Atten x 8

Flow: He 24 cc/min

Sample: 50 microliters

Valco valve

1. Hydrogen
2. Nitrogen
3. Oxygen
4. Argon

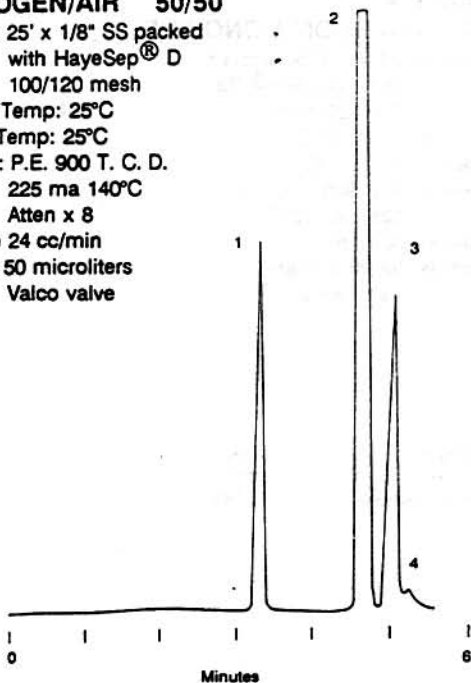


Figure 37

93.6% OXYGEN

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

Injector Temp: 22°C

Detector: P.E. 900 T. C. D.

225 ma 140°C

Flow: He 20 cc/min

Sample: 100 microliters

Valco valve

- | | |
|-------------|---------|
| 1. Nitrogen | 0.4% |
| 2. Oxygen | Balance |

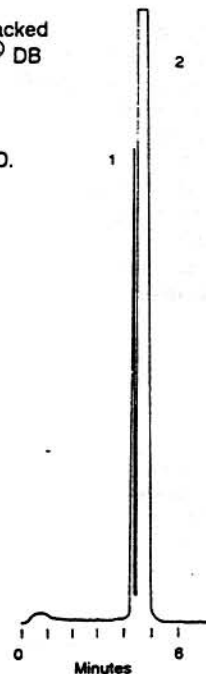


Figure 38

99.995% ARGON

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

Injector Temp: 22°C

Detector: P.E. 900 T. C. D.

225 ma 140°C

Flow: He 30 cc/min

Sample: 100 microliters

Valco valve

- | | |
|-------------|---------|
| 1. Nitrogen | 50 ppm |
| 2. Argon | Balance |

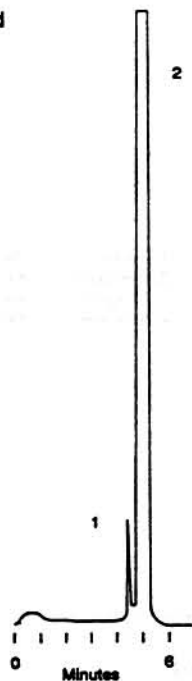


Figure 39

IMPURITIES IN NITROGEN TRIFLUORIDE

Column: 25' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 25°C

Injector Temp: 25°C

Detector: P.E. 900 T. C. D.

275 ma 140°C

Flow: He 30 cc/min

Sample: 250 microliters

Valco valve

Chart Speed: 1 cm/min

- | | |
|-------------------------|---------|
| 1. Nitrogen | 50 ppm |
| 2. Oxygen | 250 ppm |
| 3. Carbon Tetrafluoride | 400 ppm |
| 4. Nitrogen Trifluoride | Balance |

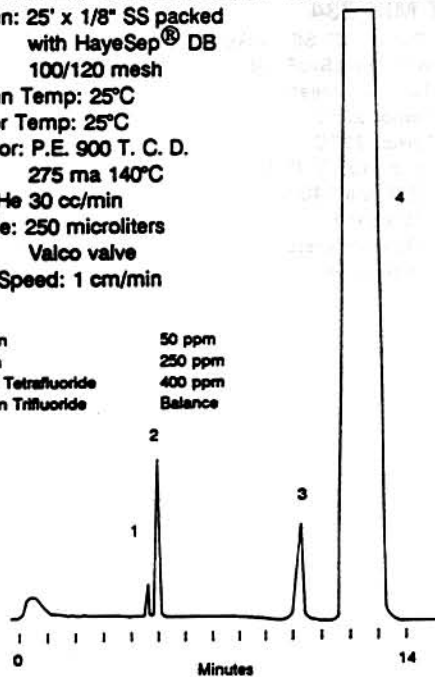


Figure 40
FAST ANALYSIS

Column: 48" x 1/16" x .04" ID SS
packed with HayeSep® DB
100/120 mesh

Column Temp: 70°C

Injector Temp: 70°C

Detector: P.E. 900 T. C. D.

225 ma 140°C

Flow: He 16 cc/min

Sample: 10 microliters

Valco valve

- | | |
|-------------------|---------|
| 1. Nitrogen | 2.7% |
| 2. Methane | Balance |
| 3. Carbon Dioxide | 3.0% |
| 4. Ethane | 3.5% |



Figure 41
99.7% CARBON MONOXIDE

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

Injector Temp: 22°C

Detector: P.E. 900 T. C. D.

225 ma 140°C

Flow: He 30 cc/min

Sample: 100 microliters

Valco valve

- | | |
|--------------------|---------|
| 1. Nitrogen | 0.28% |
| 2. Oxygen | 0.02% |
| 4. Carbon Monoxide | Balance |

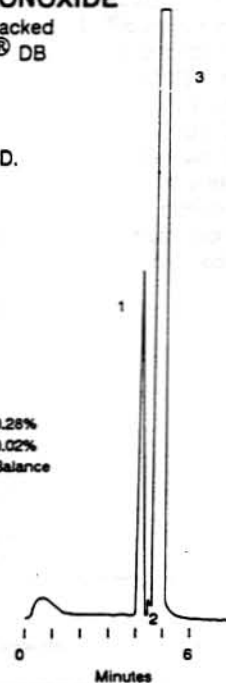


Figure 42
SCOTT MIX 234

Column: 30' x 1/8" SS packed
with HayeSep® DB
100/120 mesh

Column Temp: 22°C

Injector Temp: 22°C

Detector: P.E. 900 T. C. D.

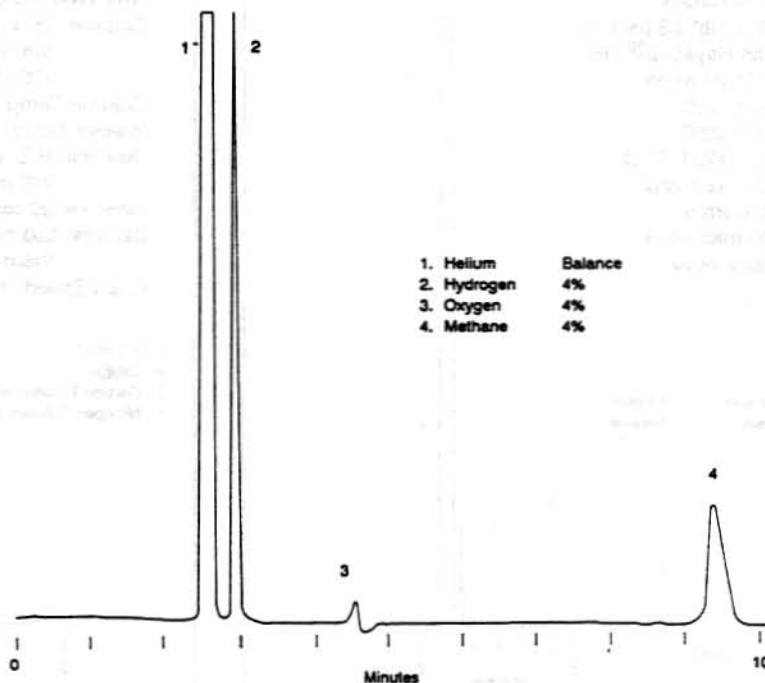
225 ma 140°C

Flow: N₂ 30 cc/min

Sample: 100 microliters

Valco valve

- | | |
|-------------|---------|
| 1. Helium | Balance |
| 2. Hydrogen | 4% |
| 3. Oxygen | 4% |
| 4. Methane | 4% |



POROUS POLYMER MICROPACKED COLUMNS

Hayes Separations, Inc. has been making porous polymers for ten years. We have occasionally made micropacked columns for various customers as well as supplied packings for this purpose. A few examples of these are listed on the following pages. Of interest is the elution time of acetylene relative to ethylene and ethane. Figure 43 shows acetylene between ethylene and ethane; Figure 47 shows acetylene behind ethylene and ethane; Figure 46 shows acetylene in front of ethylene and ethane; Figure 48 shows rapid elution of impurities in methane. References for the production of porous polymers are listed below.

- 1) Hollis, O.L., Analytical Chemistry 38:309-316 (1966).
- 2) Hollis, O.L. and Hayes, W.V., J. Gas Chrom. 4:235-239 (1966).
- 3) Hollis, O.L. and Hayes, W.V., Gas Chrom., A.B. Littlewood, editor, The Institute of Petroleum, Rome, 1966, p. 57-74.
- 4) U.S. Patent - 3,357,158 December 12, 1967.
- 5) U.S. Patent - 3,458,976 August 5, 1969.
- 6) 1966 IR 100 Award (R&D magazine).

Figure 43

SCOTT MIX 216 + MAPP GAS

Column: 10' x 1/16" x .04"
packed with HayeSep® S
100/120 mesh
Column Temp: 60°C 3min up to
90°C at 8°C/min
Detector: P.E. 990 T. C. D. 225 ma
Flow: He 14 cc/min
Chart Speed: 1 cm/min
Sample: 50 microliters

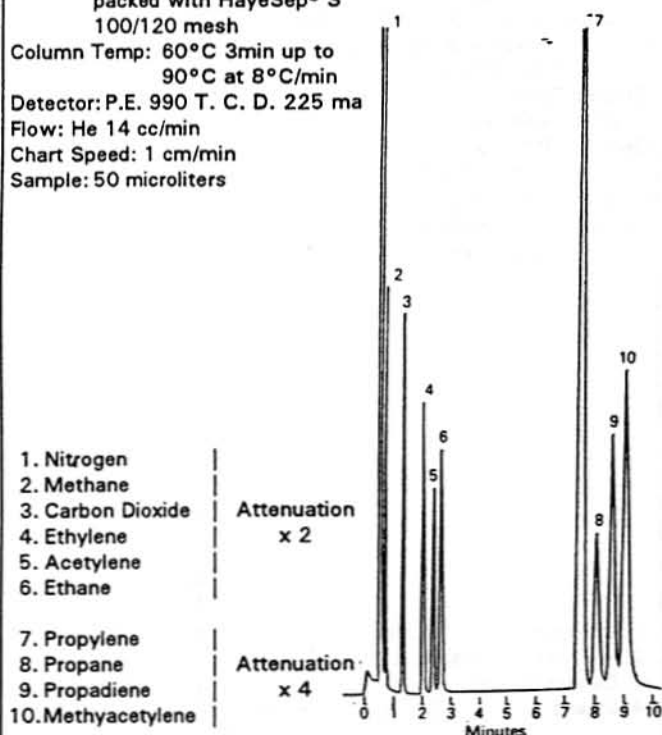


Figure 44

SCOTT MIX 234

Column: 20' x 1/16" x .04" SS
packed with HayeSep® D
100/120 mesh
Column Temp: 25°C
Detector: P.E. 900 T. C. D. 225 ma
140°C
Att. x 4
Flow: 13.3 cc/min He
Chart Speed: 1 cm/min
Sample Size: 25 microliters

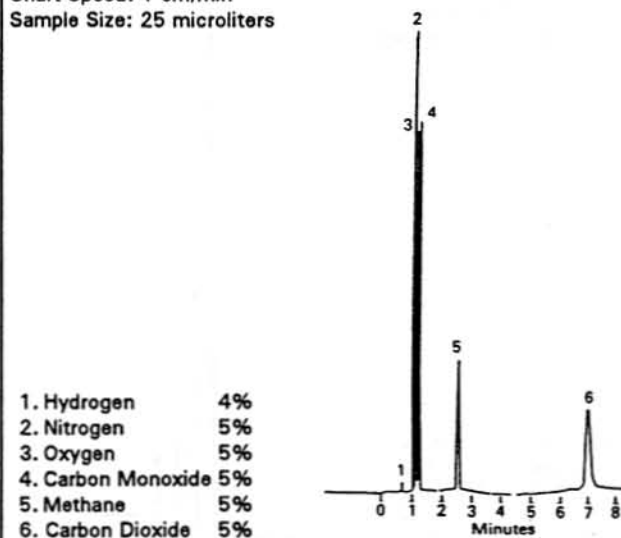


Figure 45

MIX 216 PROGRAMMED

Column: 20' x 1/16" x .04" SS
packed with HayeSep® D
100/120 mesh
Column Temp: 25°C 2min up to
110°C at 8°C/min
Detector: P.E. 900 T. C. D. at
225 ma 140°C
Att. x 4
Flow: 13.33 cc/min He
Chart Speed: 1 cm/min
Sample Size: 25 microliters

- | | |
|--------------------|----|
| 1. Nitrogen | 1% |
| 2. Carbon Monoxide | 1% |
| 3. Methane | 1% |
| 4. Carbon Dioxide | 1% |
| 5. Acetylene | 1% |
| 6. Ethylene | 1% |
| 7. Ethane | 1% |

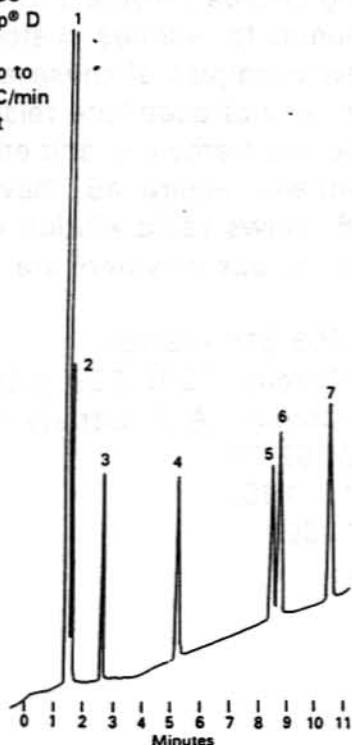


Figure 46

SCOTT MIX 216

Column: 20' x 1/16" x .04" SS
packed with HayeSep® D
100/120 mesh
Column Temp: 70° Isothermal
Detector: P.E. 900 T. C. D. at 225 ma
140°C
Att. x 4
Flow: 13.3 cc/min He
Chart Speed: 1 cm/min
Sample Size: 10 microliters

- | | |
|-------------------|-----------|
| 1. Nitrogen | (balance) |
| 2. Methane | 1% |
| 3. Carbon Dioxide | 1% |
| 4. Acetylene | 1% |
| 5. Ethylene | 1% |
| 6. Ethane | 1% |

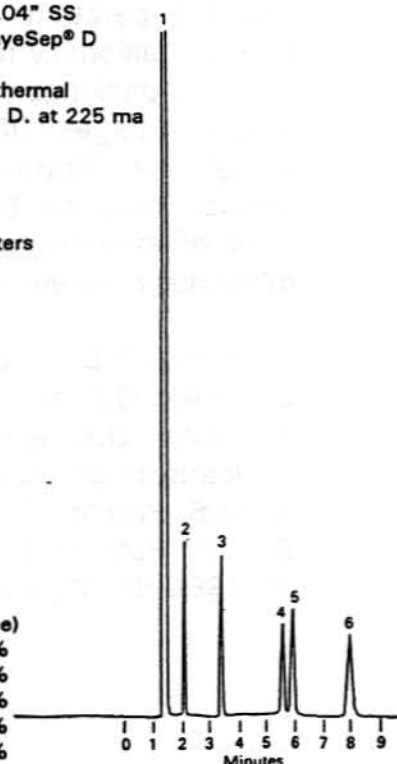


Figure 47

SCOTT MIX 216

Column: 15' x 1/16" x .03"
packed with HayeSep® A
120/140 mesh
Column Temp: 45°C
Detector: VICI micro T. C. D. 120°C
Att. x 8
Flow: He 8 cc/min
Chart Speed: 1 cm/min
Sample Size: 10 microliters

- | | |
|-------------------|-----------|
| 1. Nitrogen | (balance) |
| 2. Methane | 1% |
| 3. Carbon Dioxide | 1% |
| 4. Ethylene | 1% |
| 5. Ethane | 1% |
| 6. Acetylene | 1% |

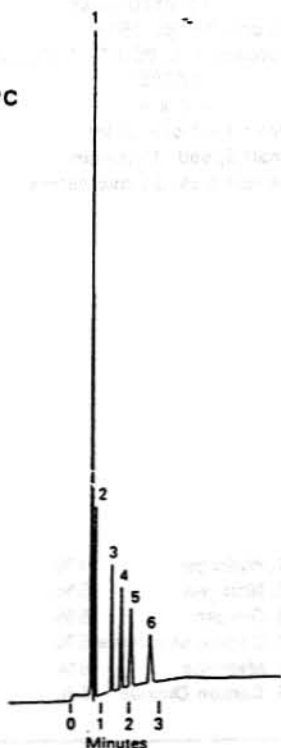
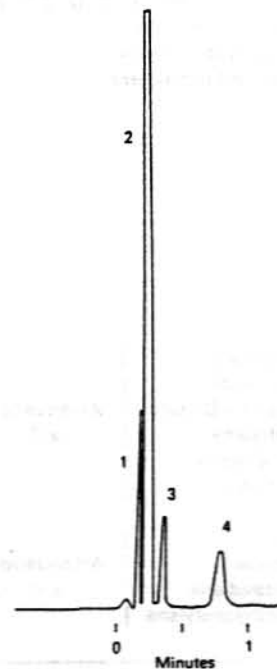


Figure 48

FAST ANALYSIS

Column: 48' x 1/16" x .04" ID SS
packed with HayeSep® DB
100/120 mesh
Column Temp: 70°C
Injector Temp: 70°C
Detector: P.E. 900 T. C. D.
225 ma 140°C
Flow: He 16 cc/min
Sample: 10 microliters
Valco valve

- | | |
|-------------------|-----------|
| 1. Nitrogen | 2.7% |
| 2. Methane | (balance) |
| 3. Carbon Dioxide | 3.0% |
| 4. Ethane | 3.5% |



HAYESEP RT'S RELATIVE TO C₂H₆

6' X 1/8" ss Columns

HayeSep 80/100 mesh

60°C - 30 cc/min. Helium

Courtesy: Brian Thompson, Varian

Compound	A	B	C	D	DB	DIP
CF ₄	.13	.10	.08	.10	.10	.10
CH ₄	.09	.12	.11	.11	.11	.12
CO ₂	.54	.32	.47	.31	.30	.30
N ₂ O	.62	.44	.59	.42	.42	.43
F116	.63	.49	.45	.51	.52	.53
C ₂ H ₂	1.29	.65	1.03	.64	.62	.64
SF ₆	.81	.65	.64	.68	.68	.68
C ₂ H ₄	.81	.71	.75	.70	.70	.70
NH ₃	1.58	.71	1.21	.90	.65	.98
F13	1.10	.90	.84	.87	.88	.90
C ₂ H ₆	1.00	1.00	1.00	1.00	1.00	1.00
H ₂ O	7.06	1.14	5.31	1.08	.95	1.10
H ₂ S	2.06	1.45	2.20	1.36	1.35	1.39
COS	3.02	2.59	3.10	2.56	2.58	2.59
F22	7.63	3.11	6.34	3.30	3.31	3.35
C ₃ H ₆	4.70	3.75	4.41	4.01	4.11	4.10
C ₃ H ₈	4.94	4.43	4.70	4.73	4.84	4.84
SO ₂	10.23	3.84	5.31	3.67	3.82	3.68
PD	6.19	4.63	5.80	4.79	4.87	4.90
MA	8.09	4.70	7.03	4.92	4.96	5.00
CP	5.90	5.02	5.89	5.18	5.32	5.26
F12	7.83	5.71	6.79	6.38	6.47	6.46
IC ₄	17.92	14.73	16.21	16.74	17.18	17.32
1, 3 BD	24.94	16.00	22.93	18.62	19.10	19.48
F114	27.35	18.35	21.46	21.48	22.11	22.88
NC ₄	23.60	19.33	21.21	22.26	22.66	23.01

RELATIVE RETENTION TIMES

Ethane = 1.00

Columns 6' x 1/8" SS 65°C

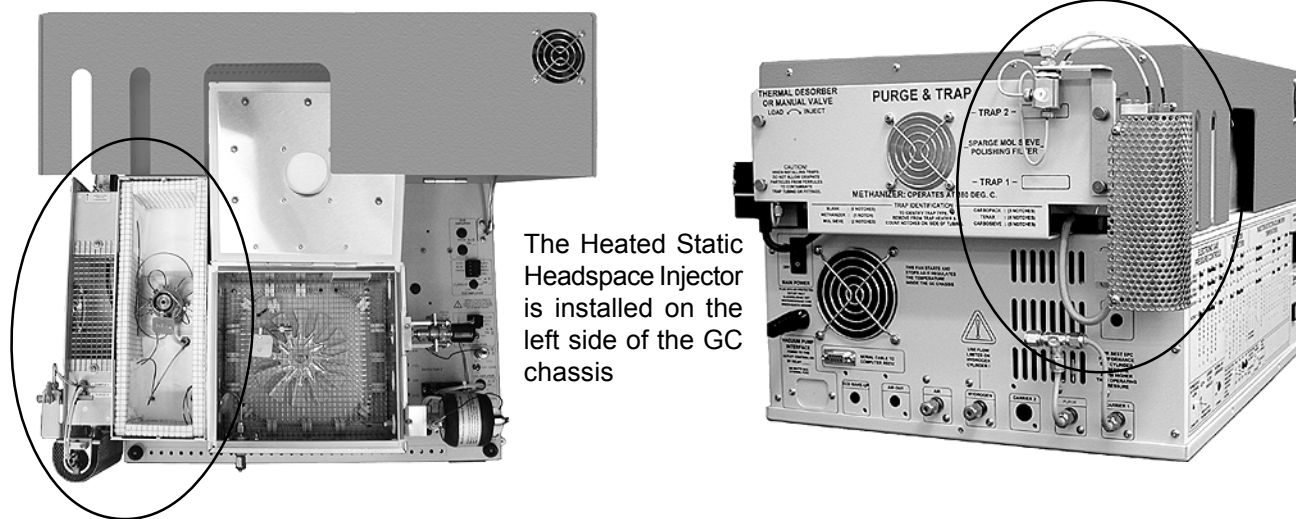
He 30 cc/min

Compound	N	Q	R	S	T
Hydrogen	0.19	.143	0.17	.19	.21
Air	0.23	.186	0.2	.21	.25
Nitric oxide	0.25	.217	0.21	.23	.33
Methane	0.30	.256	0.28	0.3	.35
Carbon dioxide	0.71	0.45	0.50	0.52	0.85
Nitrous oxide	0.80	0.57	0.59	0.59	—
Ethylene	0.83	0.74	0.78	0.78	0.9
Acetylene	1.41	0.74	1.0	0.87	2.11
Ethane	1.0	1.0	1.0	1.0	1.0
Water	10.1	1.45	6.8	4.12	19.1
Hydrogen sulphide	2.1	1.40	1.73	1.87	2.88
Hydrogen cyanide	19.3	2.31	15.6	8.26	28.8
Carbonyl sulphide	2.82	2.33	2.46	2.63	3.4
Sulphur dioxide	12.0	3.05	9.78	17.8	19.0
Propylene	4.66	3.20	3.45	3.65	4.91
Propane	4.66	3.67	3.88	4.1	4.63
Propadiene	6.50	4.12	4.39	4.7	7.55
Methylacetylene	9.5	4.12	4.84	5.14	11.3
Methyl chloride	7.43	3.93	4.67	4.92	9.2
Vinyl chloride	14.9	6.04	9.04	9.7	17.3
Ethylene oxide	17.7	6.06	8.78	9.7	23.3
Ethyl chloride	35.0	12.25	19.3	20.7	43.2
Carbon disulphide	—	32.4	—	—	40.7

INJECTORS

Heated Static Headspace Injector

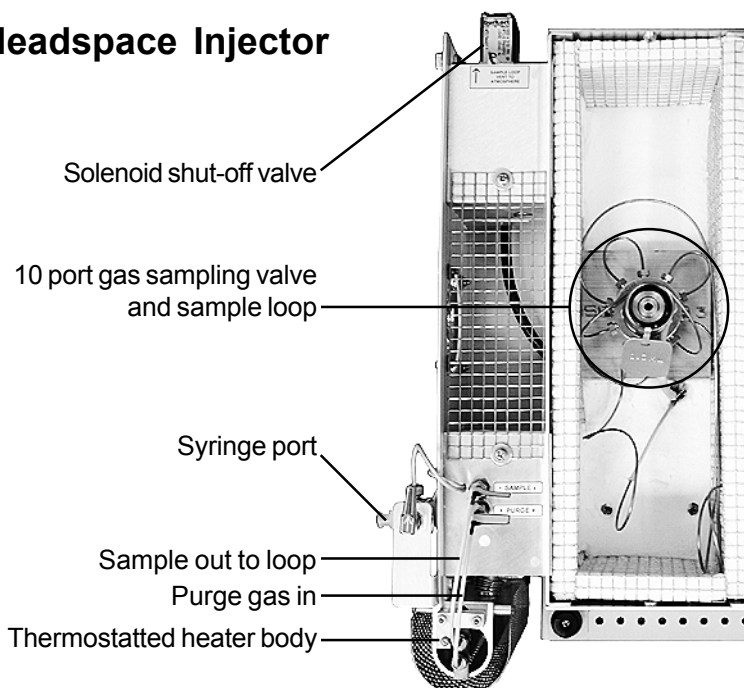
Overview



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

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

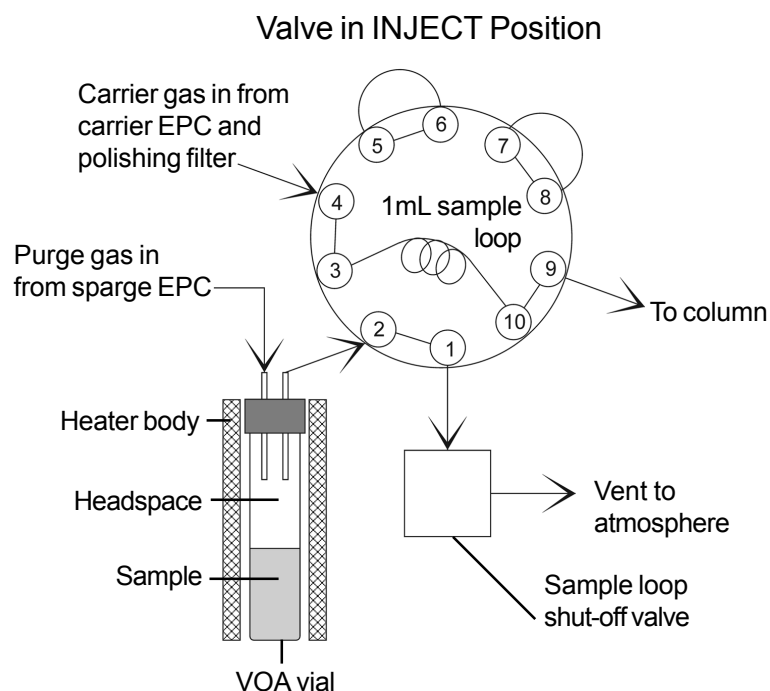
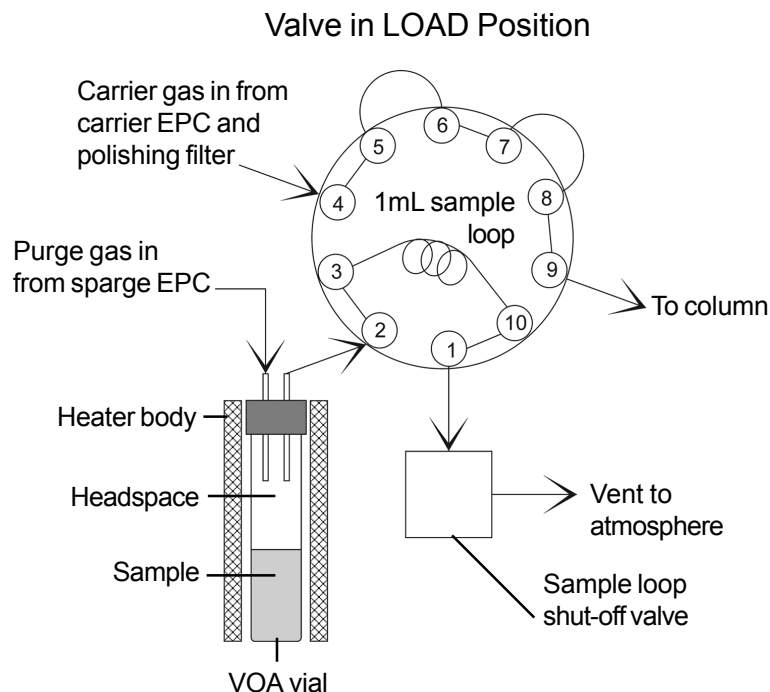
Heated Static Headspace Injector



INJECTORS

Heated Static Headspace Injector

Theory of Operation



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

INJECTORS

Heated Static Headspace Injector

General Operating Procedures

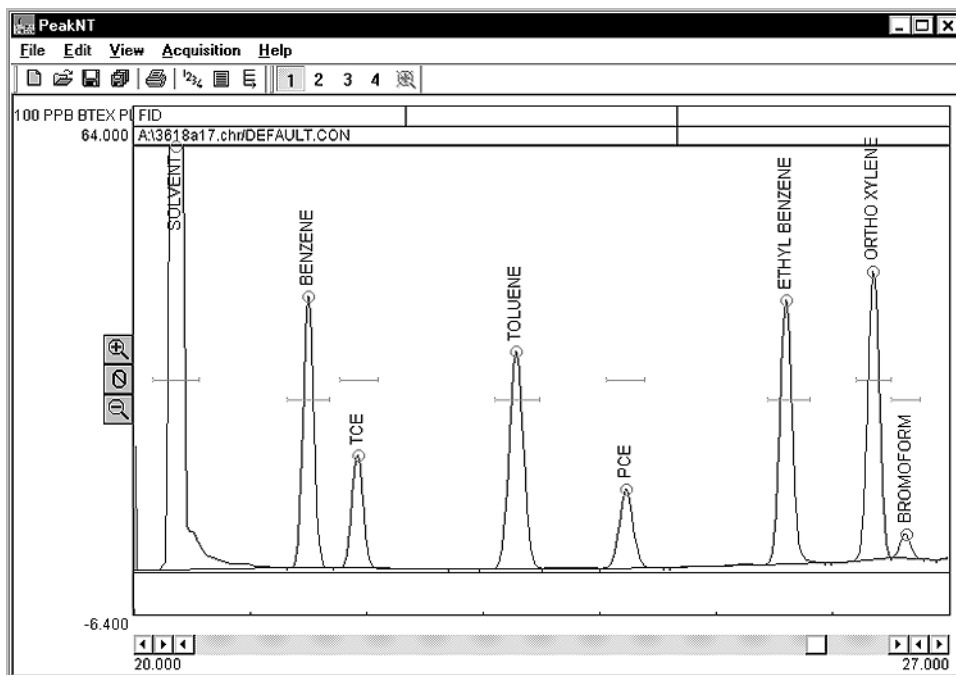
1. The Headspace injection technique requires little sample preparation, since just the sample headspace, not the sample itself, is run through the GC. Insert 10-20mL of sample into a clean VOA vial and seal it.
2. The purge gas pressure is controlled with an Electronic Pressure Controller (EPC). Set the purge gas to 0-10psi, depending on the liquid content of the sample. SRI recommends helium purge gas.
3. Using the trimpot on the top edge of the GC's front control panel, set the heater body temperature between ambient and 90°C. Pressure builds as the vial is heated. The temperature setting depends upon the target analytes and the liquid content of the sample.
4. Create or load an event table. **Hdspace.evt**, shown at right and on the **Expected Performance** page, is included in version 2.66 (and higher) of the PeakSimple software. A typical event table heats and agitates the vial for 20 minutes; it may take more or less time to achieve headspace equilibration.
5. Create or load a temperature program. The column oven is typically held at the initial temperature (usually 40°C) for the duration of the sample equilibration period, plus 2-4 more minutes.
6. Set the valve oven temperature to 100°C or higher to avoid water condensation (120°C is a typical setting).
7. Activate and energize your detectors as necessary. Consult the manual sections for your particular detector(s).
8. Insert a VOA vial filled with 10-20mL of sample into the headspace heater body: slide the vial into the heater body from the bottom. You will feel some resistance as the needles meet the vial septum lid, and once the needles have penetrated the septum, the vial will stop against the top of the heater body interior. The needles will hold the vial in place. Begin the analysis by pressing the RUN button on the GC or the spacebar on your computer keyboard.

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

INJECTORS

Heated Static Headspace Injector

Expected Performance



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

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

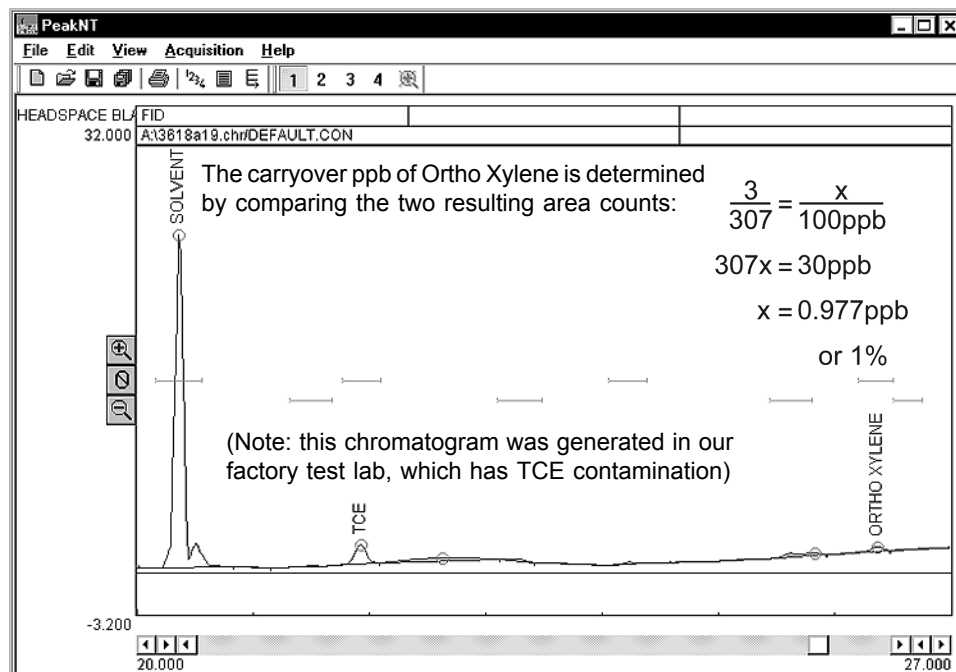
Column oven temperature program
 (Hdspace.tem):

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

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

Events:

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



BTEX sample results:

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

Water blank results:

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

DETECTORS

Helium Ionization Detector - HID

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 NO_x, CO, CO₂, O₂, N₂, H₂S and H₂. It is a robust detector that, unlike the TCD, has no filaments to burn out. The SRI HID consists of a detector body, a collector electrode, an arc electrode assembly, and a thermostatted heater block which can be heated to 375°C. In SRI GCs, the HID is mounted on the right-hand side of the Column Oven.

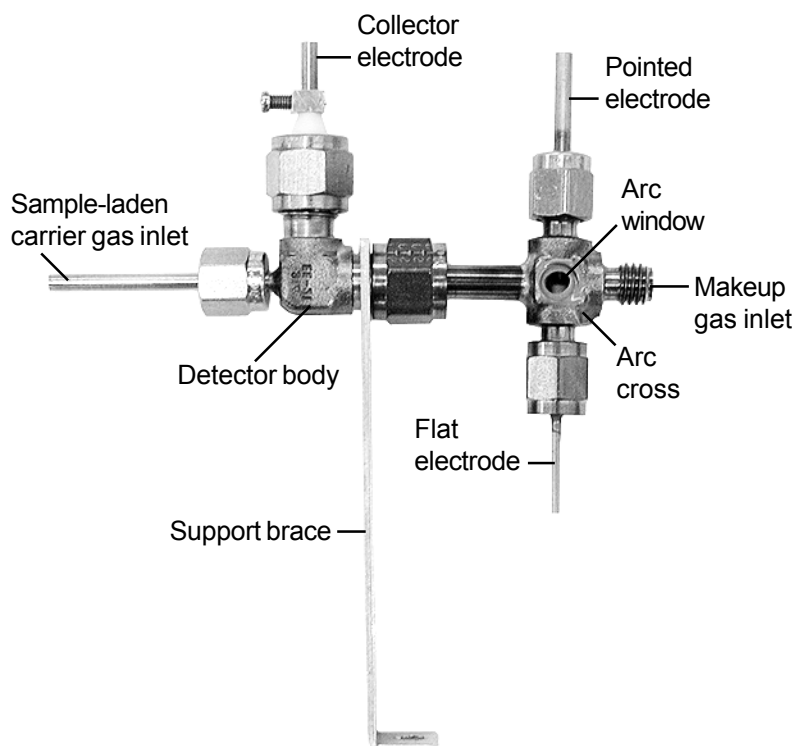


HID detector between TCD and FID detectors on an SRI GC

Close-up of the same HID detector



HID detector removed from GC and heater block

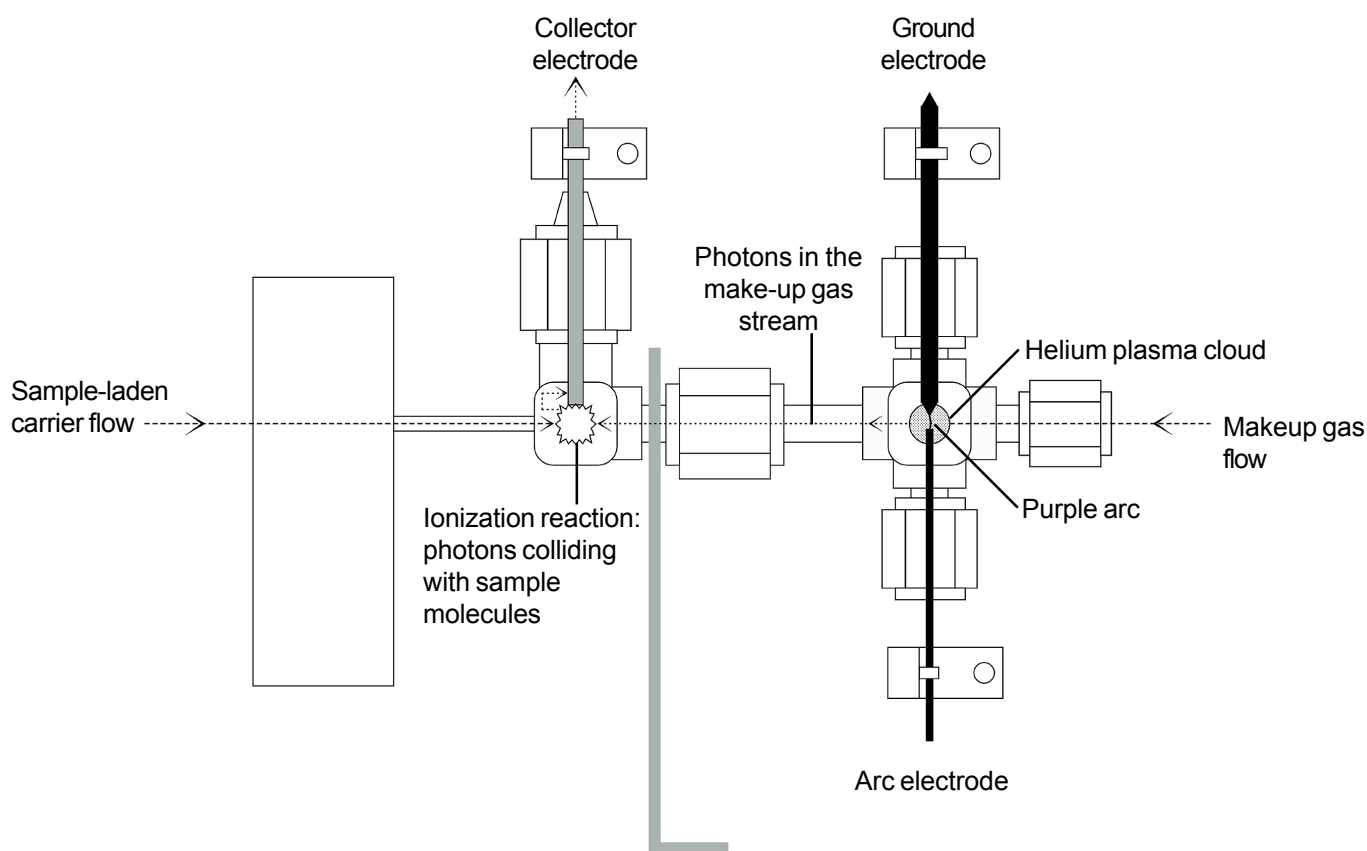


DETECTORS

HID - Helium Ionization Detector

Theory of Operation

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

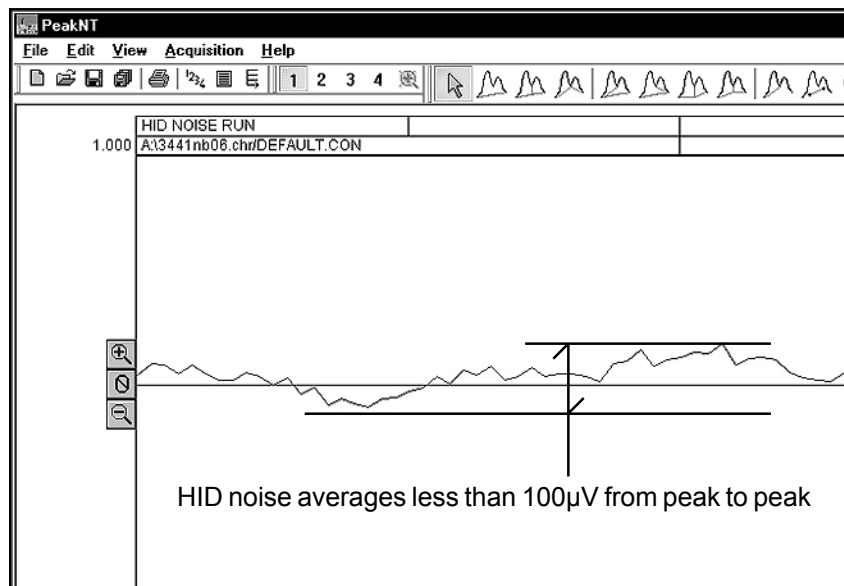


NOTE: If the arc electrode is covered with Teflon™ (translucent) insulation, it should leave 1mm of its tip exposed. If the flat electrode is covered with ceramic (white) insulation, then the tip should be flush with the edge of the insulation sleeve. There should be a 1-2mm gap between the arc electrodes,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

DETECTORS

Helium Ionization Detector - HID

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

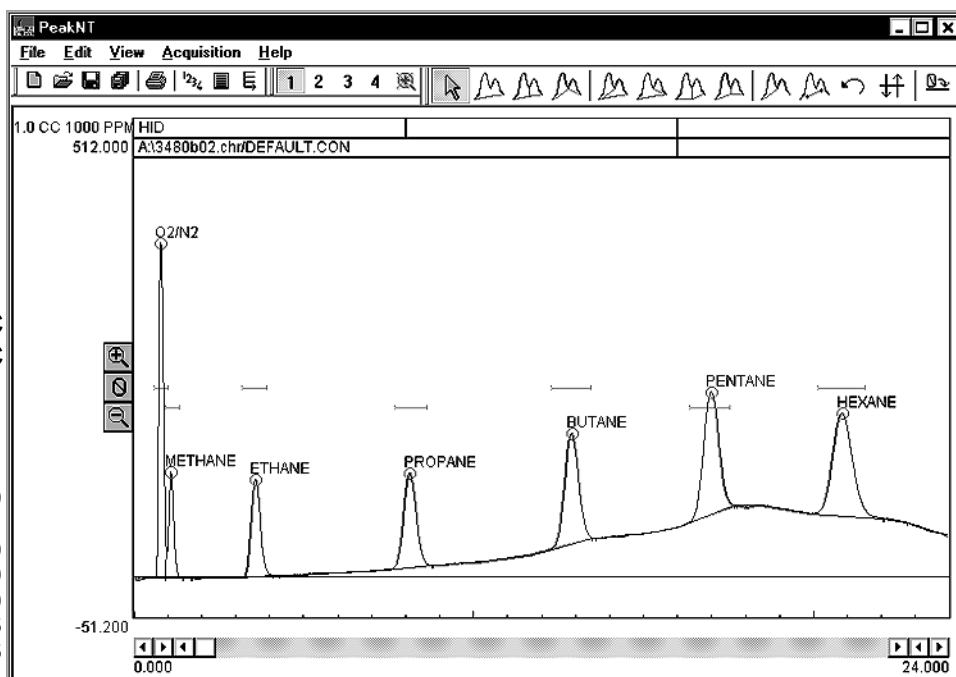
Test Analysis of 1cc 1000ppm C₁-C₆

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
O ₂ /N ₂	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.

DETECTORS

Helium Ionization Detector - HID

HID Make-up Gas Flow

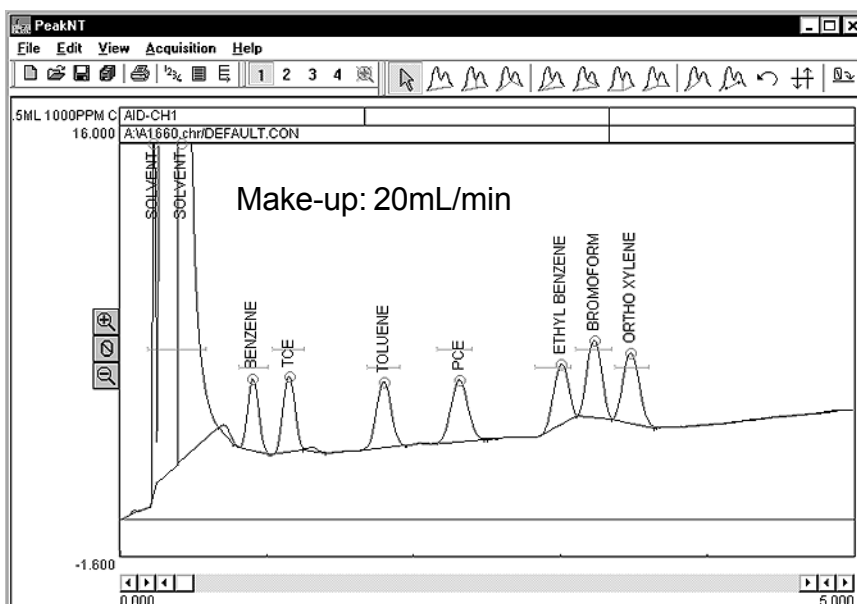
The following chromatograms were produced by an SRI HID equipped GC. Excepting the make-up gas flows, all run conditions are identical. The first chromatogram resulted from a make-up gas flow of 20mL/min. Drastically different in appearance from the first, the second chromatogram was produced with a 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 C₁-C₆
 Column: 30m MXT1-5
 Carrier: Helium @ 10mL/min

Temperature program:
 Initial Hold Ramp Final
 50°C 1.00 10.00 140°C

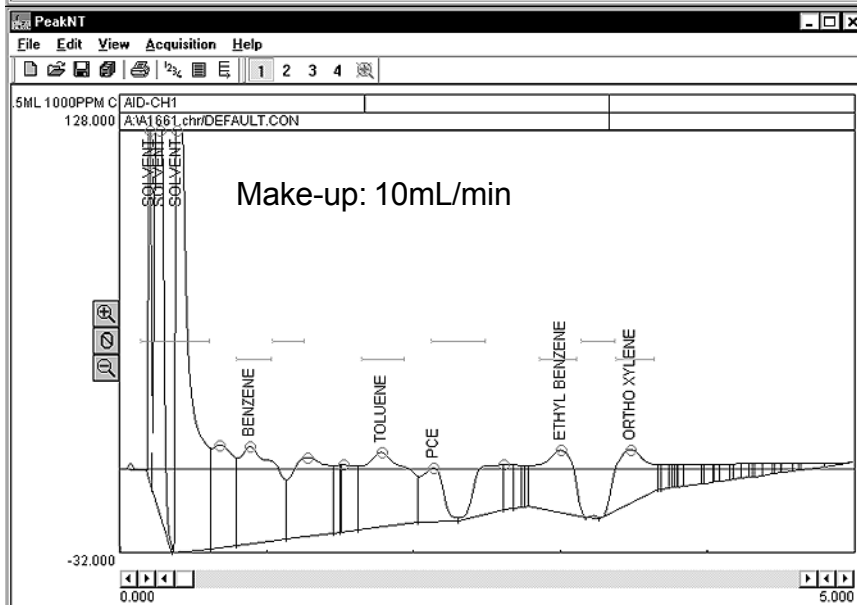
Results:

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

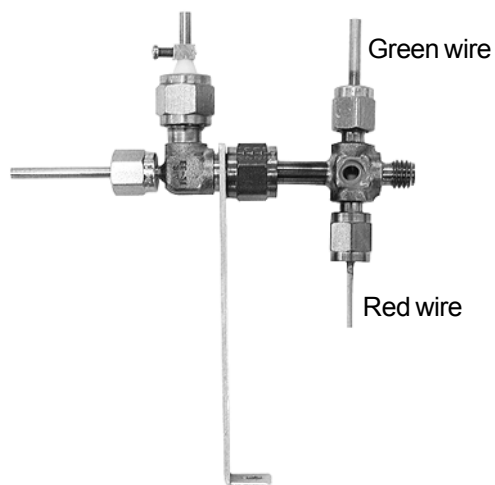


DETECTORS

HID - Helium Ionization Detector

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.



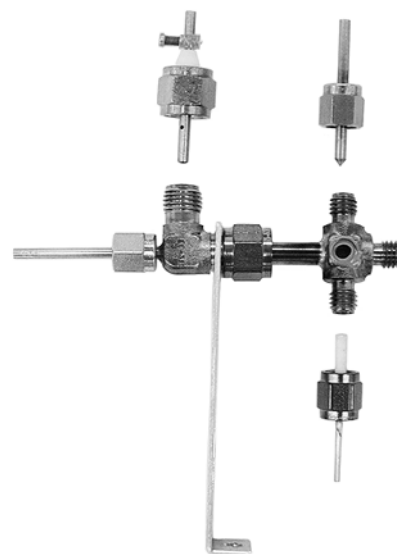
1. Unclip the amplifier lead and slide it off the collector electrode. Unclip and remove the leads from the pointed and flat electrodes (note that the green wire is connected to the pointed electrode, and the red wire is connected to the flat electrode).

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

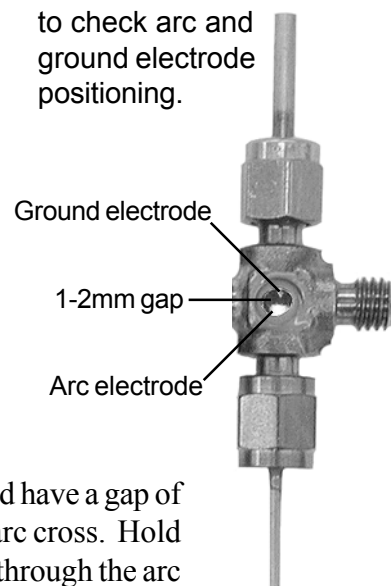
3. Remove the collector electrode by loosening the 1/4" fitting that secures it in the detector body.

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.

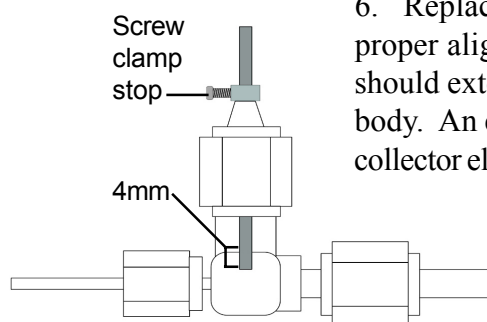
5. Remove any sanding residue from the electrodes using a paper towel optionally moistened with methanol or another quick-evaporating solvent.



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



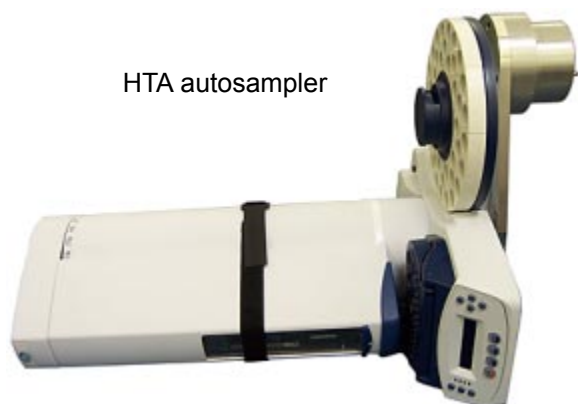
6. Replace the electrodes and check for proper alignment. The collector electrode should extend about 4mm into the detector body. An existing screw clamp stop on the collector electrode should allow replacement without readjustment. Should adjustment be required, loosen the screw clamp to position the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



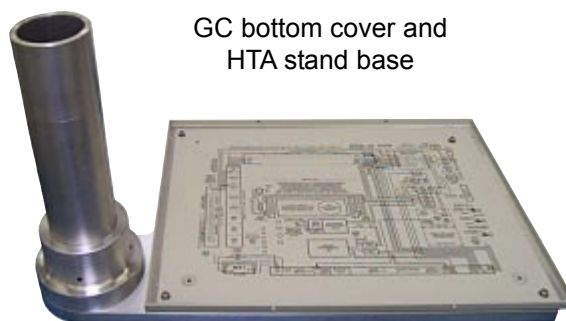
and ground electrodes, remove the arc cross from the detector body by loosening the 1/4" fitting connecting the two parts of the detector (this fitting also secures the support brace). The ground and arc electrodes should have a gap of about 1-2mm (0.040-0.080") between them, with the gap centered in 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.

GC INJECTORS HTA Autosampler

1. The HTA and the GC will arrive in two separate packages: the GC in one crate, the HTA autosampler and base (including a new GC bottom cover) in another. Unpack them, but leave the black strap on the HTA (as shown below) until it is set up and ready to operate. Lay the HTA on it's side as shown. Place the GC on a level surface so you can work on it.

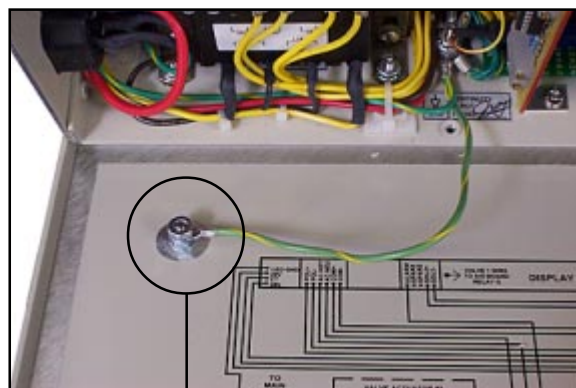


HTA autosampler



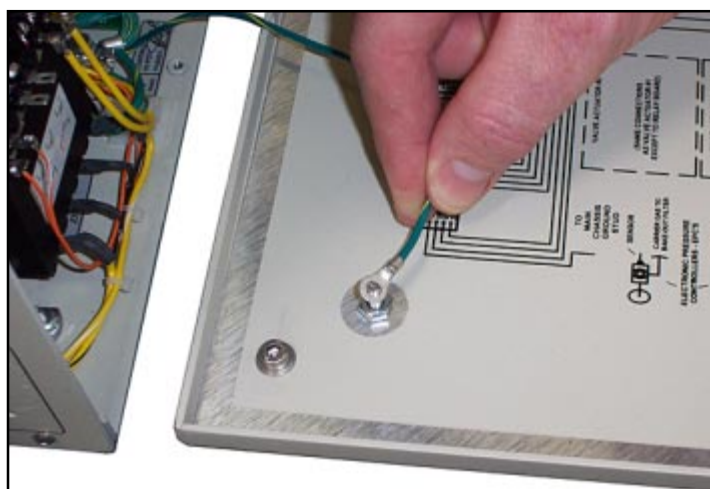
GC bottom cover and
HTA stand base

2. Remove the bottom cover from the GC by unscrewing and removing the six phillip's head screws that secure it to the GC. Rock the GC gently onto it's back and pull the bottom cover off toward you, as shown. While the GC is in this position, remove the green and yellow ground wire from the bottom cover.



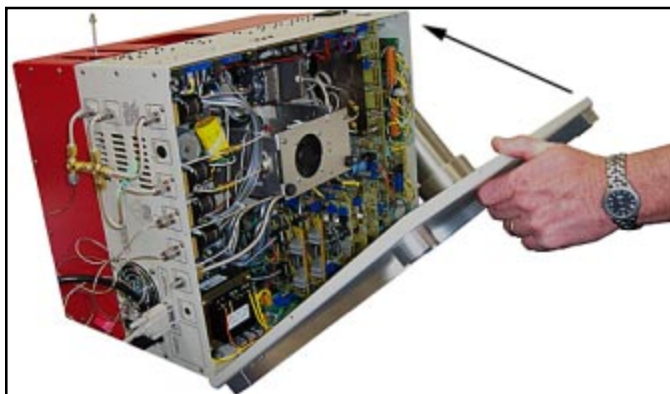
The GC ground wire is on the left-hand side of
the bottom cover

3. Place the new bottom cover and HTA stand base next to the GC, and attach the green and yellow wire to the bolt with the washer and nut from the old GC bottom cover.



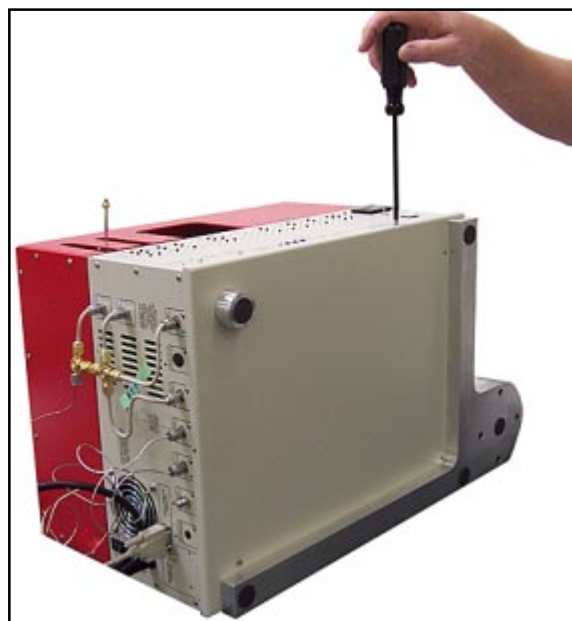
GC INJECTORS

HTA Autosampler



4. Attach the new bottom cover and HTA stand base to the GC.

5. Secure the new bottom cover and HTA stand base to the GC with the six phillip's head screws from the old GC bottom cover.



6. Rock the GC back up onto its base. It is now ready to attach the HTA and the top part of the stand.

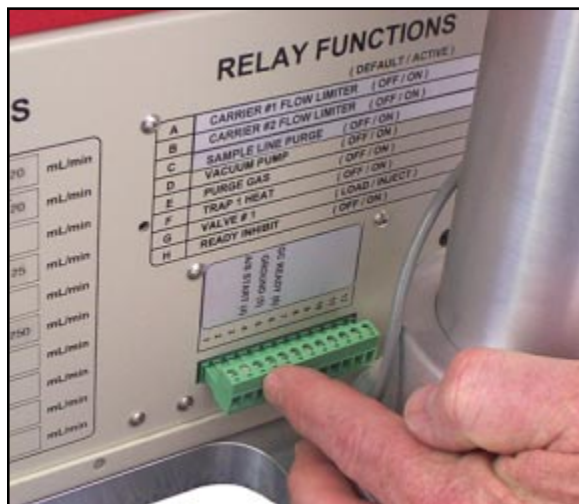


GC INJECTORS HTA Autosampler

7. The top of the stand is bolted to the HTA. Align the top of the stand with the base, and lower it onto the pole. It should slide right into place. Swivel the HTA into place over the GC injection port as shown. There is a latch on the red GC lid to hold the HTA in place; gently push it toward the back of the GC until you hear a “click.”



8. Plug the power and interface cables into the rear of the HTA as shown at right. Remove the black strap from the HTA.



9. Plug the other end of the HTA interface cable into the left-hand side of the GC under RELAY FUNCTIONS as shown at left.

10. The HTA comes with its own manual from the manufacturer. A separate document provides instructions for a process called “Border Markout” to align the HTA with the GC injection port. This process should be run one time after shipping to make sure the HTA and the GC injector port are aligned.

Upon receiving the chromatograph and data system from the freight carrier, immediately inspect the containers for visible signs of damage. If any external damage is observed, notify the delivery person immediately. If no external signs of damage are present, proceed to inspect the contents of the containers. If the materials appear to have been damaged in shipping, immediately contact the carrier and submit a written report describing the extent of the damage. All packing materials and containers should be retained if damage is discovered until the carrier has been able to inspect the damaged goods. If no damage is discovered, packing should be retained until proper unit operation has been established. The chromatograph, serial data interface cable and manual are shipped in one container, along with the GC accessory kit. This container is a reusable plastic shipping container. These containers are rugged and shipped easily via freight carriers. Most importantly, the plastic container protects larger, more complex and delicate instruments from costly damage to external accessories. Save the packing materials after removing the chromatograph, for future transportation.

The contents of the containers should be checked against the packing slip accompanying the shipment. Verify that all specified accessory items ordered such as columns, syringes and the like have actually been shipped. If any items have been omitted or are missing from the shipment, contact SRI Instruments for location and/or replacement of the item.

The SRI model 8610C gas chromatograph requires either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, dependent on which AC power supply was specified when ordered. Both AC power supplies support the 3-prong grounded outlet. Proper grounding is required to minimize AC line interference and eliminate ground loops. The 220 VAC plug is keyed so that it cannot be inserted into a 110 VAC receptacle. A generator or high-current inverter may be used for operation from a vehicular power source. If an AC power generator is used, as is done in the field, line voltage and/or current may fluctuate. Appropriate steps should be taken to minimize any inconveniences caused by line noise or an irregular AC waveform.

A standard model 8610C gas chromatograph measures approximately 18.5" x 14.5" x 12.5" and requires a counter surface space of about 32" x 22". Eight inches of clearance are needed in front of the left side control panel for the fan, gas line access and the AC power switch. Another six inches of clearance are suggested in front of the right control panel and to the rear of the unit for safe operation and ease of access during routine service. The red oven cover requires a clearance of at least 24" (measured from the counter top) in order to provide adequate access to the column oven for service. If the chromatograph is equipped with optional accessories such as the 10 station purge and trap autosampler for the optional built-in EPA Style purge and trap, the access to the left side of the chromatograph must be increased by a minimum of an additional 12". The compact footprint of the system is economical on lab counter space and is ideal for mobile environmental installations.

Prior to placing the chromatograph into service, the gas supply and related plumbing should be installed and routed. The gas cylinders should be located outside the lab where possible, with only the lines plumbed inside to the chromatograph. Gas cylinders should be secured in place with chain or nylon strap to prevent a cylinder from falling and snapping off the valve. **A gas cylinder contains up to 2700psi and can become a deadly projectile if the valve stem were snapped off.** A regulator should be used to set the supply a gas pressure reduced to a value appropriate for introduction into the GC. Gas pressures at each cylinder pressure regulator should be maintained reasonably above the carrier gas regulator setting in order to provide a range of control (a supply pressure set to no more than 20psi greater than the EPC setpoint is recommended). A block valve should be inserted on the output side of the regulator to permit line service when needed, and to permit immediate shut-off in case of emergency.

Refrigeration-grade copper tubing may be used for all of the gas supply lines to the chromatograph. Plastic tubing should never be used as it permits contaminants, including oxygen, to permeate and this can cause damage to thermal conductivity detectors (TCDs) and capillary columns, in addition to degrading the performance of the electron capture detector (ECD) system. Except in the case of the ECD detector, copper tubing destined for gas supply lines may be rinsed out with methylene chloride, followed by methanol. If the tubing is destined for use with the ECD, do not use methylene chloride or any other halogenated solvent as this would wreak havoc upon the detector indefinitely. It is preferable to switch to 1/16" stainless steel tubing, if available, for the ECD gas lines. It is also a good idea to flame the tubing with a torch while running clean carrier gas through it so that any possible pre-existing contaminants will be eliminated from the tubing run. The tubing is heated until it changes color.

In order to eliminate moisture from the gas supply lines, it is recommended that molecular sieve filters be installed in all of the gas supply lines. SRI 8610C gas chromatographs are factory-equipped with electrically heated 1/8" x 3" molecular sieve-filters on the carrier and sparge gas lines. Although not indispensable, an oxygen filter is a worthwhile optional addition to an ECD carrier gas supply line. Extremely pure gas should be used exclusively on the ECD detector (99.9995% purity).

When routing the gas lines, care should be taken to avoid creating spots where moisture can gravitate and accumulate. Also, gas lines should not be routed near electrical outlets due to the potential for short circuiting created if the bare tubing were to come into contact with exposed electrical contacts, instantly melting the tubing at the short circuit site and releasing gas into the area. If the gas were flammable, a torch-like flame might be produced. If the gas did not ignite immediately, an explosion hazard would be created.

Once the gas line connections have been made and leak-tested, and the gas chromatograph has been located in the counter-top position where it will be used, plug the GC into a properly grounded AC outlet, and energize the unit. Gas pressures may then be adjusted to proper operating conditions by means of the gas pressure setpoint trimpots located under the red protective oven cover. Please review the section regarding the setting of these setpoints for specific information regarding their use. Connect the 6' DB-9 serial cable to the RS-232 connector on the left side control panel of the GC, and connect the opposite end of the cable to the COM port to be used for communications on the PC. At this point, start the PeakSimple program and wait for the main chromatogram screen to appear.

Once the PeakSimple program is running, select the FILE- CONTROLS - CHANNELS menu (CONTROLS - CHANNELS - DETAILS menu in the MS-DOS version) and observe what temperature the default temperature is programmed to. This temperature should also be displayed on the chromatograph's LED display when digital display has been toggled on to OVEN ACTUAL position. If these two figures do not match within two degrees after a few minutes, select the CHANNELS - TEMPERATURE menu again and verify that if there is a temperature program loaded into memory, that it meet your requirements. Otherwise you may edit, replace or clear the displayed temperature program. Return to the main screen. If the temperatures match, then the data system is communicating with the chromatograph.

If there is no response from the chromatograph data system to the PC, the port address (and/or data acquisition type in the MS-DOS version) information may be set incorrectly in the OVERALL screen (DETAILS screen in MS-DOS) for each channel. This will typically produce the "Channel 1 not functioning" message. Verify that the proper hardware settings have been implemented. Once this has been done, communication between the chromatograph and the data system is typically established by activating the channel in the CHANNELS screen. Now the system may be adjusted to operating conditions.

8610C Power Consumption

7/16/2002

Basal Power

With no zones heating, Power Usage = 50W

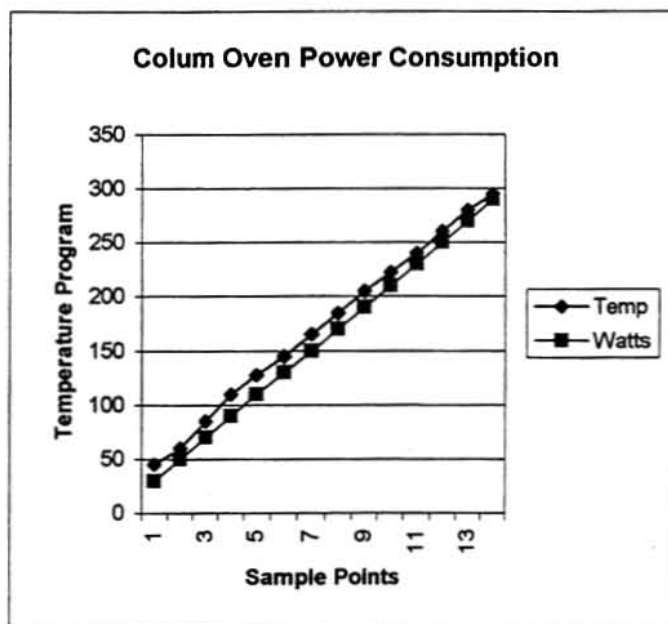
With 2 Detector zones heating = 150W

With Detector Zones Stabilized, Total Basal Power = 100W

Column Oven

Temperature Program 40C to 300C @ 5C/min

Average Temp	Watts	Temp Range
45	30	40-50
60	50	50-70
85	70	70-100
110	90	100-120
127.5	110	120-135
145	130	135-155
165	150	155-175
185	170	175-195
205	190	195-215
222.5	210	215-230
240	230	230-250
260	250	250-270
280	270	270-290
295	290	290-300



Maximum Power Usage

Ballistic Heating to 300C = 675W

Total Power = (Basal + Detector + Column Oven) = 825W

Isothermal Power Usage

Column Oven Stabilized @ 300C

2 detectors @ 150C

Total Power = (Basal + Detectors + Column Oven) = 400W

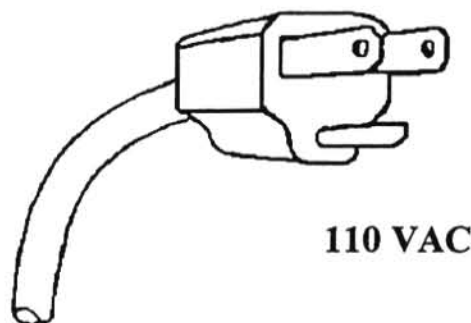
Chapter: INSTALLATION

Topic: Power Supplies and Space Requirements

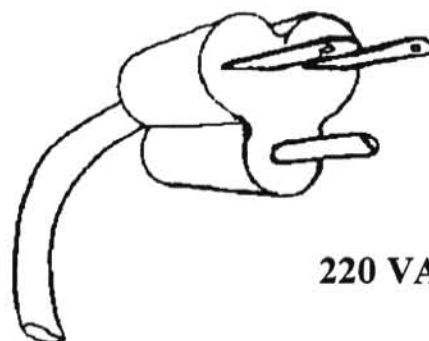
Once the equipment has been removed from all the packing material, check the contents of the container against the packing slip and make sure everything listed is included. If any item(s) have been omitted or are missing, contact SRI Instruments for location and or replacement of the item(s).

The SRI model 8610C gas chromatograph requires AC power at either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, depending on the AC power ordered. Both AC power supplies are equipped with a three prong grounded outlet (see diagrams to the right). Proper grounding is required for safe operation. Do not disable the ground prong under any circumstance. These plug configurations are for EIA standard U.S. outlets. It may be necessary to replace the plug provided with a local standard plug.

A standard SRI 8610C GC measures 18.5" X 14.5" X 12.5" and requires a minimum counter space of 28" X 22" X 23.5" for proper operation (see diagram to the right). Roughly 8" of clearance beside the left side control panel is needed for data cable, gas line and power switch access. 6" of clearance to the rear of the GC and 11" of clearance above the GC is required. This will provide adequate access to the column oven for maintenance and provide space for proper GC ventilation. To the front and right side, 1.5" of clearance should be adequate to prevent the GC from coming into contact with surrounding objects or falling off the counter. The right side of the GC does contain general information on your instrument and some operators may want additional clearance for easy reference. The front control panel of the GC should be easily accessible in order to properly monitor digital display and control operating conditions, as well as providing access to the injection port for sample injections.

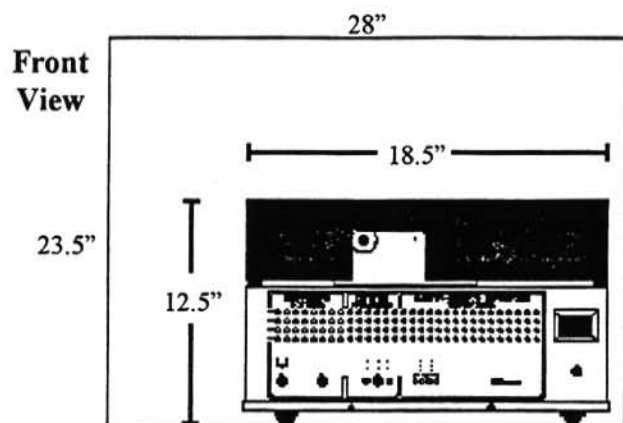
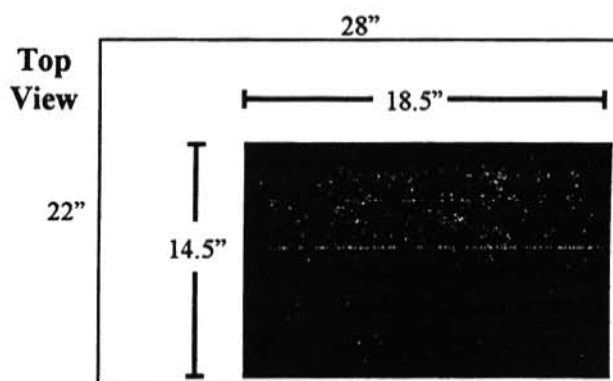


110 VAC



220 VAC

Required Operating Counter Space



Product Bulletin 209

Models 7725/7725i and 9725/9725i Sample Injectors for HPLC

Rheodyne 7725/7725i and 9725/9725i front loading HPLC Sample Injectors are easy to use, and provide chromatographers with the Rheodyne Hallmark of Excellence... unparalleled performance and exceptional product life time.

- Patented MBB™ "Make-Before-Break" architecture virtually eliminates transient pressure shocks for extended column life.
- Ceramic stator face.
- Available in stainless steel and PEEK for sample compatibility.
- Zero sample loss.
- Complete and partial loop filling accommodates variable sample volumes.
- 2 μ L internal sample loop accessory.
- Front-end pressure adjustment.
- Wide 30° port angles for easy access.
- Position sensing switch built into "i" models.
- Small diameter internal flow paths assure low internal volume and minimal dispersion.

MBB Flow Design for Pressure Shock Reduction

Rheodyne's exclusive MBB flow architecture virtually eliminates pressure transients. The high pressure flow from the pump is not interrupted when the injector is switched between LOAD and INJECT, a benefit when using flow sensitive detectors, fragile columns, or pumps that are disturbed by flow or pressure transients.

Fig. 2 shows column pressure vs. time for MBB (upper curve) in comparison to non-MBB sample injectors (lower curve).



Figure 1. Rheodyne 7725/7725i stainless steel (left) and 9725/9725i PEEK (right) front loading sample injection valves for HPLC.

A passage in the stator face makes new connections before old ones break. This patented "Make-Before-Break" or "MBB" design is an improvement over injectors which use a bypass; it is easy to troubleshoot, and does not dilute sample.

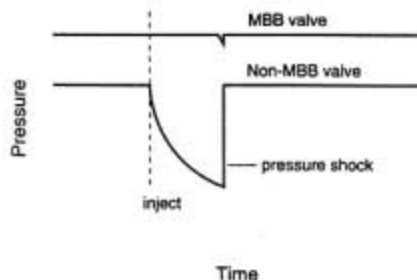


Figure 2. Rheodyne's MBB 7725 and 9725 series sample injectors virtually eliminate pressure transients characteristic non-MBB sample injectors.

Mechanical Design

The interface between the rotor and stator face is the location of the flow switching and high-pressure sealing. This interface is a flat surface, and consists of a

rotor seal made of an inert polymer, and an alumina ceramic stator face assembly. The highly-polished alumina ceramic does not scratch or wear under normal use.

As a result, a rotor seal should last for tens of thousands of injections in normal use, and shed very few particles.

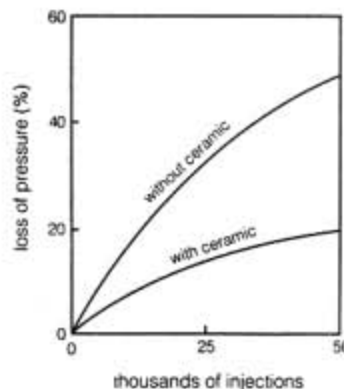
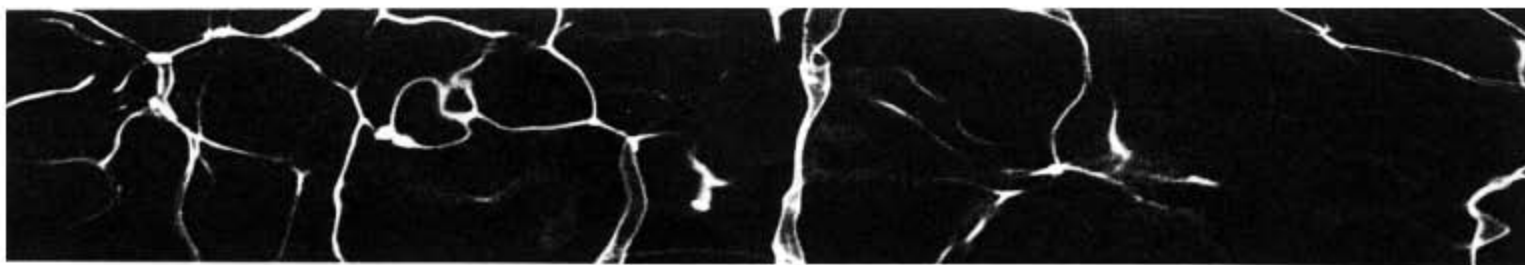


Figure 3. Pressure loss of stainless steel/polymer valve (top) and Rheodyne ceramic/polymer valve (bottom) vs. thousands of injections.



Position Sensing "i" Injectors

In the 7725i and 9725i valves, turning the valve to INJECT closes a built-in position sensing switch, which remains closed until the valve is returned to LOAD. When the position sensing switch wires are attached to a chromatograph, the switch provides the system with a reproducible start signal.

Zero Sample Waste

A syringe needle with a flat end (square cut) **must** be used. When the needle is inserted into the needle port, the tip of the needle passes through a seal inside the rotor seal, and then contacts the ceramic stator face. This direct connection between the tip of the needle and the end of the loop eliminates sample holdup. All sample leaving the needle enters the loop, so there is zero sample waste.

Leak Tight Needle Seal

The needle seal is a Teflon sleeve that is built into the rotor seal. It grasps the tip of the needle, aligning it with the loop passage, and assures that all sample dispensed from the syringe enters the loop. The seal is under compression from an internal spring, which maintains a leak-tight, self-adjusting seal around the needle.

Sample Injection

The needle port of each injector is built into the valve's handle. When the injector is mounted on a panel, the handle and needle port are in front (Fig. 4A). In back of the panel are the body and the stator. The stator contains six tubing ports (Fig. 4B) for connecting the pump, column, sample loop, and vent lines.

Fig. 4A shows a cut-away view of the valve in both the LOAD and INJECT positions. Below the cut-away view is an illustration of the internal flow passages. The six circles represent the ports in the stator. The arcs represent the connecting passages in the rotor. The needle port is shown aligned with port 4.

In the LOAD position mobile phase flows to the column via port 2, a rotor passage, and port 3. The loop, which contains mobile phase trapped when the injector was returned to LOAD, can be partially or

completely filled with sample from a syringe via the needle port, which aligns with port 4. The mobile phase displaced by the sample exits the loop via drain at port 6.

With the syringe still in the needle port the valve is turned to clockwise through 60° to INJECT. Channels in the rotor seal now direct the mobile phase into the end opposite from where the sample entered the loop. Note that the sample travels in a direction opposite to the direction during loading; it does not have to pass through the entire loop. The needle port and syringe now align with drain at port 5. When the needle port is flushed, the flushing solvent exits directly out this port without entering the sample loop. The needle port and syringe are never exposed to high pressure. The loop is self-cleaning, being continuously flushed by mobile phase during analysis.

VERSATILE INJECTION METHODS

Rheodyne front loading injection valves operate as follows: Sample is first **LOADED** into the sample loop by dispensing it from a syringe (Fig. 5) or by pulling it through a dip tube using a Suction Needle Adapter (Fig. 6). Sample is then **INJECTED** onto the column by turning the handle, which connects the loop to the high-pressure mobile phase stream. These versatile injectors offer a choice of three loading methods: Complete, Partial, and Suction.

Sample Injection using Complete Filling Method

When injection volumes do not often change, or when sample conservation is not required, complete filling is the method of choice. The complete fill method produces excellent volumetric precision, typically about 0.1% relative standard deviation. Sample is dispensed from a syringe, using an excess amount

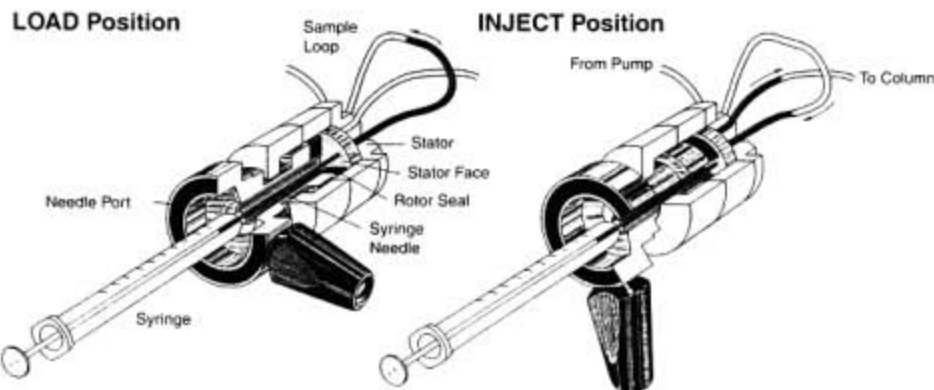


Figure 4A. Cut-away illustrations (front view) of valve showing sample **LOAD** to the sample loop and sample **INJECT** onto the column.

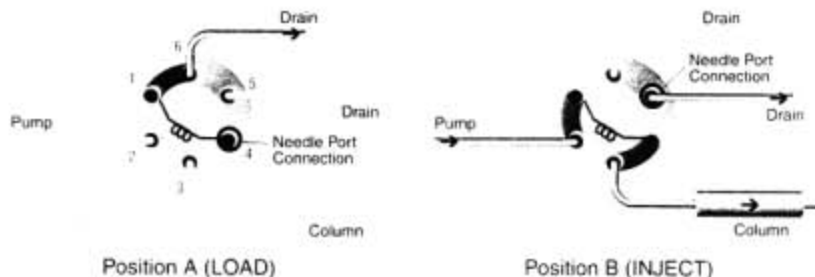
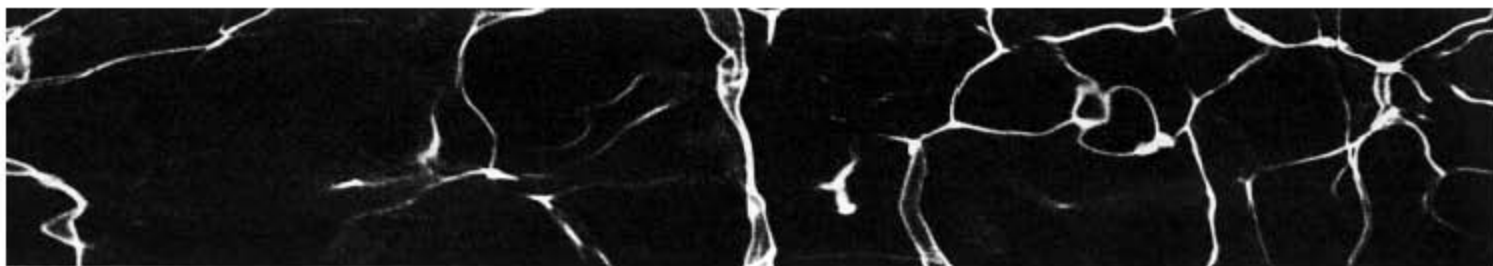


Figure 4B. Illustrations of flow paths during **LOAD** and **INJECT** as viewed from the rear of the valve. Flow passages at the interface of the rotor seal and ceramic stator face are shown as grooves. The needle port connection is shown as a dark circle. MBB passages are below the plane of the interface and are not shown.



(at least 3 times the loop volume) to displace all the mobile phase in the loop. The loop sets the volume injected; the volume is varied by changing the loop size. Since the loop sets the volume, this method does not require precise use of the syringe.

Sample Injection using Partial Filling Method

Applications where injection volumes change frequently, or when sample conservation is important, partial filling is the method of choice. Volumetric accuracy and precision depend on the operator's ability to read and use the syringe, typically about 1%. The volume injected is determined by the amount dispensed into the loop by the syringe. The amount dispensed must be less than half of the total loop volume. The partial fill method does not waste sample, and allows the volume to be continuously varied without changing the loop.

In partial filling (load no more than 50% of the loop volume), all of the sample dispensed from the syringe needle enters the loop, even with samples as small as 0.1 μL . There is zero sample waste.

Sample Injection using Suction Method

Rheodyne 9725 (PEEK) valves have no metal in contact with the flow stream. The preceding methods expose sample to the metal needle of the syringe. Though the exposure is small, metal can be completely avoided by using a syringe to suck sample into the loop via a PEEK tube at port 6. Using this method the tube end is placed into the sample vial. An empty syringe is inserted into the needle port, and used to pull sample into the loop. Model 9125-076 Suction Needle Adapter, an accessory listed under ordering information, is recommended for the suction method. As shown in Fig. 6, the adapter makes suction loading easier.



Figure 5. Sample loading by dispensing from a syringe. The sample injection valve is shown mounted to a Model 7160-010 Valve Angle Bracket. A Model 7200 2.5 μL syringe is shown.



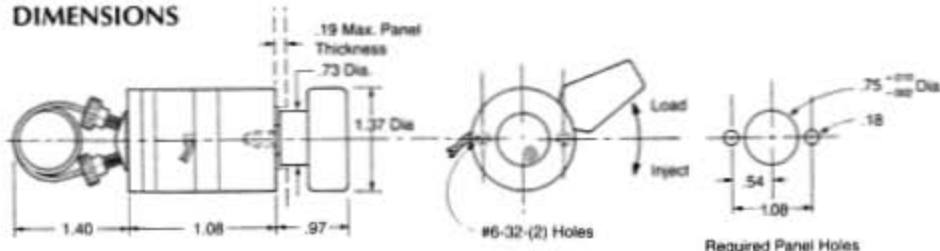
Figure 6. Sample loading by suction through a dip tube. A Model 7252 2.5 mL syringe is shown connected to the valve needle port using a Model 9125-076 Needle Port Adapter.

7725 and 9725 Sample Injectors for HPLC



TM

DIMENSIONS



RheBuild™ Kits

RheBuild Kits are available for all Rheodyne valves... all parts, tools, and instructions to maintain precision performance.



SPECIFICATIONS

Stainless Steel Model: 7725 and 7725i

Pressure Set at 345 bar (5000 psi).
Adjustable to 482 bar (7000 psi).

Wetted Surfaces 316 stainless steel, alumina ceramic, and an inert polymer.

pH Range 0-10 (pH >10, ask factory)

Temp. 80°C max.

Supplied 20 µL sample loop, fitting set for tubing connections, hex keys (2), needle port cleaner, mounting screws (2), and vent tubes (2).

PEEK Model: 9725 and 9725i

Pressure Set to 354 bar (5000 psi).

Wetted Surfaces PEEK, alumina ceramic, and an inert polymer.

pH Range 0-14 (Tefzel)

Temp. 50°C max.

ORDERING INFORMATION

Stainless Steel Injection Valves

Part Number	Description
7725	Sample Injector
7725i	Sample Injector with position sensing switch

Use only genuine Rheodyne fittings

Stainless Steel Sample Loops for Stainless Steel Valves

Part Number	Description
7755-015	2 µL
7755-020	5 µL
7755-021	10 µL
7755-022	20 µL
7755-023	50 µL
7755-024	100 µL
7755-025	200 µL
7755-026	500 µL
7755-027	1 mL
7755-028	2 mL
7755-029	5 mL

PEEK Injection Valves

Part Number	Description
9725	Sample Injector
9725i	Sample Injector with position sensing switch

Use only genuine Rheodyne fittings

PEEK Sample Loops for PEEK Valves

Part Number	Description
7755-015	2 µL
9055-020	5 µL
9055-021	10 µL
9055-022	20 µL
9055-023	50 µL
9055-024	100 µL
9055-025	200 µL
9055-026	500 µL
9055-027	1 mL
9055-028	2 mL
9055-029	5 mL
9055-033	10 mL

Replacement Parts, Fittings* and Accessories

Part Number	Description
-------------	-------------

RheBuild Kits

7725-999
for Models 7725/7725i
9725-999
for Models 9725/9725i

Stainless Steel Fittings*

6000-109
Nuts and Ferrules (5/pk)
6000-110
Ferrules (5/pk)
6000-111
Long Nuts and Ferrules (5/pk)

PEEK RheFlex™ Fittings*

6000-054
Fittings Set (5/pk)
6000-051
Ferrules (5/pk)

Accessories

9125-076
Suction Needle Adapter
7160-010
Valve Angle Bracket
7160
Mounting Panel
7160-029
Ring Stand Bracket Assembly

*Use only genuine Rheodyne fittings



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1.0 DESCRIPTION

Models 7725 and 9725 are seven-port sample injection valves in which the sample is loaded through a built-in needle port in the front of the valve. Model 7725 is the stainless steel version, and Model 9725 is the PEEK version.

Figure 1 shows the flow diagram of the valve. The six circles represent the ports in the valve stator. The triangle represents the make-before-break (MBB) hole located on the stator face. The MBB passage is shown as the dashed line slot. The connecting passages in the rotor seal are shown as the solid line slots.

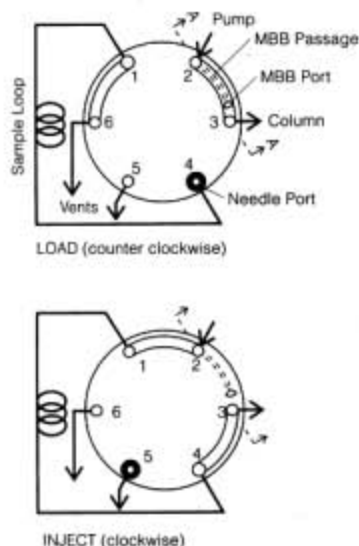


Figure 1: Models 7725/9725 Flow Diagram

A detailed cut-away view (cross section A-A) of the MBB design is shown in Fig. 2.

The sample loop is loaded through the needle port in the LOAD position. Rotation of the knob 60°, switches the valve from LOAD to INJECT. In INJECT the mobile phase flows through the loop. The flow is not interrupted when switched from LOAD to INJECT, or back again. This is due to the MBB flow passages in the stator face which make the new connection before the old one breaks.

There is a 2- μ L internal sample loop (P/N 7755-015) available. It can be installed inside the injector, in place of the stator face assembly.

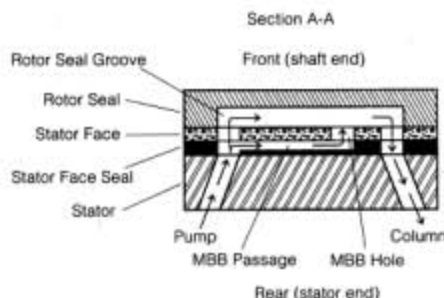
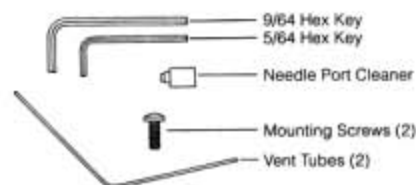


Figure 2: Cut-away View of MBB Design.

2.0 SUPPLIED WITH THE VALVE

Supplied with the valve in a separate bag are fitting sets for tubing connections and the items shown below.



The 22 gauge needle supplied in the valve should be removed from the needle port before using the valve.

3.0 SPECIFICATIONS

- Maximum temperature is 80°C for stainless steel valves, 50°C for PEEK valves.
- Built-in position sensing switch ("i" models only).
- The PEEK valve is set to hold 207 bar (3000 psi). It can be adjusted to hold up to 276 bar (4000 psi).
- The stainless steel valve is set to hold 345 (5000 psi) bar. It can be adjusted to hold up to 482 bar (7000 psi).
- In the PEEK valve the wetted surfaces are PEEK, alumina ceramic, and an inert polymer.
- In the stainless steel valve the wetted surfaces are 316 stainless steel, alumina ceramic, and inert polymer.

- Flow is not interrupted when switched from LOAD to INJECT.
- 2- μ L internal loop is available.

4.0 IMPORTANT SAFETY NOTICES

4.1 Warning: When using sample loops larger than 100 μ L, shield yourself from mobile phase coming out of the needle port when the valve is turned from INJECT to LOAD. Example: 1-mL loop ejects 20 μ L upon decompression from 200 bar.

4.2 Warning: When using the Needle Port Cleaner, empty the syringe slowly to prevent solvent from squirting back at yourself.

4.3 Caution: Use the correct size syringe needle to prevent damage to the valve.

4.4 Caution: When using the PEEK valve use only plastic ferrules in the stator ports. Metal ferrules can cause irreparable damage to the plastic stator.

4.5 Caution: Rinse the valve after using buffer solutions to prevent crystals from forming, which can cause scratches on the rotor seal.

4.6 Caution: Due to the 30° ports, sample loops for Model 7725/7725i are not interchangeable with loops of many other stainless steel Rheodyne valves.

5.0 USING PROPER SYRINGES

Use syringes with 0.028-inch O.D. (22 gauge) x 2-inch long needle, without electro taper and with 90° point style (square end). Using the incorrect needle size will damage the injector.

6.0 INSTALLATION

- To mount the valve on a panel, remove the handle by loosening the two handle set screws. Use the two screws supplied to fasten the valve to a panel.
- Replace the handle by tightening the two set screws on the two flats of the shaft.
- Connect the two vent tubes (supplied) to ports 5 and 6. Place the outlet ends of both at the same horizontal level as that of the needle port to avoid siphoning. See Fig. 3.

d) Connect the pump to port 2 and the column to port 3. Leave the column disconnected from the valve during initial flushing.

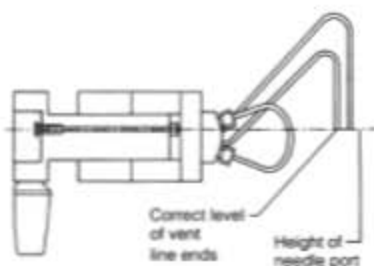


Figure 3: Correct Position of Vent Lines

CAUTION: Use only plastic ferrules in the connections to all the ports of the PEEK stator. Metal ferrules will damage the plastic stator irreparably and void the warranty.

7.0 FLUSHING THE INJECTOR

In INJECT, flush the needle port with 1-mL of mobile phase, using the needle port cleaner as shown in Fig. 4. At this time, the pump flushes the loop.

CAUTION: To avoid liquid squirting back at you, discharge syringe slowly.

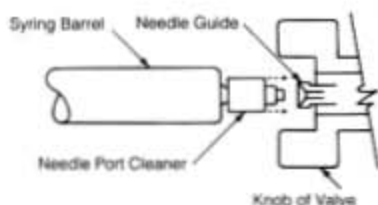


Figure 4: Use of Needle Port Cleaner

8.0 MAKING AN INJECTION

There are basically two methods of loading the sample loop—complete or partial filling. The third method described is for those using the PEEK valve and need to avoid contact with metal.

8.1 COMPLETE LOOP FILLING

In complete filling, the volume of sample injected is set by the volume of the loop (this includes the valve passages). This method produces the highest precision.

Overfill the loop with at least two to three loop volumes of sample. Five to ten loop volumes will provide even better precision.

An excess of sample is needed because mobile phase near the wall of the loop is displaced slowly due to the laminar flow effect shown in Fig. 5.

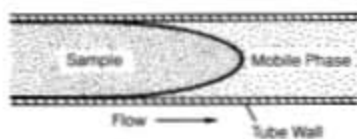


Figure 5: Laminar Flow Effect.

To completely fill the loop:

- See **Warning 4.1** and turn to **LOAD**.
- Insert the syringe into the needle port. You will feel tightness during the last 2-3 mm of travel as the needle passes through the needle seal, it then stops against the stator face.
- Load the sample.
- Leave the syringe in and turn to **INJECT**.

8.2 PARTIAL LOOP FILLING

If you only have small quantities of sample, this is the method of choice. In the partial filling method the volume of sample injected is set by the syringe. In this method, no more than half a loop volume of sample is loaded into the loop. For example, load no more than 10 μ L into a 20- μ L loop. With larger volumes some of the sample is lost out vent tube 6. This is because sample flows down the center of the loop at twice the average velocity due to the laminar flow effect shown in Fig. 5.

To partially load the loop:

- In **INJECT**, use the Needle Port Cleaner to flush out the needle port with about 1 mL of mobile phase. This will flush out contamination from the earlier injection. This liquid will exit out vent tube 5.
- Follow steps a-d in Section 8.1.

8.3 LOADING BY SUCTION

The two methods above expose the sample to the metal needle of the loading syringe which might need to be avoided for those using the PEEK valve. Metal can be completely avoided by using a syringe to suck sample into the loop.

The steps are as follows:

- In **LOAD**, dip the tube attached to port 6 into the sample.
- Insert an empty syringe into the needle port and suck up sample into the loop. Alternatively, use the Suction Needle Adapter (P/N 9125-076, not supplied). Use of this needle adapter is shown in Fig. 6.
- Leave the syringe in position and turn to **INJECT**.

The metal needle of the syringe will contact the sample if an excess of sample is drawn into the syringe, but this excess sample is out of the sample loop and will not be injected. The syringe can be used many times before it needs to be emptied.

To load the loop with all of the available sample and not waste any, the loop should be at least four times the volume of sample loaded. The loop is first filled with mobile phase via the dip tube, then the whole sample is drawn into the dip tube and loop, followed by more mobile phase. The sample is now sandwiched between two zones of mobile phase in the loop.

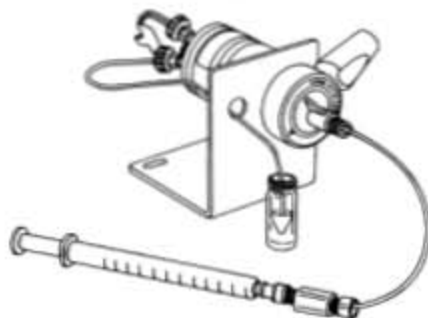


Figure 6: Loading By Suction Using the Suction Needle Adapter.

9.0 ADJUSTING FOR HIGHER PRESSURE OPERATION/LEAKAGE

There is a single pressure adjusting screw at the handle end of the valve body. The handle assembly is designed to be used as a tool for adjusting this screw. If you need operation up to a higher pressure or if there is a leak between the stator and stator ring, loosen the two set screws so that the handle slips down the shaft and the two protrusions fit into the slots on the adjusting screw. Tighten the adjusting screw about 1/20th turn. Use the 20 dial markings on the body and the painted spot on the adjusting screw as guides, see Fig. 8.

Note: When the valve is not panel mounted, the adjusting screw can be hard to turn. In this case, loosen the three stator screws 1/4 turn prior to adjusting the screw. Retighten the stator screws before testing for leaks.

If there is still leakage at this new setting, repeat the process. Finish by retightening the handle set screws onto the flats of the shaft. Replace the rotor seal if the leak continues.

Note: If the vent tubes from ports 5 and 6 do not have their outlet ends at the same horizontal level as the needle port, siphoning can result, which is often misinterpreted as a leak. A siphoning leak will stop when the vent tubes and needle port tube are empty. A leak due to a damaged rotor seal will continue.

10.0 MAINTENANCE

With normal use the valve will give many thousands of injections without trouble. The rotor seal wears with use and is the only part that needs routine replacement.

The main causes of early failure are:

- the wrong needle tip can chip the ceramic stator face which then causes deep scratching of the rotor seal surface or
- abrasive particles in the sample can scratch the rotor seal surface.

10.1 CHANGING THE ROTOR SEAL

To change the rotor seal refer to Fig. 7 and proceed as follows:

- Remove the three stator screws.
- Remove the stator, stator face assembly and stator ring from body.
- Pull the rotor seal off the pins.
- Leave the isolation seal and bearing ring in place.

See Section 10.2 for reassembly.

10.2 REASSEMBLY

CAUTION: On reassembly, loosen the pressure adjusting screw **BEFORE** tightening the stator screws.

To reassemble the valve proceed as follows and refer to Fig. 7:

- Using the handle as described in Section 9.0 loosen the pressure adjusting screw about $1\frac{1}{2}$ turn. Note the original position of the two red dots, see Fig. 8.
- Line up the rotor seal as shown in Fig. 9. The rotor seal slots face the stator.
- Replace the stator ring so that the pin in the 60° stop ring enters the mating hole in the stator ring.
- Put the stator face assembly on the stator. The three pins on the assembly fit into the mating holes in the stator only one way.
- Replace the stator and stator face assembly on the valve so that the pin in the stator ring enters the mating hole in the stator.
- Add the three stator screws. Tighten each an equal amount and until screws are tight.
- Retighten the pressure adjusting screw.
- Replace the handle and tighten the two set screws on the two flats of the shaft.

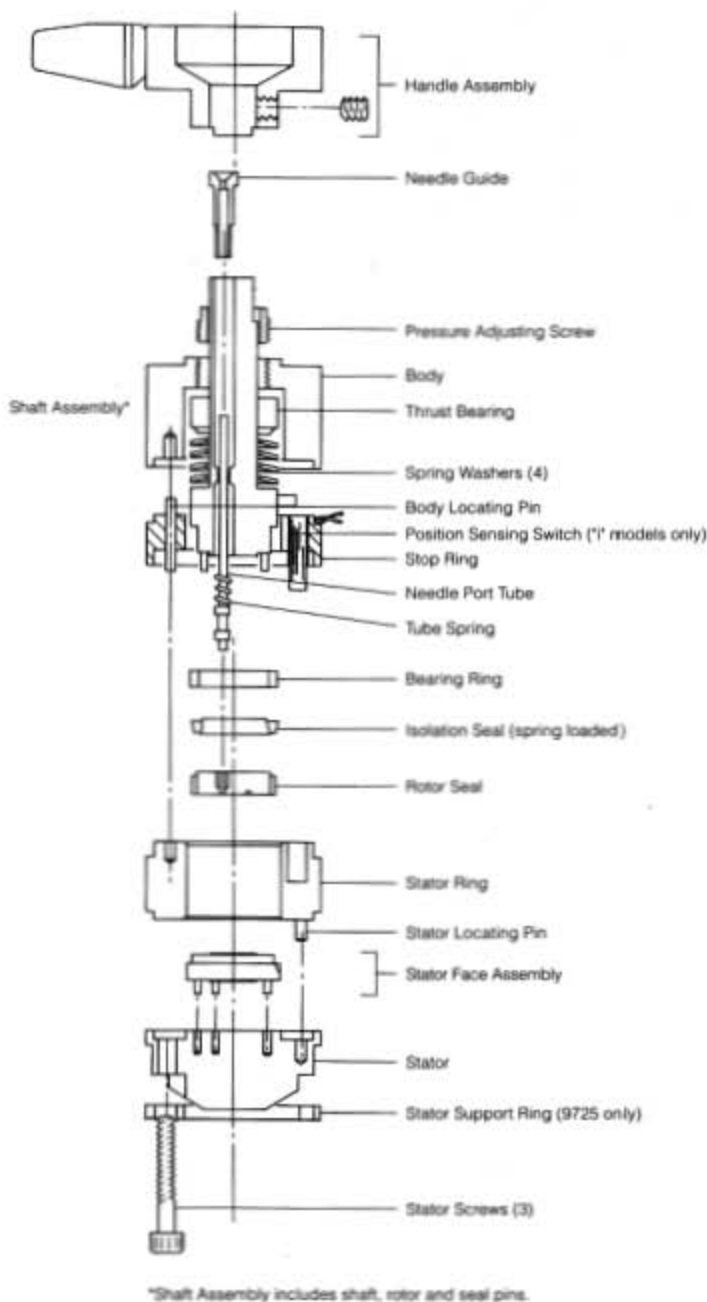


Figure 7: Model 7725/9725 Exploded View



Figure 8: Guides on Adjusting Screw and Body.

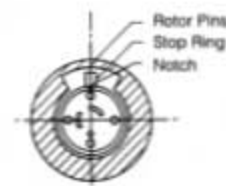


Figure 9: Correct Position of Rotor Seal (slots face the stator).

10.3 POSITION SENSING SWITCH

The position sensing switch is standard on the 7725i and 9725i. It is a magnetic reed switch actuated by a magnet sealed inside the shaft. The switch is rated for 100 volts at 200 milliamperes.

To replace or remove the switch:

- Remove stator, ring and stop ring.
- Pull the switch out of the stop ring.
- Replace with a new switch.
- Follow Section 10.2 to reassemble.

10.4 SPARE PARTS

7125-047 Vespel Rotor Seal (standard for 7725/7725i)

7125-079 Tefzel Rotor Seal (high pH for 7725/7725i)

9125-082 Tefzel Rotor Seal (standard for 9725/9725i)

11.0 OPERATING SUGGESTIONS AND TROUBLESHOOTING

11.1 LEAKAGE

If you see liquid between the stator and stator ring, or from the needle port or a vent tube, tighten the pressure adjusting screw as explained in Section 9.0. If this fails to stop the leak then replace the rotor seal.

11.2 NEEDLE SEAL LEAKAGE

Since the outside diameter of syringe needles can vary, the needle seal (Teflon sleeve in the rotor seal) may not seal correctly around a needle which is smaller than average. This will result in a loss of accuracy in loading the sample.

To make a good seal, remove the needle from the needle port, push in on the plastic needle guide with the eraser end of a pencil. Repeat if necessary.

11.3 USE OF AQUEOUS BUFFERS OR SALT SOLUTIONS

To prevent the formation of salt crystals in the valve which can scratch the rotor seal, flush out the flow passages and the needle port with water after using salt solutions.

11.4 USE OF HIGH pH SOLUTIONS

The standard rotor seal in Models 7725/7725i is Vespel, a polyimide with good wear resistance. It is sensitive to alkaline attack, so avoid solutions having a pH of 10 or more. Use the Tefzel rotor seal for alkaline solutions.

The standard rotor seal in Models 9725/9725i is Tefzel. This rotor seal tolerates the entire pH range, from 1 to 14.

11.5 ACCURACY OF SAMPLE LOOPS

Sample loop sizes are not actual values. The actual volume can differ by $\pm 10\%$ for a 20- μL loop. There is a greater difference for smaller loops. Use partial filling if you must know the actual volume injected.

12.0 WARRANTY

All Rheodyne products are warranted against defects in materials and workmanship for a period of one year following the date of shipment by Rheodyne. Rheodyne will repair or replace any Rheodyne product that fails during the warranty period due to a defect in materials or workmanship at no charge to the customer. The product must be returned to Rheodyne's factory, transportation prepaid. This limited warranty is Rheodyne's sole warranty of its products, and all other warranties, express or implied, including any implied warranties of merchantability or fitness for any particular purpose are hereby disclaimed. Under no circumstances will Rheodyne be liable for any consequential or incidental damages attributable to a claimed failure of a Rheodyne product, even if Rheodyne has been placed on notice of possibility of such damages.

Chapter: Troubleshooting

Topic: Leak Checking your GC

There are 3 ways to check for gas leaks in a GC.

The 1st method of leak checking is called "**looking for bubbles**" or "snooping the fittings". Snoop® is a specific brand of leak check solution, but SRI suggests a mixture of isopropyl alcohol (IPA) and water. The alcohol reduces the surface tension of the water so it flows into the cracks between the tubing and the fitting, otherwise water alone would be fine. Don't use soapy water because if the leak check solution gets inside the GC tubing or fittings, it will contaminate the system.

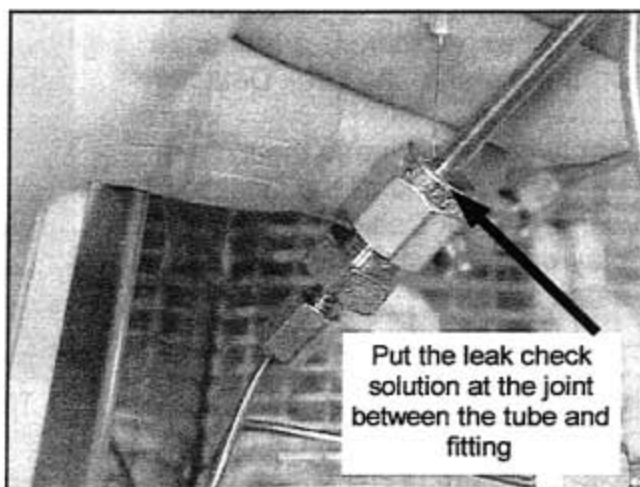
Apply gas pressure to the system then place a droplet or two of leak check solution on the tube connections. If tiny little bubbles are visible then the fitting is leaking.

Using the liquid leak check solution can be difficult however when there are many fittings to test or when some of the fittings are hot, (this will rapidly boil off the leak check solution) making it impossible to tell if there are bubbles from a leak or bubbles from the water boiling away.

Liquid leak checking is the least effective way to check for gas leaks in a GC system.



A low cost disposable medical syringe is perfect for placing a drop of leak check solution at the joint where the tube and the fitting meet.
A 3ml leak check syringe is provided with every SRI GC



Put the leak check solution at the joint between the tube and fitting



Some GC systems may have many places where leaks can occur.

Chapter: Troubleshooting

Topic: Leak Checking your GC

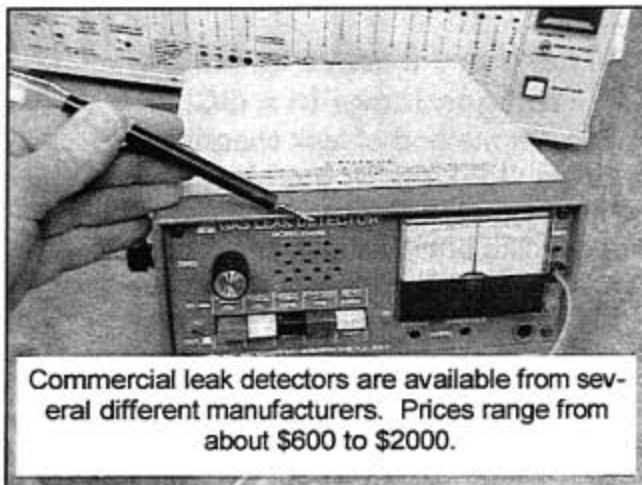
The second method of leak checking is to use a **leak detector**.

Leak Detectors are made by several different manufacturers, but in most cases they consist of a vacuum pump and a thermistor detector which measures the thermal conductivity of the gas that is sucked up through the hand held probe. When helium or hydrogen flows through the thermistor, the thermal conductivity is a little greater than the thermal conductivity of air, so there is a response on the meter of the Leak Detector.

Apply gas pressure to the system then sniff around all the fittings with the Leak Detector. The display on the Leak Detector indicates a leak.

Unlike the liquid leak check solution, the Leak Detector can be used on hot fittings, but is difficult to use if there is any airflow around the fittings (such as in a GC oven with the fan running).

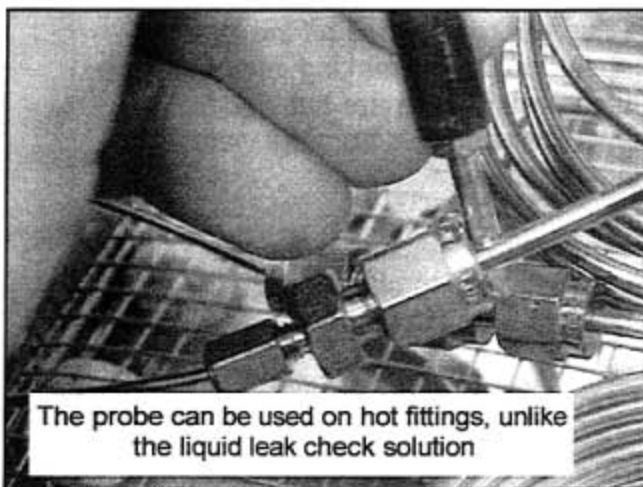
Some leaks may be too small to detect, and some fittings may be in-accessible. Be careful with the probe around live electrical circuits or heaters



Commercial leak detectors are available from several different manufacturers. Prices range from about \$600 to \$2000.



The hand-held probe is used to sniff around the fitting for leaking helium or hydrogen



The probe can be used on hot fittings, unlike the liquid leak check solution

Chapter: Troubleshooting

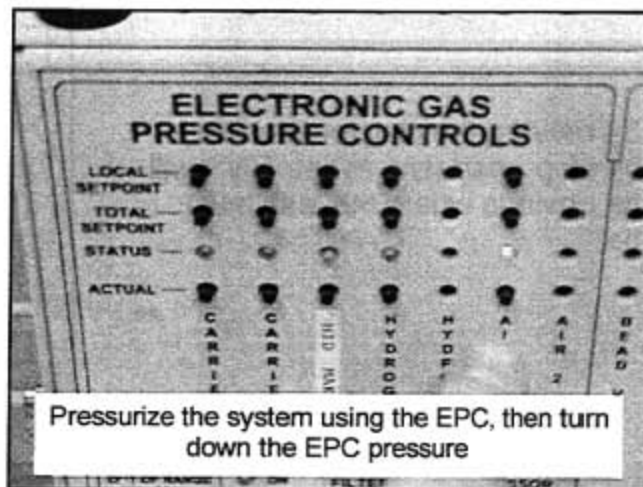
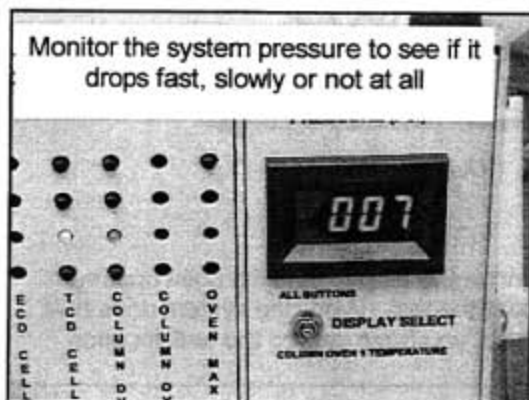
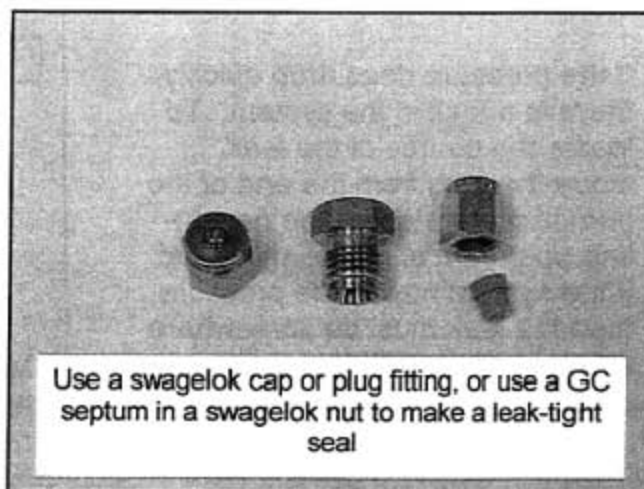
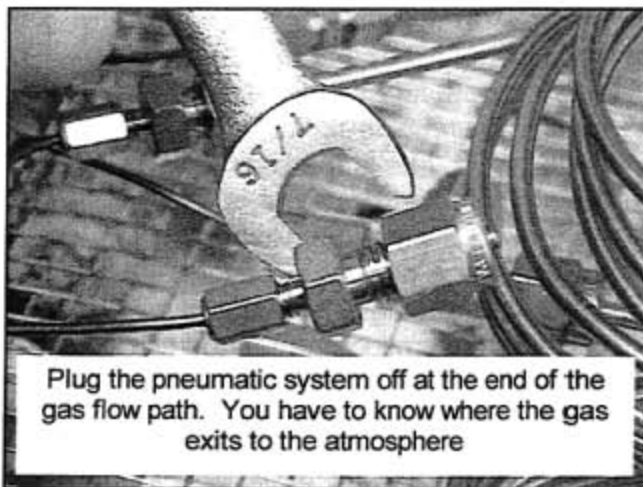
Topic: Leak Checking your GC

The third method of leak checking is called the **"pressure drop"** test.

The pneumatic system is plugged at the end of the gas flow path. This may be the outlet of the detector or the end of the column.

Use a swagelok fitting or a swagelok nut with a GC septum to make a gas tight seal. If the plug leaks, the test will not work.

The system is then pressurized using the EPC (electronic pressure regulator) built-in to the SRI GC. The EPC is then turned down (or off). Because the end of the gas flow path is plugged, the gas is trapped (under pressure) in the pneumatic system. If there is a leak, the gas pressure will drop. If the entire system is leak free, the pressure will remain for many minutes before it slowly drops. The rate at which the pressure drops is indicative of the magnitude of the leak



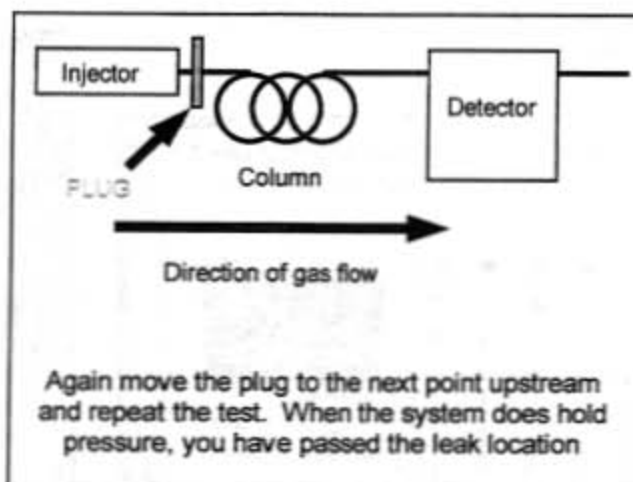
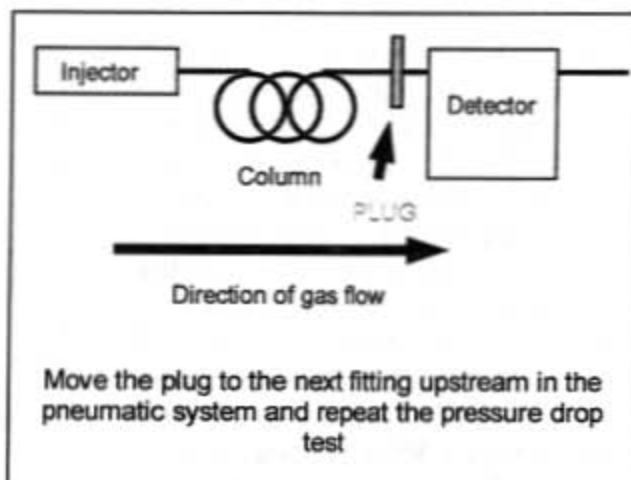
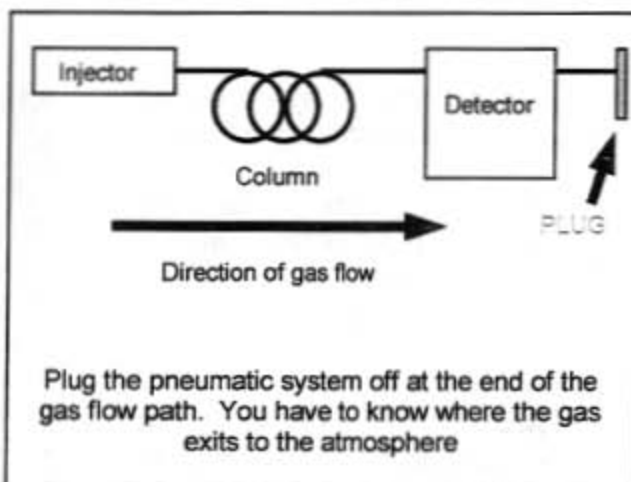
Chapter: Troubleshooting

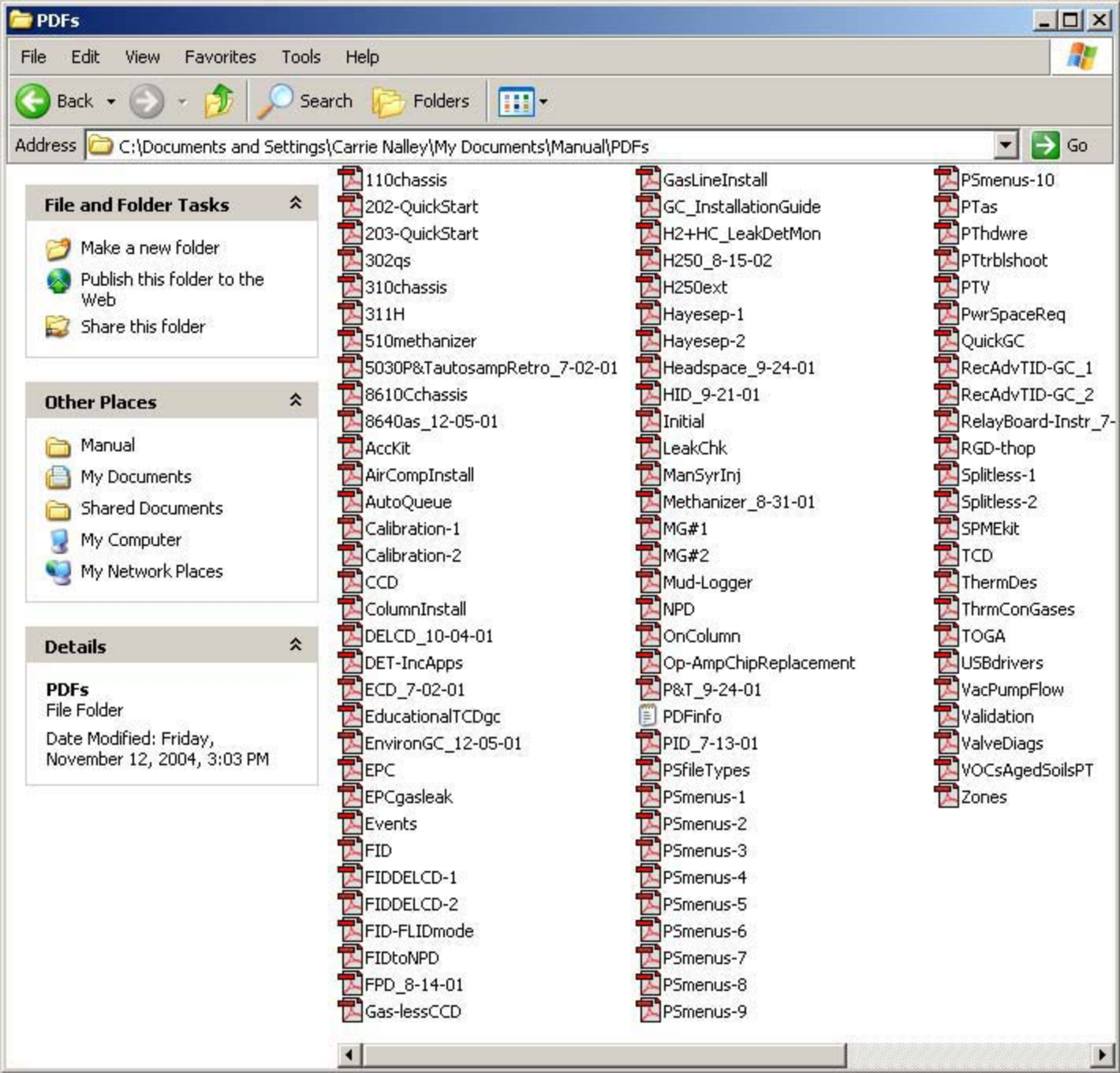
Topic: Leak Checking your GC

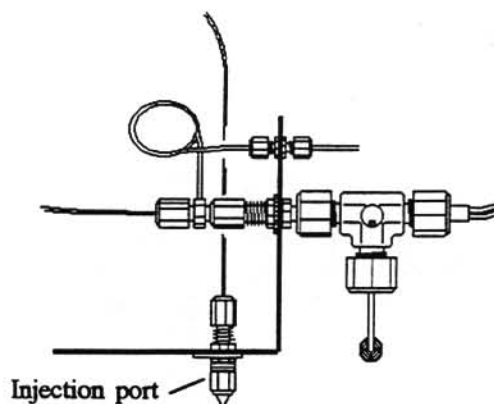
If the pressure does not drop at all, or drops very slowly, then the entire system is leak-tight. In some ways, this is the best way to check for leaks, because one test verifies that every connection in the system is holding pressure. With a complex gas system, or one where some of the fittings are in-accessible or hot it may be difficult to use the leak check liquid or meter. Also, the pressure drop test can detect leaks that are too small for the other methods to see.

If the pressure does drop quickly, there is a leak in the system. To locate the source of the leak, move the plug from the end of the pneumatic system to the next fitting upstream and repeat the test. If the system now holds pressure, then the leak must be somewhere between the location of the previous plug and the current plug.

By moving the plug location step by step upstream, eventually it will be obvious where the leak must be.

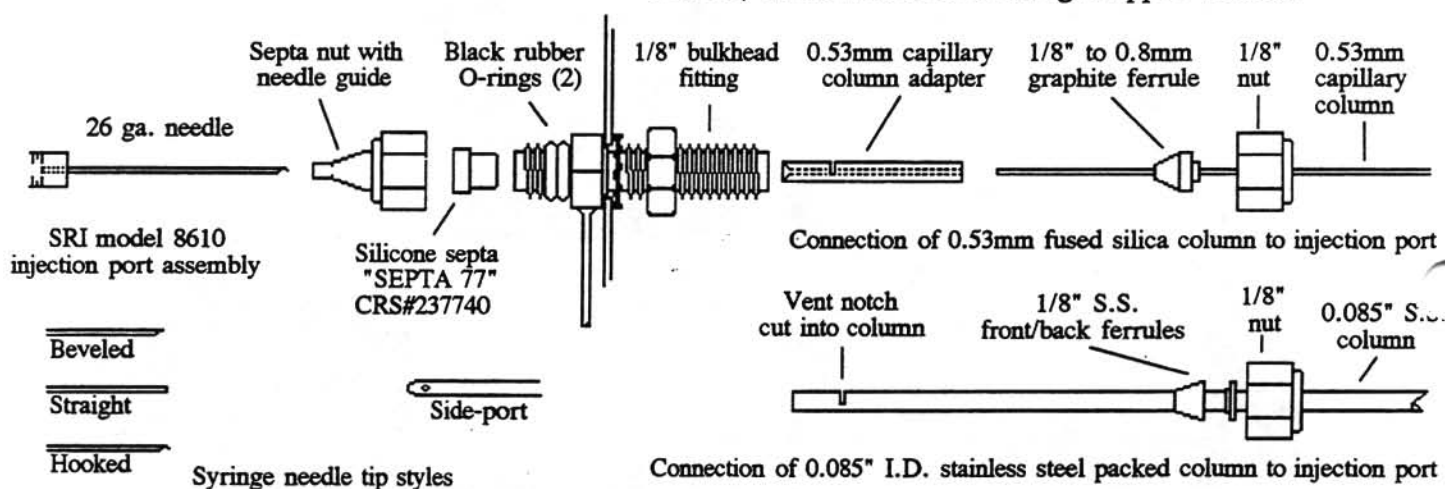




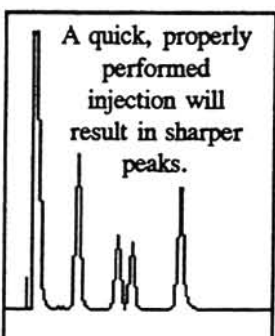
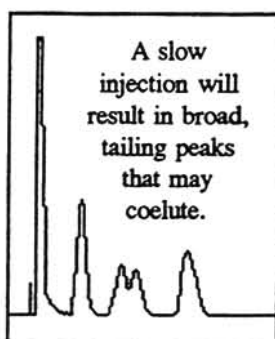


Location of injection port in typical FID system

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

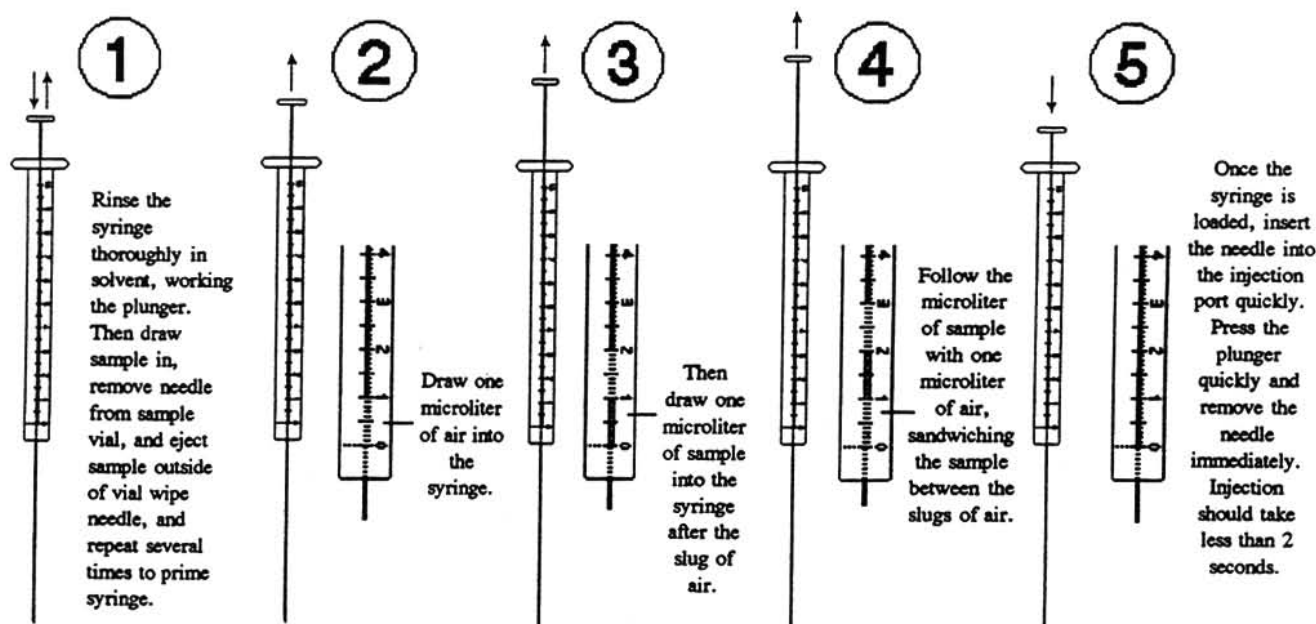


Therefore, they are recommended. "Coring" punches shards of septum into the injection port and may result in plugging of the syringe needle and failure to deliver sample. Over an extended period of time, these shards could migrate into the column. In a packed column, this accumulation of septum shards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



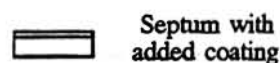
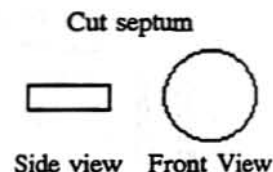
When performing analyses using manual direct injection, the method or technique used to prepare the syringe and perform the injection can mean the difference between obtaining chromatograms that are either poorly resolved or clean and sharp. Reproducibility can also be affected if the amount injected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

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

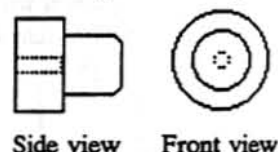


Properly prepared, the syringe needle is inserted completely into the injection port in one smooth, quick motion. Then the plunger is driven home immediately. As soon as the plunger tip hits the end of the sample chamber, the syringe needle is withdrawn from the injection port in a quick, smooth motion.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

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



Molded septum used by SRI

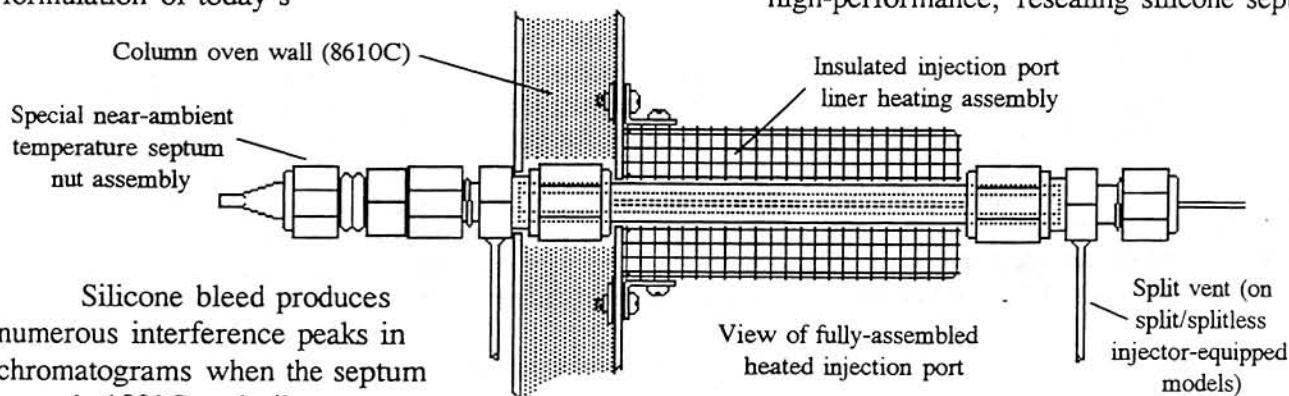


All septa are made of silicone rubber that contains polymerized silicone gums. A catalyst is used to obtain the polymerization that produces the elasticity sought from the septum material. Unfortunately, some silicone oils remain unpolymerized in the septum and freely diffuse out of the silicone septum.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

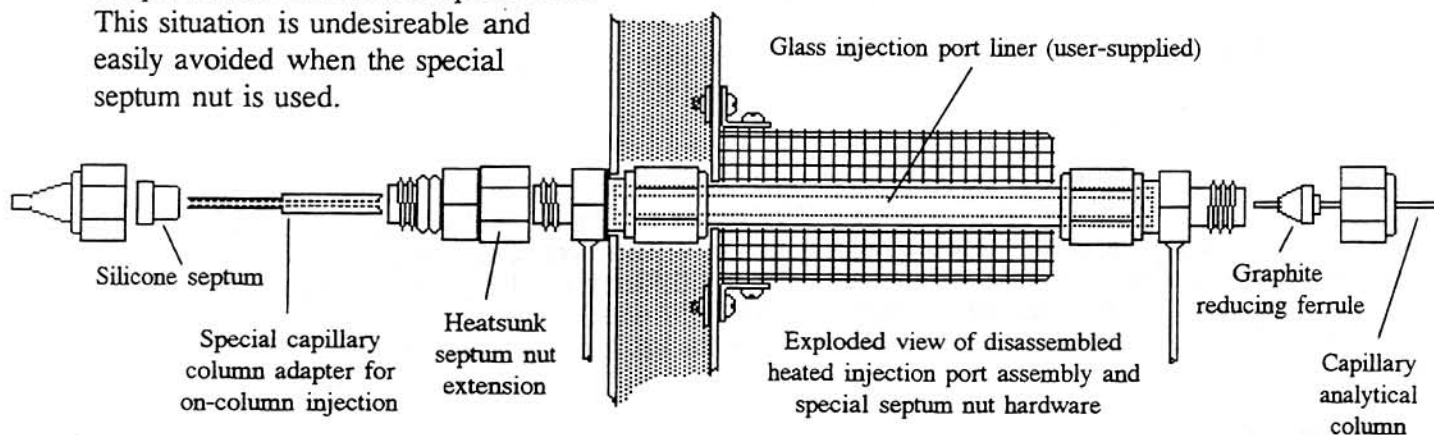
When silicone oils bleed into the column over time, the efficiency and performance of the column is degraded. Columns with a silicone liquid phase, such as OV-1 or SE-30 types, will not display the effects of septa bleed as readily as would a phase such as Carbowax 20M, which would be adversely affected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

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

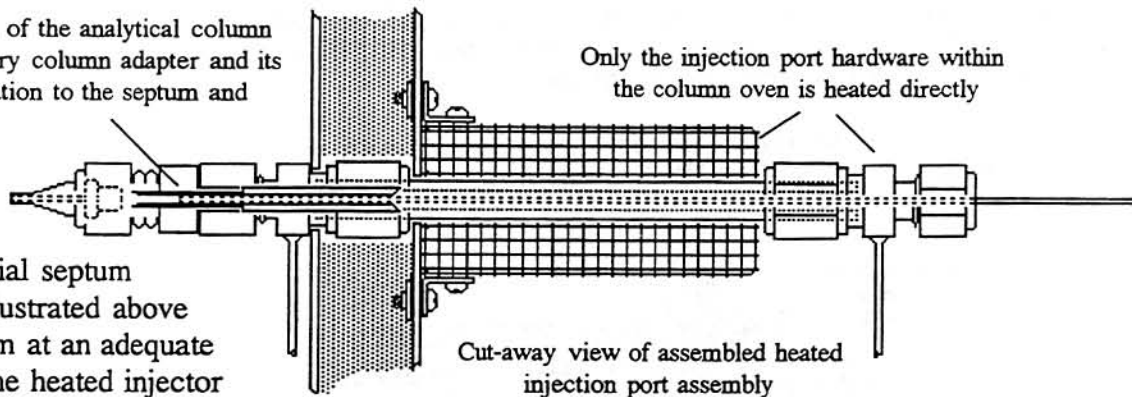
All SRI Instruments heated injection ports are equipped with a specially-designed septum nut which dissipates any heat that could be transferred from the heated injection port body (including split-splitless configurations), to the septum nut and septum by contact. Experience indicates that when injection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



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



Note the position of the analytical column within the capillary column adapter and its position with relation to the septum and injector liner

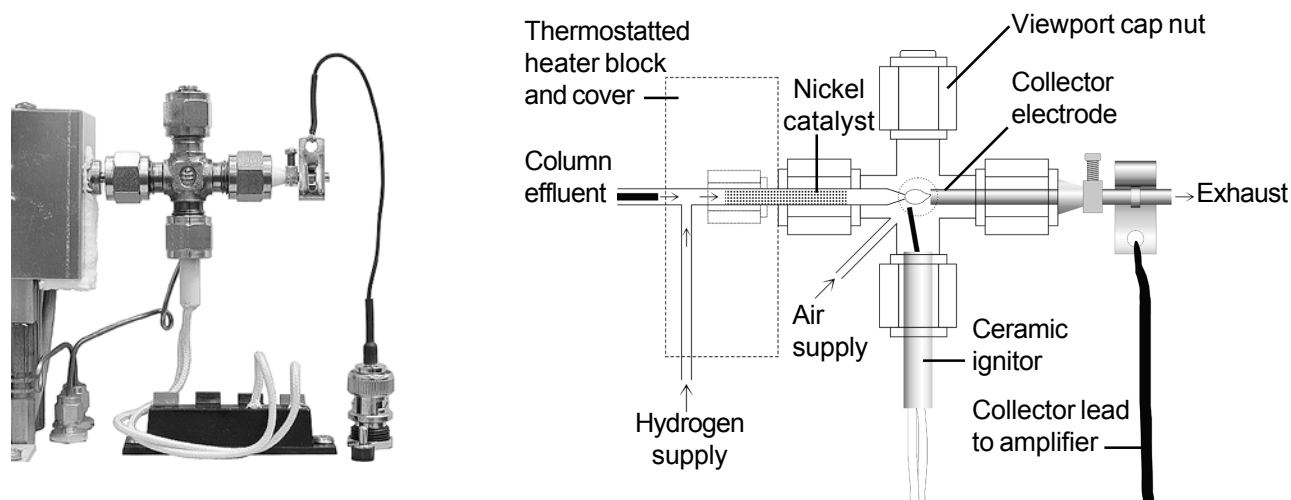


The special septum nut assembly illustrated above keeps the septum at an adequate distance from the heated injector assembly to maintain the septum at or near ambient temperature with the assistance of the additional mass of the septum nut extension. This prevents any silicone oils present in the septum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

GC ACCESSORIES

Methanizer

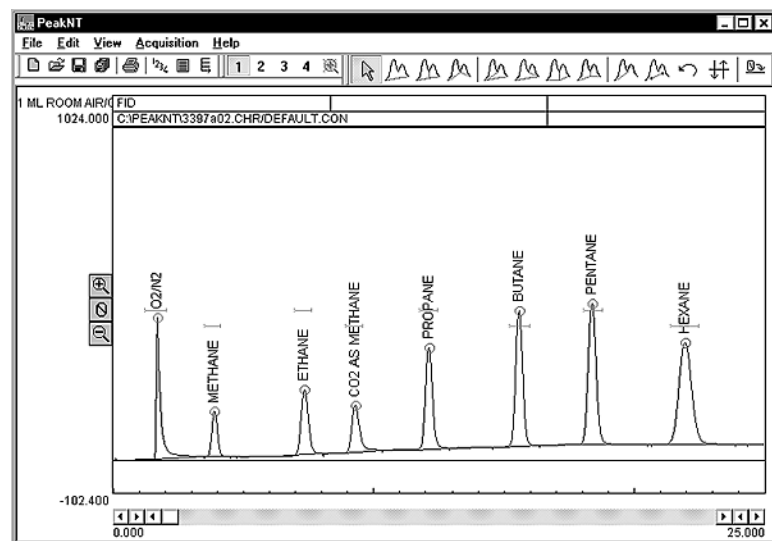
Methanizer-equipped FID Detector



The Methanizer option enables the Flame Ionization Detector to detect low levels of CO and CO₂. 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 CO₂ are converted to methane. Since the conversion of CO and CO₂ 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 SRI Multiple Gas Analyzer #1 equipped with a Methanizer.



Sample: 41cc room air + 15cc 1000ppm C₁-C₆ injected into the 1mL sample loop = 250ppm CO₂ and C₁-C₆

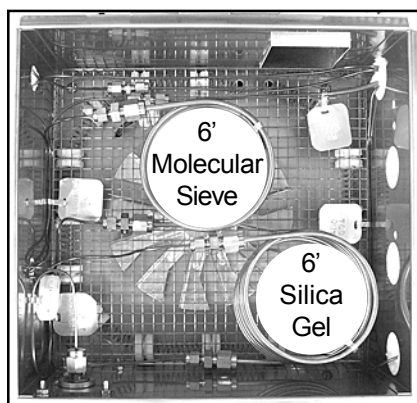
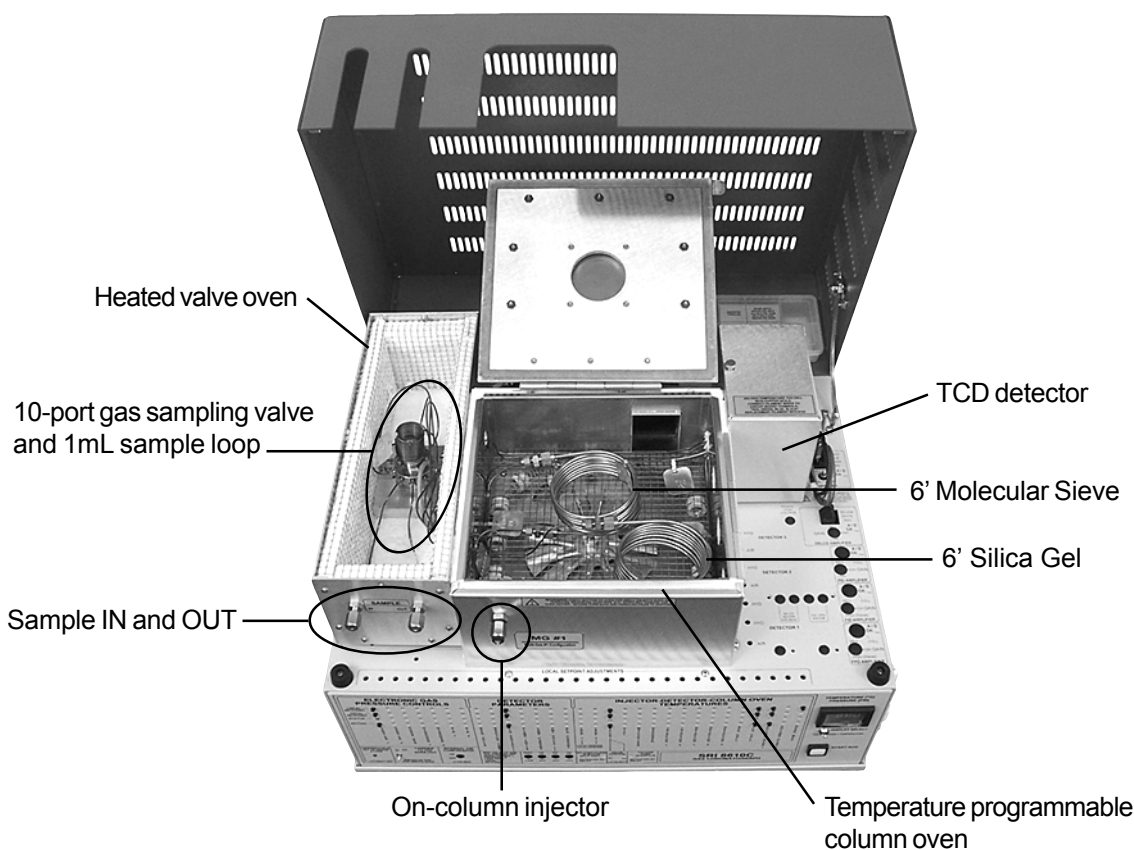
Results:

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

POPULAR CONFIGURATION GCs Multiple Gas Analyzer #1

System Overview

Your SRI Multiple Gas Analyzer #1 (MG#1) GC is pre-plumbed and ready to resolve H₂, O₂, N₂, Methane, CO, Ethane, CO₂, Ethylene, NO_x, Acetylene, Propane, Butanes, Pentanes, and C₆ through C₈. 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

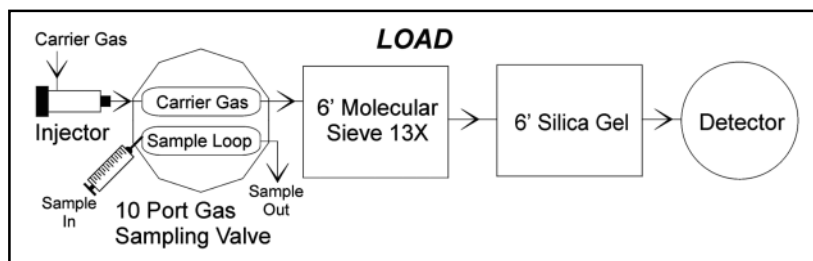
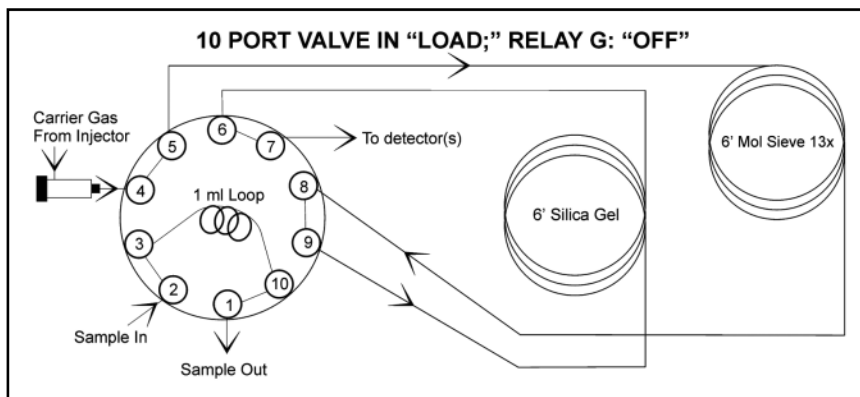
Theory of Operation

10-Port Gas Sampling Valve Plumbing Connections

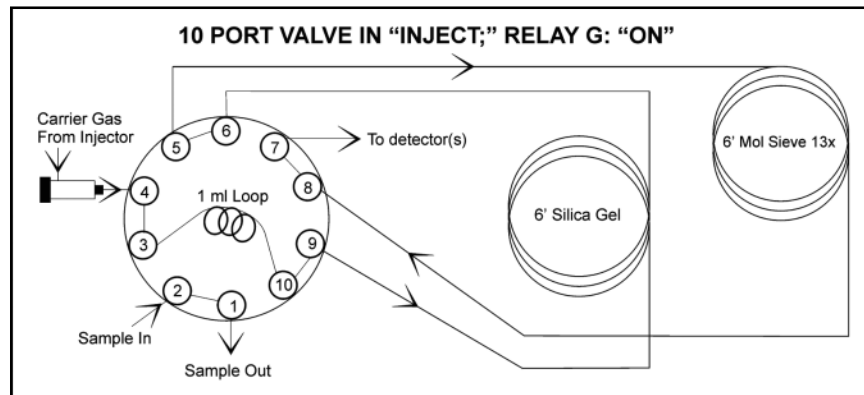
The valve, sample loop, and column combination is plumbed in a specific way to allow the MG#1 to separate hydrogen, oxygen, nitrogen, methane, ethane, propanes, butanes, pentanes, carbon monoxide, and carbon dioxide with a single injection.

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).

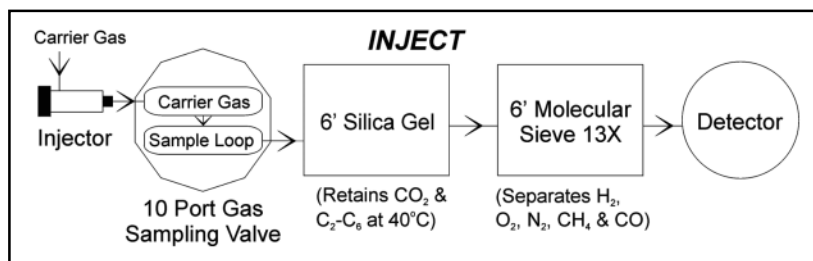


10-Port Gas Sampling Valve in the INJECT Position



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.



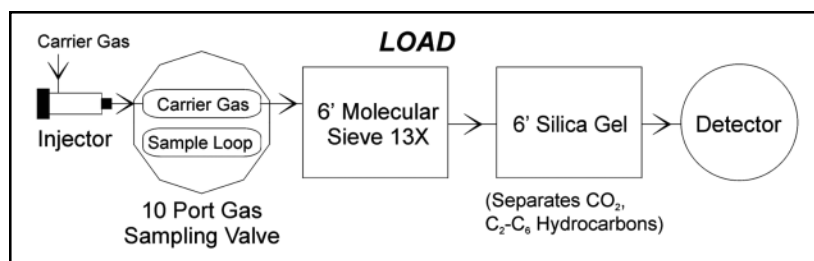
POPULAR CONFIGURATION GCs

Multiple Gas Analyzer #1

Theory of Operation

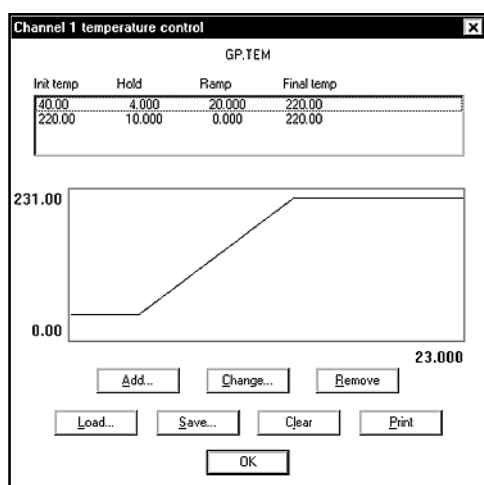
10-Port Gas Sampling Valve Plumbing Connections

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 H₂ (or helium, whichever is not being used as a carrier), O₂, N₂, methane, and CO, continues on to the Molecular Sieve column. During a chromatographic run with the sampling valve in the INJECT position, the H₂ or helium, O₂, N₂, 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 C₂, CO₂ 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.



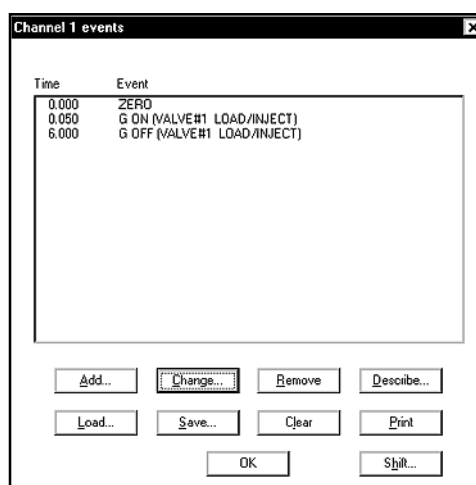
After the elution of the CO peak, the valve is switched back into the LOAD position, and the C₂, CO₂, and higher hydrocarbons come off the Silica Gel column.

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

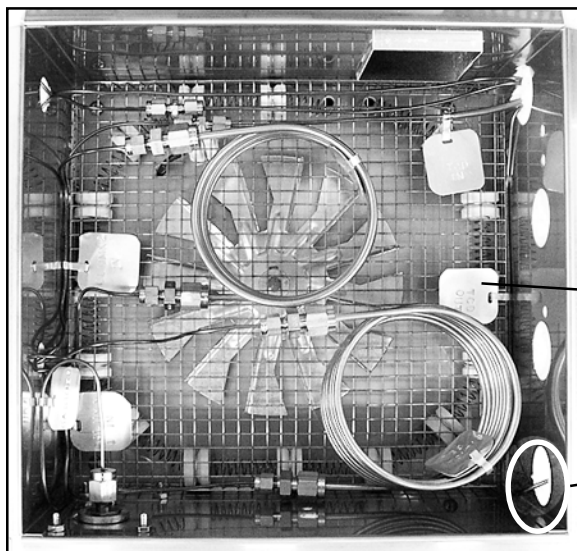
1. Set the cylinder head pressure 15-20psi higher than the head pressure. The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC.

GAS FLOW RATES				
CARRIER 1:		:	29	PSI = 20 ml/min

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

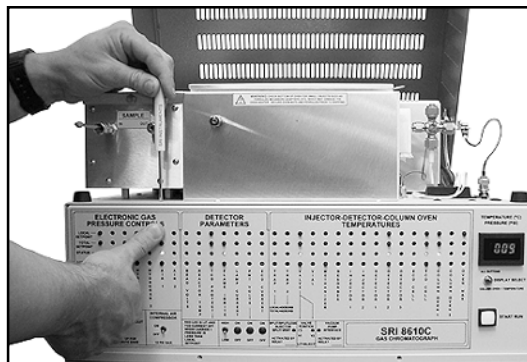
if carrier gas flow is not detectable. A filament protection circuit shuts OFF the TCD current if the column head pressure drops below 3psi, but it cannot prevent filament damage under all circumstances. Any lack of carrier gas flow should be corrected before proceeding. If necessary, reconnect the TCD outlet tubing to the FID or HID when you are finished testing the carrier flow.



The TCD carrier outlet tubing is tagged inside the column oven.

Unless connected to another detector, the end of the TCD outlet tubing will be on the outside of the column oven wall.

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.

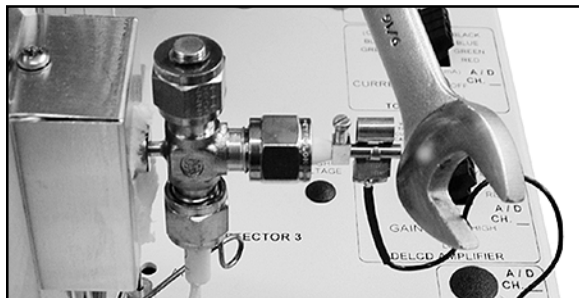


4. Set the valve oven temperature to 90°C. If present: set the FID/Methanizer temperature to 380°C; set the HID temperature to 200°C.

POPULAR CONFIGURATION GCs Multiple Gas Analyzer #1

General Operating Procedures continued

5. Turn the TCD current ON to LOW. Ignite the FID, if present, by holding up the ignitor switch (labeled “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 SRI manual for more information.

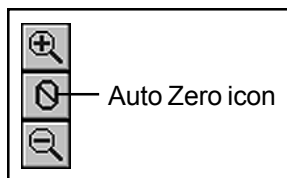
6. Ethane is the first peak to elute from the Silica Gel column after the H_2 , O_2 , N_2 , CH_4 , and CO , which are separated by the Molecular Sieve column. The ethane and CO_2 will get stuck in the Molecular Sieve column if the gas sampling valve is not rotated back into the LOAD position (by turning 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:

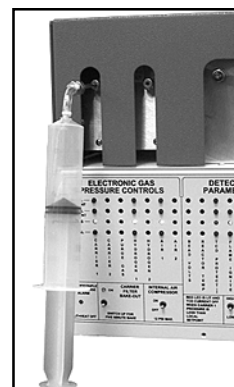
Initial	Hold	Ramp	Final
40°C	6.00	10.00	200°C



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.

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



Injection by syringe of gas sample into valve

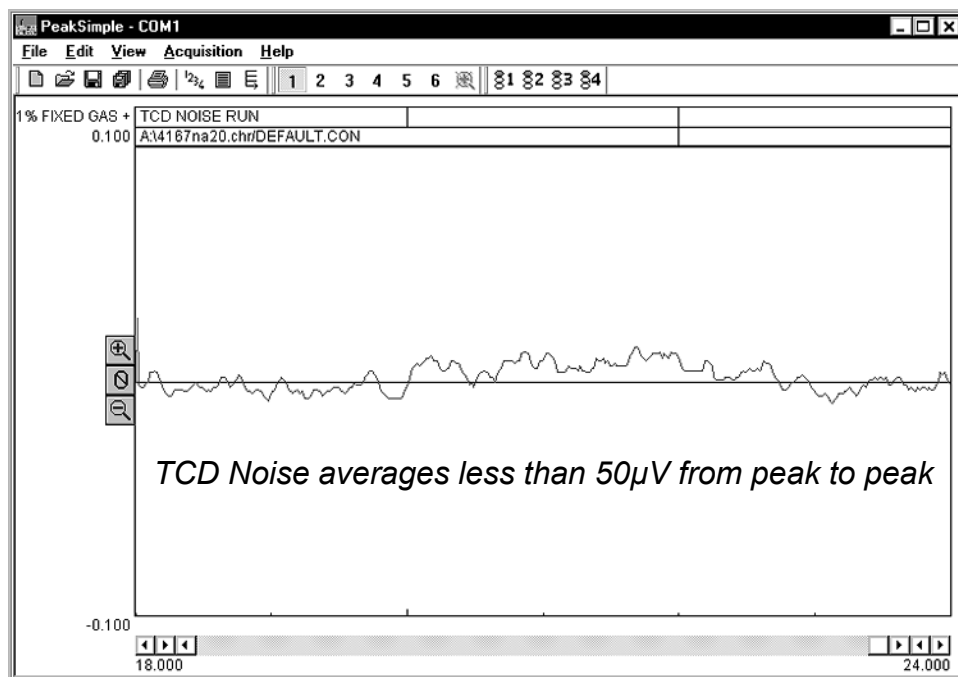
10. Revise the temperature program if necessary. The temperature program used for the test chromatogram on the Expected Performance page works well with the above event program.

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:

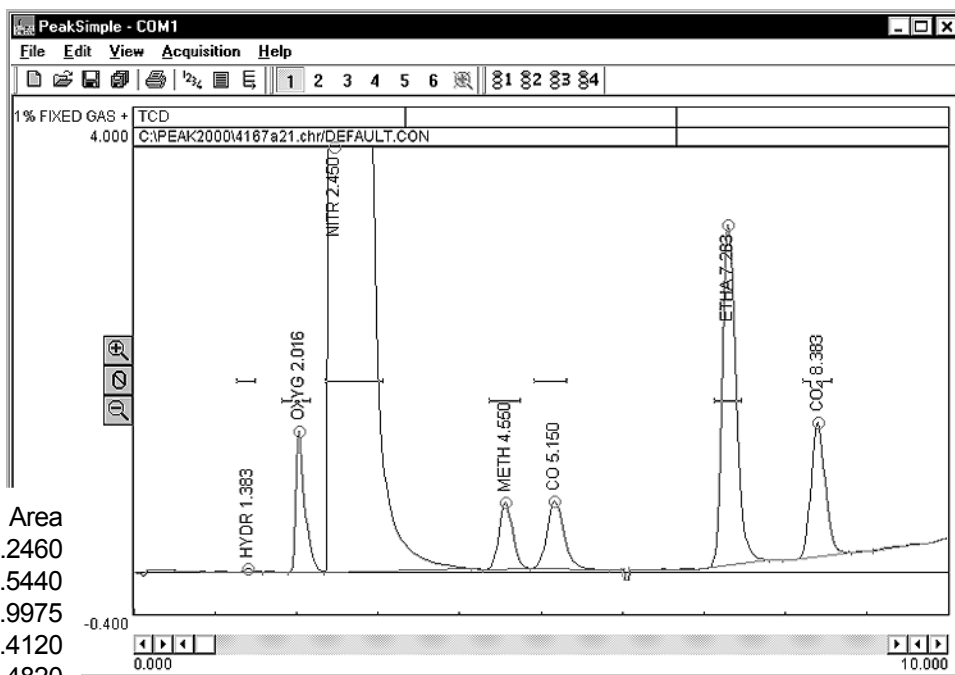
Initial Hold Ramp Final
40°C 4.00 20.00 220°C

Events:

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

Results:

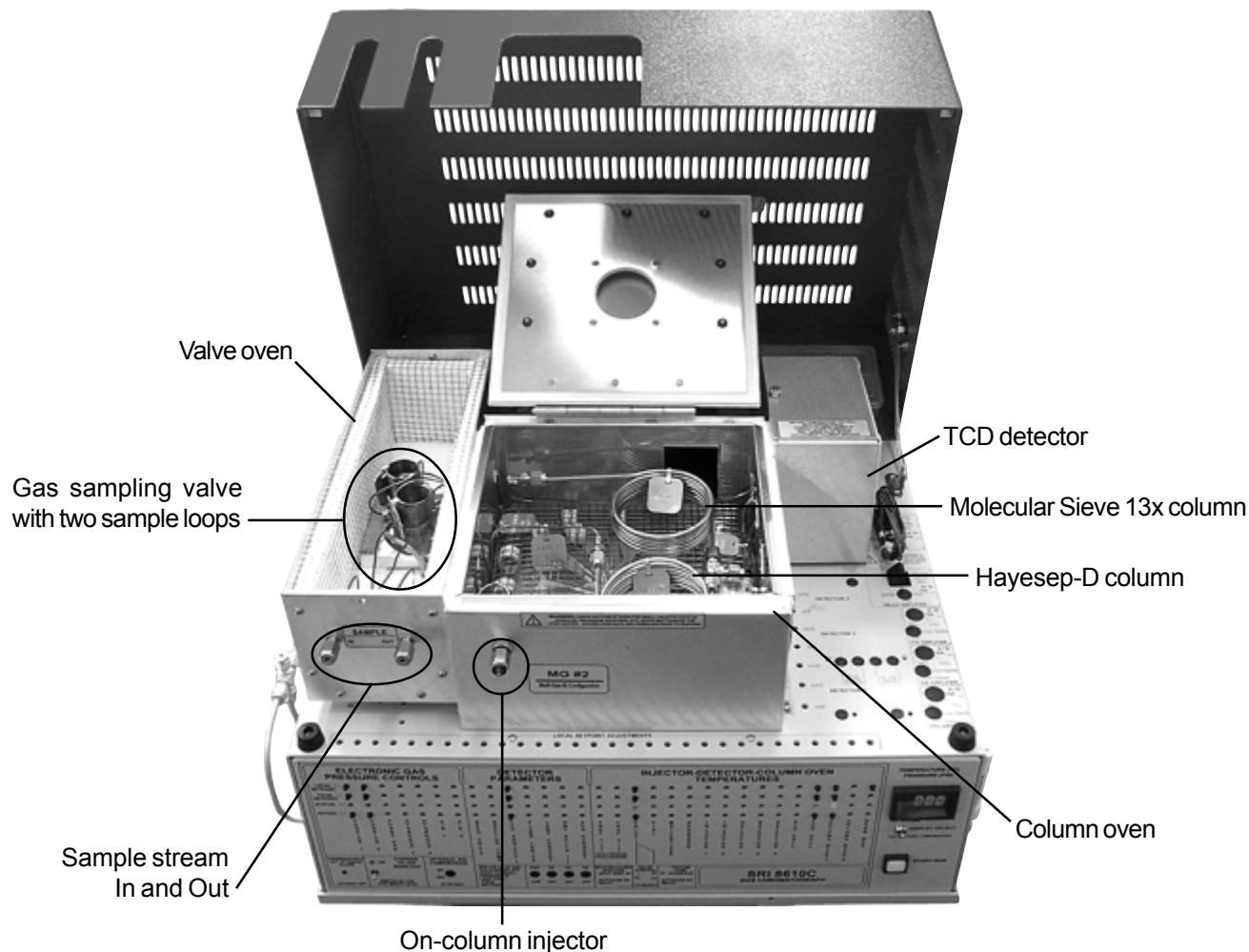
Component	Retention	Area
Hydrogen	1.383	0.2460
Oxygen	2.016	10.5440
Nitrogen	2.450	924.9975
Methane	4.550	7.4120
CO	5.510	9.4820
Ethane	7.283	38.2725
CO ₂	8.383	15.4100
TOTAL		1006.3640



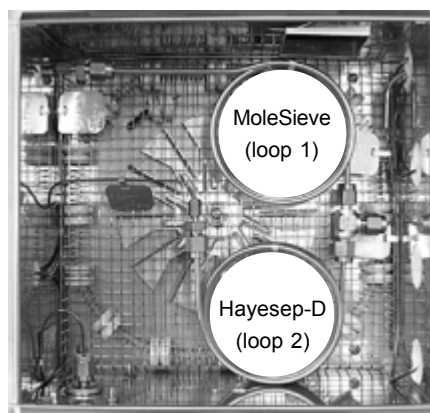
POPULAR CONFIGURATION GCs Multiple Gas Analyzer #2

System Overview

The SRI Multiple Gas Analyzer #2 (MG#2) is configured on the 8610C chassis. It is equipped with a gas sampling valve plumbed with dual sample loops in a heated valve oven, and two packed columns in the column oven. The basic model, shown below, comes with a TCD detector. The MG#2 may optionally be equipped with a FID/methanizer or HID detectors in addition to the TCD. A capillary column in parallel with the Hayesep-D column is an option for separating out hydrocarbons through C_{20} .



The MG#2 separates a wide variety of peaks without co-elution by turning the carrier gas flow to the two packed columns ON and OFF individually at different times during the run. The carrier to the Molecular Sieve 13x column (carrier #1) is turned ON first to complete the separation of H_2 , O_2 , N_2 , CH_4 and CO. At this point, the MoleSieve carrier flow is turned OFF and the Hayesep-D carrier (carrier #2) is turned ON. All compounds in the C_1 - C_6 range are then separated by the Hayesep-D column. The MoleSieve column is connected to sample loop 1, and the Hayesep-D to loop 2.



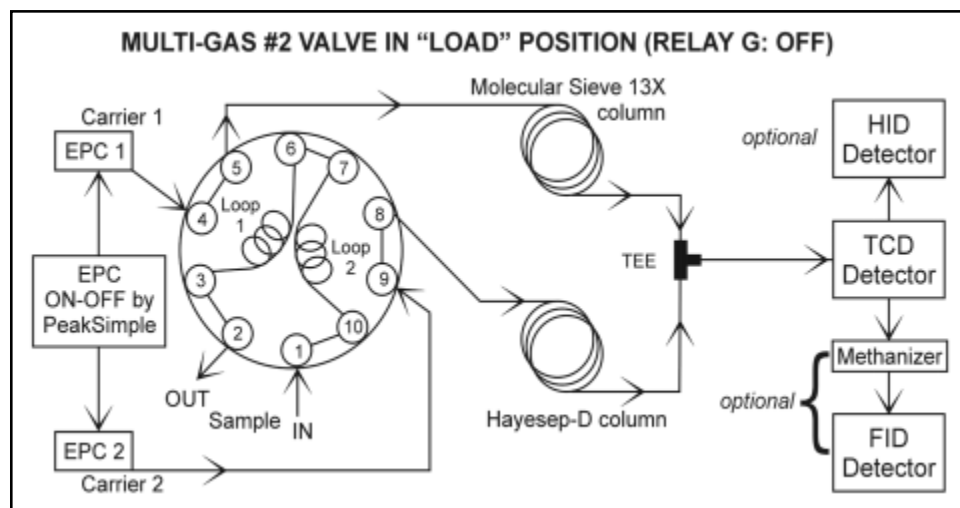
POPULAR CONFIGURATION GCs

Multiple Gas Analyzer #2

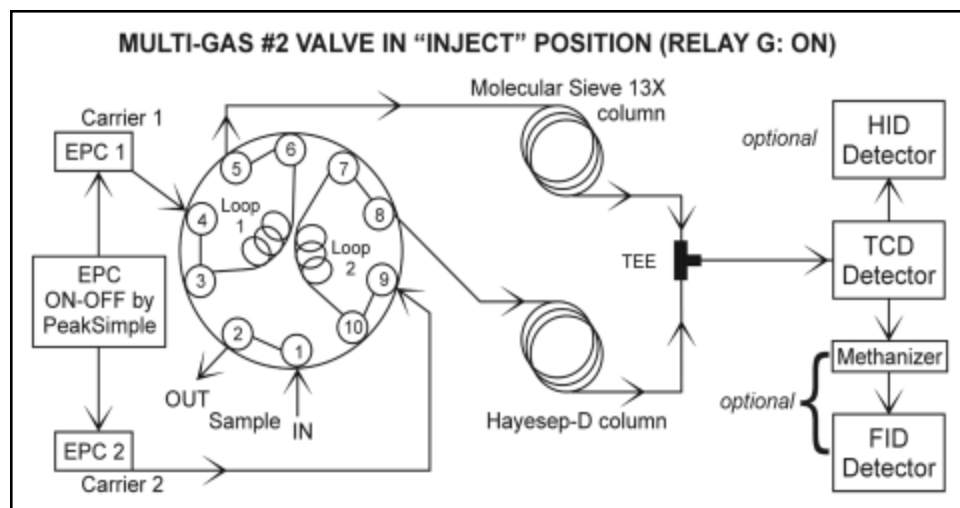
Theory of Operation

The MG#2 GC uses a single automated 10 port Gas Sampling Valve and multiple columns to separate a wide variety of compounds. It achieves this by turning the carrier gas flow to each column on at different times during the run. This procedure allows the Molecular Sieve 13X column to completely separate H_2 , O_2 , N_2 , CH_4 and CO before the carrier flow to the Hayesep-D column is turned on. The Hayesep-D column then separates all compounds in the C_1 - C_6 range. An optional 30-meter MXT-1 capillary column separates the remaining hydrocarbons through C_{20} , using the same carrier gas flow as the Hayesep-D column and an FID or HID detector.

The MG#2 is plumbed with two separate carrier gas flows, each regulated by Electronic Pressure Control (EPC) through the PeakSimple data system. Carrier 1 flows through sample loop #1 to the MoleSieve column, then on through the "Tee" to the TCD detector. Carrier 2 flows through sample loop #2 to the Hayesep-D column, then through the "Tee" to the TCD detector. Carrier #1 and #2 flows are turned ON and OFF by PeakSimple, controlled by the user with an Event table.



When the MG#2 valve is in the LOAD position, loops #1 and #2 are loaded with the sample gas stream while carrier flows #1 and #2 bypass the loops and travel on to the columns.



When the MG#2 valve is in the INJECT position, carriers #1 and #2 flow through the sample loops, sweeping their contents to the columns.

POPULAR CONFIGURATION GCs Multiple Gas Analyzer #2

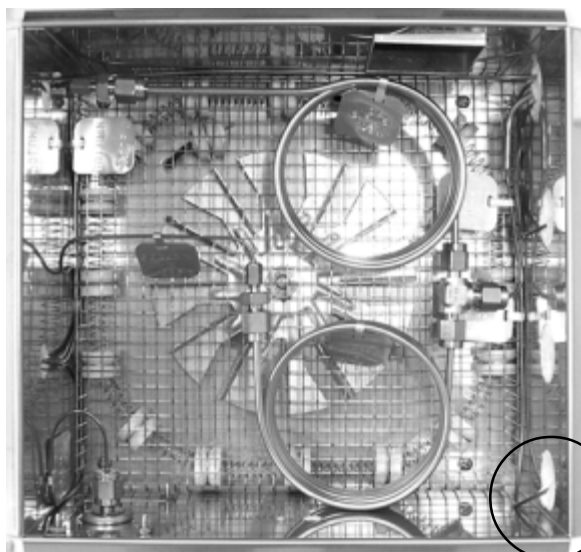
General Operating Procedure

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right-hand side of your GC. Verify that with carrier gas turned off at the cylinder, that the actual GC pressure reads ZERO.

GAS FLOW RATES					
CARRIER 1:	MOL. SIEVE	:	10	PSI =	10 ml/min
CARRIER 2:	HAYESEP-D	:	7	PSI =	10 ml/min
P&T PURGE:		:		PSI =	ml/min
HYDROGEN 1:		:		PSI =	ml/min
HYDROGEN 2:		:		PSI =	ml/min

2. 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 ON the TCD current. Labelled for identification, the TCD carrier gas outlet tubing is located inside the column oven. Place the end of the tubing in 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

prevents filament damage by shutting OFF the TCD current when the column head pressure is below 3psi. Because this protect circuit cannot prevent filament damage under all circumstances, any lack of carrier gas flow should be corrected before proceeding. NEVER turn both carrier #1 and carrier #2 OFF at the same time. Please see the TCD manual section for more information about the detector.



The TCD carrier gas outlet tubing is located inside the column oven. If there is also an FID detector on your MG#2, the TCD carrier gas outlet tubing is connected to the FID inlet bulkhead in the column oven wall. If your MG#2 has a TCD only, the end tubing will be on the outside of the column oven, on the detector side.

Use the trimpot directly above the "VALVE" zone to set or adjust the valve oven temperature.



3. Set the valve oven temperature to 90°C using the trimpot on the top edge of the GC front control panel.

4. Turn the TCD current ON to LOW. If present, ignite the FID/methanizer and set the temperature to 380°C. If present, turn ON the HID current.

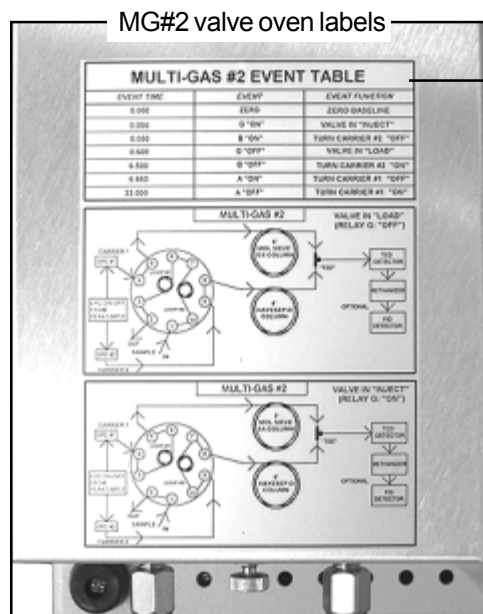
5. Set the column oven temperature program in PeakSimple as follows. (This is an example; your analysis may require a different temperature program.)

Initial	Hold	Ramp	Final
50.00	3.00	20.00	220.00
220.00	25.00	0.00	220.00

POPULAR CONFIGURATION GCs

Multiple Gas Analyzer #2

General Operating Procedure continued



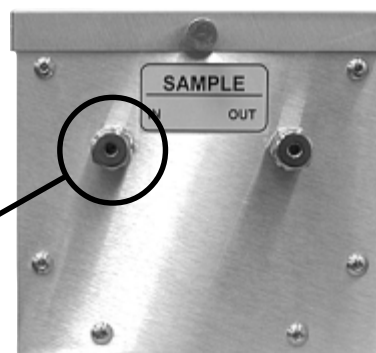
Example event table:

Time	Event
0.000	ZERO (zero data system signal)
0.050	B ON (carrier #2 OFF)
0.500	G ON (valve INJECT)
3.500	B OFF (carrier #2 ON)
3.600	A ON (carrier #1 OFF)
18.000	A OFF (carrier #1 ON- MoleSieve Bake Out phase)

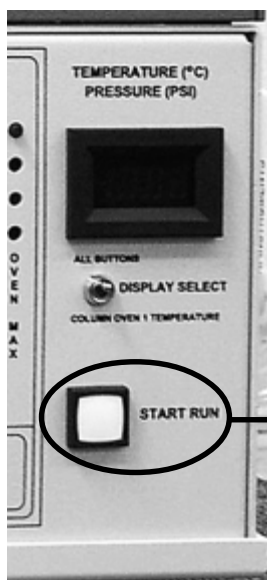
6. Type in an Event table. The example shown is labeled on the MG#2 valve oven. The event table should allow for the elution of CO from the molecular sieve column before carrier #2 is turned back ON. The column oven temperature may be increased to speed the elution of the H₂, O₂, N₂, CH₄, and CO. Hydrocarbons like ethane and propane end up on the Molecular Sieve 13x column after its carrier is turned OFF and the HayeSep-D carrier is turned

ON. The example Event table also turns Carrier #1 ON at the end of the run, while the column is still hot enough to bake the hydrocarbons out of the MoleSieve column. This Bake Out phase is required to get rid of any residual peaks, so that following analyses are not compromised. Keep Carrier #1 ON and the column oven hot long enough for any contamination peaks to elute. Click the Edit drop down menu in the main PeakSimple window, then choose Overall, then make sure that the "Reset relays at end of run" checkbox is selected. Otherwise, you will have to include G OFF at the end of the event table.

7. Load your sample gas stream by connecting the flow to the sample inlet port ("SAMPLE IN") on the front of the valve oven with the provided 1/8" swagelok nut and brass ferrule.



Sample inlet port



8. Start the analysis by pressing the START RUN button on the front of your GC, or by pressing your computer keyboard spacebar.

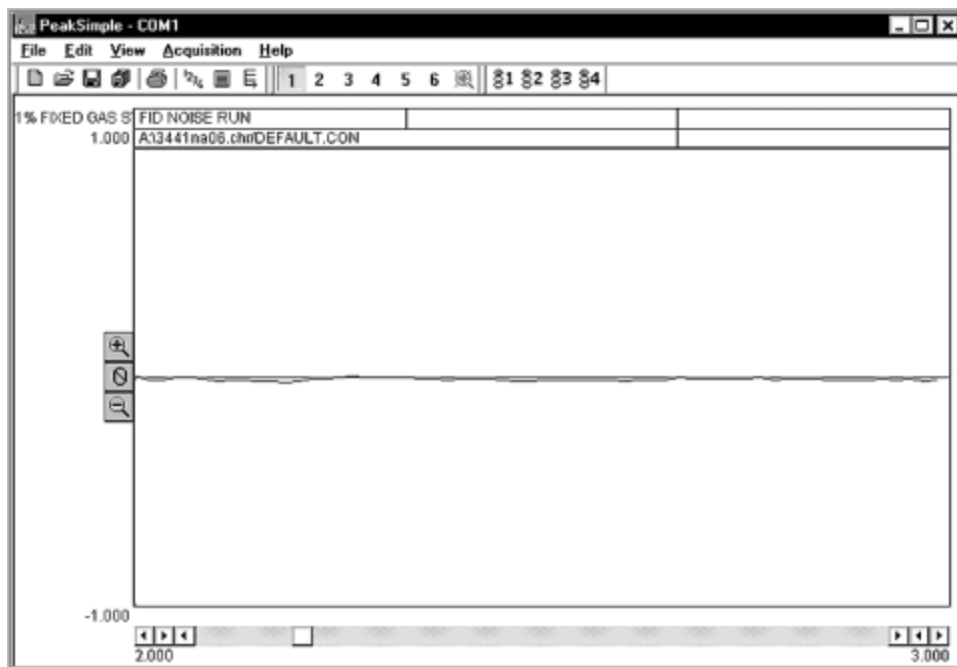
The START RUN button is on the lower right hand corner of the GC's front control panel.

POPULAR CONFIGURATION GCs

Multiple Gas Analyzer #2

Expected Performance

These two noise runs were made with identical parameters (carrier flow, columns, temperature program) on a Multiple Gas Analyzer #2 GC equipped with FID and TCD detectors. The only differences are the detector particulars, which are listed next to the appropriate chromatogram.



FID noise run

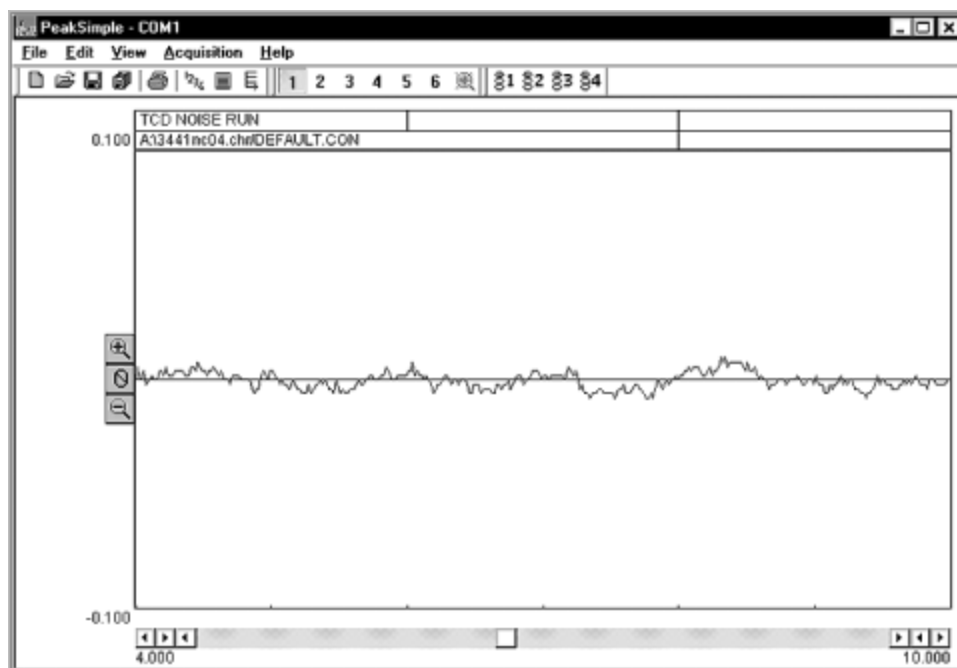
FID gain = HIGH
FID temp = 380°C
FID ignitor = -400
Methanizer in FID
detector body

Valve temp = 90°C
Carrier #1 Mol. Sieve 13x = 20mL/min
Carrier #2 Hayesep-D = 20ml/min
Total carrier flow = 40mL/min

Temperature program:
Initial Hold Ramp Final
80°C 20.00 0.00 80°C

TCD noise run

TCD current = LOW
TCD temp = 150°C



POPULAR CONFIGURATION GCs

Multiple Gas Analyzer #2

Expected Performance

The first chromatogram shows the TCD response to a 1% Fixed Gas Standard sample. Using the same valve temperature, column oven temperature program, carrier flow and event table, the second chromatogram shows the TCD response to a Natural Gas Standard sample. The event table used is shown on the **General Operating Procedure continued** page.

Columns: 2-meter Hayesep-D, 2-meter
Molecular Sieve 13x
TCD current = LOW; TCD temp = 150°C

Carrier: Helium at 40mL/minute combined
(20mL/minute through each column)
Valve temp = 90°C

Column Oven

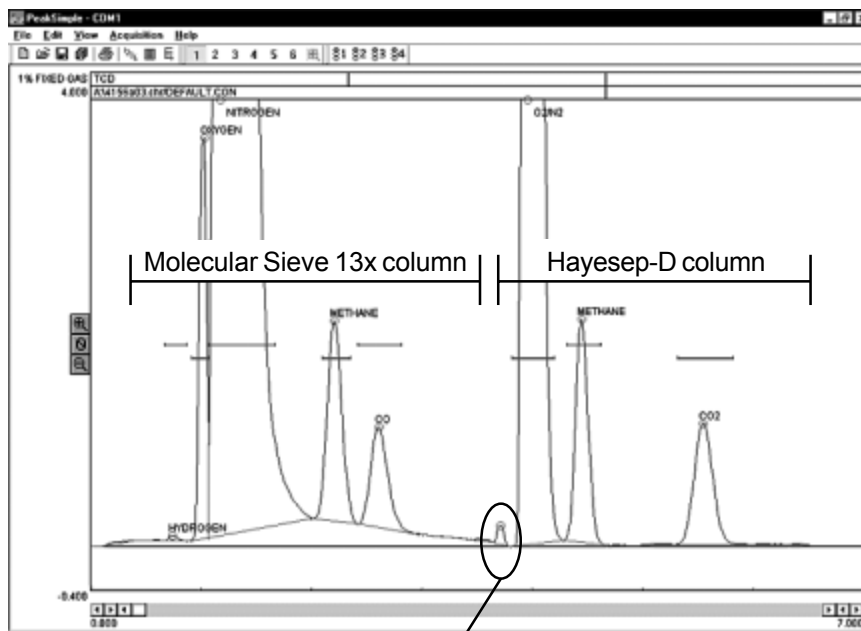
Temperature program:

Initial	Hold	Ramp	Final
50°C	3.00	20.00	220°C
220°C	25.00	0.00	220°C

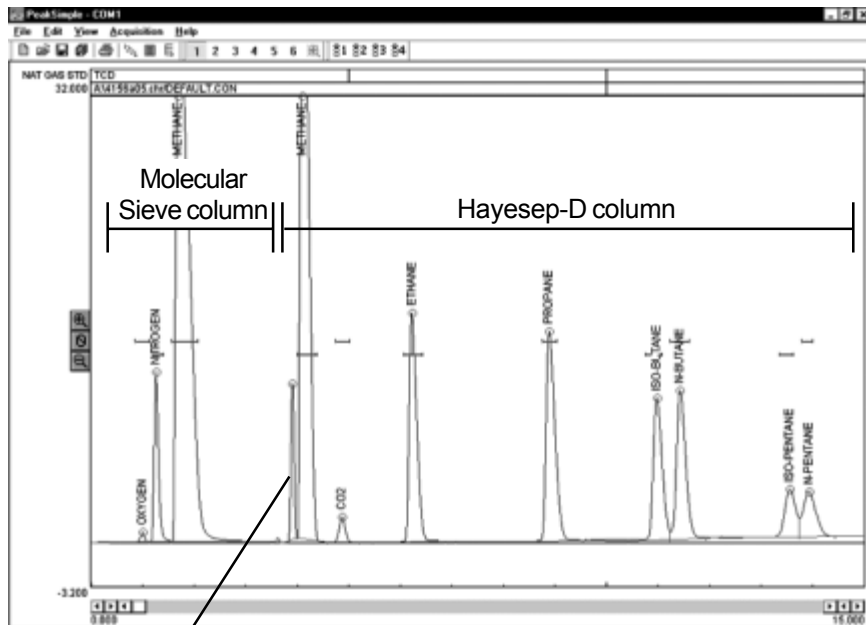
1% Fixed Gas Standard

RESULTS:

Component	Retention	Area
Hydrogen MS	0.733	00.2510
Oxygen MS	1.016	16.0495
Nitrogen MS	1.166	1108.7680
Methane MS	2.200	16.5050
CO MS	2.600	09.7370
O ₂ /N ₂ Hay-D	3.950	863.6340
Methane Hay-D	4.433	15.7300
CO ₂ Hay-D	5.533	12.9205
TOTAL		2043.5950



Carrier switch



Carrier switch

Natural Gas Standard

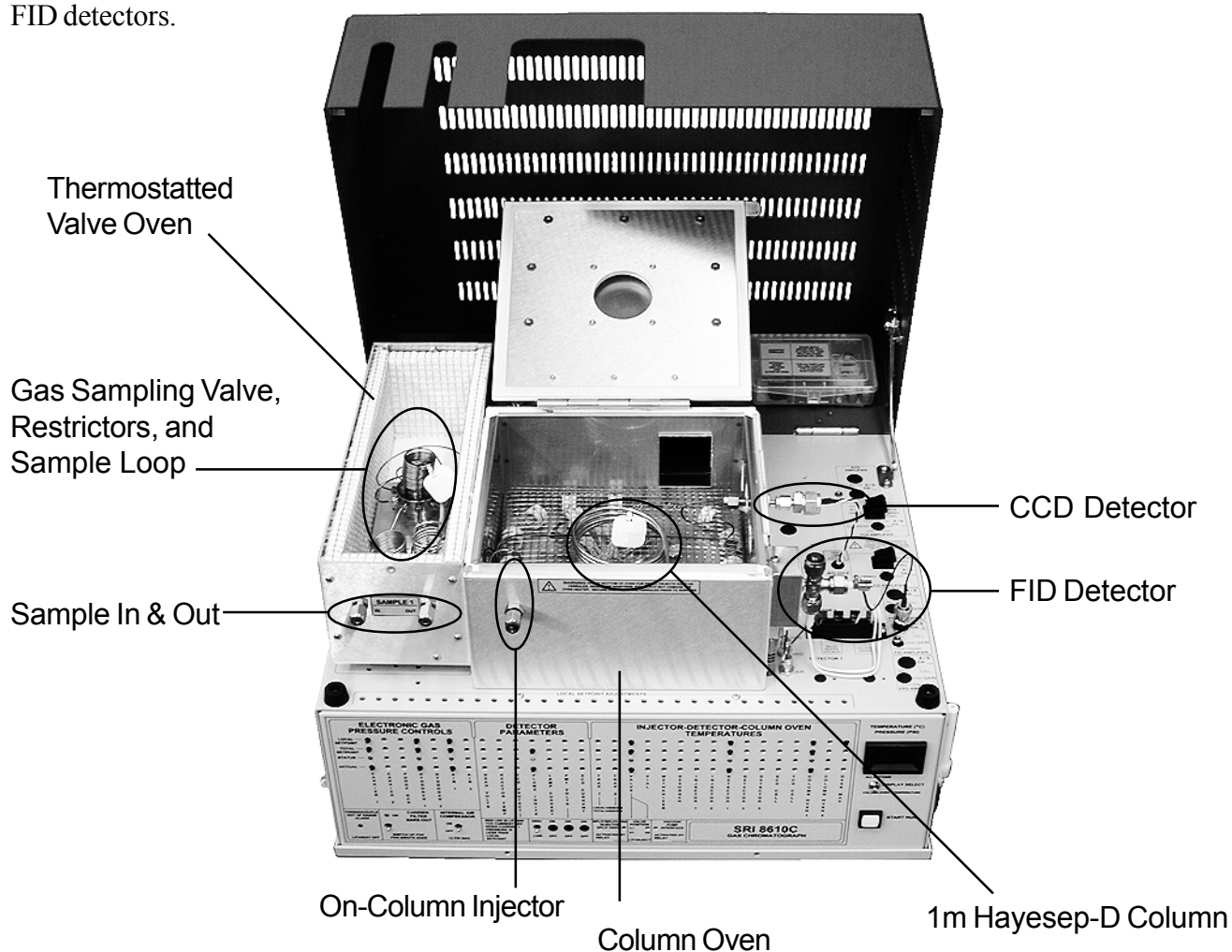
RESULTS:

Component	Retention	Area
Oxygen MS	0.983	3.4190
Nitrogen MS	1.250	72.5450
Methane MS	1.683	706.7920
Methane Hay-D	4.083	587.7140
CO ₂ Hay-D	4.850	14.7710
Ethane Hay-D	6.216	169.1275
Propane Hay-D	8.866	180.2660
Iso-Butane Hay-D	10.966	126.6950
N-Butane Hay-D	11.400	134.1470
Iso-Pentane Hay-D	13.533	50.1540
N-Pentane Hay-D	13.916	54.4740
TOTAL		2099.1045

POPULAR CONFIGURATION GCs Mud-Logger

System Overview

The Mud-Logging GC system is designed to provide a continuous reading of total hydrocarbons in a gas stream, while periodically performing a chromatographic separation of the sample to determine the composition of the sample gas stream. This is accomplished using a 10 port Gas Sampling Valve with a 25 μ L Sample Loop in a thermostatted Valve Oven, a 1m (3') Hayesep D packed column in a temperature programmable Column Oven, a CCD detector, an FID detector and a built-in Air Compressor. This GC can be modified to incorporate a second FID instead of the CCD for total hydrocarbon monitoring. The model shown below has CCD and FID detectors.



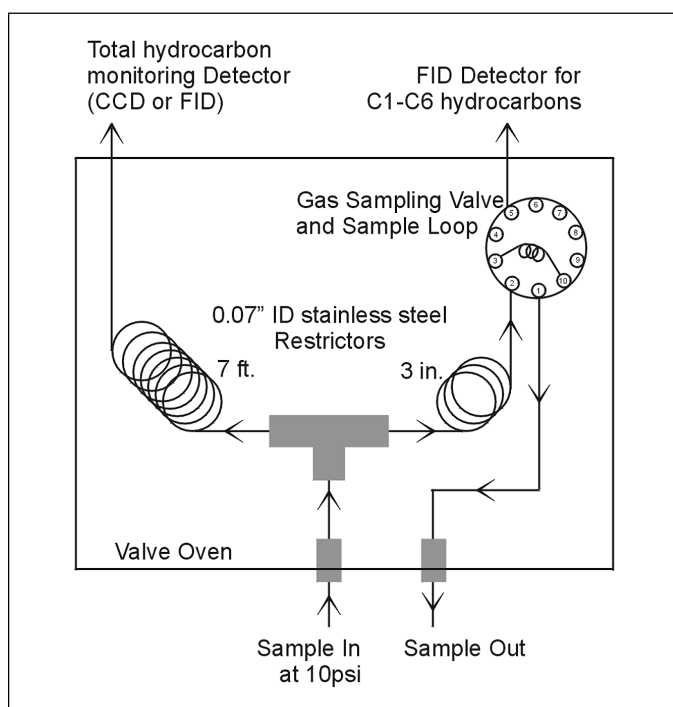
Speciation of C₁-C₆ hydrocarbons is handled by the Gas Sampling Valve, Hayesep-D column, and FID while the CCD provides continuous, total hydrocarbon monitoring. Detection limits for this system are 0.1% to 100% for the continuous total hydrocarbon monitor, and 0.005% to 100% for speciated hydrocarbons using the FID. The Air Compressor supplies combustion air for the FID, and the air make-up for the CCD. The built-in PeakSimple data system displays both the continuous total hydrocarbon reading, using the Data Logger mode, and the separated peaks. When the system receives out-of-range readings, an alarm function may alert the user.

POPULAR CONFIGURATION GCs

Mud-Logger

Theory of Operation

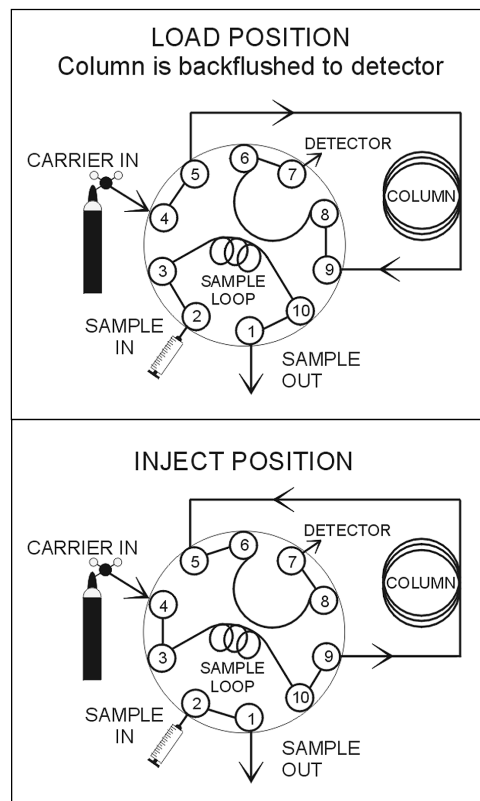
The sample gas stream is connected to a bulkhead fitting on the system's thermostatted Valve Oven where it flows through the sampling loop of the 10 port Gas Sampling Valve, and also to the CCD detector. The fitting labelled "Sample In" (pictured at right) on the front of the Valve Oven is the sample gas stream inlet. The user must regulate the pressure of the sample stream so that it enters this inlet at 10psi. The instrument is factory preset to deliver 5mL/min to the CCD at 10psi. The remainder of the flow, approximately 100mL/min, passes through the Sample Loop. This relatively high flow rate gets the sample from the sampling point into the GC with minimal delay.



Once the sample enters the inlet, its path is T'd through two restrictors and on to the detectors. To avoid damaging the CCD, the maximum pure hydrocarbon flow to reach this detector is 5mL/min. The restrictors regulate the flow to the CCD when the sample inlet pressure is 10psi. The remainder of the sample stream (approximately 100mL/min) flows through the Gas Sampling Valve's loop and is periodically injected into the Hayesep-D column, then detected by the FID.

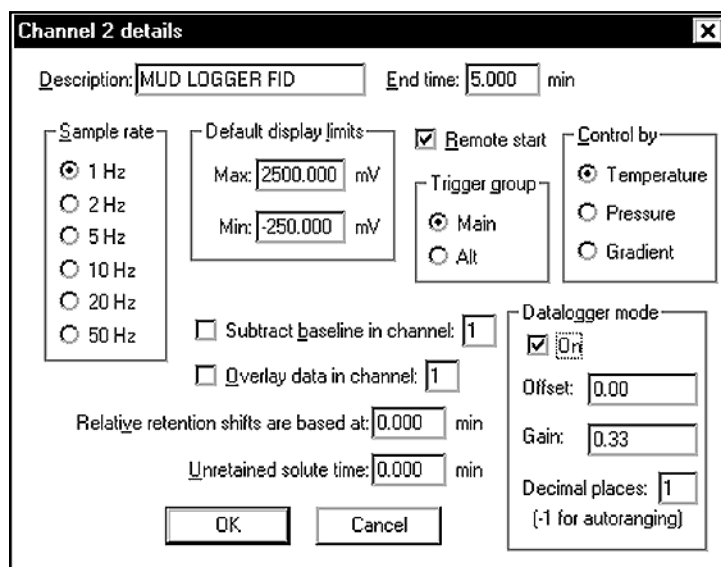
10 Port Gas Sampling Valve

At an automatically repeating time interval controlled by the user with the built-in PeakSimple data system, the Gas Sampling Valve injects the contents of its sample loop into the Hayesep D packed column where it is separated into the constituent hydrocarbon (C_1 - C_6) peaks and detected by the FID detector. Between automatic sample injections into the column, the 10 port Gas Sampling Valve is in LOAD position (top right schematic). In this position, the carrier gas flows into the column while sample gas flows through the 25 μ L Sample Loop and to vent. When PeakSimple automatically moves the valve to the INJECT position (bottom right schematic), the carrier gas flows through the Sample Loop first, then sweeps the sample into the Hayesep-D column.



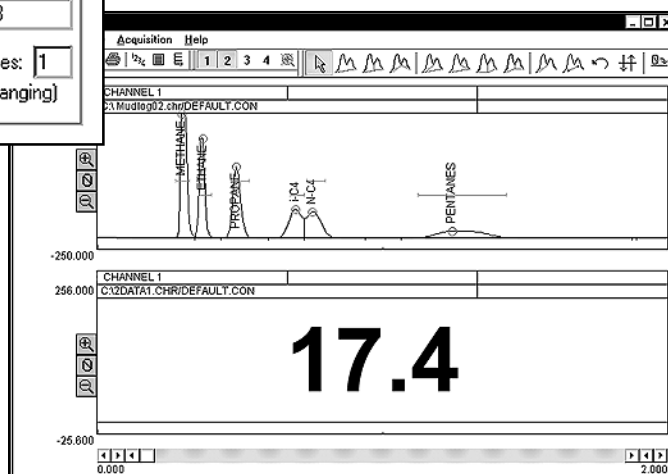
General Operating Procedure Part 1: Total Hydrocarbons Using the CCD Detector

1. Connect zero gas to sample inlet at 10psi. Zero gas has no hydrocarbons.
2. Zero the CCD detector signal using the Auto Zero button for its channel (typically channel 2).
3. Connect calibration gas standard to the sample inlet at 10psi. Calibration gas is typically 100% methane.
4. The CCD signal will increase approximately 300 millivolts while running 100% methane.
5. In PeakSimple, open the CCD Channel Details dialog box by right-clicking in that channel's chromatogram window. Enter the gain factor which will multiply the 300 millivolt signal to produce the desired concentration unit. For example: $300 \times .33 = 100$ if the desired unit is percent.
 $300 \times 3333 = 1,000,000$ if the desired unit is parts per million
6. Also in the Channel Details dialog box, select Data Logger mode by clicking in the appropriate checkbox. The CCD signal times the gain factor will be displayed on the screen in large numbers.



PeakSimple
Channel Details dialog box

Chromatogram with
channel 2 in Data Logger mode



POPULAR CONFIGURATION GCs

Mud-Logger

General Operating Procedure Part 2: Speciated Hydrocarbons Using the FID Detector

1. Connect the calibration gas standard to the sample inlet at 10psi.
2. Set the Valve Oven temperature to 90°C.
3. Ignite the FID.
4. Set an isothermal Column Oven temperature program as follows:

Initial Temp	Hold	Ramp	Final Temp
200°C	5.00	0.00	200°C

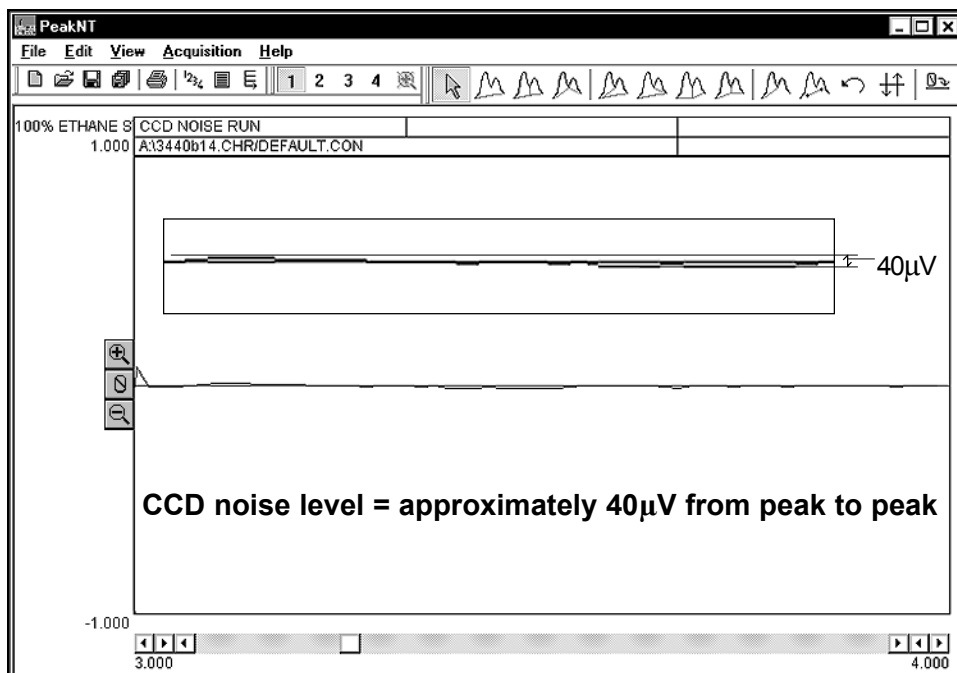
5. Type in an even table as follows:

Time	Event
0.00	Zero
0.050	G ON
1.5	G OFF

6. Set the FID gain to MEDIUM.
7. Start the analysis by hitting the spacebar on the computer keyboard.
8. In PeakSimple, input the retention windows to identify the individual hydrocarbon components (methane, ethane, propane, butane, etc).
9. Calibrate the individual hydrocarbon peaks.
10. This instrument is plumbed for backflush. This gives the user the option to set the valve program to backflush the heavier hydrocarbons after the desired peaks have been separated. For instance, if your application required separation of hydrocabons up to C₅, you could set the valve to backflush after the elution of the C₅ component(s), and all the heavier hydrocarbons would together produce one large peak.

POPULAR CONFIGURATION GCs Mud-Logger

Expected Performance



CCD Noise

Column: 1m Hayesep-D
Carrier: Air @ 10mL/min
Air make-up = 100mL/min

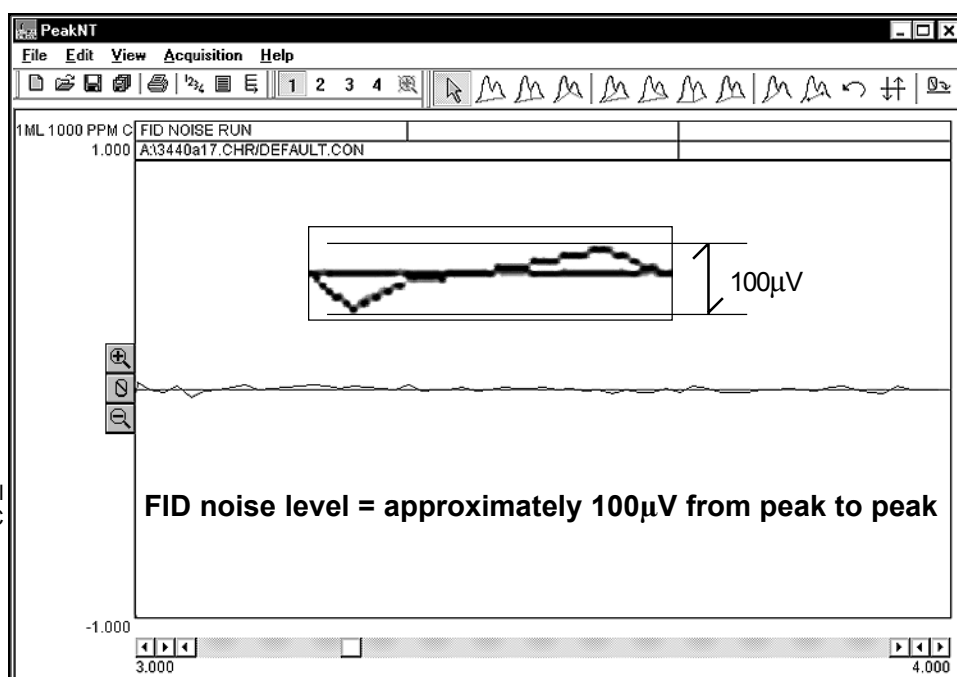
Temperature program:
Initial Hold Ramp Final
80°C 5.00 0.00 80°C

CCD noise level = approximately 40µV from peak to peak

FID Noise

Column: 1m Hayesep-D
Carrier: Helium @ 10mL/min
FID gain = High
FID temp = 150°C
FID ignitor = -400
Valve temp = 90°C

Temperature program:
Initial Hold Ramp Final
80°C 5.00 0.00 80°C



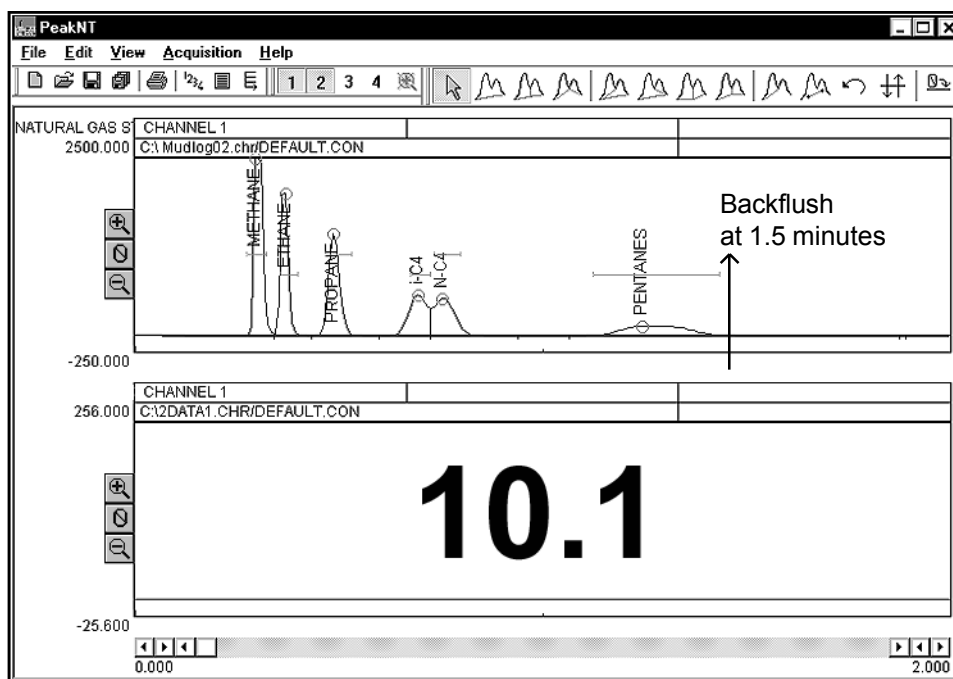
FID noise level = approximately 100µV from peak to peak

POPULAR CONFIGURATION GCs

Mud-Logger

Expected Performance

Factory Test Run of a Standard Mud-Logging System (FID and CCD)



Column: 1m Hayesep-D
Carrier: Helium @10psi
Sample: Natural Gas standard
Method: Valve injection
FID H2 = 30, FID air = 6
FID temp = 150°C
FID ignitor = -750
FID gain = MEDIUM
Valve temp = 90°C

Temperature program:

Initial	Hold	Ramp	Final
200°C	5.00	0.00	200°C

Events:

Time	Event
0.000	Zero
0.050	G ON
1.500	G OFF

Results:

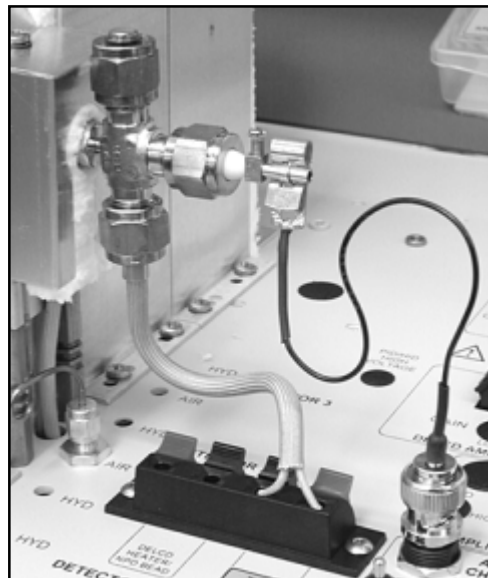
Component	Retention	Area
Methane	0.291	6664.1410
Ethane	0.366	2770.3785
Propane	0.483	2762.6450
i-C4	0.691	1754.0118
N-C4	0.750	1913.8415
Pentanes	1.241	1580.4310
Total		17445.4488

DETECTORS

Nitrogen/Phosphorus Detector - NPD

Overview

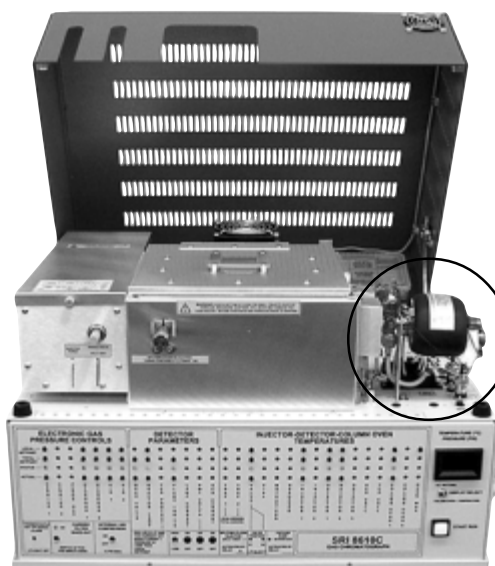
The SRI Nitrogen-Phosphorus Detector (NPD) has a linear response selective to organic compounds containing nitrogen and/or phosphorus. The NPD also responds to normal hydrocarbons, but approximately 100,000 times less than nitrogen or phosphorus containing compounds. Due to its selectivity and sensitivity, 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.



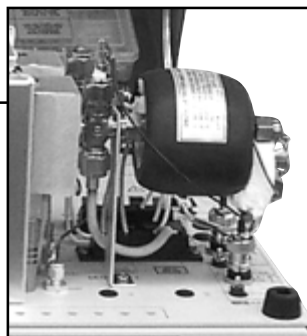
The NPD is similar in design to the FID, except it uses a thermionic NPD bead to generate ions in a hydrogen and air plasma. Like the FID, the NPD uses a stainless steel jet to deliver sample-laden carrier gas and hydrogen gas to the detector, and a positively charged collector electrode that also serves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

The thermionic NPD bead is coated with an alkali salt.



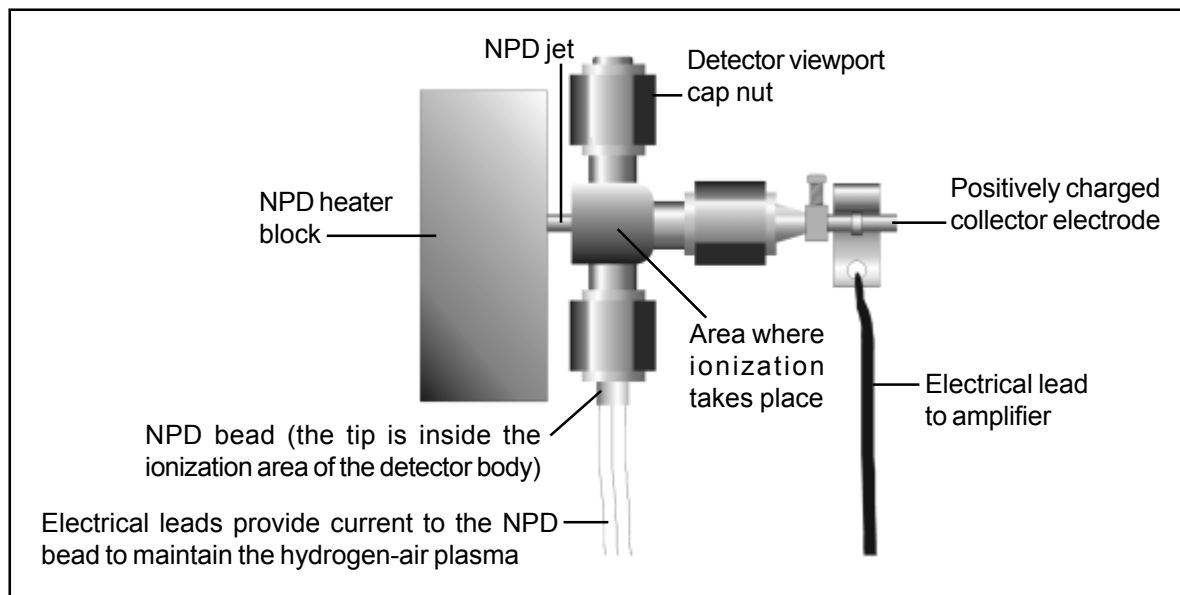
The NPD may be combined with the DELCD for pesticide screening. The NPD detects Organo-phosphate pesticides and the DELCD detects the chlorinated pesticides.



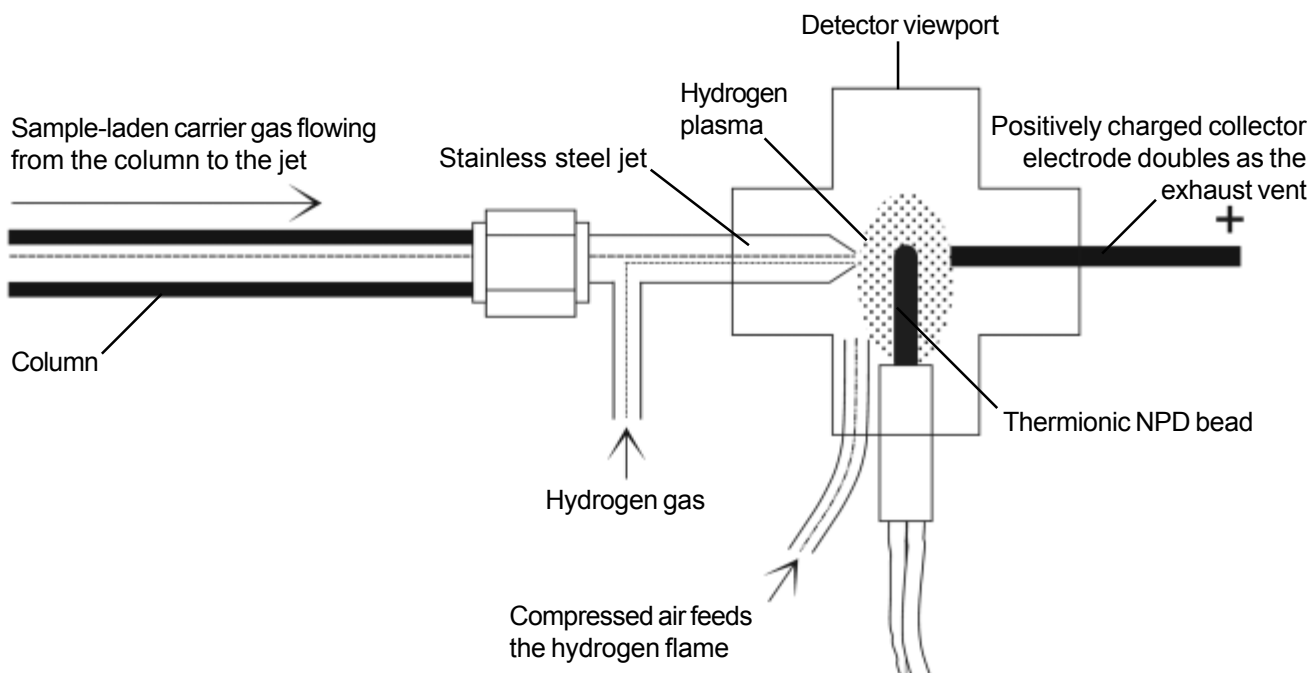
DETECTORS

Nitrogen/Phosphorus 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.



DETECTORS

Nitrogen/Phosphorus Detector - NPD

Expected Performance

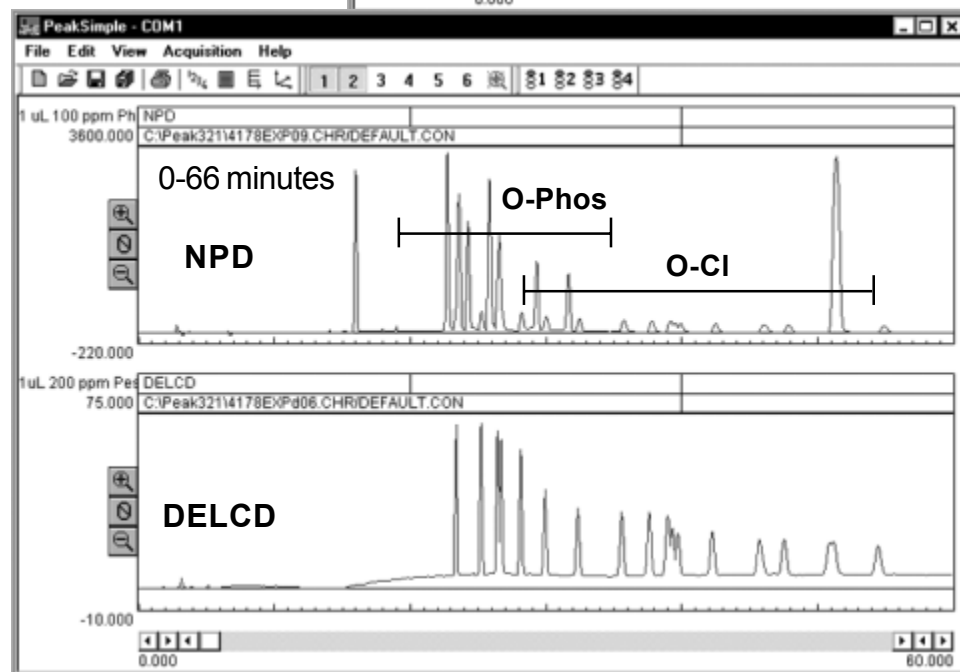
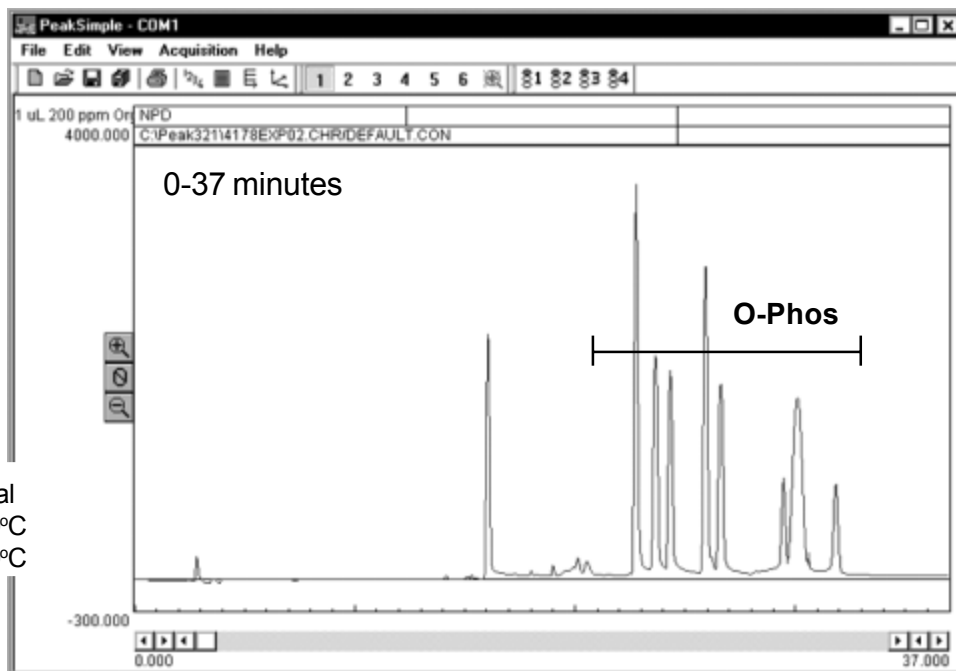
The following chromatograms are from an SRI GC equipped with NPD, DELCD and FID detectors. Since the NPD is not the only detector, helium carrier gas was used instead of nitrogen. The first chromatogram shows a separation of 200ppm Organophosphorus pesticide standard, Mix 8270. The second chromatogram 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: 1 μ L 200ppm
Organophosphorus 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



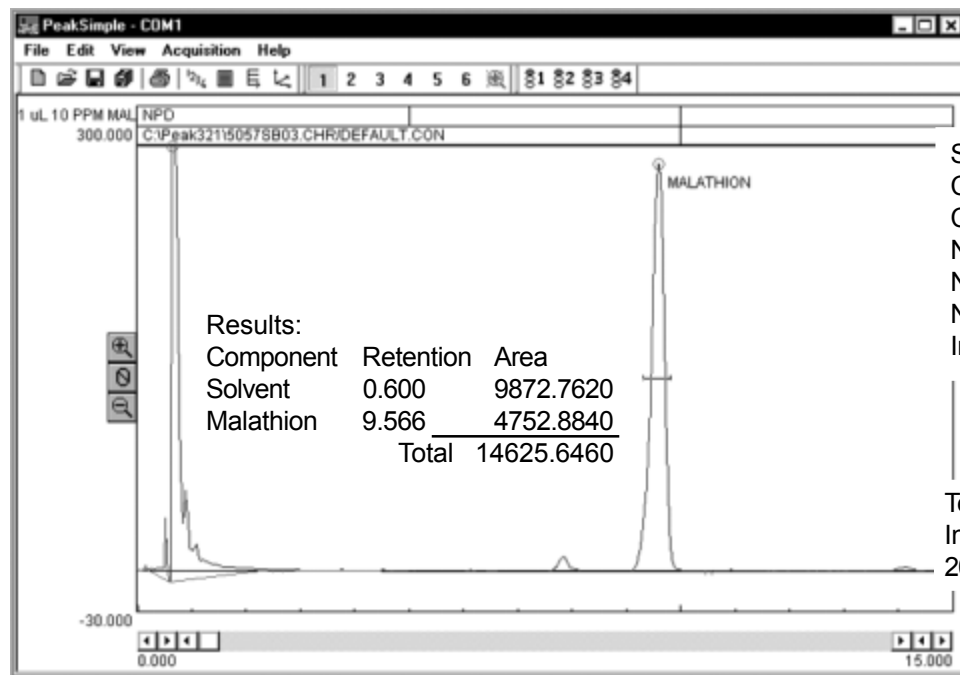
Sample: 1 μ L 100ppm
Organophosphorus pesticide
standard mix 8270 +
100ppm Organochlorine
pesticide standard mix 8081
Method: direct injection
Injector temperature: 150°C
Column: 60-meter MXT-VOL
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -370
DELCD gain: LOW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

DETECTORS

Nitrogen/Phosphorus Detector - NPD

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.

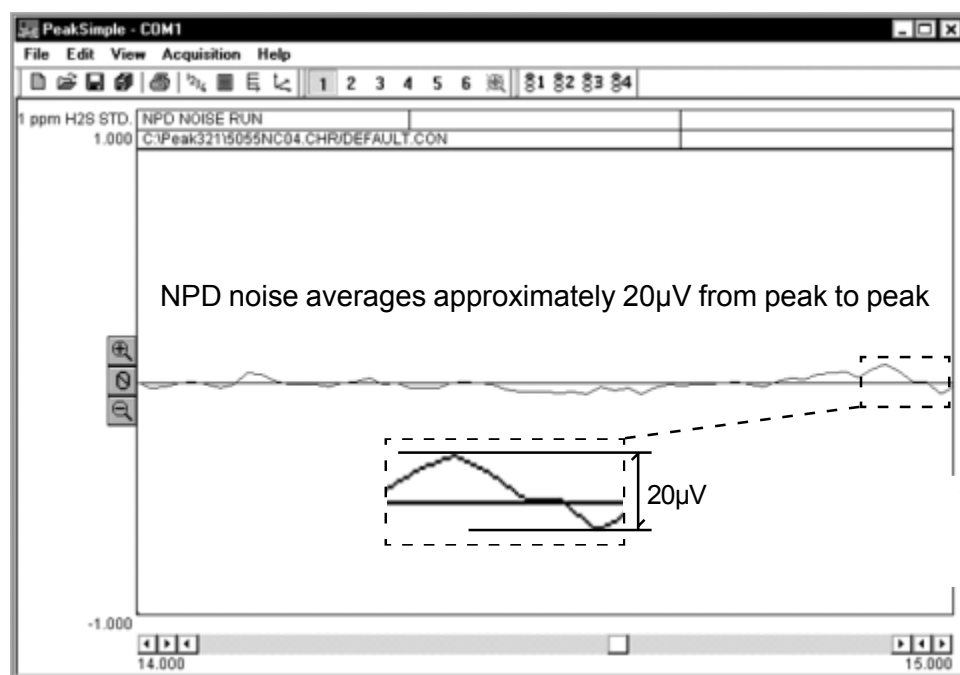


Sample: 1 μ L 10ppm malathion
Column: 15-meter MXT-5
Carrier: helium at 10mL/min
NPD gain: HIGH
NPD temperature: 250°C
NPD bead current: -380
Injector temperature: 200°C

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

DETECTORS

Nitrogen/Phosphorus Detector - NPD

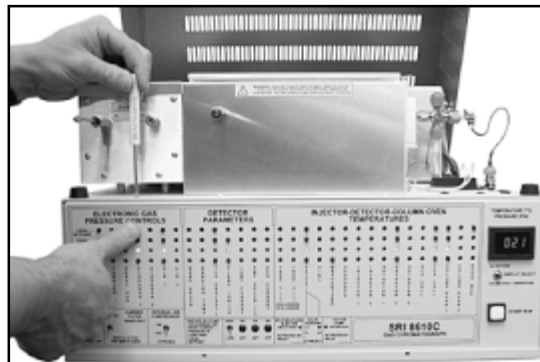
Note: Most SRI NPDs are installed on a GC with one or more other detectors. Therefore, SRI tests its NPD detectors with helium carrier gas.

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 temperature 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.

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

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

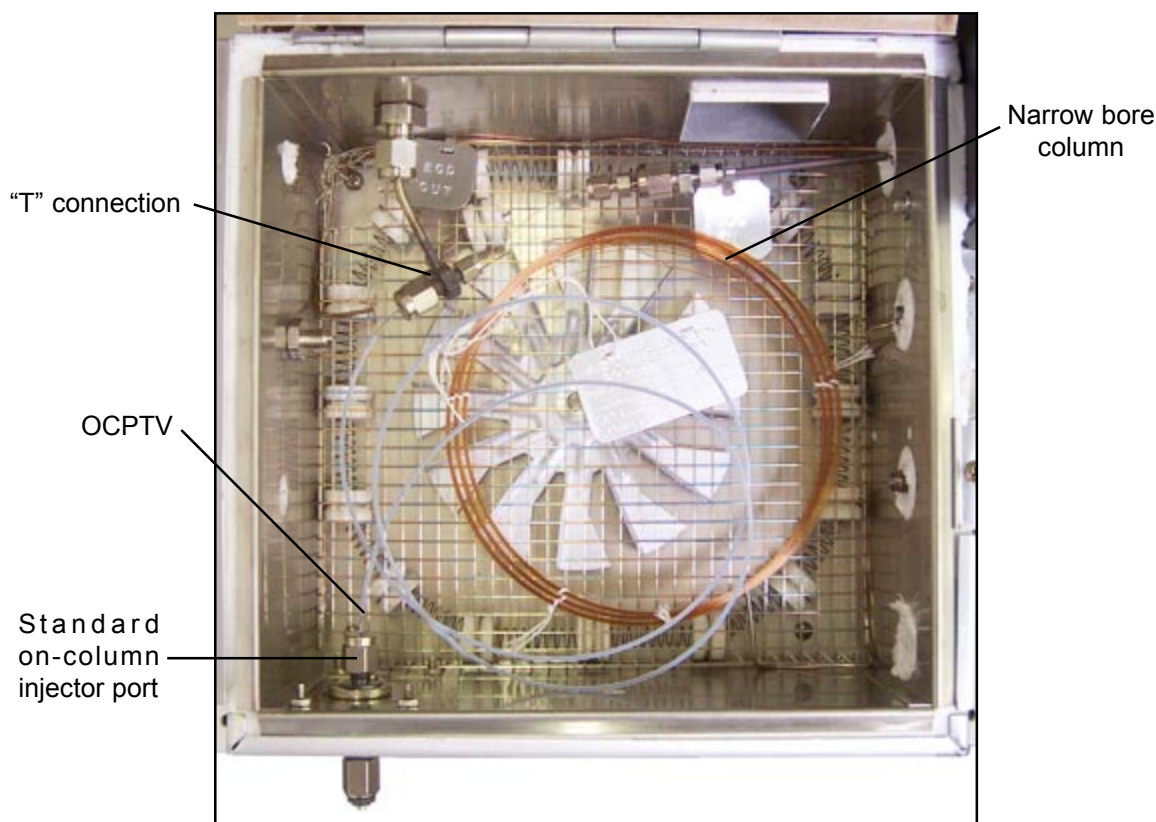
Example Gas Flow Rates table: this particular GC is equipped with NPD/DELCD and FID detectors.

INJECTORS

OCPTV - On-Column Programmed Temperature Vaporization Injector

Overview

The On-Column PTV is a resistively heated pre-column, consisting of a 1-meter long segment of 0.53ID metal capillary column (5µm film thickness), and insulating teflon sleeve. The pre-column is connected to the user's narrow bore column with a special, electrically insulated split "T" inside the GC column oven.



Like the Split/Splitless injector, the OCPTV has a split vent and needle valve for venting solvent while concentrating sample.

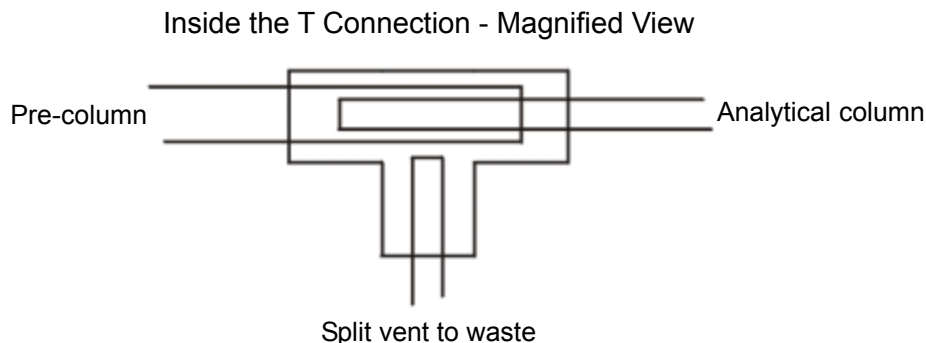
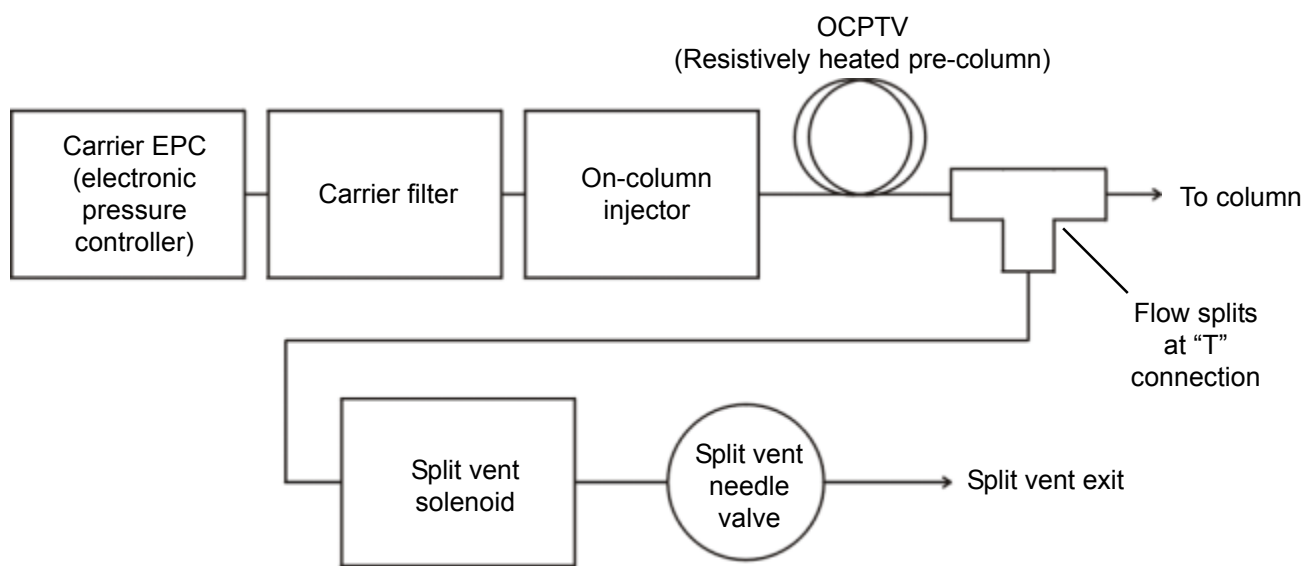
The pre-column will be factory installed. Should you need to disconnect it, simply loosen the injector port fitting inside the column oven and pull the pre-column out. Loosen the "T" fittings and remove both the pre-column and narrow bore column from the "T." Connect your replacement column to the injector port fitting inside the column oven.

INJECTORS

OCPTV - On-Column Programmed Temperature Vaporization Injector

Theory of Operation

The GC operator injects sample via syringe through the on-column injection port with the split vent open to vent the solvent. After injection and solvent venting, the pre-column heats up while the carrier gas flows through it to sweep focused analytes from the pre-column to the analytical column. At this point, the pre-column is hotter than the column oven. The temperature difference between the hotter pre-column and cooler analytical column causes the analytes to focus on the analytical column, resulting in sharp peaks on the chromatogram.



The 5 micron non-polar phase in the OCPTV pre-column has a high capacity to absorb high boiling compounds, and is stable at high temperatures. Like in-tube SPME, the pre-column discriminates in favor of high boiling semi-volatile analytes, concentrating them in the phase. Unlike a normal heated split/splitless injector, the OCPTV vents the solvent without expanding it to a gas. Therefore, the OCPTV can accommodate larger liquid injections than heated split/splitless injector.

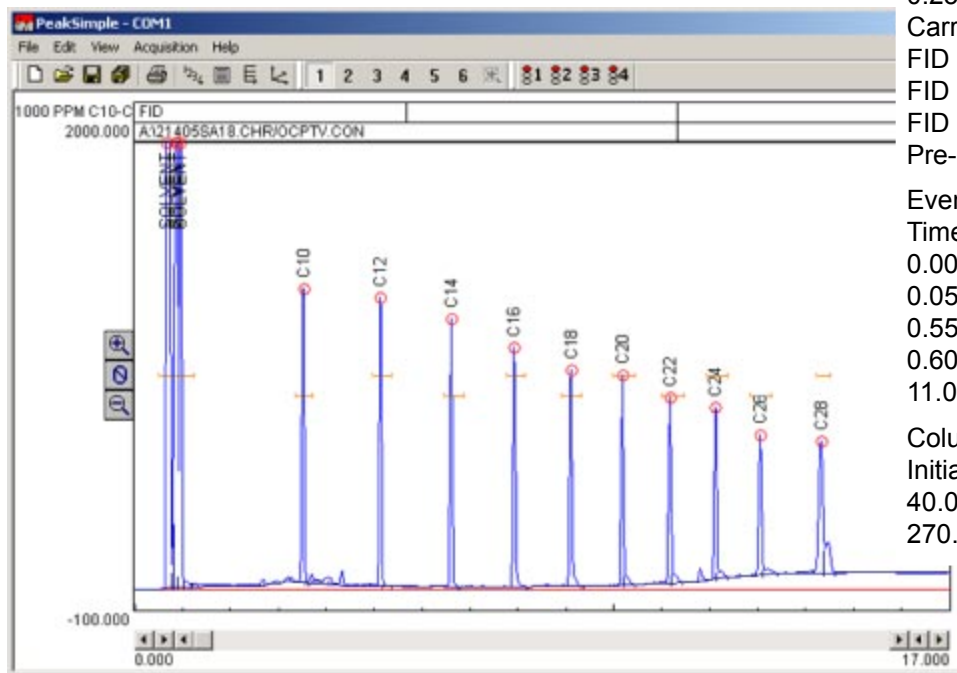
INJECTORS

OCPTV - On-Column Programmed Temperature Vaporization Injector

Expected Performance

The following chromatogram from a GC equipped with an FID detector and OCPTV injector demonstrates the sharp peaks attainable from high boiling analytes with the OCPTV.

Sample: 2 μ L 100ppm diesel range organics in hexane
 Column: 25-meter RXT-1 0.25mm 0.25 μ m
 Carrier: hydrogen at 20psi
 FID gain: HIGH
 FID temperature: 300°C
 FID ignitor: -400
 Pre-column vent flow: 100mLs/minute



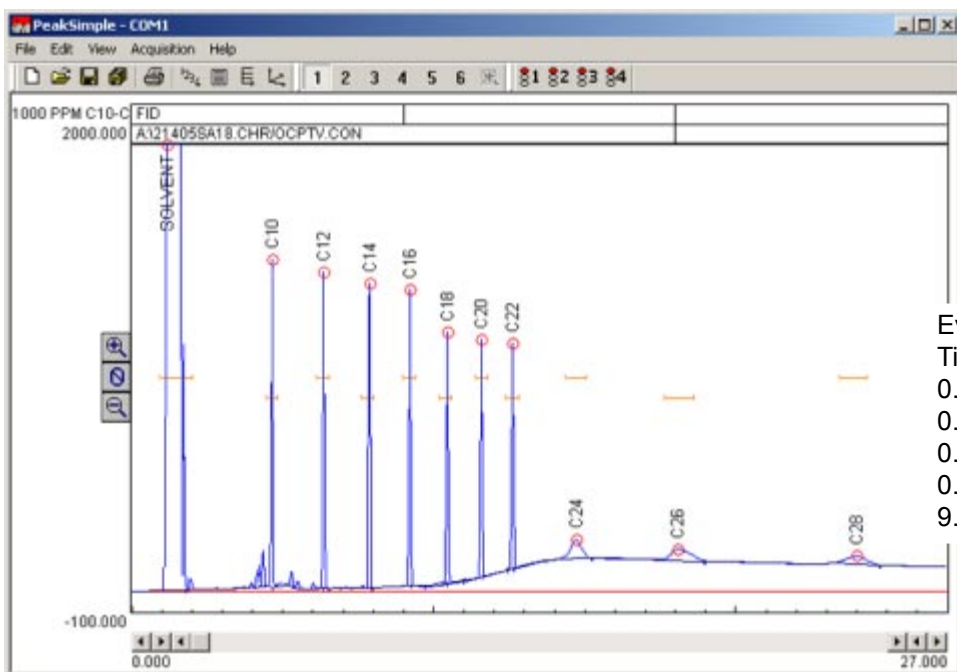
Event Table:

Time	Event
0.000	Zero signal
0.050	A ON (pre-column vent)
0.550	A OFF
0.600	F ON (pre-column heat)
11.000	F OFF

Column oven temperature program:

Initial	Hold	Ramp	Final
40.00	1.00	20.00	270.00
270.00	20.00	0.00	270.00

This chromatogram is from the same GC. It was made with the same analytical parameters, except a slightly different event table was used. In this event table, the pre-column heat was not kept on long enough to transfer the highest boiling analytes to the analytical column. The last three peaks are broad



because the column oven heat is what drove them from the OCPTV pre-column, rather than the OCPTV heat itself, as in the first chromatogram.

Event Table:

Time	Event
0.000	Zero signal
0.100	A ON (pre-column vent)
0.450	A OFF
0.500	F ON (pre-column heat)
9.000	F OFF

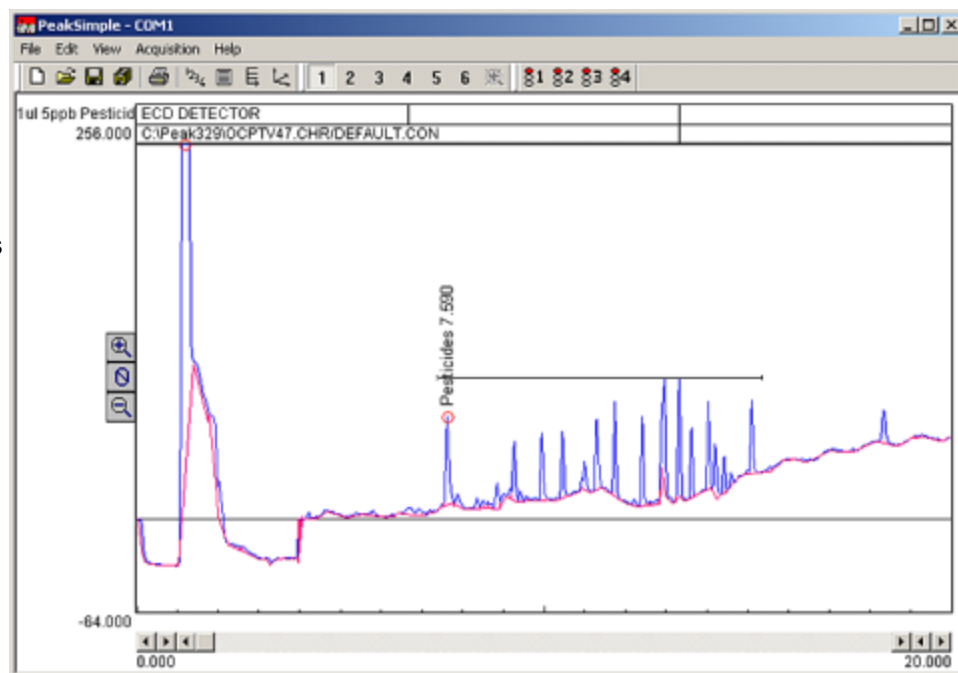
INJECTORS

OCPTV - On-Column Programmed Temperature Vaporization Injector

Expected Performance

The following chromatogram, from an SRI GC equipped with an OCPTV injector and an ECD detector, demonstrates the low detection levels achievable with the OCPTV. **NOTE:** Depending on the configuration, your GC may have different relay assignments than the examples used here.

Sample: 1 μ L 5ppb pesticides mix
Column: 15-meter XLB
0.25mm 0.25 μ m
Carrier: helium at 20psi
ECD temperature: 300°C
ECD current: 200



Event Table:

Time	Event
------	-------

0.000	Zero signal
-------	-------------

0.050	A ON (pre-column vent)
-------	------------------------

0.980	A OFF
-------	-------

1.000	B ON (pre-column heat)
-------	------------------------

11.000	B OFF
--------	-------

Column oven temperature program:

Initial	Hold	Ramp	Final
50.00	1.00	15.00	260.00
260.00	5.00	0.00	260.00

Results:

Component	Retention	Area
Pesticides	7.590	1864.6387

Chapter: Injectors

Topic: On-column Injector Operation

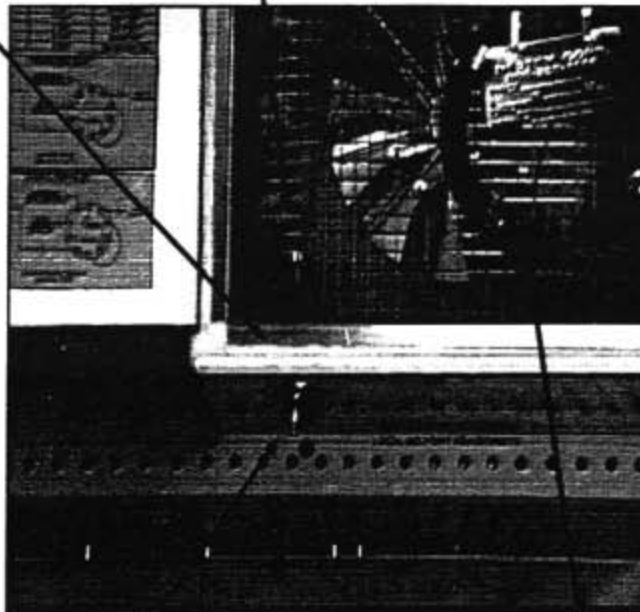
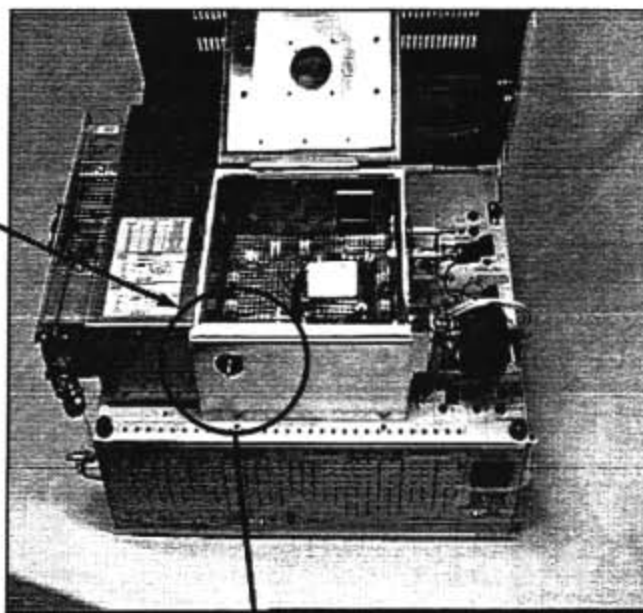
The On-column injector is designed for .53mm (wide-bore) capillary columns and 1/8" packed columns. One or two on-column injectors can be mounted on the 8610C GC, while a single on-column injector can fit on the Model 310 GC. The photo at right shows a single on-column injector mounted on the 8610C GC.

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

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

For most applications, the on-column injector is the best way to inject a liquid sample because the syringe deposits the sample into the bore of the column itself.

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



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

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

Chapter: Injectors

Topic: On-column Injector Operation

The On-column injector consists of:

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

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

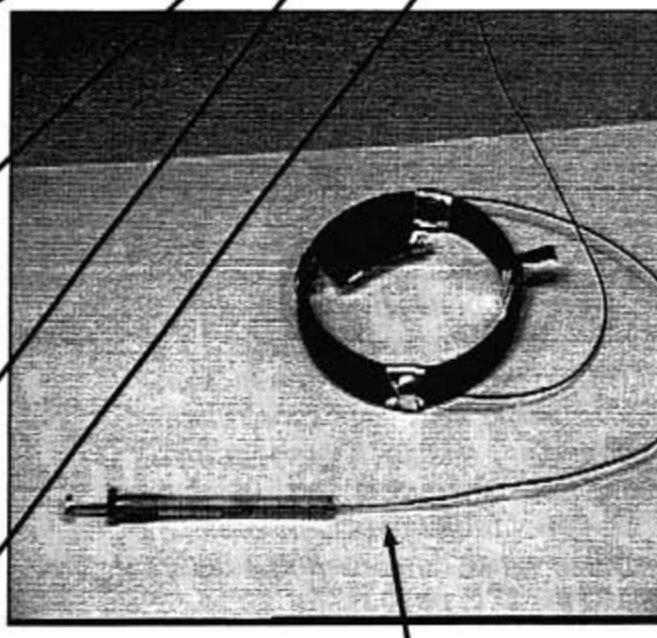
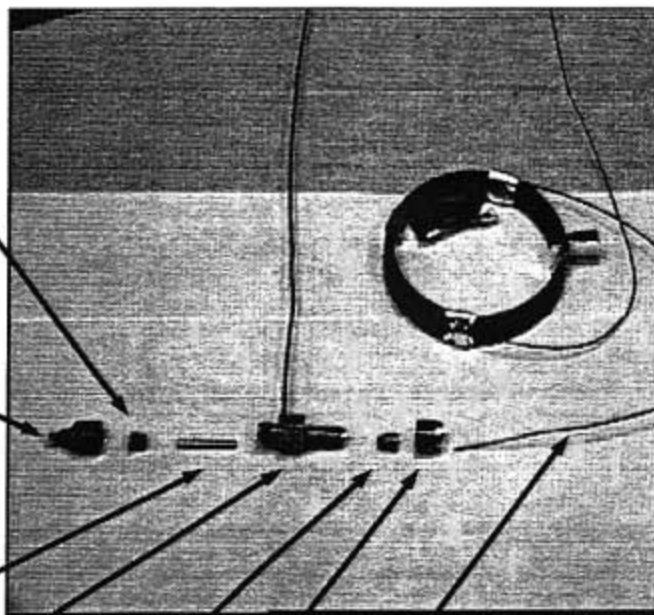
3) The wide-bore capillary column adapter (part#8690-9093) which aligns the syringe needle and the column inside the on-column injector body.

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

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

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

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



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

Chapter: Injectors

Topic: On-column Injector Operation

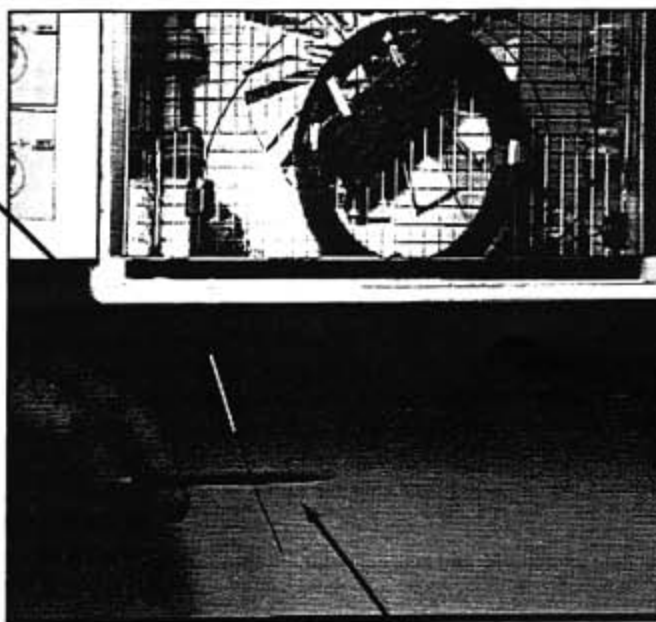
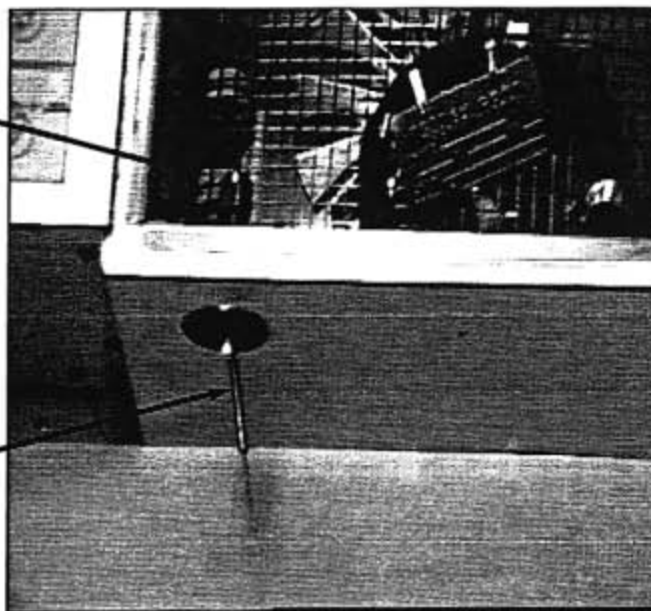
To install the column in the injector:

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

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

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

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

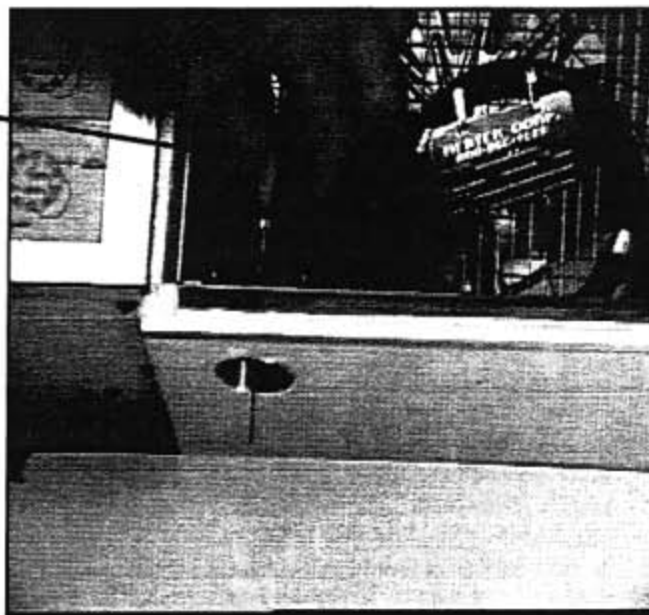


As shown above, a sharp triangle file is used to score the metal capillary column a few inches from the end which may have picked up graphite or other debris during the installation process.

Chapter: Injectors

Topic: On-column Injector Operation

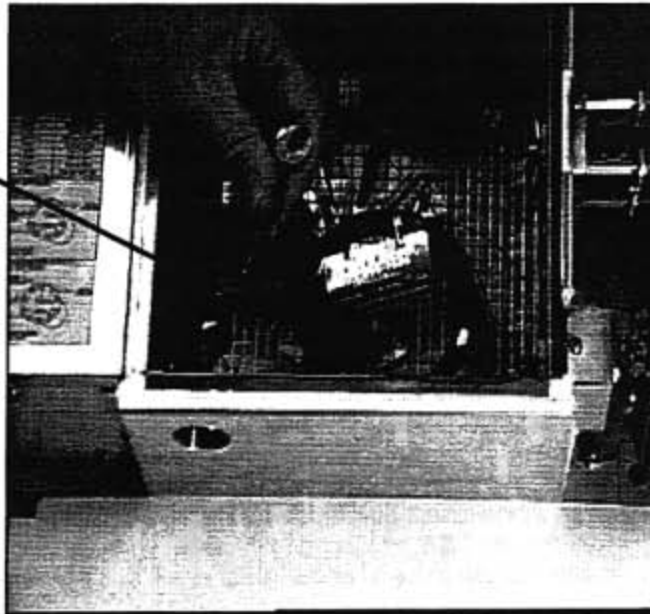
4) Pull the column and wide-bore adapter back into the injector fitting through the partially tightened nut and graphite ferrule. As you pull, the column will gradually disappear from view inside the injector fitting. Pull the column until the open end is about halfway into the fitting. The exact distance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



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

NOTE:

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

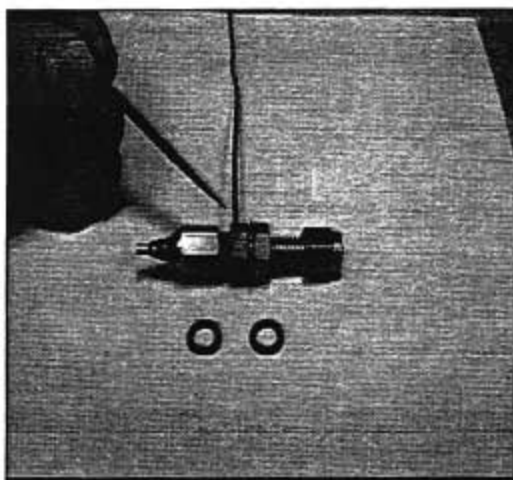


A 7/16" wrench is used to snug up the nut and graphite ferrule securing the column to the injector.

Chapter: Injectors

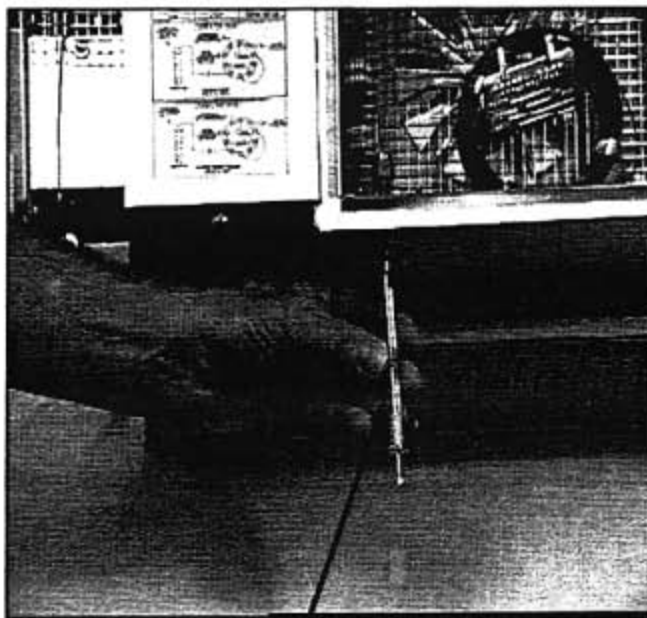
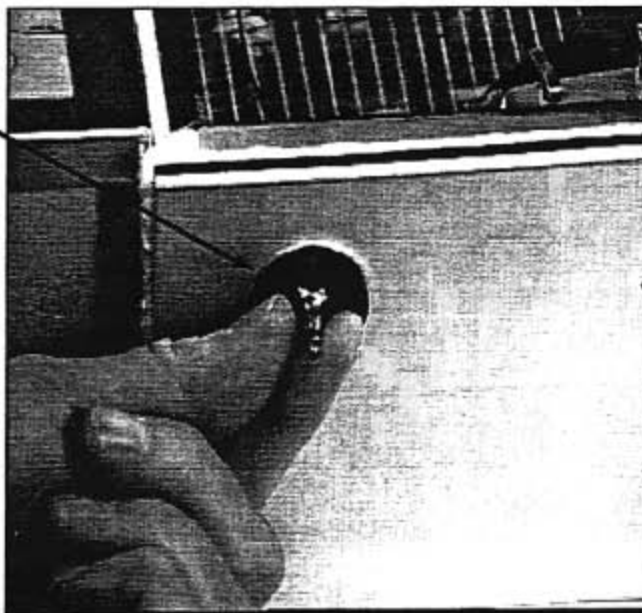
Topic: On-column Injector Operation

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



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

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



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

ELECTRONICS

Replacing the OP-Amp Chip in Your SRI GC

Testing the OP80 or LF356 Amplifier Chip(s)

The parts kit in the plastic tackle box under the red lid of your SRI GC contains a spare OP80 or LF356 amplifier chip. Additional OP-Amp chips are available under SRI part number 8690-7000. FID, NPD, HID, TID, PID, FPD, and DELCD detectors use the OP-Amp chip. The OP-Amp chip amplifies the analog detector signal. You should replace a detector's OP-Amp chip when you're not getting the signal response that is otherwise consistently observed from your detector, when the detector signal is pegged up (5000mV) or down (0), or when the detector has failed the Wet Finger test.

If your detector signal is pegged up or down, try the following:

1. Turn OFF the GC power (for at least 10 seconds).
2. Shut down the PeakSimple software program.
3. Re-launch PeakSimple.
4. Turn the GC power ON.
5. Without zeroing the data system signal, observe the milliVolt reading. If it is still pegged at 5000mV, replace the OP-Amp chip. Sometimes the signal will be pegged at or near 0, but 5000mV is much more common with a faulty OP-Amp chip.



Wet Finger Test:

1. Make a "V" sign with the first 2 fingers of your hand.
2. Moisten those fingers (lick them).
3. Place one finger on the collector electrode, and simultaneously place the other on bare metal, like the column oven lid. Make your contact brief, and observe the milliVolt reading.
4. The data system signal should jump from 0 to 5000mV (max voltage), and come back down when you remove your fingers. If your contact does not produce a similarly significant change in the milliVolt reading, then you should replace the OP-Amp chip.



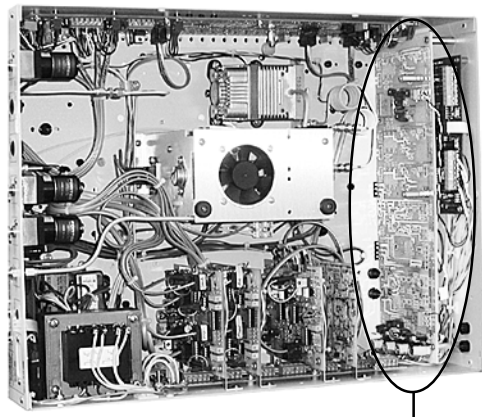
If you have dummy loads or the means to create them at your disposal, you may use the method outlined in the table below to test the OP-Amp chip(s). If the readings are not within the target range, you should replace the OP-Amp chip(s).

DETECTOR GAIN TEST		
DETECTOR(S)	DUMMY LOAD	TARGET GAIN(S) READINGS
FID / NPD / HID / TID	16000MOhm resistor connected to center of BNC jack and ground	HIGH (+2000mV), MED (100mV), HI FILT (+2000mV)
PID	16000MOhm resistor connected to center of BNC jack and ground	HIGH (+1000mV), MED (100mV), LOW (10mV)
FPD	16000MOhm resistor connected to center of BNC jack and ground	HIGH (+1000mV), MED (100mV), HI FILT (+1000mV)
DELCD	100MOhm resistor connected to red & white wire screw terminals	HIGH (+1400mV), MED (140mV), LOW (14mV)

ELECTRONICS

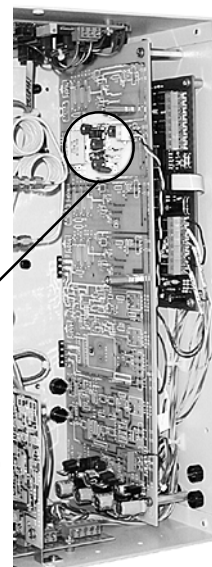
Replacing the OP-Amp Chip in Your SRI GC

Replacing the OP-Amp Chip



The amp board is the long board on the right hand side of the GC chassis interior

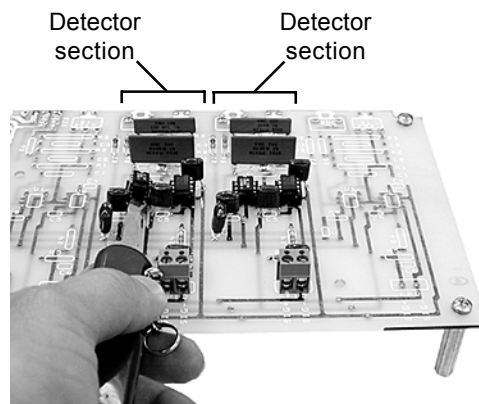
1. Turn OFF and unplug the GC.
2. Remove the 6 screws holding the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its back, then lower the panel to your working surface to access the chassis interior.
3. Locate the Amp board inside the GC chassis on the right hand side. Remove the protective steel plate by loosening the two thumbscrews that secure it to the aluminum stand-offs, and set it aside.



Pictured here is an amp board with two OP80 amp chips on it.

4. There are four possible places on the Amp board for the OP-Amp chip, depending on the detector to which it is assigned. From the top (or front, with the GC in normal operating position), the Amp board sections read FPD, FID, PID, and DELCD. If present, the NPD will be at the FID position, as would a TID if present. However, because the circuits are identical, this is just a general guide. The Amp board will be populated according to the detectors installed on the GC. Note that each circuit has a pair of chips, almost identical in appearance, installed side by side; the OP-Amp chip is the one on the left.

5. Use a small flat blade screwdriver or similar tool to pry the OP-Amp chip out of its socket and off the Amp board. (A pocket knife nail file was used in the picture).



OP80 Amp Chip

Marker



6. Note the semi-circular depression on one end of the OP-Amp chip; this is a marker for proper orientation of the chip, and it corresponds with a similar mark on the socket. The end of the chip bearing the marker faces the top of the amp board, so you will install it with that end facing away from you. Carefully position the chip over the socket so that each of the eight pins occupies a hole and press it into place.

7. Test the new OP-Amp chip with the methods described on the previous page.

GC MAINTENANCE

Replacing the Column Oven Fan Motor

If the column oven fan motor stops turning or begins to make noise, then either the bearings or the entire motor need to be replaced. A wobbly fan or noisy operation are indications that you need to replace the bearings. In most cases, you can swap out the bearings without having to replace the entire oven fan motor. You must first obtain a replacement motor and fan (impeller) from SRI: 8670-6501 for 8610 GCs, and 8670-6502 for 310 and 410 GCs; add a “-1” to the part number for 120VAC, or a “-2” for 220VAC.

Replacing the Fan Motor Bearings

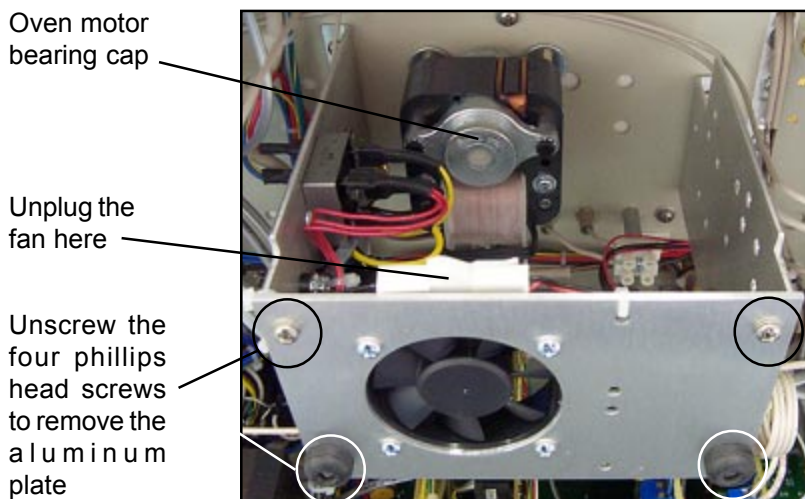
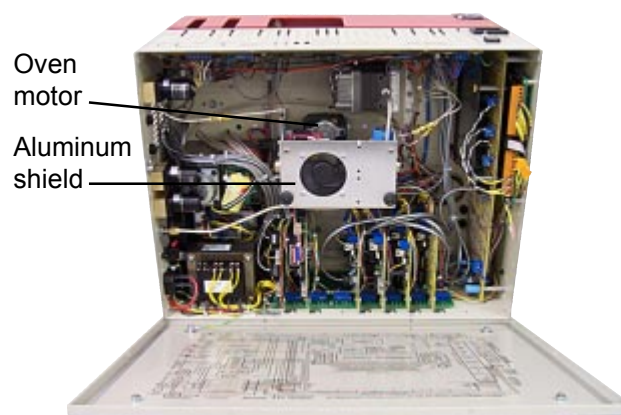
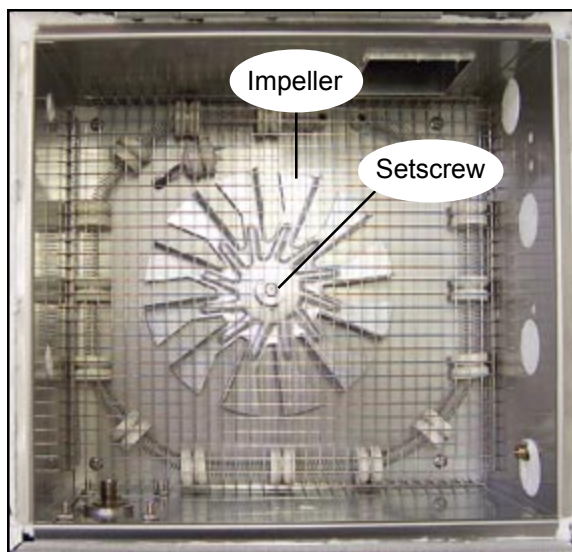
1. Remove the screen covering the fan (impeller) and heating elements by pulling it straight upward. If you need to bend it a little to remove it, that's OK, you can reshape it before you replace it.

2. The fan (impeller) is delicate and will likely sustain damage from this procedure; therefore, you should not attempt this procedure without a replacement impeller. Squirt some WD-40 on the fan motor shaft near the impeller and let it soak in for 30 seconds. Use an allen wrench (3/32) to unscrew the setscrew that secures the impeller to the oven motor shaft, then pull the impeller up and off of the shaft.

IF YOU ARE SURE YOU MUST REPLACE THE ENTIRE MOTOR, SKIP AHEAD TO “REPLACING THE ENTIRE OVEN MOTOR.”

3. To access the GC interior, unscrew the six phillip's head screws that secure the bottom panel to the GC. Rock the GC onto it's back and pull the bottom cover off toward you, as shown. The oven motor is behind an aluminum plate near the center of the GC chassis interior.

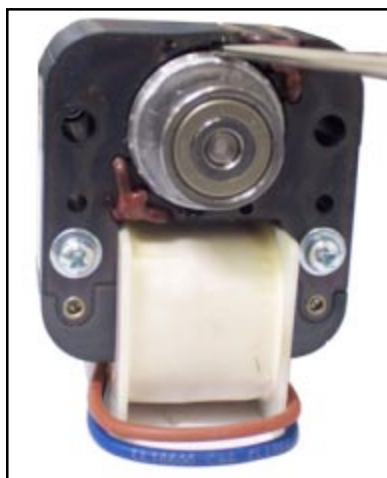
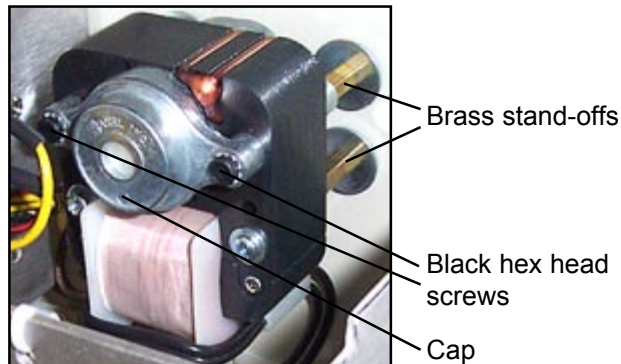
4. Unplug the fan, and unscrew the four screws to remove the aluminum plate.



GC MAINTENANCE

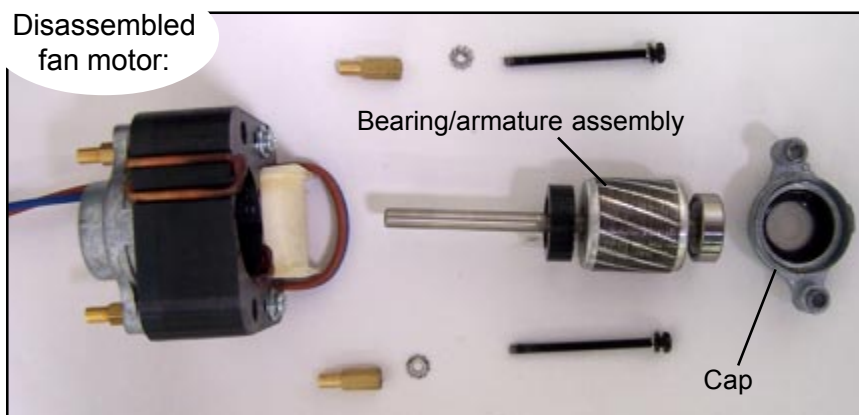
Replacing the Column Oven Fan Motor

5. The oven motor is secured to the chassis with four brass stand-offs. The bearing/armature assembly is secured to the oven motor by two black hex head screws through a cap. Use a 1/4" wrench to hold the stand-offs while unscrewing the black hex head screws to remove the bearing/armature assembly. Take care not to break or twist the screws. Once the screws are loose enough, the cap comes off and you can see the bearing/armature assembly.



6. Use your SRI screwdriver to pry the bearing/armature assembly from the fan motor.

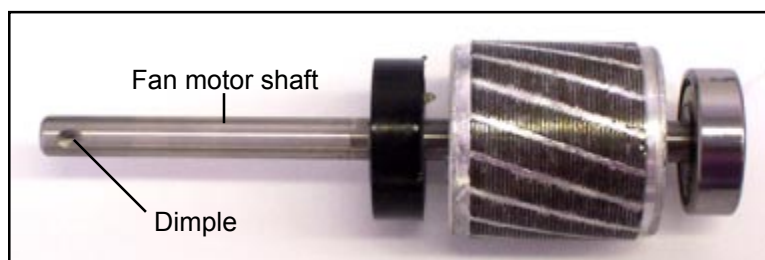
Disassembled fan motor:



7. Remove the new bearing/armature assembly from the new motor. Insert it into the center of the existing motor and press it into place. Replace the cap and tighten the two black screws.

8. Replace the aluminum plate and plug in the fan.

9. Replace the GC bottom cover and rock the GC up onto its base. Secure the bottom panel with its six screws.



10. Lubricate the new fan impeller and setscrew with anti-seize compound. Attach the new impeller to the motor shaft protruding through the oven floor. When the setscrew is tightened, it locks into a dimple on the shaft. Replace the screen, reshaping it if necessary.

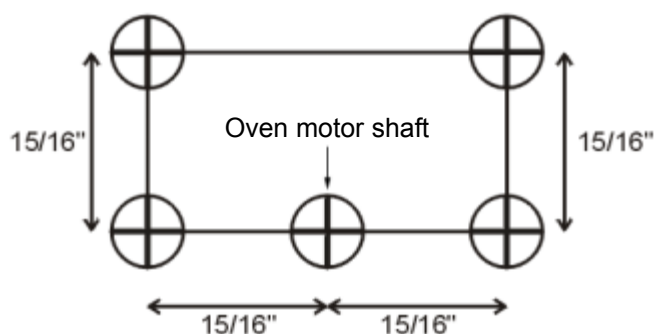
GC MAINTENANCE

Replacing the Column Oven Fan Motor

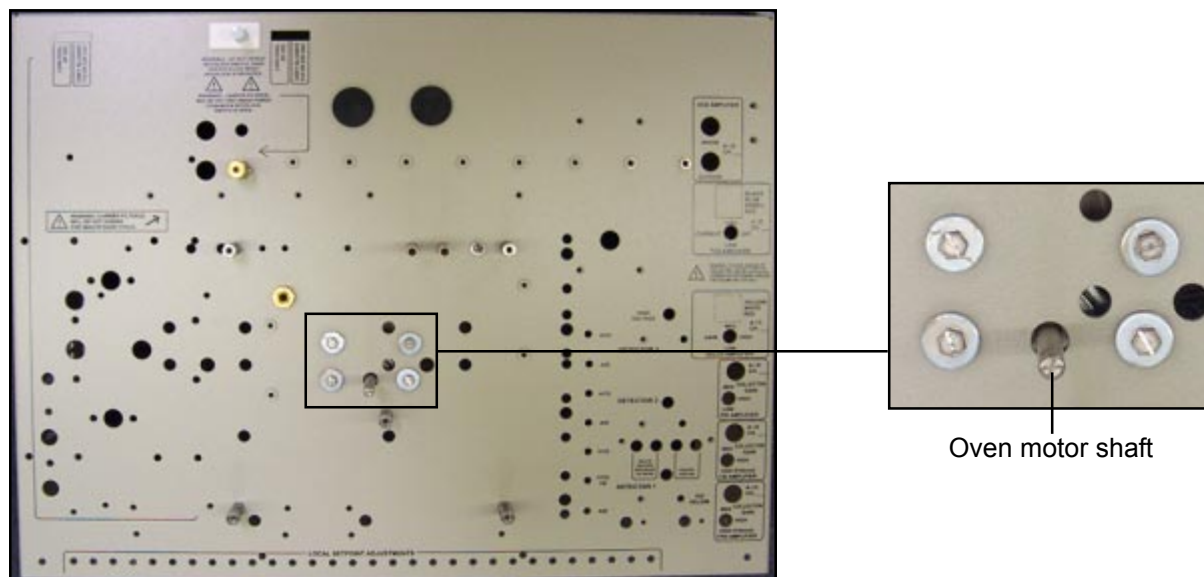
Replacing the Entire Oven Motor

1. Complete steps 1 and 2 on page 1.

2. You must drill 4 holes into the bottom of the oven. Measure $15/16''$ from the center of the oven motor shaft on each side, as shown in the diagram at right, then $15/16''$ up from the outer end of each horizontal segment. Or, you can use the diagram as a template; just center the bottom middle circle over the oven motor shaft. The picture below shows what the fan motor looks like underneath the column oven.



The following picture shows an SRI GC chassis in production with the fan motor installed:



3. Use a little oil on the drill bit because the oven is constructed of stainless steel. Drill $1/16''$ starter holes in the center of each of the four corner circles. Gradually increase the drill bit size to $3/8''$.

4. Insert a flat blade screwdriver through each of the corner holes and loosen the screws holding the oven fan motor to the chassis.

GC MAINTENANCE

Replacing the Column Fan Oven Motor

5. You must access the GC interior to remove the old oven motor and replace it with the new one. Unscrew the six phillip's head screws that secure the bottom panel to the GC. Rock the GC onto it's back and pull the bottom cover off toward you, as shown. The oven motor is behind an aluminum plate near the center of the GC chassis interior.

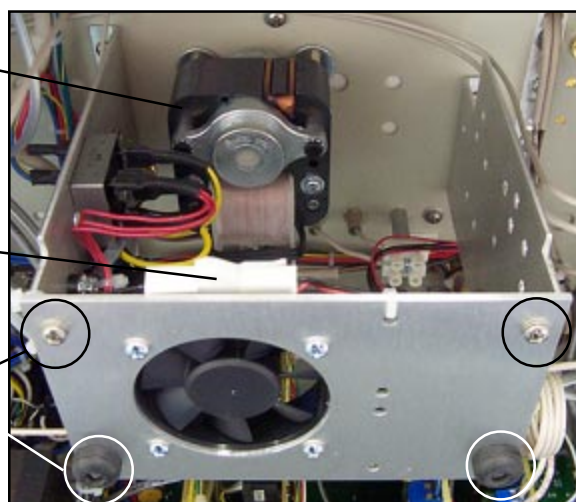


6. Unplug the fan, and unscrew the four screws to remove the aluminum shield so you can pull out the old fan motor (leave the black rubber grommets in the GC chassis). Cut the two black wires that provide power to the motor (crimp connectors are provided to connect the new motor to the black power wires).

Oven motor

Unplug the fan here

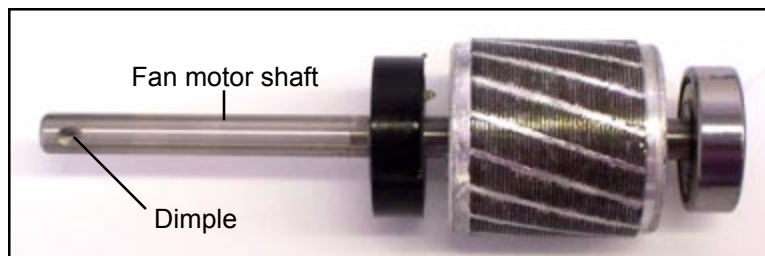
Unscrew the four phillips head screws to remove the aluminum plate



7. Install the new oven motor in place with the four brass stand-offs and stainless steel washers. Tighten the screws to center the fan shaft perpendicular to the oven floor, so that the fan can spin freely. Trim the new fan motor power wires, and connect them to the existing black power wires with the provided crimp connectors.

8. Replace the aluminum plate and plug in the fan.

9. Replace the GC bottom cover and rock the GC back onto its base. Secure the bottom cover with the six screws.



10. Lubricate the new fan impeller and setscrew with anti-seize compound. Attach the new impeller to the motor shaft protruding through the oven floor. When the setscrew is tightened, it locks into a dimple on the fan motor shaft. Replace the screen, reshaping it if necessary.

11. Fill the four holes you drilled in the oven floor with insulation (you can harvest some from another part of the oven; it doesn't take much).

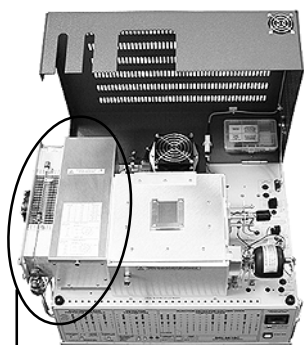
INJECTORS

Purge & Trap

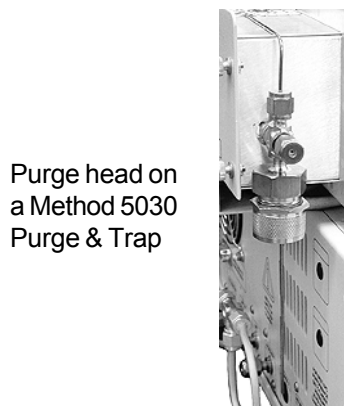
Overview

Built-in to the 8610C gas chromatograph, the SRI Purge & Trap is designed for compliance with EPA Methods 5030 and/or 5035 for the extraction of volatile organic compounds from water or soil samples. The purge and trap technique is applicable to a range of molecules from C_3 to C_{12} . The Purge & Trap hardware consists of a 10 port valve in a heated, ducted valve oven, two traps, a cooling fan, and the purge head(s). The unique dual trap design enables the simultaneous trapping of compounds with different boiling points. Each trap has its own heater, and the ends of the traps are enclosed in the valve oven ducts to prevent cold spots. The cooling fan maintains the adsorption temperature and rapidly lowers trap heat after desorption. The trap in the lower position (TRAP 1) is usually packed with TenaxTM-GR at the factory, while the upper trap (TRAP 2) is left empty for the user to pack with the desired adsorbent. A CarbosieveTM packed trap is also shipped with the GC for optional installation in the TRAP 2 position. The Carbosieve trap is used only when the analysis includes light gaseous VOC's, the most common being vinyl chloride. The Method 5030 Purge & Trap is the standard model with a fixed purge head that uses disposable 16mm test tubes for ambient temperature purging. There is a built-in septum port on this purge head through which gas standards may be spiked. The

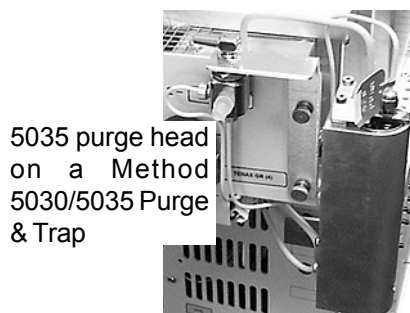
Method 5030/5035 Purge & Trap features interchangeable purge heads. The 5035 purge head is a thermostatted heater body (from ambient to 50°C) which accepts standard 40mL VOA vials. Inside the heater body are two needles which puncture the septum: the longer one bubbles helium purge gas through the sample, while the shorter needle exhausts sample-laden gas to the adsorbent traps. In compliance with EPA Method 5035, the purge head is mechanically agitated while the sample is being purged. There is a syringe port on the Method 5030/5035 Purge & Trap that allows water and internal standard to be added to the sample in the vial without puncturing the septum again. Operation of the Purge & Trap is automated by the PeakSimple data system.



SRI GC equipped with
Method 5030 Purge & Trap

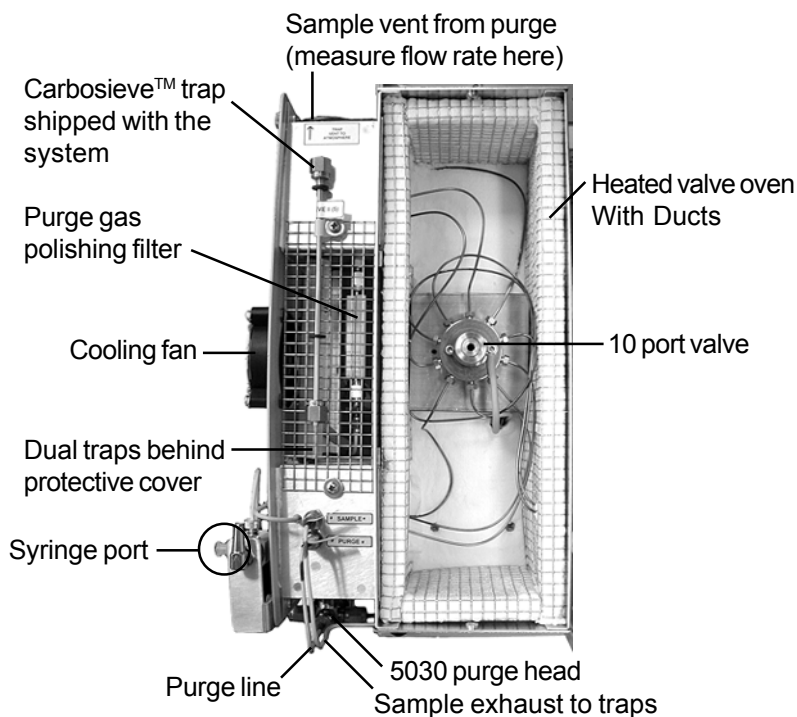


Purge head on
a Method 5030
Purge & Trap



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

Method 5030/5035 Purge & Trap

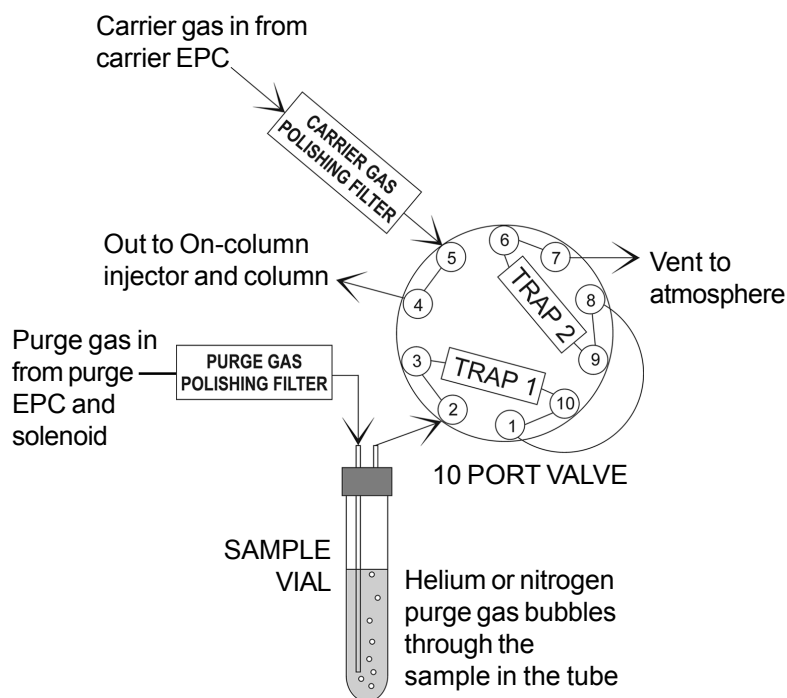


INJECTORS

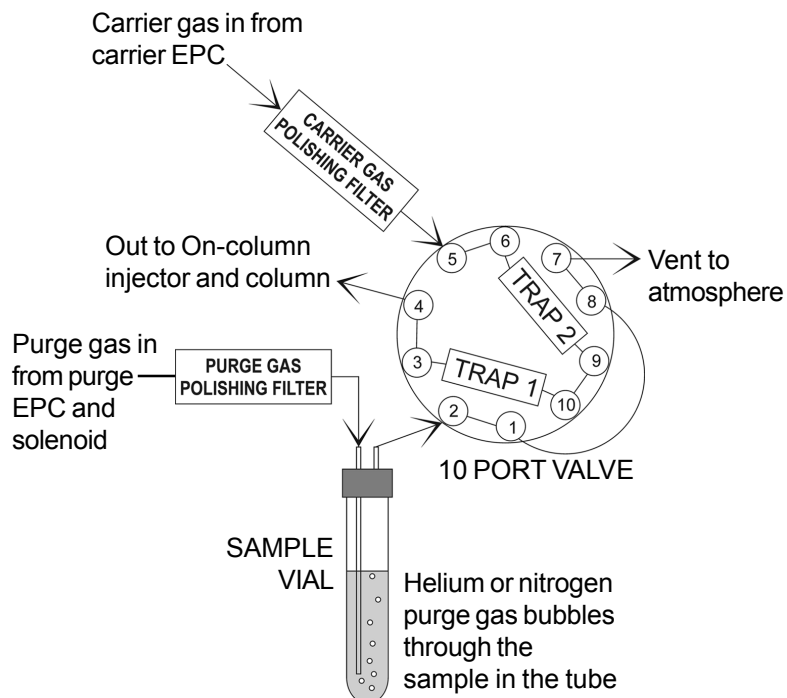
Purge & Trap

Theory of Operation

Purge & Trap Valve in the LOAD Position



Purge & Trap Valve in the INJECT Position



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

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

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

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

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

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

Sample Preparation

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

Method 5030

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

For aqueous samples:

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

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

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



Always use clean sample containers

Method 5035

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

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

INJECTORS

Purge & Trap

General Operating Procedures

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

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

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

Typical 5030/5035 Event Table		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
0.200	(D "ON")	Shaker "ON"
5.000	(D "OFF")	Shaker "OFF"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.050	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
12.900	B "ON"	Trap set "ON" (+50°C)
13.000	G "OFF"	Valve in "LOAD"
14.900	F "OFF"	Trap 1 "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.300	E "OFF"	Purge "OFF"
15.500	B "OFF"	Trap set "OFF" (+0)

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

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

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

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

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

General Operating Procedures Continued

Using Two Traps

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

Tenax™-GR Properties

Composite of Tenax TA and 30% graphite

Low water affinity

350°C temperature limit

200nm average pore size

60/80 mesh size

Available from Alltech

2051 Waukegan Road

Deerfield, IL 60015 USA

908-788-5550

www.alltechweb.com

Carbosieve™ S111 Properties

Carbon molecular sieve

Moderate water affinity

400°C temperature limit

15-40 angstrom average pore size

60/80 mesh size

Available from Supelco

Supelco Park

Bellefonte PA 16823

800-247-6628

www.sigma-aldrich.com

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

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

INJECTORS

Purge & Trap

General Operating Procedures continued

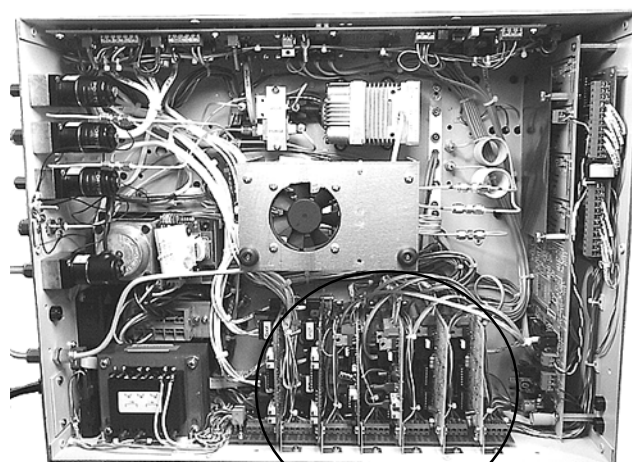
Adjusting Trap Adsorption Temperatures

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

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

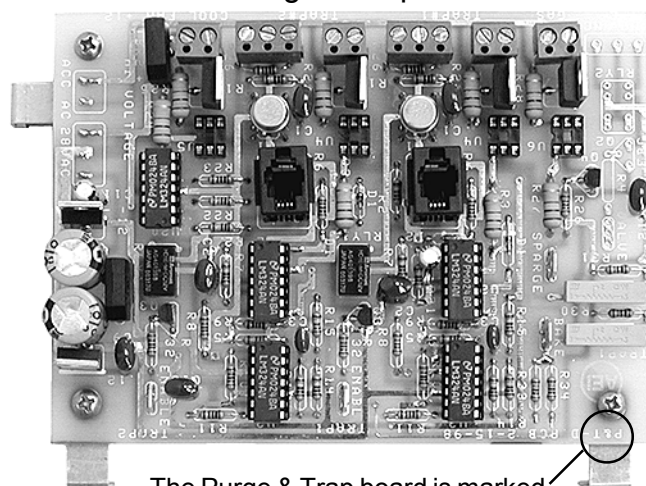
2. Locate the Purge & Trap board; it is one of a group of similar-looking boards installed along the back and top walls of the GC interior. The Purge & Trap board has two trimpots right next to each other, and it is marked with an upside-down “P&T” on the lower outer corner.

GC Chassis Interior



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

The Purge & Trap Board



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

—Trap 2 trimpot setpoint
—Trap 1 trimpot setpoint

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

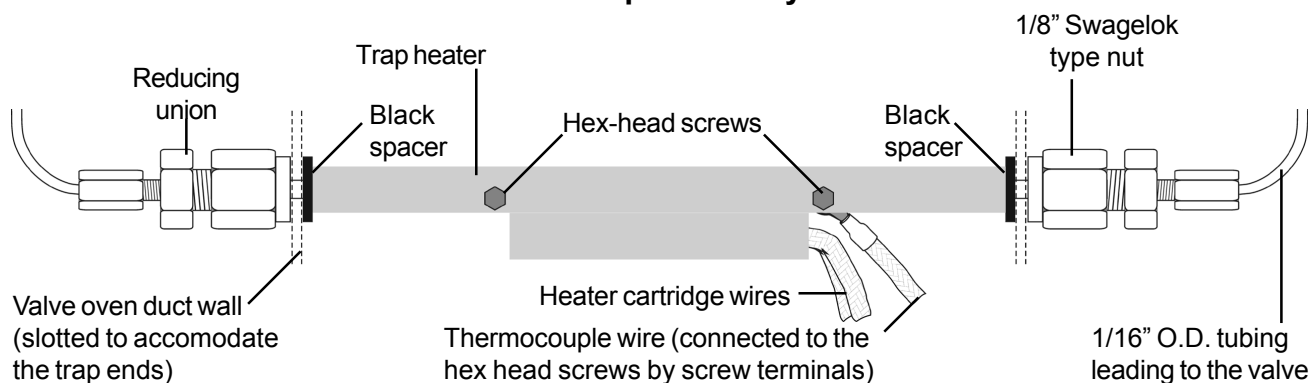
4. When you are finished adjusting both trap adsorption temperatures, place the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its base. Secure the base with its 6 screws.

Switching / Replacing Traps

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

1. With the red protective GC cover raised, remove the Purge & Trap cover plate by loosening the four brass thumbscrews at its corners.
2. Carefully remove the two squares of white insulation from each valve oven duct to expose the fittings that secure the traps ends to the 1/16" O.D. tubing leading to the 10 port valve.
3. Gently slide the trap assembly out of the slots in the valve oven ducts (there is enough slack in the heater and thermocouple wires to pull either trap about 1 inch outside the duct).
4. Use two wrenches to loosen the 1/8" Swagelok type nuts that secure the traps ends to stainless steel 1/8"-1/16" reducing unions.
5. The trap heater is a clamshell design, consisting of two halves. To remove the heater from the trap, loosen but do not remove the two securing hex head screws. The two halves of the clamshell heater will open enough to let the trap drop out.
6. Attach the replacement trap to the reducing unions with the trap's two 1/8" nuts. Use stainless steel nuts and brass ferrules when replacing traps. DO NOT use graphite ferrules, as graphite has some adsorption properties and may interfere with your analysis.
7. Slip the trap into the clamshell heater and tighten the two hex head screws.
8. Gently push the trap ends back into the slots in the two interior duct walls, making sure that the black spacers are between the duct walls and the trap heater. TO AVOID DAMAGE, ARRANGE THE TRAPS SO THAT ONE TRAP'S HEATER WIRES DO NOT LAY ACROSS THE OTHER TRAP'S HEATER.
9. Repeat the process with the other trap if necessary. Replace the white duct insulation squares, then replace and secure the Purge & Trap cover plate.

SRI Trap Assembly



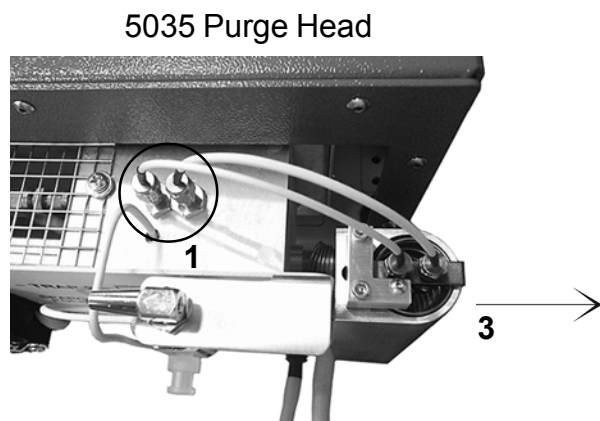
INJECTORS

Purge & Trap

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

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

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

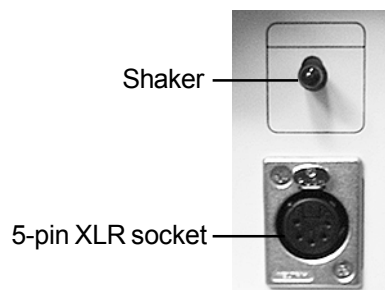
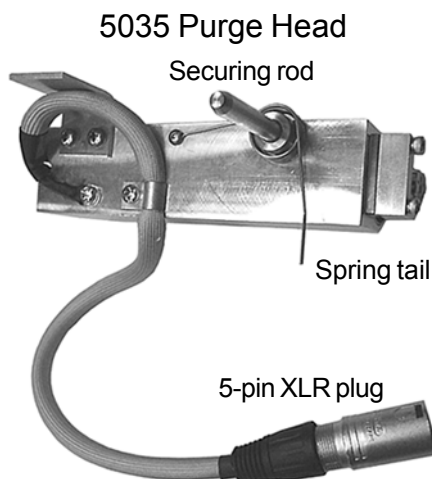


3. If you are removing the 5035 purge head, squeeze the protruding tail of the spring toward the heater body with your thumb as you pull the purge head out toward the front of the GC. Unplug the cord from the socket on the GC.



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

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



INJECTORS Purge & Trap

Expected Performance

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

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

FID Results:

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

PID Results:

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

DELCD Results:

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

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

FID Results:

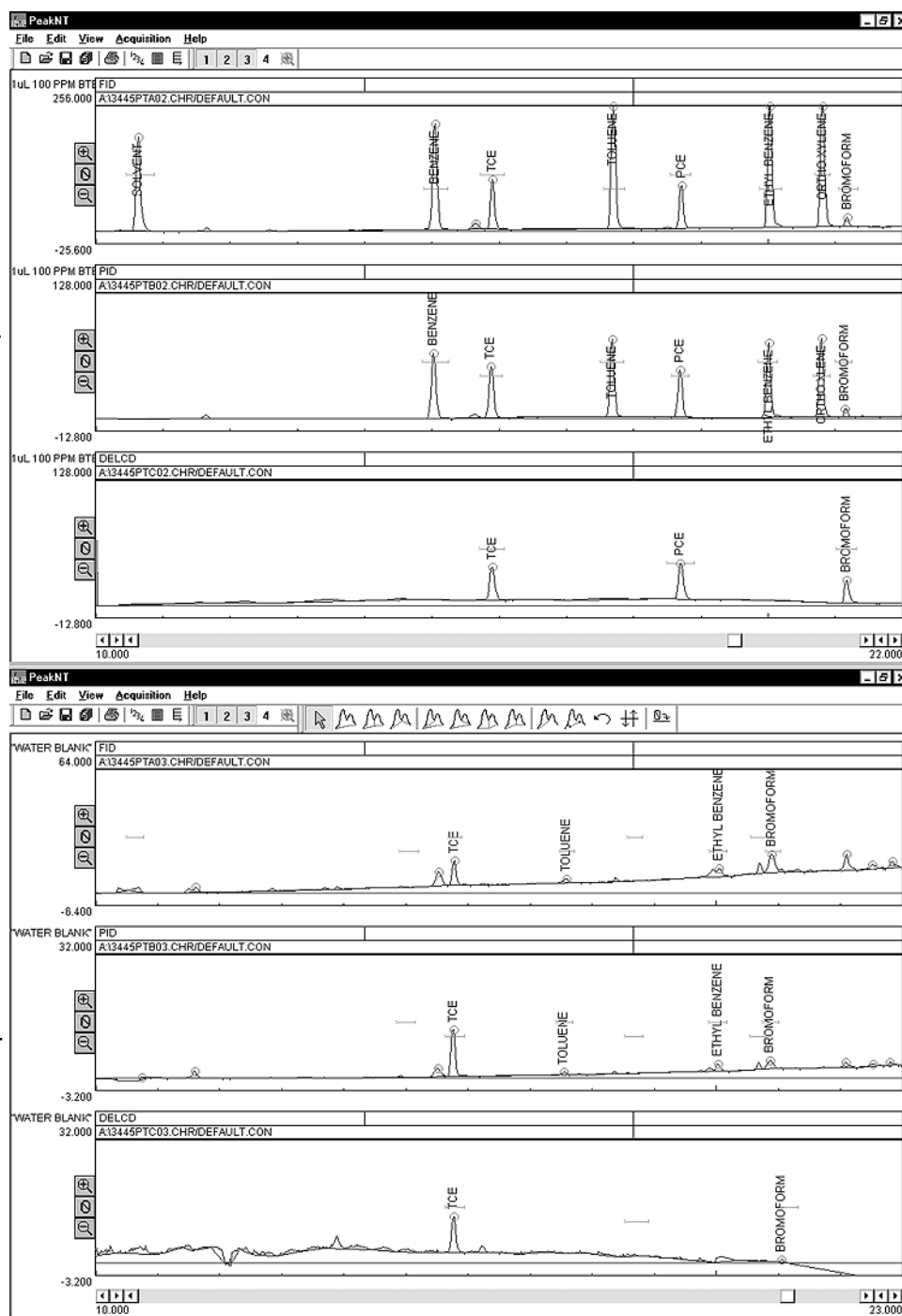
Component	Retention	Area
TCE	15.883	441.8700
Toluene	17.566	17.4000
Ethyl Benzene	20.016	1247.3420
Ortho Xylene	20.800	1258.9260
Total		6546.8080

PID Results:

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

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340



INJECTORS

Purge & Trap

Troubleshooting and Maintenance

Carryover

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

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

$$\frac{4}{353} = \frac{x}{10\text{ppb}}$$
$$353x = 40\text{ppb}$$
$$x = 0.1133\text{ppb}$$

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

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

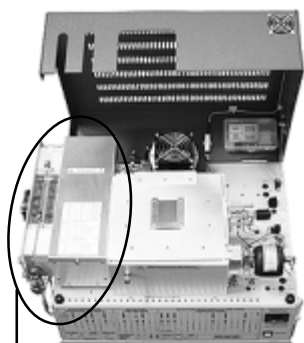
- ◆ Method 5021, in which an automated headspace sampler is used with a PID and DELCD equipped GC
- ◆ Method 3820, in which a hexadecane extraction of the sample is analyzed by a FID and/or ECD equipped GC.

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

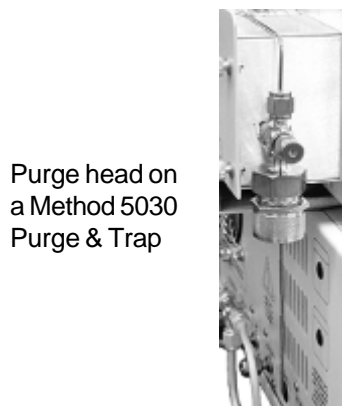
Overview

Built-in to the 8610C gas chromatograph, the SRI Purge & Trap is designed for compliance with EPA Methods 5030 and/or 5035 for the extraction of volatile organic compounds from water or soil samples. The purge and trap technique is applicable to a range of molecules from C_3 to C_{12} . The Purge & Trap hardware consists of a 10 port valve in a heated, ducted valve oven, two traps, a cooling fan, and the purge head(s). The unique dual trap design enables the simultaneous trapping of compounds with different boiling points. Each trap has its own heater, and the ends of the traps are enclosed in the valve oven ducts to prevent cold spots. The cooling fan maintains the adsorption temperature and rapidly lowers trap heat after desorption. The trap in the lower position (TRAP 1) is usually packed with TenaxTM-GR at the factory, while the upper trap (TRAP 2) is left empty for the user to pack with the desired adsorbent. A CarbosieveTM packed trap is also shipped with the GC for optional installation in the TRAP 2 position. The Carbosieve trap is used only when the analysis includes light gaseous VOC's, the most common being vinyl chloride. The Method 5030 Purge & Trap is the standard model with a fixed purge head that uses disposable 16mm test tubes for ambient temperature purging. There is a built-in septum port on this purge head through which gas standards may be spiked. The

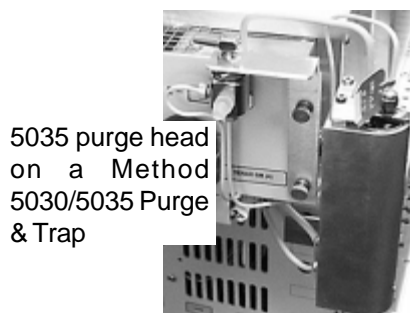
Method 5030/5035 Purge & Trap features interchangeable purge heads. The 5035 purge head is a thermostatted heater body (from ambient to 50°C) which accepts standard 40mL VOA vials. Inside the heater body are two needles which puncture the septum: the longer one bubbles helium purge gas through the sample, while the shorter needle exhausts sample-laden gas to the adsorbent traps. In compliance with EPA Method 5035, the purge head is mechanically agitated while the sample is being purged. There is a syringe port on the Method 5030/5035 Purge & Trap that allows water and internal standard to be added to the sample in the vial without puncturing the septum again. Operation of the Purge & Trap is automated by the PeakSimple data system.



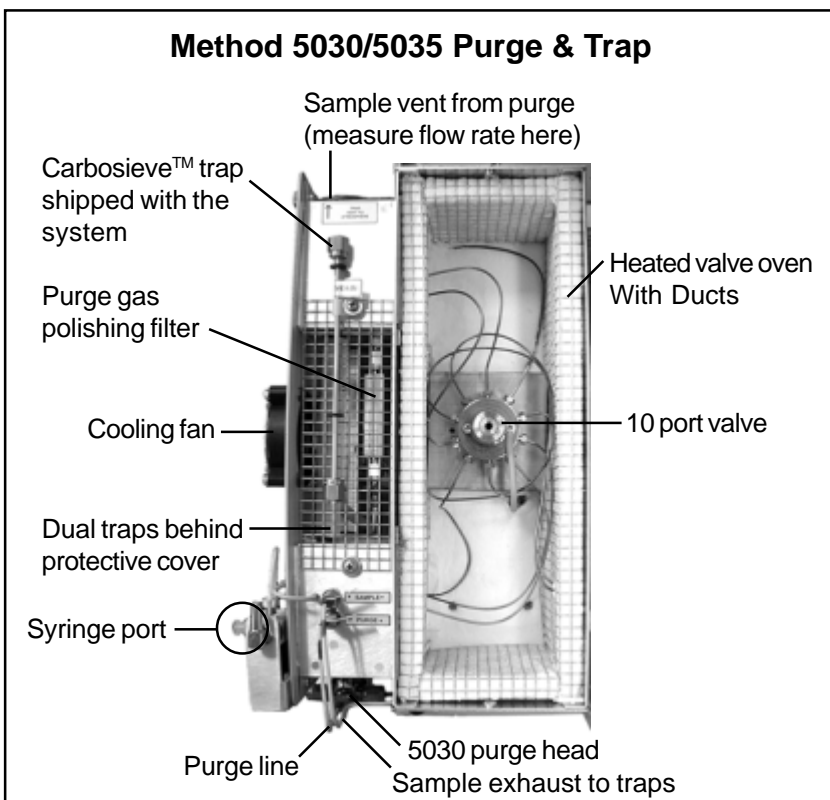
SRI GC equipped with Method 5030 Purge & Trap



Purge head on a Method 5030 Purge & Trap



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

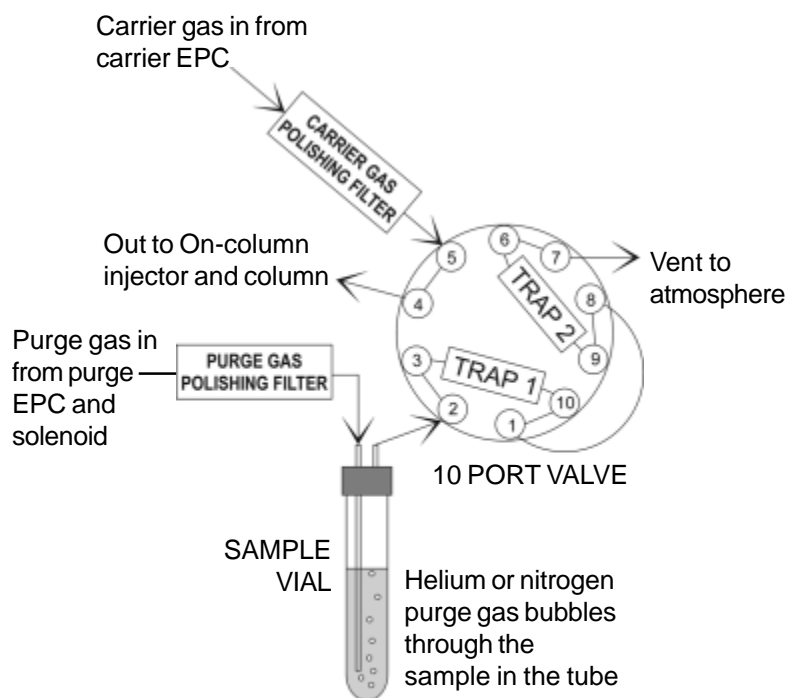


INJECTORS

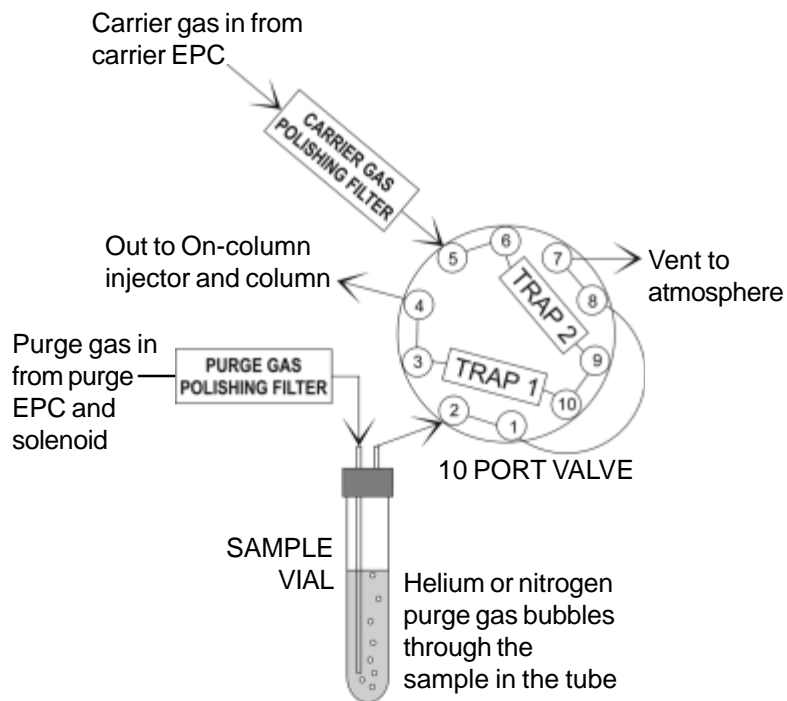
Purge & Trap

Theory of Operation

Purge & Trap Valve in the LOAD Position



Purge & Trap Valve in the INJECT Position



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

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

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

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

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

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

Sample Preparation

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

Method 5030

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

For aqueous samples:

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

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

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



Always use clean sample containers

Method 5035

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

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

INJECTORS

Purge & Trap

General Operating Procedures

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

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

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

Typical 5030/5035 Event Table		
EVENT TIME	EVENT	EVENT FUNCTION
0.000	ZERO	Zero signal
0.100	E "ON"	Purge "ON"
0.200	(D "ON")	Shaker "ON"
5.000	(D "OFF")	Shaker "OFF"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.050	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
12.900	B "ON"	Trap set "ON" (+50°C)
13.000	G "OFF"	Valve in "LOAD"
14.900	F "OFF"	Trap 1 "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.300	E "OFF"	Purge "OFF"
15.500	B "OFF"	Trap set "OFF" (+0)

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

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

5. Load or create an appropriate temperature program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software. As a basic rule for good separation, the column oven should be kept at 40°C for 10-12 minutes: 6 minutes while the sample is purging plus 4-6 more minutes after the valve actuates to the INJECT position.

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

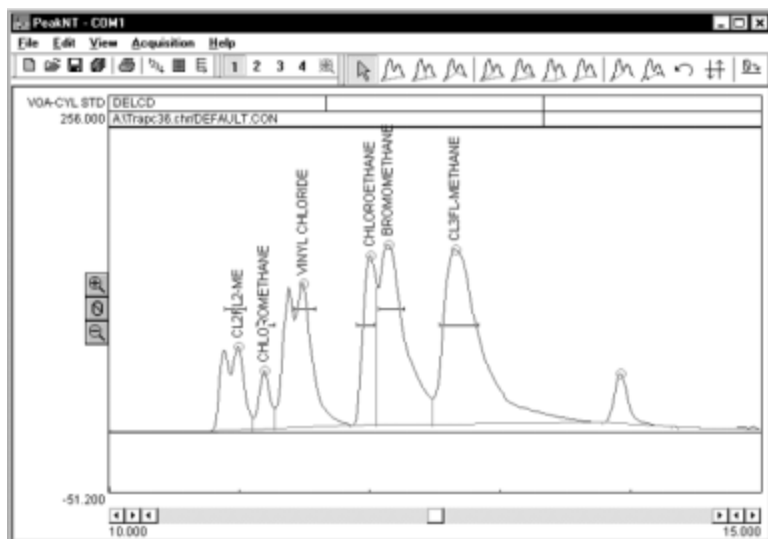
7. When the system is at temperature and displaying a stable signal, insert the sample test tube or VOA vial into the purge head and begin the analysis.

INJECTORS

Purge & Trap

General Operating Procedures Continued Using Two Traps

The SRI dual trap design gives the Purge & Trap user many options to effectively trap and release analytes from a particular adsorbent. Due to its low affinity for water, Tenax™-GR is especially useful for the purging of VOCs from aqueous samples, making it a good general purpose trap for EPA style purge and trap techniques. The Carbosieve™ packed trap is very retentive for light hydrocarbons, but since it tends to retain water and smear the other peaks, it should only be used when vinyl chloride is among the target analytes. This tendency to smear may be reduced by manipulating the desorption times for the two traps. If the Carbosieve™ trap (TRAP 2) is desorbed while the Tenax™-GR trap (TRAP 1) is still cold, the components will refocus on the Tenax™-GR. The Tenax™-GR trap is then heated to desorb all the components, which results in sharper peaks on the chromatogram. These two chromatograms are from an EPA style purge and trap analysis. The first one was made using Epa&t1c.evt for one trap. The peak separation is much better on the second chromatogram, which was made using Epa&t2c.evt for two traps.

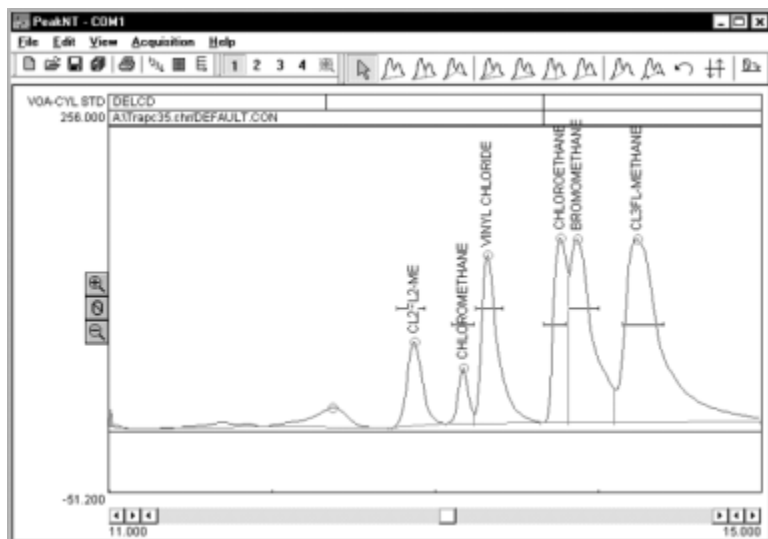


Sample: VOA-CYL standard
Method: 5030 P&T injection
Column: 60m MXT-1

DELCD gain: MED
DELCD temp: 260

Epa&t1c.evt		
EVENT TIME	EVENT	EVENT FUNCTION
0.100	E "ON"	Purge "ON"
5.100	E "OFF"	Purge "OFF"
6.000	C "ON"	Trap 2 (heat) "ON"
6.100	F "ON"	Trap 1 (heat) "ON"
8.000	G "ON"	Valve in "INJECT"
12.000	E "ON"	Purge "ON"
13.000	G "OFF"	Valve in "LOAD"
13.100	B "ON"	Trap set "ON" (+50°C)
14.900	F "OFF"	Trap 1 "OFF"
15.050	E "OFF"	Purge "OFF"
15.100	C "OFF"	Trap 2 "OFF"
15.200	B "OFF"	Trap set "OFF" (+0)

Temperature program:
Initial Hold Ramp Final
35°C 25.00 0.00 35°C



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

INJECTORS

Purge & Trap

General Operating Procedures continued

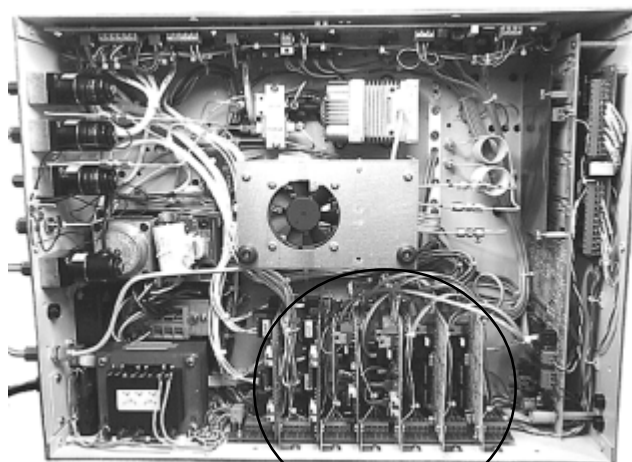
Adjusting Trap Adsorption Temperatures

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

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

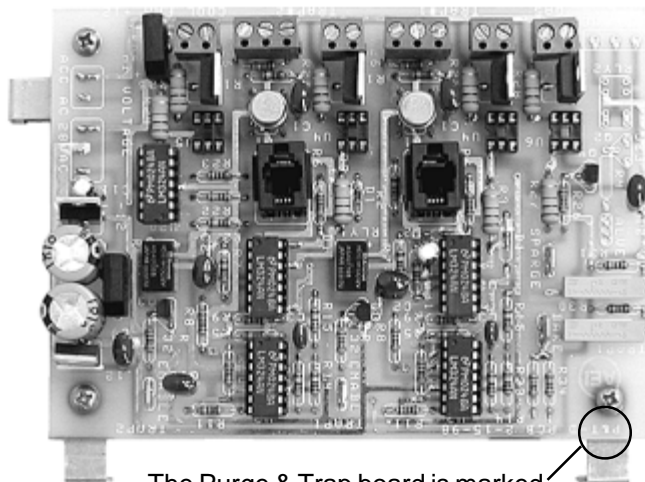
2. Locate the Purge & Trap board; it is one of a group of similar-looking boards installed along the back and top walls of the GC interior. The Purge & Trap board has two trimpots right next to each other, and it is marked with an upside-down “P&T” on the lower outer corner.

GC Chassis Interior



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

The Purge & Trap Board



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

—Trap 2 trimpot setpoint
—Trap 1 trimpot setpoint

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

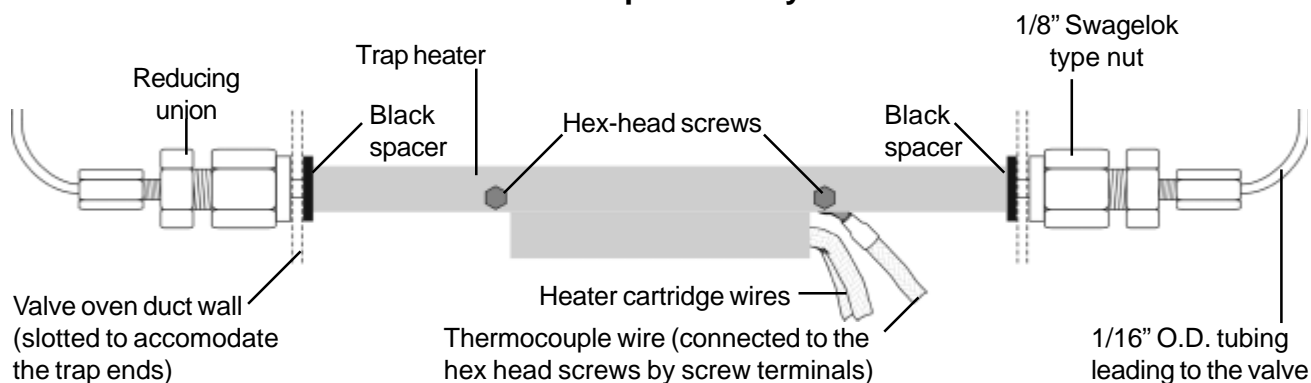
4. When you are finished adjusting both trap adsorption temperatures, place the bottom panel on the GC chassis. Support the panel while you gently rock the GC onto its base. Secure the base with its 6 screws.

Switching / Replacing Traps

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

1. With the red protective GC cover raised, remove the Purge & Trap cover plate by loosening the four brass thumbscrews at its corners.
2. Carefully remove the two squares of white insulation from each valve oven duct to expose the fittings that secure the traps ends to the 1/16" O.D. tubing leading to the 10 port valve.
3. Gently slide the trap assembly out of the slots in the valve oven ducts (there is enough slack in the heater and thermocouple wires to pull either trap about 1 inch outside the duct).
4. Use two wrenches to loosen the 1/8" Swagelok type nuts that secure the traps ends to stainless steel 1/8"-1/16" reducing unions.
5. The trap heater is a clamshell design, consisting of two halves. To remove the heater from the trap, loosen but do not remove the two securing hex head screws. The two halves of the clamshell heater will open enough to let the trap drop out.
6. Attach the replacement trap to the reducing unions with the trap's two 1/8" nuts. Use stainless steel nuts and brass ferrules when replacing traps. **DO NOT** use graphite ferrules, as graphite has some adsorption properties and may interfere with your analysis.
7. Slip the trap into the clamshell heater and tighten the two hex head screws.
8. Gently push the trap ends back into the slots in the two interior duct walls, making sure that the black spacers are between the duct walls and the trap heater. **TO AVOID DAMAGE, ARRANGE THE TRAPS SO THAT ONE TRAP'S HEATER WIRES DO NOT LAY ACROSS THE OTHER TRAP'S HEATER.**
9. Repeat the process with the other trap if necessary. Replace the white duct insulation squares, then replace and secure the Purge & Trap cover plate.

SRI Trap Assembly



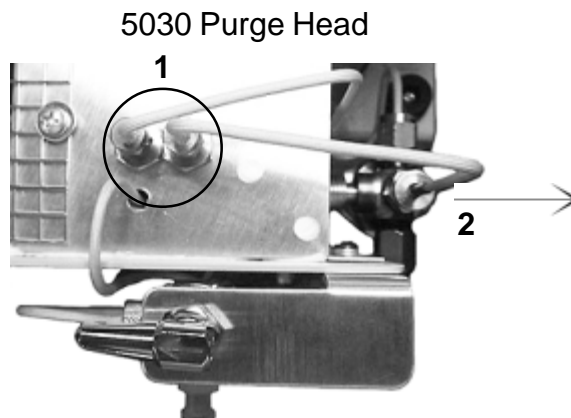
INJECTORS

Purge & Trap

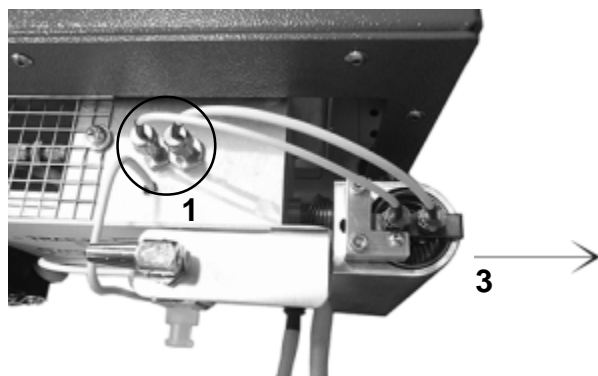
Method 5030/5035 Purge & Trap: Changing the Purge Heads

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

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



5035 Purge Head

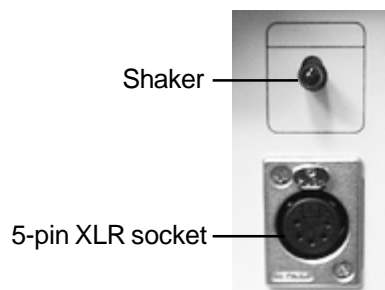
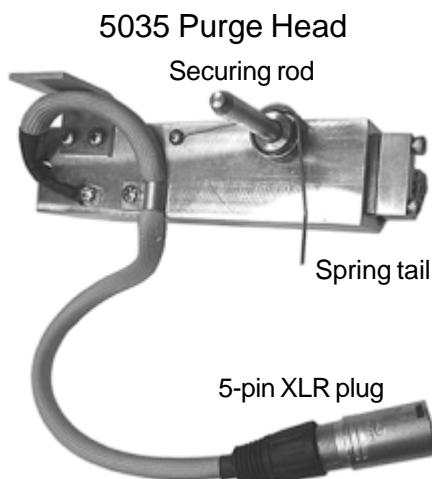


3. If you are removing the 5035 purge head, squeeze the protruding tail of the spring toward the heater body with your thumb as you pull the purge head out toward the front of the GC. Unplug the cord from the socket on the GC.



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

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



Expected Performance

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

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

FID Results:

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

PID Results:

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

DELCD Results:

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

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

FID Results:

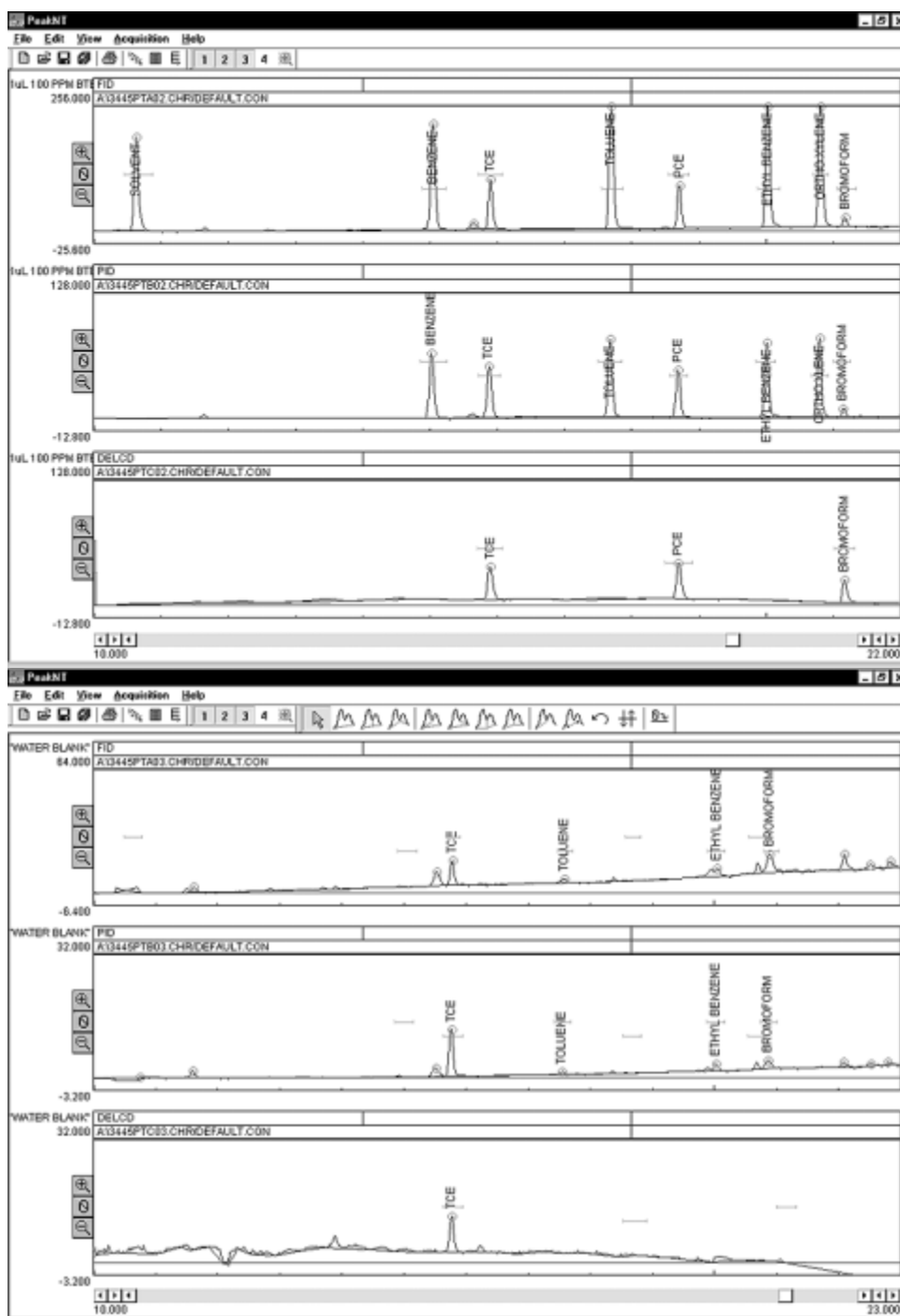
Component	Retention	Area
TCE	15.766	58.9100
Toluene	17.566	17.4000
Ethyl Benzene	20.033	51.9080
Ortho Xylene	20.833	91.5290
Total		219.7470

PID Results:

Component	Retention	Area
TCE	15.750	58.1920
Toluene	17.533	4.3400
Ortho Xylene	20.850	20.8720
Total		609.1300

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340



INJECTORS

Purge & Trap

Troubleshooting and Maintenance

Carryover

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

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

$$\frac{4}{353} = \frac{x}{10\text{ppb}}$$
$$353x = 40\text{ppb}$$
$$x = 0.1133\text{ppb}$$

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

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

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

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

PeakSimple Pull-Down Menus - part 4

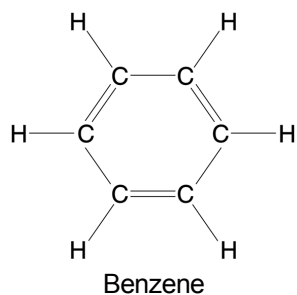
Operation of PeakSimple chromatography software

SRI Instruments

chromatography acquisition software, peaksimple, peak simple, calibration, manual, operation

DETECTORS

Photo Ionization Detector - PID



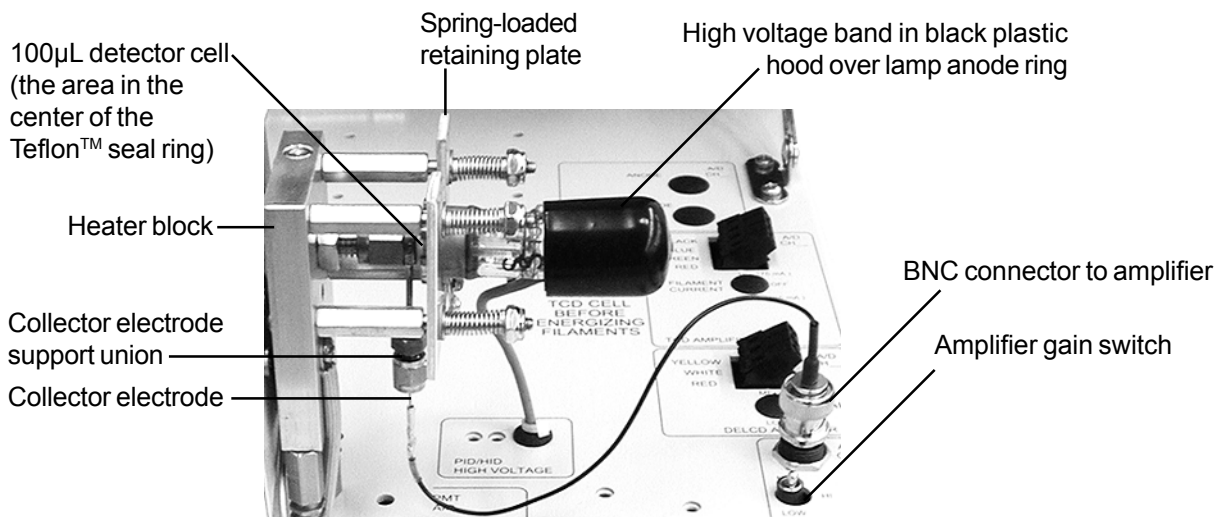
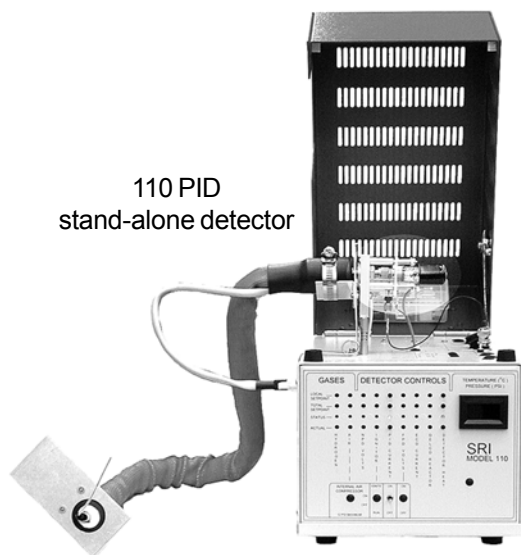
Overview

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 (100 μ L), 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 an SRI 8610C chassis

110 PID stand-alone detector

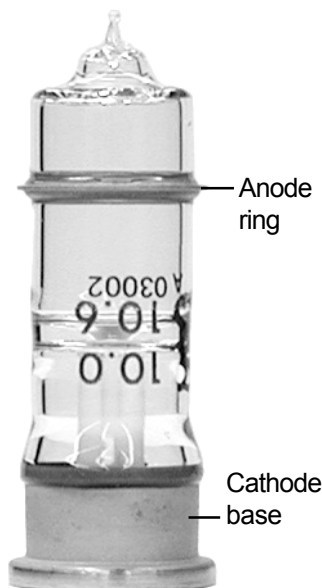


DETECTORS

Photo Ionization Detector - PID

Theory of Operation

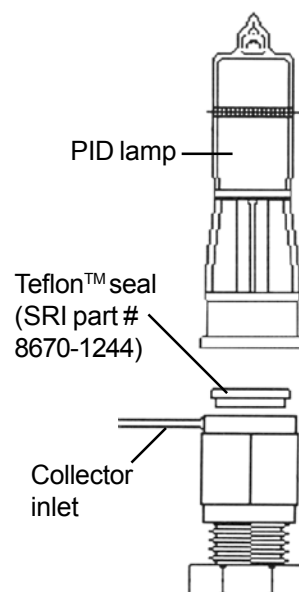
10.6eV PID Lamp (SRI Part # 8670-1242)



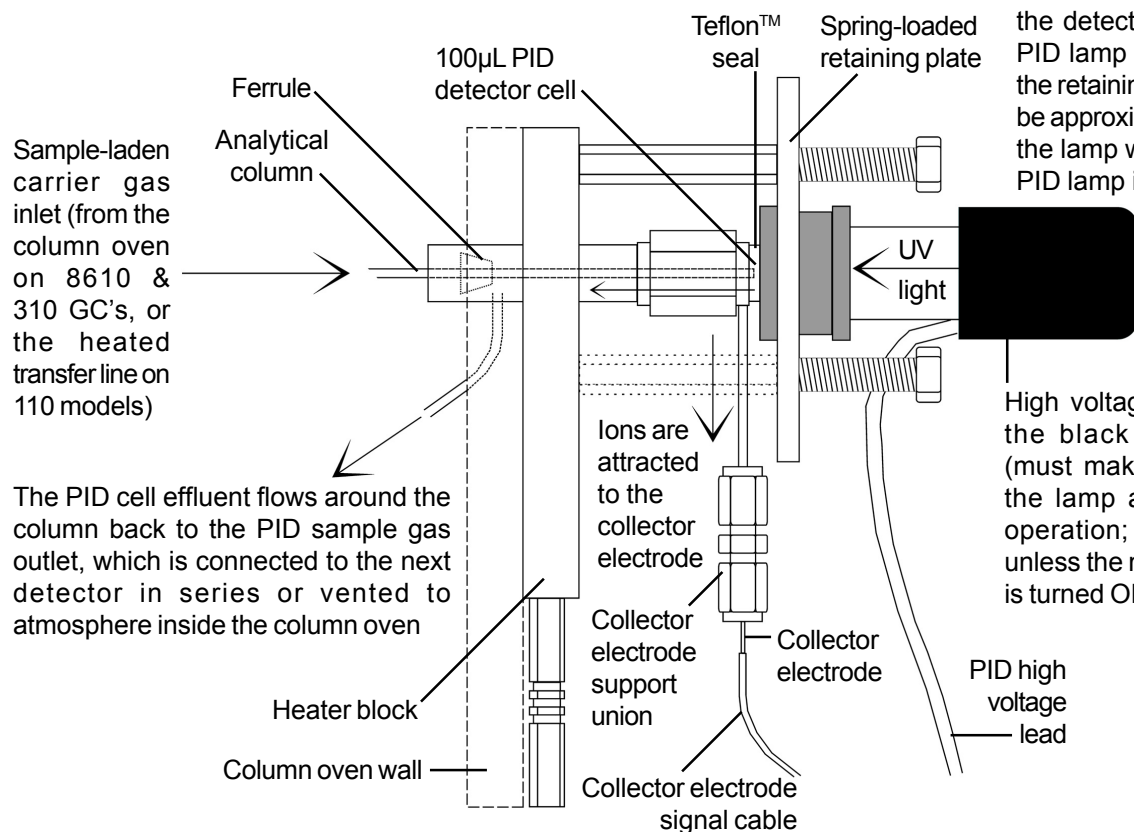
The SRI PID design uses a 10.6eV lamp with a high voltage power supply. Sample laden carrier gas flows from the analytical column into the PID sample inlet, where it is streamed through a 100 μ L flow-through cell. When sample molecules flow into the cell, they are bombarded by the UV light beam. Molecules 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 SRI PID detectors.

Partial PID Assembly - Exploded View



Simplified PID Operational Diagram



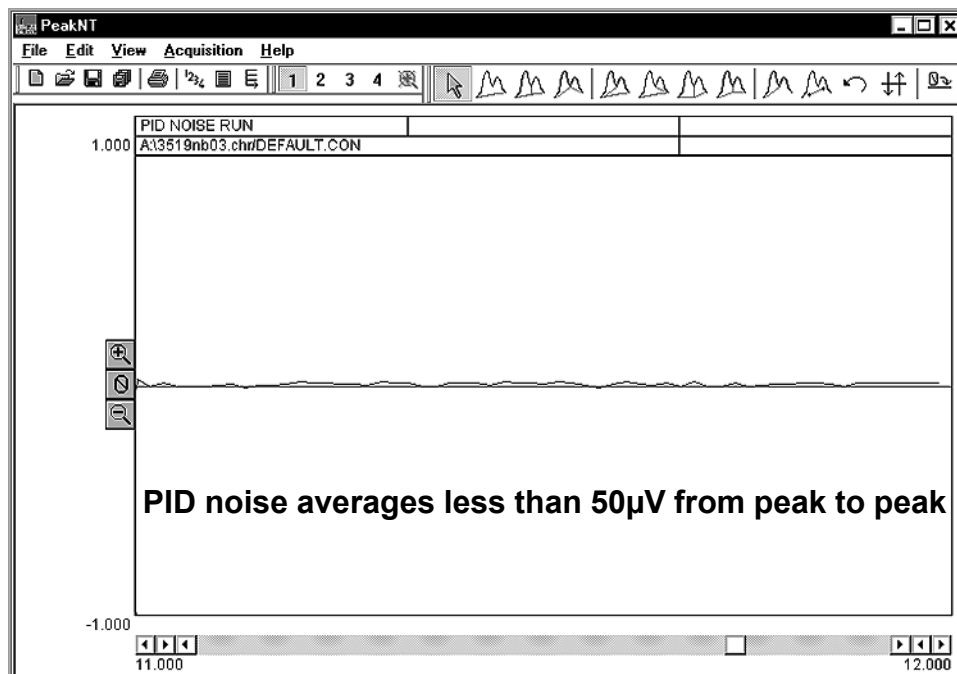
NOTE: The end of the column must be visible in the detector cell when the PID lamp is removed from the retaining plate. It should be approximately 1mm from the lamp window when the PID lamp is in place.

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)

DETECTORS

Photo Ionization Detector - PID

Expected Performance



PID Noise Run

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

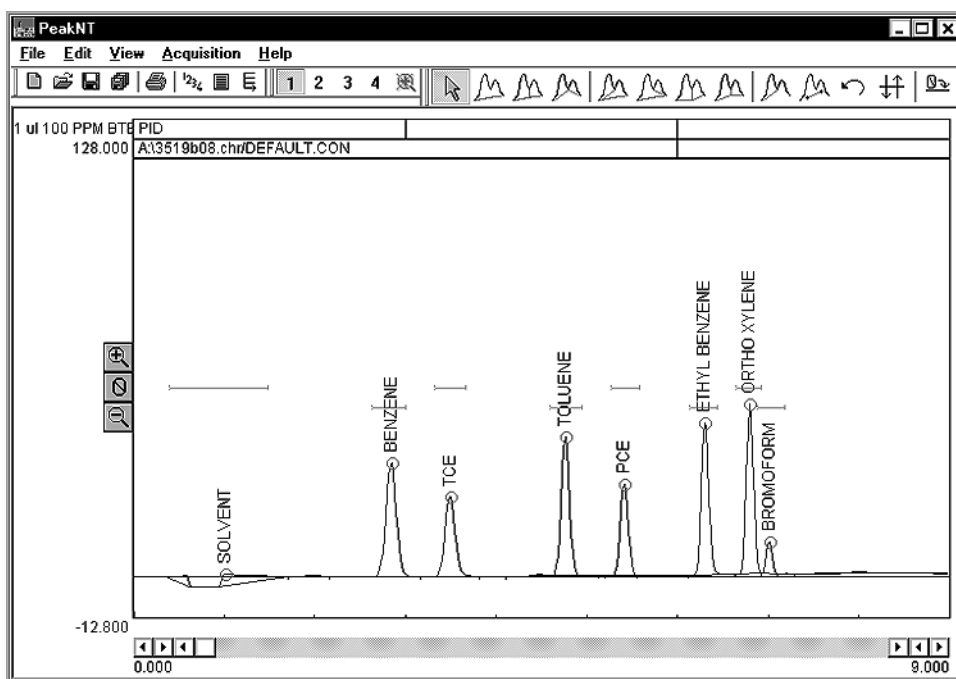
PID BTEX Analysis (in series with FID and DELCD)

Sample: 1 μ L 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



DETECTORS

Photo Ionization Detector - PID

General Operating Procedure

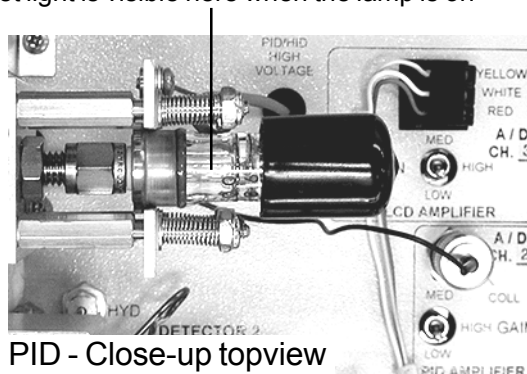
The capillary column enters the PID cell from inside the column oven through the bulkhead fitting in the insulated oven wall. The column may be installed with the lamp in place. Insert the capillary column into the PID detector inlet until the column stops at the lamp window inside the PID cell, then 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.

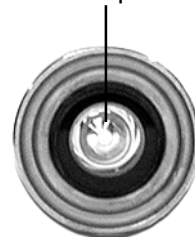
NOTE: Lamps are a consumable part of the PID detector. It is recommended to have a spare lamp available if critical analyses are being performed at remote field sites. Spare and replacement 10.6eV PID lamps are available under SRI part number 8670-1242. Teflon seals are available under SRI part number 8670-1244.

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.

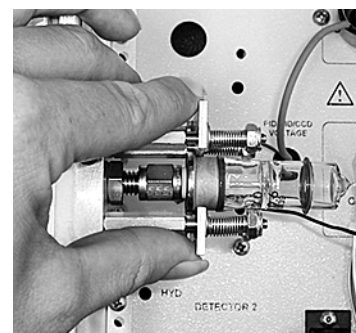
The PID lamp window



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.



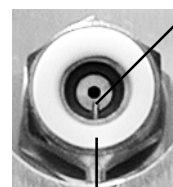
4. Clean the lamp window using a mild abrasive cleanser like Bon Ami or Comet. Wet your finger, and make a paste with a small amount of cleanser. Scrub the lamp window clean in a circular motion with your finger.

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.

The collector electrode protrudes into the cell

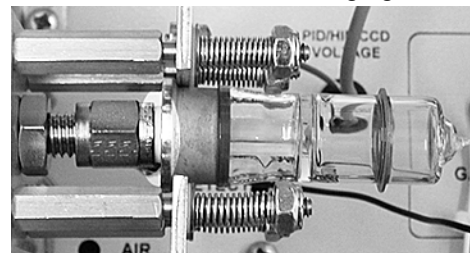


Teflon™ seal

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.

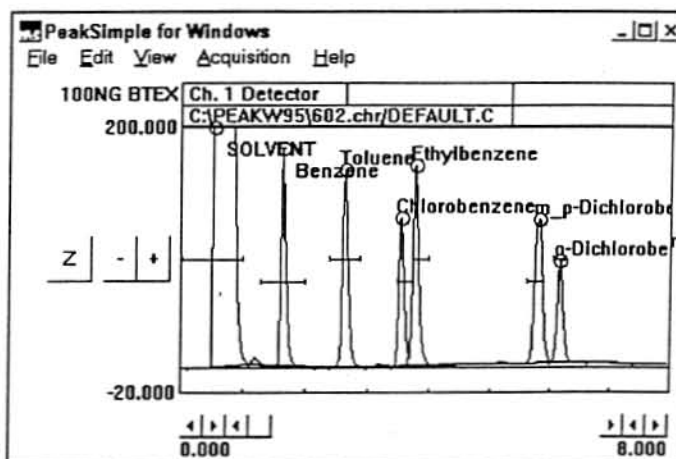
Make sure the lamp window is centered over the Teflon seal and snug against it



Getting Started

In this section, we will cover the basic information needed to set up proper communication with your G.C. or Data System hardware.

The Windows version of PeakSimple requires the use of the serial port interface that is built into most 8610-C and Model 310 gas chromatographs. This data acquisition and interface unit permits you to acquire up to four separate channels of data simultaneously without the need for additional hardware or acquisition boards.



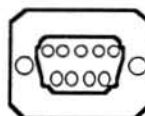
The earlier IBM PC-compatible ISA expansion bus data acquisition cards (AD100 and AD110) used by PeakSimple II and PeakSimple III data systems are *not supported* by PeakSimple for Windows. However, all chromatograms acquired using DOS-based PeakSimple II and PeakSimple III continue to be compatible with this Windows version and may be imported as native files.

Identifying Your COM Port

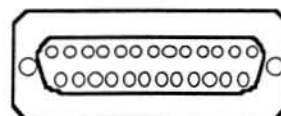
Before attempting to establish communication between your G.C. and the serial data system interface, be sure to check that all the necessary electrical connections have been made, including the connection of any optional remote start cables.

Select an unused serial port on your PC and identify the COM port number assigned to it. It is important that this port NOT SHARE AN INTERRUPT with any other device used in your computer. Typical PCs are equipped with two COM (or serial) ports. COM 1 is typically used by the mouse or some other pointing device. COM 2 may be open (unused) or shared with another device, such as a fax modem, scanner or other peripheral. Determine which COM port you will use and remove any other device that may be in contention with that specific COM port number. Refer to your PC's hardware manual for instructions on changing COM port addresses and device drivers.

Most COM ports are provided with DB-9 connectors (nine pins configured in two rows - 5 pins over 4 pins - within a D-shaped plug or chassis connector). If your PC has a DB-25 serial port (25-pin connector), you will require a DB-25 to DB-9 adapter.



DB-9
Serial Port
Connector



DB-25
Serial Port
Connector

Establishing Communication

The cable provided with your G.C. or data system has a male DB-9 plug on one end and a female DB-9 connector on the other end. Plug one end of this cable into your available computer COM port and plug the other end into the G.C. or Data System DB-9 connector and tighten the retaining screws.

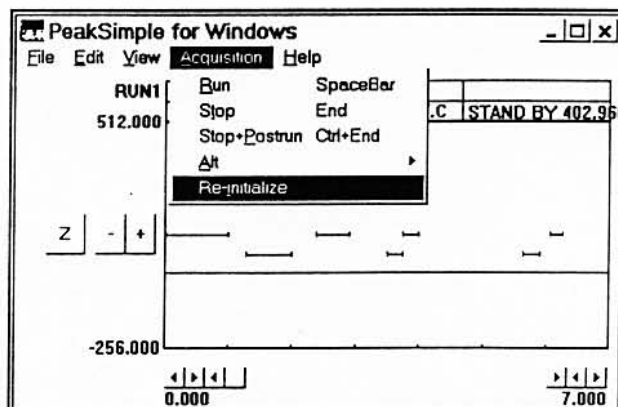
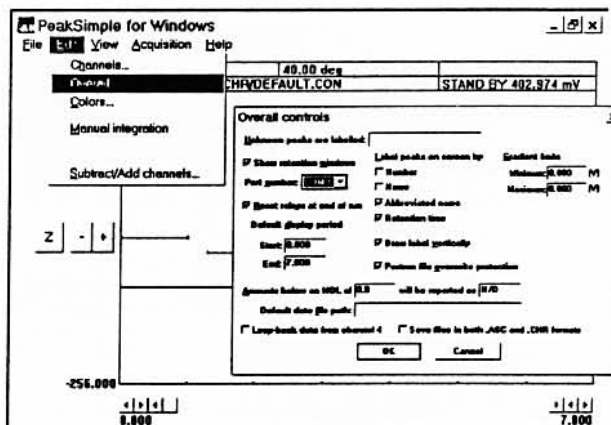
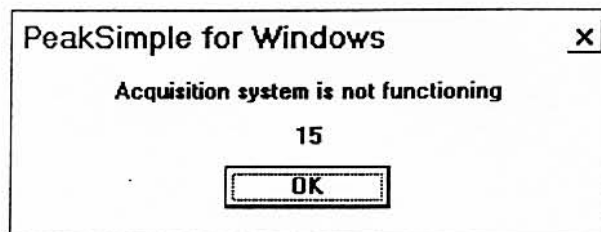
Plug your G.C. or Data System into an approved GFCI protected outlet and turn the power switch to the 'ON' position. Start PeakSimple by double-clicking on the PeakSimple icon (or by clicking 'Start', 'Programs', 'PeakSimple' in Windows 95). When PeakSimple loads, it will automatically attempt communication with your G.C. or Data System using *COM 1 as the default COM port*.

If the serial port interface does not respond, you will see the following error messages appear on the screen: "**Can't wake- check power and cable**" followed by the message "**Acquisition system is not functioning**".

These messages indicate that your computer cannot communicate with your G.C. or Data System through the default COM port, COM 1. You will need to set up the correct COM port in PeakSimple. To do this, click on the **EDIT** pull-down menu and select **OVERALL**. Change the **PORT NUMBER** to the COM port into which you chose to plug your interface cable. Click **O.K.**.

If at anytime you wish to force PeakSimple to reinitialize communication, click on the **ACQUISITION** pull-down menu and select **RE-INITIALIZE**. If the COM port information is correct and communication errors still appear when the computer attempts to activate the serial port interface, check the serial port connections at both ends of the interface cable for loose connections. Also, visually check the serial cable for nicks or cuts.

It is important to understand that in order for PeakSimple to communicate with your G.C. or Data System, at least ONE channel must be **ACTIVE**. To determine which channels are active, click on the **EDIT** pull-down menu and select **CHANNELS**. A channel is active if the box next to **ACTIVE** is marked with a checkmark. The **EDIT-CHANNELS** menu is described in greater detail in the **EDIT** section in this manual.



PEAKSIMPLE SOFTWARE

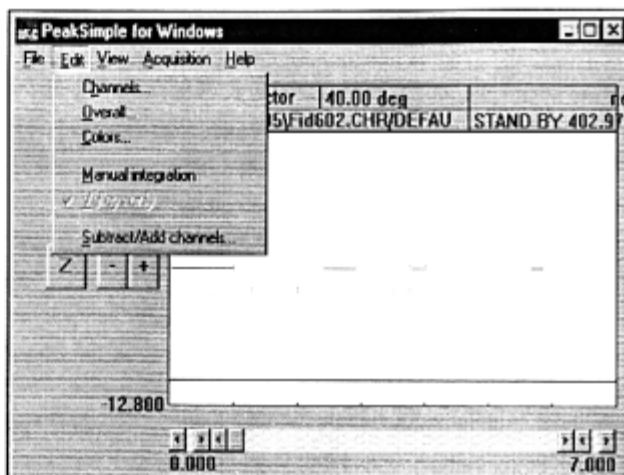
File types used by PeakSimple

- .CHR = chromatogram or data file. The chromatogram is the graphic depiction of raw analytical data in binary format.
- .ASC = ASCII file. You can save your raw data in binary format (.CHR), ASCII format (.ASC), or both.
- .THU = thumbnail file. PeakSimple keeps thumbnail snapshots of your .CHR files to facilitate browsing. In PeakSimple version 3.21 and newer, you can elect to not save thumbnails to save disk space.
- .3D = three dimensional file. Load multiple chromatograms (.CHR files) for viewing in the 3D display window. Save the 3D display in a .3D file for later viewing.
- .TEM = temperature file. TEM files contain the column oven temperature program information: initial and final temperatures, hold times and ramping rates. .TEM, .GRA, and .FLO files perform the same function for different applications; the only real difference between them is their extension. The user selects which application and file extension to use in the Channel details window: on the right side under "Control by," are three radio buttons with the choices Temperature, Pressure, or Gradient.
- .GRA = gradient file for HPLC. Gradient files contain the solvent and sample mixing percentages for one or two pumps. PeakSimple uses this extension when the "Gradient" radio button under "Control by" in the Channel details window is selected.
- .FLO = pressure flow file. PeakSimple uses this extension when you select the "Pressure" button under "Control by" in the Channel details window. This is useful when varying the carrier gas pressure, making use of the Electronic Pressure Controllers (EPCs) in your SRI GC. The user must move a wire to switch from temperature to pressure control.
- .EVT = event files. Events are controlled by turning relays ON and OFF. Event tables allow you to automatically turn relays ON/OFF at specified times during an analysis. Integration events can also be automatically performed using an event table.
- .CPT = components file. Each channel has it's own Components table that displays the list of expected components, their retention times, and calibration files. All component information is input by the user.
- .CAL = component calibration file. The calibration curve is calculated from user-generated results obtained at several different concentrations that span the expected range to be encountered in actual samples. Calibration is required for each component you expect to be present in your sample, and for each detector to be used in the analysis. When a component calibration file is saved, it will appear in the Components window, next to the appropriate component.
- .LOG = results log file. Use the Post-run actions window to have PeakSimple add the results to the results log after the analysis.
- .RES = results file. The results file displays analytical results in ASCII format. The results window is accessed through the View menu. The results log (.LOG) may be viewed by clicking a button in the results window.
- .CON = Control files contain user-defined analytical parameters such as column oven temperature program (.TEM files), components (.CPT files), and relay event tables (.EVT files), as well as print information and many other parameters.
- .QUE = autosampler queue files. The autosampler queue lets you load multiple control files for use with an autosampler. In batch reprocessing mode, the autosampler queue is used to re-run data under the parameters of a particular control file.

Using PeakSimple: Menus

Operation of Menu Bar Pull-Down Menus

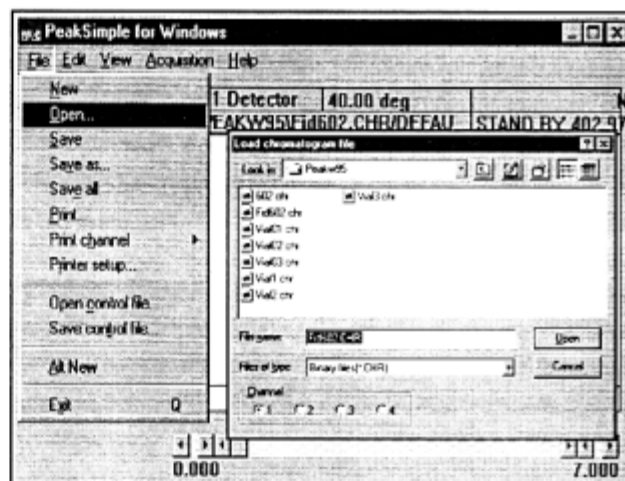
All PeakSimple for Windows features may be accessed from pull-down menus. When you click on a menu bar item, a pull-down group menu will open to permit navigation to specific group features. These pull-down menus may also be opened by pressing the <ALT> key and the letter key corresponding to the underlined letter in the menu bar item name. For example, to open the **EDIT** menu press <ALT> and the letter "E" (This is not case sensitive).



The FILE Pull-Down Menu

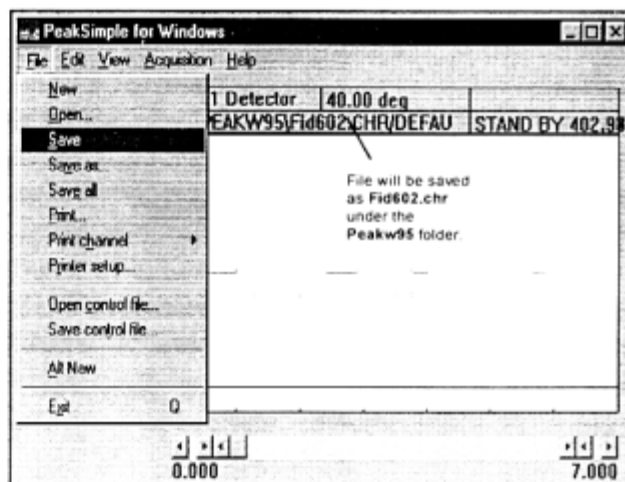
The **FILE-NEW** feature will clear the display of all active channels in the **Main** timebase without starting a new chromatographic run.

To open a previously saved chromatogram file, select **FILE-OPEN**. A **LOAD CHROMATOGRAM FILE** screen will appear which will allow you to select any file from any directory (folder) on your system. Choose the channel (1-4) in which you wish to display your saved chromatogram and then click **OPEN**.



FILE SAVE

The **FILE-SAVE** feature saves the displayed chromatograms of all active channels. The name given to the file(s) is the same name that is displayed in the Data Boxes below the menu bar and will be given the default .**CHR** extension. This file name is editable by the user by changing information in the **EDIT-CHANNELS-POSTRUN** pull-down menu. See the **EDIT** section for more information.



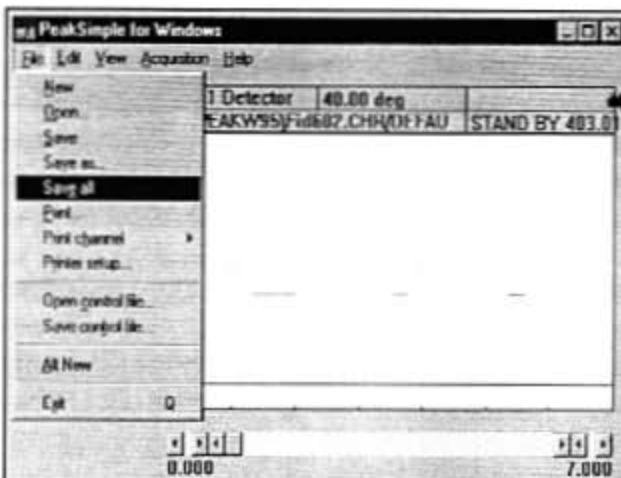
The FILE-SAVE-AS Pull-Down Menu

To save a newly created chromatogram file, select **FILE-SAVE AS**. A **SAVE CHROMATOGRAM FILE** screen will appear which will allow you to save the file in any directory (folder) on your system. Type a name up to eight characters into the **File Name** box and choose which channel (1-4) you wish to save and then click **SAVE**. The file will be saved as a **binary file** by default, with a **.CHR** extension. You may also select to save the file in **ASCII** format with a **.ASC** extension.



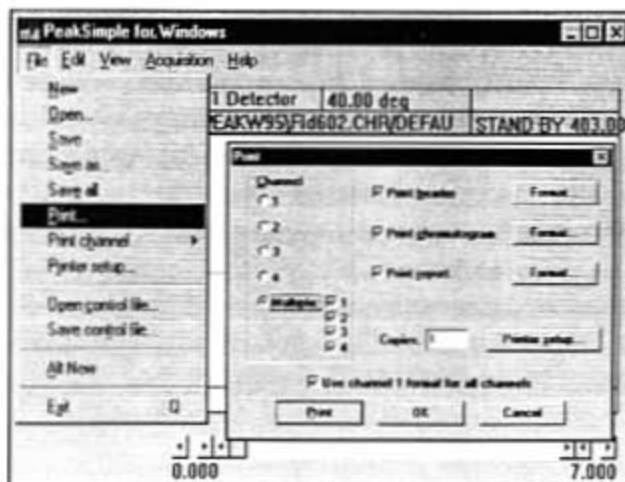
The FILE-SAVE-ALL Pull-Down Menu

The **FILE-SAVE-ALL** feature will automatically save your chromatogram as a **.CHR** file; your temperature program as a **.TEM** file; your component table as a **.CPT** file; your event table as a **.EVT** file and then saves them all under a control file (**.CON** file). **DEFAULT.CON** will be used if no other name for the **control file** is specified using the **SAVE-CONTROL FILE** feature. All print information is also saved when you save a **control file**.



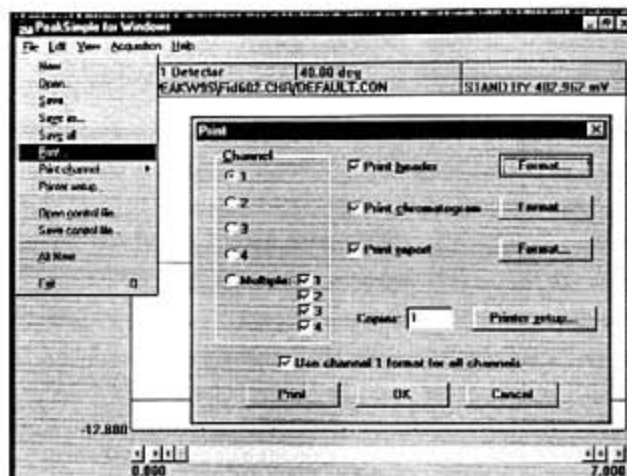
The FILE-PRINT Pull-Down Menu

Numerous fields are available for print information. When you access the **FILE-PRINT** pull-down menu you will notice that any combination of one to four channels can be printed out on a single sheet of paper simply by marking the circle next to the channel number. Print information concerning the **header**, **chromatogram** and **report** can be easily edited.



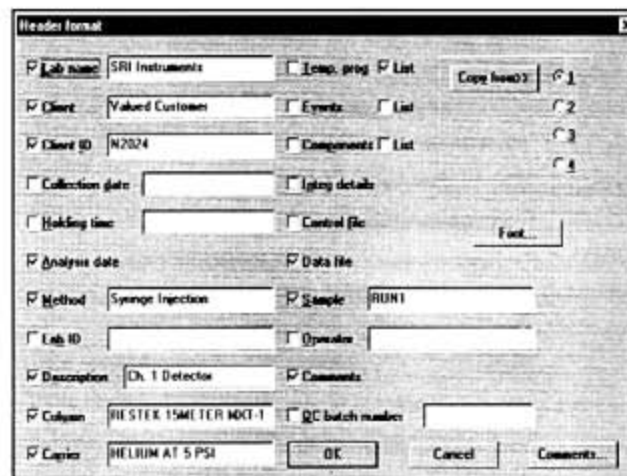
The FILE-PRINT Pull-Down Menu (CHANNEL 1)

When you access the **FILE-PRINT** pull-down menu you will notice that you can select to print any combination of **multiple** channels by clicking on the circle next to the word **multiple**. You may also choose to print individual channels by clicking on the circle next to the desired channel. **Click on Channel 1** to edit the **Channel 1** information in the **Print Header**, **Print Chromatogram** and **Print Report Format** fields. Rather than enter unique information for all four channels, you may wish to check the **Use channel 1 format for all channels** box.

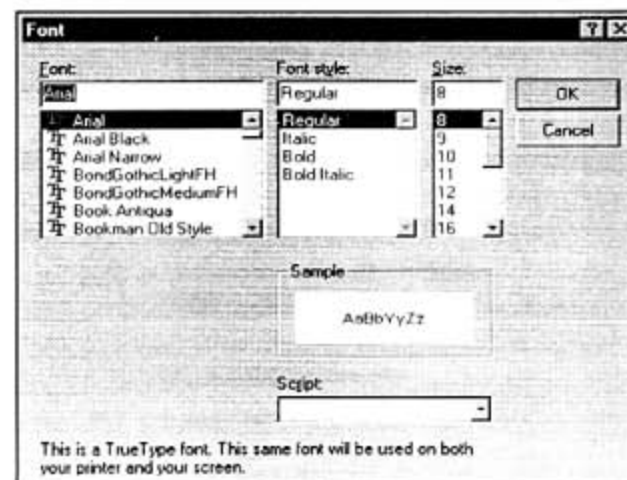


PRINT HEADER FORMAT

Clicking on the **Print Header FORMAT** button will allow you to customize the appearance of your printed chromatogram header. Input your **Laboratory name**, **Analysis method**, **Sample type**, **Column**, etc and check the box next to each field. **Analysis date** prints the date in your PC's BIOS.



Print out **Temperature Programs**, **Events** and **Components** file names by checking their boxes; or click on **List** to print the complete Temperature Program, Event Table or Component List. **Copy from:** selects which channel will provide the **List** information. Check the **Comments** box and click on **Comments...** to enter customized information about your analysis. You can change the **Font**, style and size of your printed text by clicking on the **Font** box. Select a size that will provide readable text while still leaving room for your chromatogram and report.

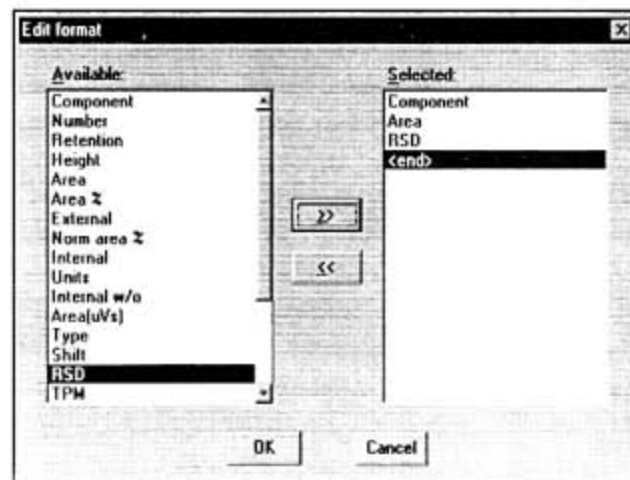
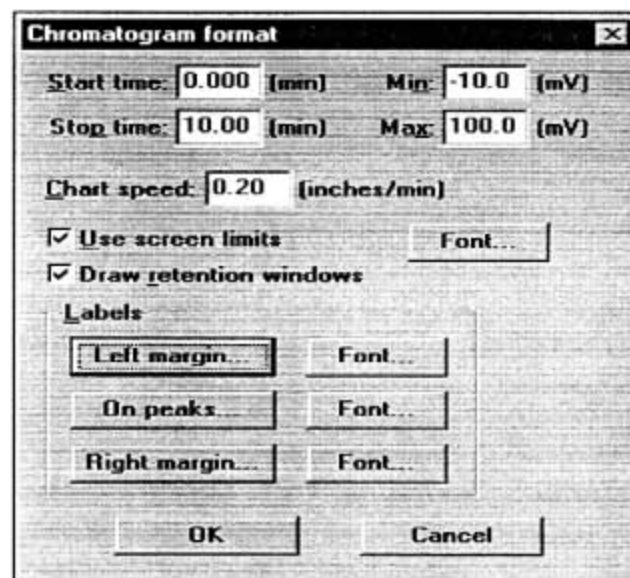
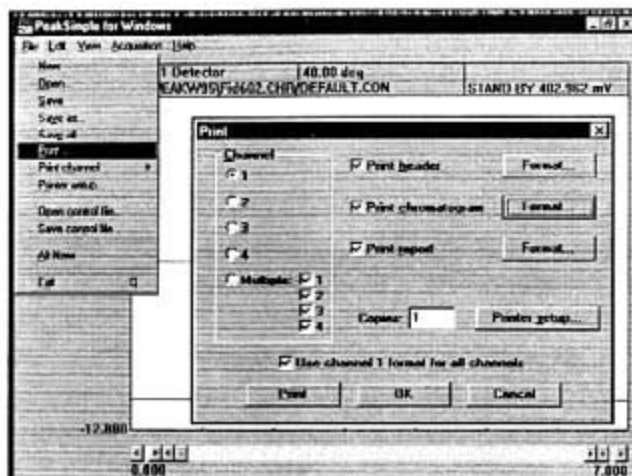


PRINT CHROMATOGRAM FORMAT

You can also edit the chromatogram print parameters when you access the **FILE-PRINT** pull-down menu. Check the **Print Chromatogram** box and select **Format**. The **Chromatogram format** screen allows editing of the chromatogram **Start time** and **Stop time** and the **Min** and **Max** millivolt levels.

The **Chart speed** setting will determine the size of the chromatogram section of your printout. A setting of **1.0 inches/minute** for a 5 minute chromatogram will produce a **5 inch** chromatogram print. You may need to experiment with this setting to fit your **header, chromatogram and report** information all on one printed page. When the **Use screen limits** box is checked only the displayed section of a chromatogram will be printed. The **Draw retention windows** box allows for retention windows to be printed as well.

The **Labels** section of the screen lets you select what useful information will be printed along the borders of the chromatogram, and above the peaks. Clicking on **Left margin**, for example, will bring up the **Edit format** screen which will allow you to select from a list of measurements which will automatically be calculated and printed in the left margin of your chromatogram. To choose **RSD**, for example, click on **RSD** from the left column and then click on the right arrows (**>>**). **RSD** will now appear in the **selected** column on the right. Click **OK** to close the window. Edit **On peaks** and **Right margin** in the same manner.



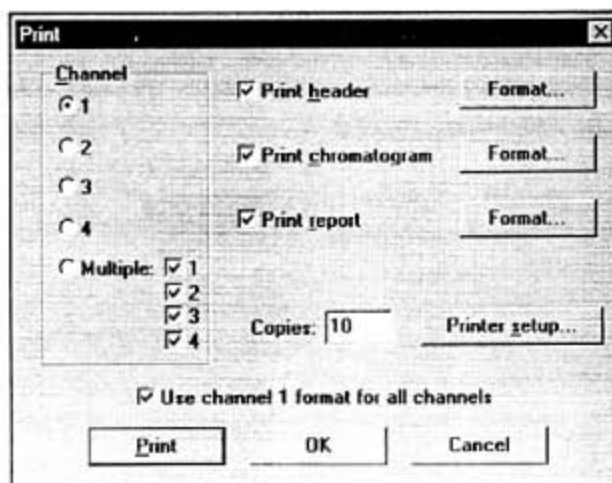
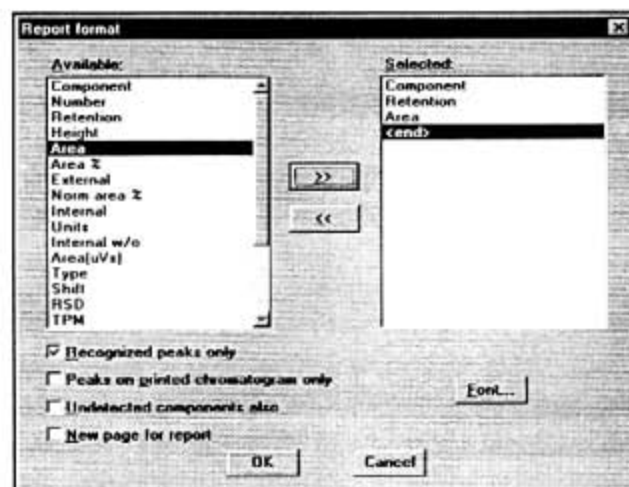
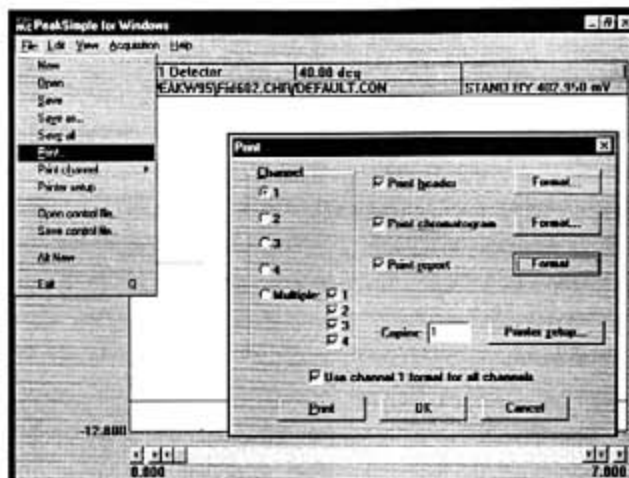
PRINT REPORT FORMAT

A report may be printed along with your chromatogram to summarize component retention time, area counts or other data. Clicking on the **View** pull-down menu and selecting **Results** will show a preview of your report.

Click on the **Print Report** box and select **Format**. The **Report Format** screen will appear which will allow you to select from a list of measurements which will automatically be calculated and printed on the bottom of your chromatogram. To choose **AREA**, for example, click on **AREA** from the left column and then click on the right arrows (>>). **AREA** will now appear in the **Selected** column on the right.

Clicking on the box next to **Recognized peaks only** will place a check mark in the box and only those peaks which integrate properly within named retention windows will be printed in the report. Checking the **Peaks on printed chromatogram only** box will allow the report to show only those peaks defined by the **Chromatogram format- Start time and Stop time**. This feature allows you to set up your report to ignore all peaks that appear outside your window of interest.

Checking the **Undetected components also** box will report information about all named peaks even if no peak is present within the retention window. Checking **New page for report** will print all report information on a separate page. Click **OK** to close the **Report format** window. You may print out as many Chromatogram **Copies** as you need by entering a number in the **Copies** box and selecting **Print**.

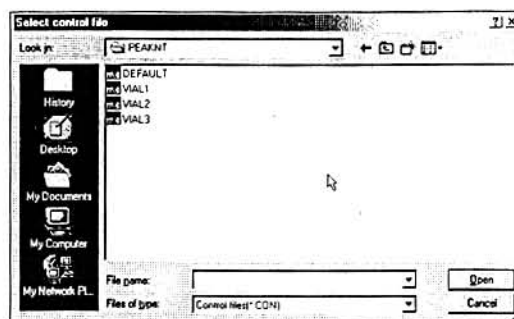


The View-Autosampler Window (cont.)

Add



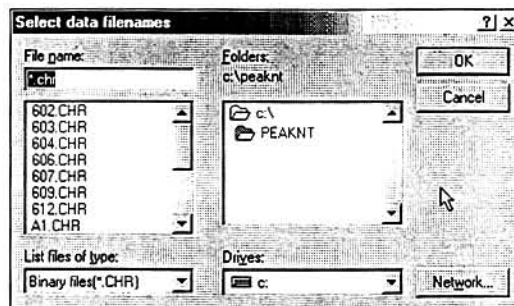
Select the Add button to add a control file to the queue. Selecting the button opens up the Select control file window where the file can be loaded into the list box. Each control file in the queue must have a different name even though almost identical actions are performed.



Add Multiple/Batch Reprocessing



The Add multiple button allows the user to load multiple data files into the list box. Click on the button to open up the Select control file window and then click on a control file name to open up the Select data filenames window. Select as many data files as needed by pressing the shift button and clicking with the mouse cursor and then click on OK to load them into the queue. The Add multiple button is only useful for use with the Batch reprocessing mode.



Delete

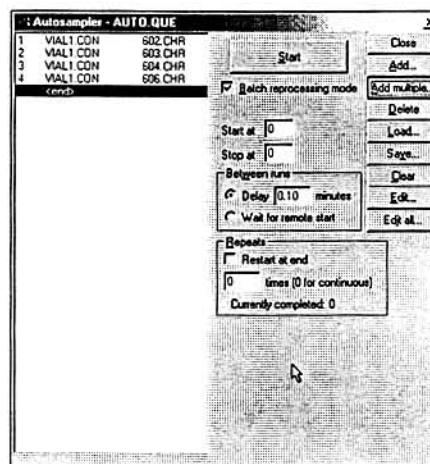


After highlighting a control file in the list box to the left select the Delete button to remove that control file from the queue.

Load



Select the Load button to open up a previously saved queue file. Clicking on the Load button opens up the Load autosampler queue window where the queue file can be selected and loaded.



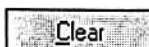
Save



Selecting the Save button opens up the Save autosampler queue window. Save the queue in the file box by naming the file and selecting save. It is recommended that all files be saved to the Peak-Simple directory.



Clear



The Clear button erases the entire queue.

The View-Autosampler Window (cont.)

Edit



After highlighting a control file select the Edit button to modify that control file. Selecting the Edit button loads the control file on the PeakSimple main screen. To make any changes click on the main screen, do all modifications, and then select Save all from the PeakSimple file menu.

Edit All



To edit all the control files in the queue at once click on the Edit all button to open up the Autosampler queue spreadsheet. Many of the commonly adjusted control file parameters are displayed in the spreadsheet enabling the user to input changes to the queue. Not all control file parameters can be modified using Edit all (only the parameters that are selected in Format) and so must be done individually with the Edit function.

Autosampler Queue Window

Close



The Close button exits the window after prompting the user to save the spreadsheet.

Cancel



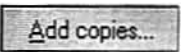
The Cancel button exits the spreadsheet window without prompting the user to save.

Add

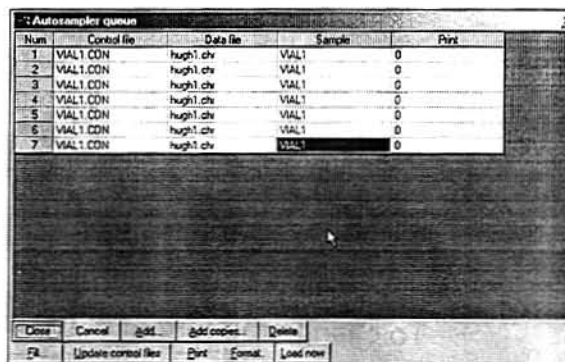


Selecting the Add button opens up the Select control file window where an existing control file can be added to the queue.

Add Copies

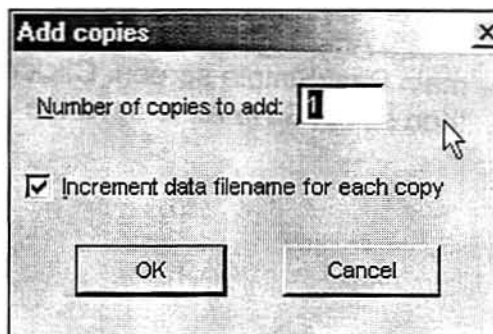


After highlighting a control file in the spreadsheet select the Add copies button to add copies of the file to the list. Once the Add copies window pops up input the number of copies to be made in the dialogue box and specify whether the file names should be incremented. The Add copies button is useful for creating a queue from scratch with a single control file.



Num	Control file	Data file	Sample	Port
1	VIAL1.CON	hugh1.chr	VIAL1	0
2	VIAL1.CON	hugh1.chr	VIAL1	0
3	VIAL1.CON	hugh1.chr	VIAL1	0
4	VIAL1.CON	hugh1.chr	VIAL1	0
5	VIAL1.CON	hugh1.chr	VIAL1	0
6	VIAL1.CON	hugh1.chr	VIAL1	0
7	VIAL1.CON	hugh1.chr	VIAL1	0

Buttons at bottom: Data, Cancel, Add, Add copies, Delete, File, Update control file, Print, Format, Load new



Add copies

Number of copies to add:

☒ Increment data filename for each copy

OK Cancel

Autosampler Queue Window (cont.)

Delete

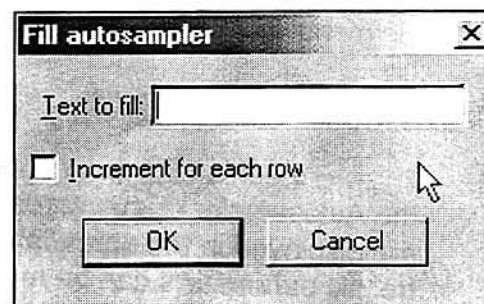


The Delete button deletes a highlighted control file off the list. If no file is highlighted then the last file will be deleted from the queue.

Fill



The Fill button fills a spreadsheet column, row, or cell with selected text. Once the desired cells are highlighted clicking the Fill button opens up the Fill autosampler options box. Input the text to fill in the information field and specify whether the text should be incremented for each row.



The 'Fill autosampler' dialog box contains a 'Text to fill:' text input field. Below it is a checkbox labeled 'Increment for each row'. At the bottom are 'OK' and 'Cancel' buttons.

Update Control Files



Selecting the Update control files button saves all changes to the control files in the list.

Print

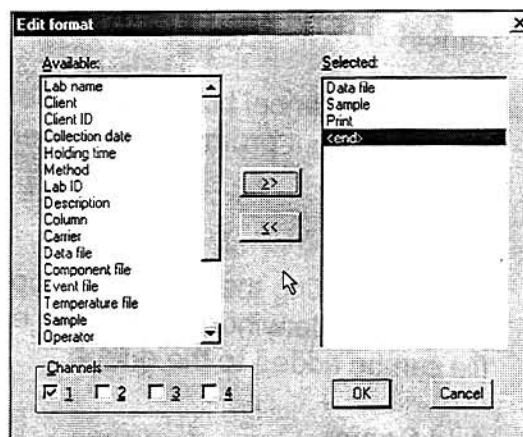


The Print button prints the queue spreadsheet.

Format



To change the format of the queue spreadsheet and open up the Edit format window select the Format button. In the Edit format window a format type can be added by selecting it in the Available window and then hitting the right facing arrow button. To remove a format type from being displayed in the spreadsheet highlight the format type in the Selected box and click on the left facing arrow.



The 'Edit format' dialog box has two list boxes: 'Available' on the left and 'Selected' on the right. The 'Available' list includes: Lab name, Client, Client ID, Collection date, Holding time, Method, Lab ID, Description, Column, Carrier, Data file, Component file, Event file, Temperature file, Sample, and Operator. The 'Selected' list includes: Data file, Sample, Print, and <end>. Between the lists are right-facing (>) and left-facing (<) arrow buttons. At the bottom are 'Channels' (1, 2, 3, 4) with checkboxes, and 'OK' and 'Cancel' buttons.

Load Now



After highlighting a control file select the Load now button to load that control file to the main PeakSimple screen. Click on the screen and make any changes to the control file and then select Save all to save the changes.

The View-Channel "X" Options

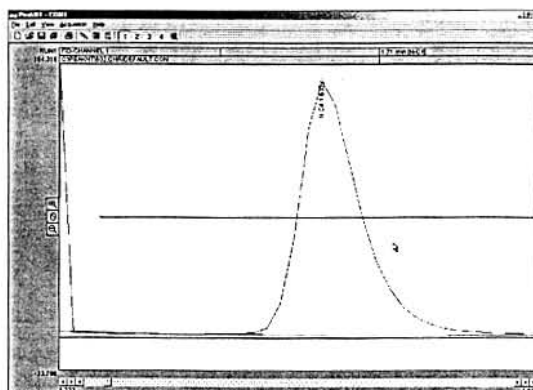
1 2 3 4

To view a specified chromatogram channel open the View menu in the PeakSimple menubar and select a channel to be viewed; either 1, 2, 3, or 4. Keyboard shortcuts can also be used to alternate viewing between chromatogram channels. Hitting F9 displays channel 1, F10 displays channel 2, F11 displays channel 3, and F12 displays channel 4. Furthermore the numerical icons in the PeakSimple toolbar can be used to toggle between chromatogram channels.

Unzoom

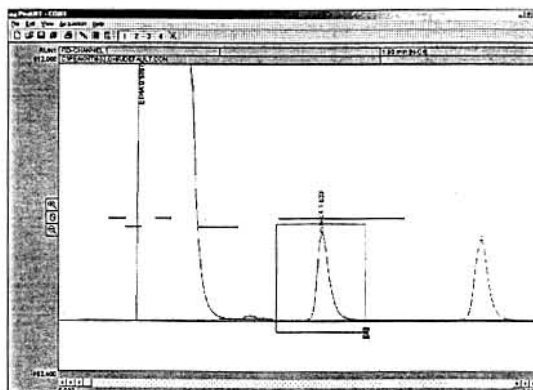


To unzoom from a close up view of a chromatogram select the Unzoom tool from the View menu or hit F6. PeakSimple will zoom out to the first level with the soriginal display units of the chromatogram when the Unzoom tool is used. The Unzoom button in the PeakSimple toolbar can also be used to unzoom a chromatogram or F6 on the keyboard.



Refresh

The Refresh tool in the View menu redraws the chromatogram screen to fix any glitches or resolve an error. Pressing Enter on the keyboard also refreshes the screen.



The Help Menu

About PeakNT

To view program information about PeakSimple click on the About PeakNT option in the Help menu. The PeakNT window will pop up and display the information.

Show Tooltips

The Show tooltips option in the Help menu toggles the PeakSimple tooltips off or on. When Show tooltips is checked a helpful text tip will appear when the mouse cursor is held over a tool or button in PeakSimple. The tooltips provide relevant information to the operation and use of the PeakSimple data system.



The Acquisition Menu

The Acquisition menu contains the commands to run a chromatogram run when hardware is connected to the PeakSimple data system. All Acquisition menu commands have corresponding keyboard hotkeys for convenience.

Run

The Run command begins a chromatogram run on the main trigger group when hardware is connected to the data system. The error message "No active channels in group" appears when no hardware is available to make a chromatograph run. The spacebar can also be used to start a run.



Stop

The Stop command is used to end a chromatogram run once it has been started. Using the Stop command ends the chromatogram run without running any of the Postrun operations. The End button can also stop a chromatogram run.

Stop+Postrun

The Stop+Postrun command ends a chromatogram run and executes the operations specified in the Postrun screen. Holding the Control button and pressing End on the keyboard is the same as the Stop+Postrun command.

Alt

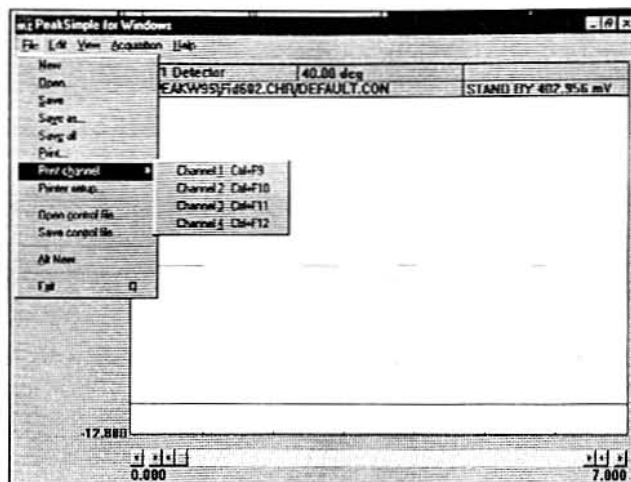
The Alt menu in the Acquisition menu controls the acquisition commands for the alternate trigger group. The + button begins the alternate trigger chromatogram run, the - button stops the alternate trigger run, and the / button on the keyboard stops the run and begins the Postrun operations for the alternate trigger group.

Re-initialize

The Re-initialize command reestablishes the connection between the hardware and the PeakSimple data system. A connection between hardware and the data system has to exist for re-initialization to occur.

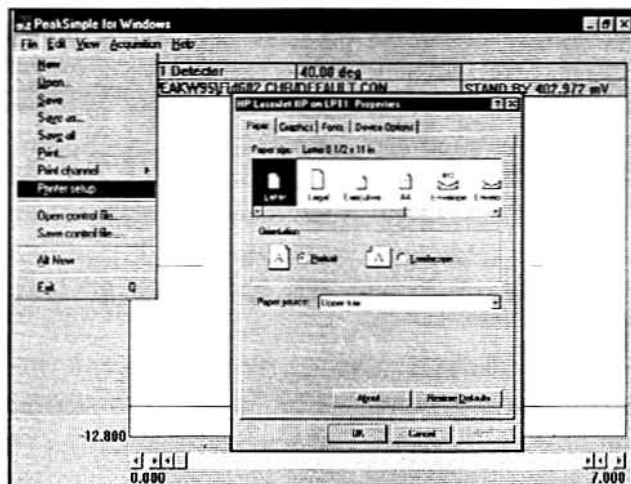
The FILE-PRINT CHANNEL Pull-Down Menu

After all **Print** parameters have been set up, the easiest way to print out a chromatogram is to use the **File-Print Channel** quick keys. Hold down the **Ctrl** (control) key and then press **F9** (function #9) to instantly print the **Channel 1** chromatogram. Press **Ctrl F10** to print **Channel 2**, **Ctrl F11** for **Channel 3** or **Ctrl F12** for **Channel 4**. Of course you may also select these commands from the pull-down menu.



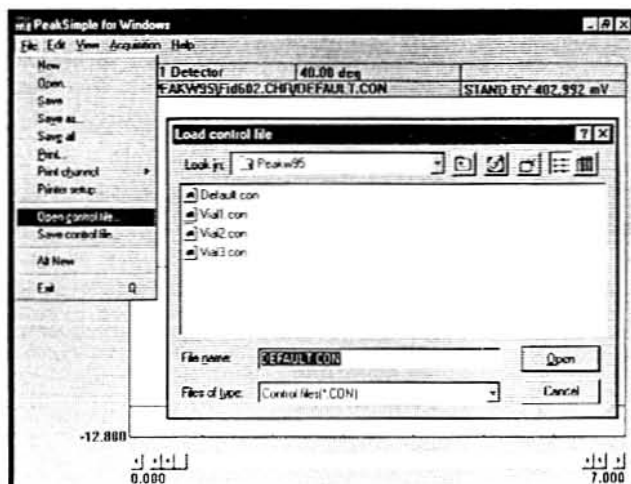
The FILE-PRINTER SETUP Pull-Down Menu

Selecting **Printer setup** from the **FILE** pull-down menu will allow you to enter the Printer Properties screen for your specific printer. This screen is similar to Windows Printer Properties screen that is accessible from the Windows Control Panel. Typically, using your printer default settings with **portrait** orientation will produce a visually appealing printout.



The FILE-OPEN CONTROL FILE Pull-Down Menu

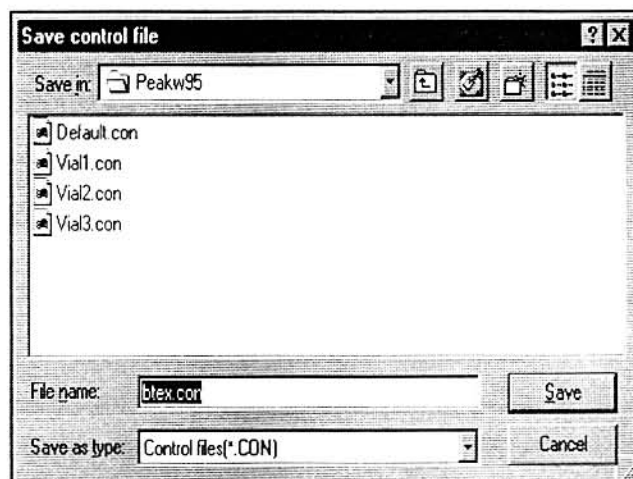
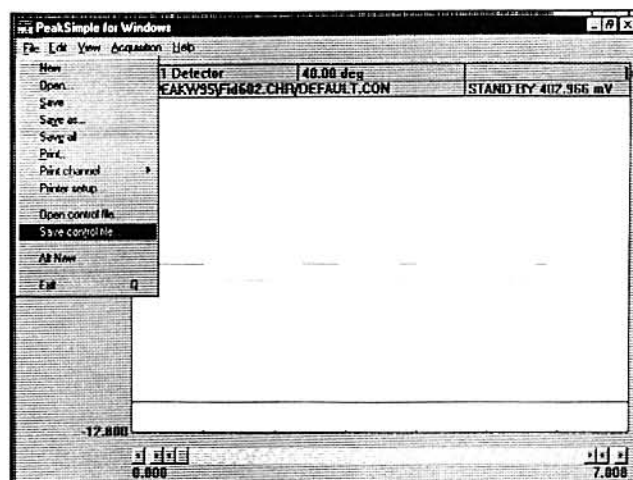
PeakSimple for Windows uses **Control Files**, identified with the **.CON** extension, to save the operating settings of specific methods. To load a **Control File**, drop down the **FILE** menu and select **OPEN CONTROL FILE**. A window will open which will allow you to use standard Windows navigating tools to select from a list of **.CON** files, located on the **Drive** or **Directory** of your choice. Click on the desired **File Name** and then click **O.K.**



The FILE-SAVE CONTROL FILE Pull-Down Menu

Once you have set up all of the user-definable parameters within PeakSimple for Windows that meet the requirements of your system and/or your specific analytical method, it is wise to save these settings for future use. PeakSimple uses **control files**, identified with a **.CON** extension, to save the operating settings of specific methods, this includes the event table, temperature program, component table, print information, calibration table, etc.

A **control file** is like a photocopy of your operating settings that you can reload for use at any time. When using **control files**, you only need to set analysis parameters once and then save them using a descriptive file-name, followed by the **.CON** extension, (for example, **BTEX.CON**). To save the **control file**, drop down the **File** menu and select **Save control file**. Enter the name for your file in the **File name** box and click **O.K.**. If you want these current settings to be loaded by default each time you start PeakSimple, name the control file **Default.con**.

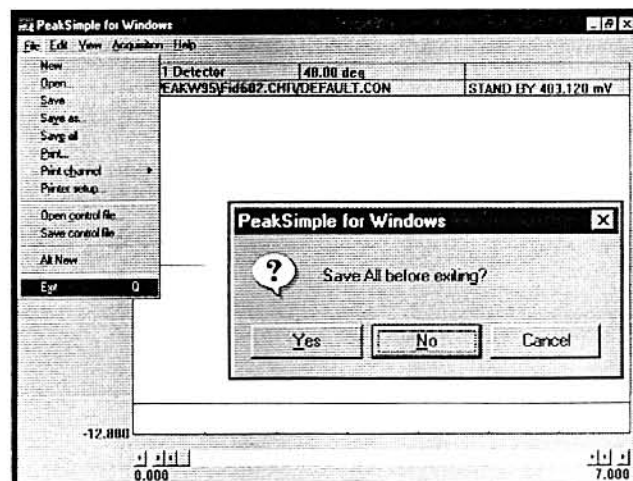


FILE-ALT NEW

The **FILE-ALT NEW** feature will clear the display of all active channels in the **Alternate** timebase without starting a new chromatographic run.

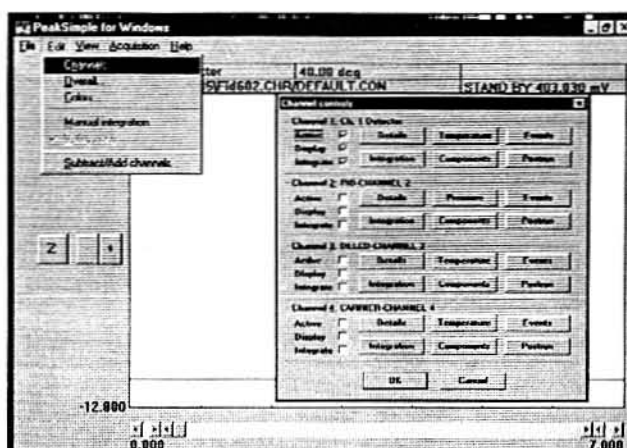
FILE-EXIT

Exits PeakSimple for Windows. Click **Yes** to save any changes made to your **control file** parameters.



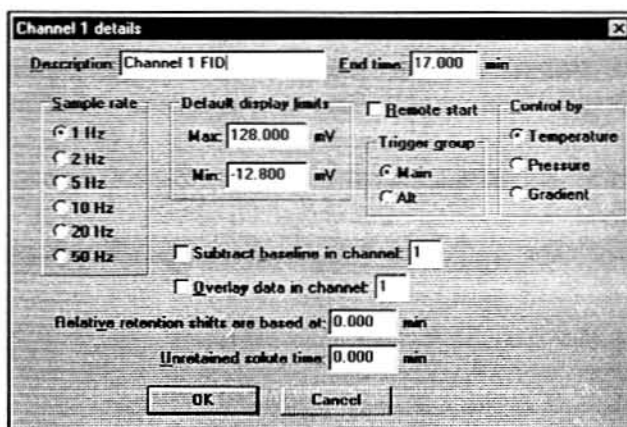
The EDIT-CHANNELS Pull-Down Menu

The **EDIT** pull-down menu allows you to modify most of the operating parameters for your specific application. Selecting **EDIT-CHANNELS** will bring up a screen which will enable you to select which of the four channels are **active**, **displayed** and **integrated**. Each channels' operating parameters such as **Details**, **Temperature**, **Events**, **Integration**, **Components** and **Postrun** information can be easily modified.



The EDIT-CHANNELS-DETAILS Screen

Clicking on the **Details** box for **Channel 1** will bring up a screen where you can enter a **Description** of your analysis. **End Time** displays the length of the chromatographic run in minutes. By default, the **End Time** is determined by the length of the temperature program but you may modify this field to end the chromatographic run at any time.



The **Sample Rate** should be set to a rate sufficient to ensure that 20 data samples are collected for each peak. For example: A **Sample Rate** of 1 Hz will allow the collection of 20 data points from a peak 20 seconds wide from base to base. And a **Sample Rate** of 10 Hz will allow the collection of 20 data points from a peak 2 seconds wide from base to base. The analog to digital converter is limited in its ability to sample high rates when many channels are active. The limits are: 50 Hz with one channel active, 10 Hz with two channels active and 5 Hz with three or four channels active.

The **Default Display Limits** can be adjusted to view data above and below the 0 mV baseline. A minus (-) setting for **minimum** will display negative going peaks. The ratio of **min./max.** display limits is maintained when you click on the Display minus and plus buttons in the main data acquisition screen.

The **Remote Start** feature allows the user to start a chromatographic run using an external signal such as a footswitch. Check the box to enable **Remote Start**. (There must be an internal connection made to the A/D board in order for this option to work.)

The EDIT-CHANNELS-DETAILS Screen (continued)

Unretained Solute Time

If resolution has been selected to be printed in the chromatogram report, then a **Unretained Solute Time** value needs to be entered to ensure correct resolution calculations. Enter the number of minutes an **Unretained Solute** takes to pass through the column. This value is used in the determination of peak resolution statistics.

Channel 1 details

Description: Channel 1 FID End time: 17.000 min

Sample rate:
☒ 1 Hz
☐ 2 Hz
☐ 5 Hz
☐ 10 Hz
☐ 20 Hz
☐ 50 Hz

Default display limits:
Max: 128.000 mV
Min: -12.800 mV

Remote start: ☐
Trigger group:
☒ Main
☐ Alt

Control by:
☒ Temperature
☐ Pressure
☐ Gradient

☐ Subtract baseline in channel: 1
☐ Overlay data in channel: 1

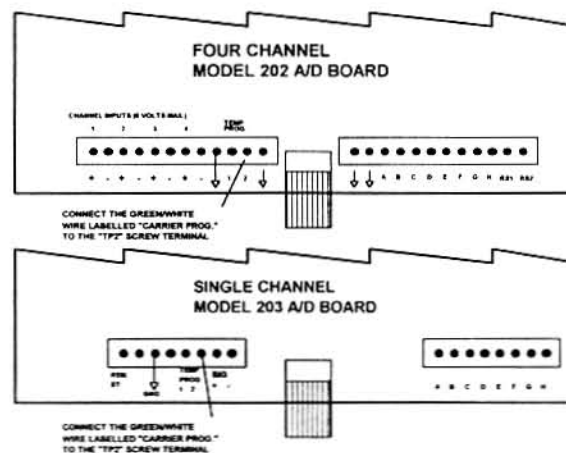
Relative retention shifts are based at: 0.000 min
Unretained solute time: 0.000 min

OK Cancel

Control By

The A/D Board that is built into the SRI gas chromatograph includes two digital-to-analog converters or DACs. DAC1 is primarily used to control the column oven #1 **temperature** ramp by introducing 10mV / °C to the oven heating circuit and is programmable by editing the **Channel 1-Temperature** control window. DAC2 is primarily used to control the column oven #2 **temperature** ramp. Carrier gas E.P.C. pressure is also programmable by editing the **Channel 2-Temperature/Pressure** control window. The DACs may be used to control **Pressure** by following the procedure described below and then selecting **Pressure** in the **Control By** window of the **Edit-Channels-Details** screen.

To avoid startup difficulties, the Carrier E.P.C. is shipped disabled. To enable the use of the DACs to set up a **Pressure Program**, only a single wire needs to be moved inside the G.C.. Unplug the G.C. and remove the six screws which secure the bottom cover. Tilt the G.C. onto its back and remove the bottom cover. The A/D Board is green in color and is mounted on the right-hand side of the G.C. chassis. Locate the Green wire with a White stripe on the A/D Board. This is the **Carrier Program** wire. Normally this wire is connected to a ground (GD) screw terminal. Unscrew the **Carrier Program** wire and connect it to the temperature/pressure #2 (TP2) screw terminal also on the A/D Board. Re-attach the bottom cover, connect power and re-establish communication between the G.C. and the computer. Select **Pressure** in the **Control By** window of the **Edit-Channels-Channel 2-Details** screen. A pressure program ramp set up in **Channel 2** will now control the Carrier Gas E.P.C. pressure by introducing 10mV for every P.S.I.. Turn the Carrier 1 **Local Setpoint** to zero. This is necessary since the Local setpoint is added to the programmed E.P.C. input in determining the Carrier 1 **total setpoint**.



The EDIT-CHANNELS-DETAILS Screen (continued)

Trigger Group

The **Trigger Group** selection assigns the channel to the **Main** or **Alt** trigger group. The picture at right shows the **Channel 1 Details** screen with the **Main Trigger Group** selected.

Channel 1 details

Description: Channel 1 FID End time: 17.000 min

Sample rate: ☒ 1 Hz ☐ 2 Hz ☐ 5 Hz ☐ 10 Hz ☐ 20 Hz ☐ 50 Hz

Default display limits: Max: 128.000 mV Min: -12.800 mV

Remote start: ☐ Trigger group: ☒ Main ☐ Alt

Control by: ☒ Temperature ☐ Pressure ☐ Gradient

☐ Subtract baseline in channel: 1

☐ Overlay data in channel: 1

Relative retention shifts are based at: 0.000 min

Unretained solute time: 0.000 min

OK Cancel

Any **Channel** with the **Main** trigger group selected will start running when the SPACEBAR is pressed and end when the END key is pressed. Any **Channel** with the **Alt** trigger group selected will start running when the + (plus) key is pressed and end when the - (minus) key is pressed. When acquiring four detector signal inputs from one gas chromatograph; verify that all four channels' **Trigger Group** is set to **Main**. This ensures that all four channels are acquiring data synchronously by using the same timebase. If two channels of data are coming from an SRI gas chromatograph, and you also wish to acquire two channels from an external input device such as an HPLC, then select the **Alt** trigger group for channels 3 and 4. This allows for asynchronous data collection.

Subtract Baseline In Channel "X"

Checking **Channel 1's** box for **Subtract Baseline In Channel "X"**, where "X" is 1,2,3 or 4, will cause the chromatogram in **Channel 1** to subtract the baseline stored in **Channel "X"**, while running in real-time. Load the baseline to be subtracted into an inactive channel to ensure that the data is not deleted by the start of a new run on that channel. (Uncheck the **active** box, see **Edit-Channels**). Baseline subtraction can also be performed using PeakSimple's **Edit-Subtract/Add Channels** feature, however, this is not a real-time function, but a post-run function, done at the end of the chromatographic run.

Overlay Data In Channel "X"

Checking **Channel 1's** box for **Overlay Data In Channel "X"**, where "X" is 1,2,3 or 4, will overlay the data stored on **Channel "X"** onto **Channel 1** using contrasting colors. The channel selected for overlay can be either an active or inactive channel. When the overlay channel is active then the overlay will be seen in real-time.

Relative Retention Shifts Are Based At "X" Minutes

Relative Retention Shifts Are Based At "X" Minutes. Enter into this box the time, in minutes, that the sample is actually injected onto the column. This is done to ensure that relative retention times are correctly calculated. See the **EDIT-CHANNEL-COMPONENTS** section of this manual for more details.

The EDIT-CHANNELS-DETAILS Screen (continued)

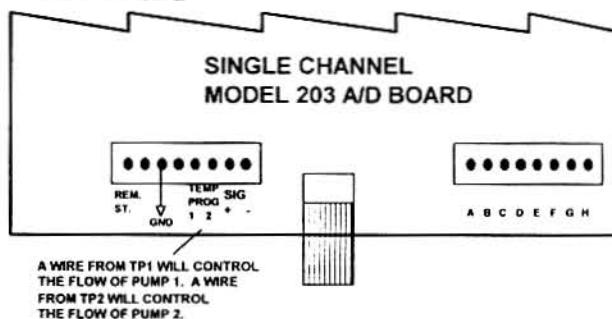
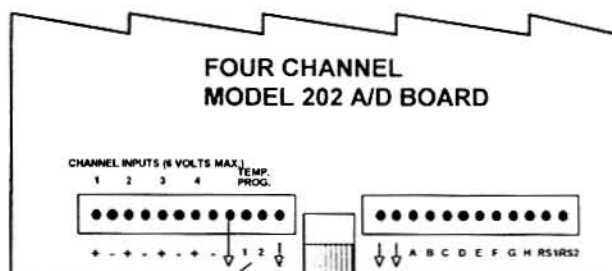
Setting Up Gradients for Liquid Chromatography

Data System users may wish to use the A/D Board DACs for setting up an HPLC solvent gradient. PeakSimple for Windows allows the user to control the flow of two pumps, provided they operate from a zero to five volt (0-5V) ramp input.

To operate the pumps, several internal connections must be made between the HPLC and the Data System. Unplug the Data System and remove the two screws which secure the top cover. Route the Pump A and Pump B control wires from the HPLC to the Data System and connect the Pump A control wire to TP1 and the Pump B control wire to TP2. Re-attach the top cover, connect power and re-establish communication between the Data System and the computer.

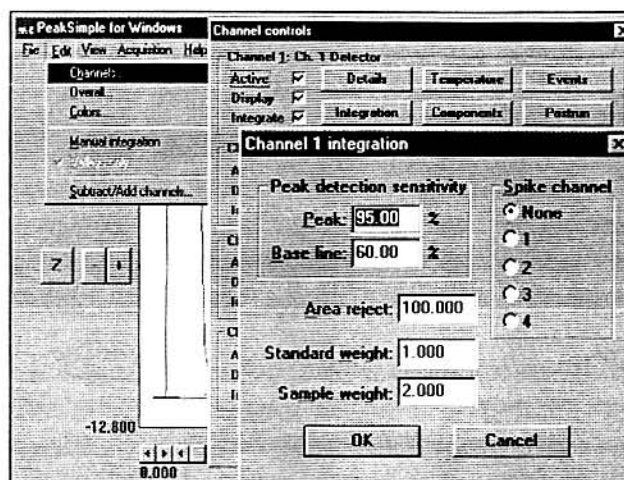
Set up the **Gradient** ramp on **channel one** (TP1) to control the flow of Pump A into the system (10mV / %) and the **Gradient** ramp on **channel two** (TP2) to control the flow of Pump B. Modifying the **Gradient** ramp program on **Channel 1** to rise from 10% to 90% will automatically create a **Gradient** ramp program on **Channel 2** that decreases proportionately from 90% to 10%.

Gradient Limits Zero and Span may be scaled in the **Edit-Overall** screen to account for any offsets. PeakSimple allows for a voltage offset and scaling factor in these fields to calibrate the voltage output to match the pump's requirements.



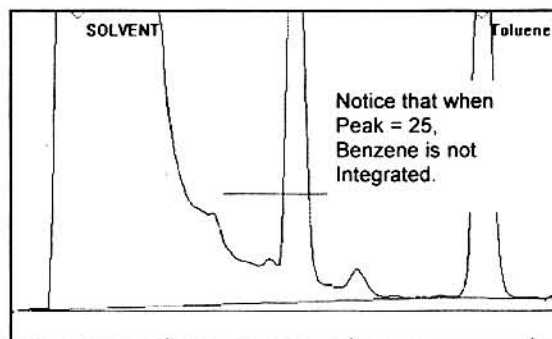
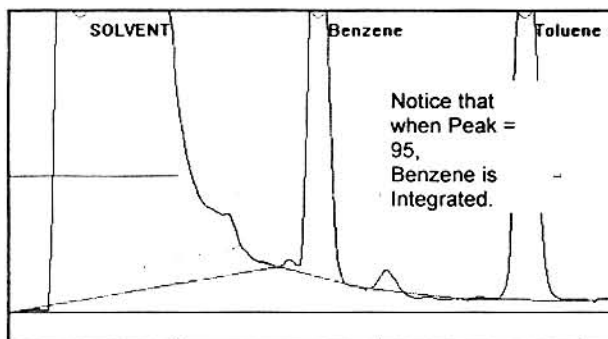
The EDIT-CHANNELS- INTEGRATION Screen

PeakSimple for Windows allows you to define specific integration parameters necessary for the proper analysis of your sample data, such as peak and baseline sensitivity and area reject. Any of the **Integration** parameters described below may be modified either before or after data collection. Pressing the **ENTER** key will update the report and the results of the chromatogram currently being displayed.

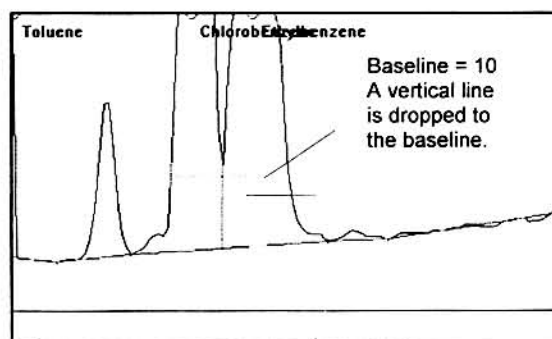
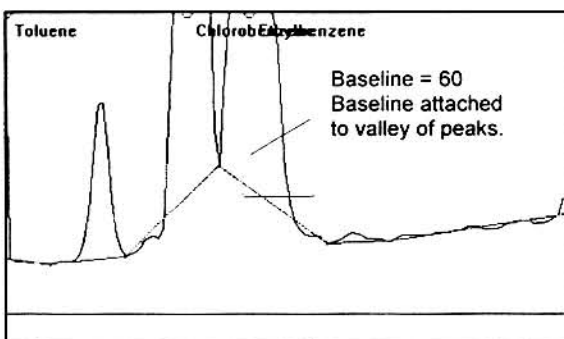


Peak Detection Sensitivity

The **Peak** sensitivity setting determines how PeakSimple detects the beginning and end of a peak. A high **Peak** number requires only a small slope change to initiate the start or end of a peak. A low **Peak** number requires a very large slope change to initiate the start or end of a peak.



The **Baseline** sensitivity setting determines how PeakSimple attaches the baseline to the data line. The larger the **Baseline** number; the more likely PeakSimple will draw the baseline to a valley between two peaks. The smaller the **Baseline** number; the more likely PeakSimple will drop a vertical line from a valley to a horizontally constructed baseline below the peak.



The EDIT-CHANNELS- INTEGRATION Screen (continued)

Area Reject

If a chromatogram contains peaks whose area counts fall below the threshold defined by the **Area Reject** for that channel, the peak will be ignored and no integration will occur. If the peak area is of interest, you can lower the **Area Reject** value until the peak in question is integrated. Integrated peaks are marked with a circle at the top of the peak.

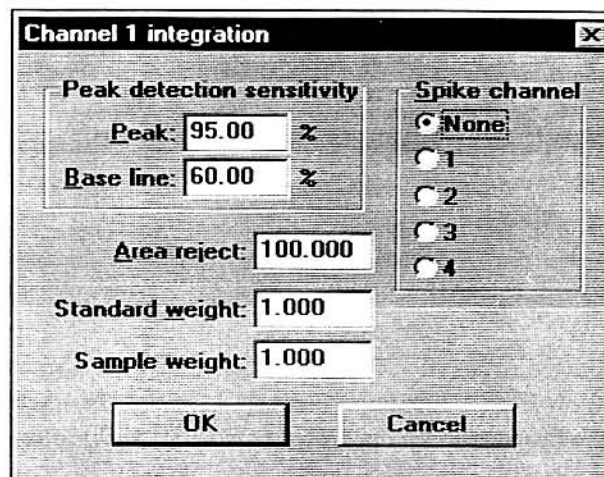
Standard Weight

PeakSimple for Windows determines the internal or external standard results by the ratio of the STANDARD divided by the SAMPLE.

The **Standard Weight** setting may be changed to adjust the channel's quantification, affecting internal or external peak results by the factor entered. For instance: A setting of 2.000 will double the weight of the standard thereby doubling the internal or external standard results. (Increased to 20.000 in the example shown.)

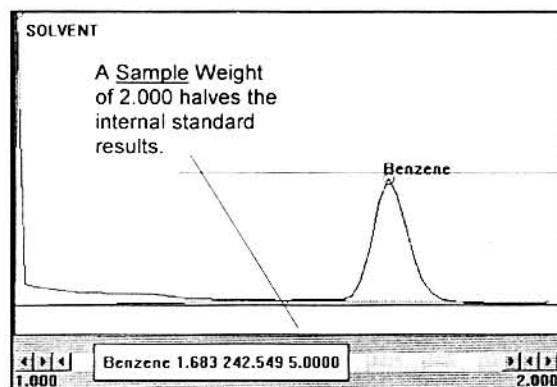
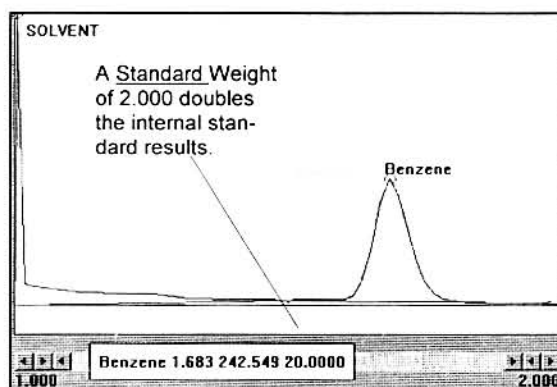
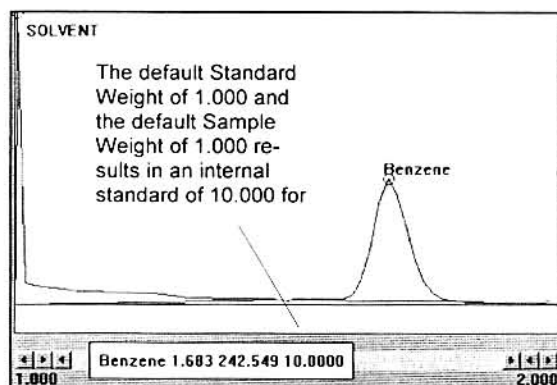
Sample Weight

The **Sample Weight** setting may also be changed to adjust the channel's quantification, affecting internal or external peak results by the factor entered. For instance: A setting of 2.000 will double the weight of the sample thereby halving the internal or external standard results. (Decreased to 5.000 in the example shown.)



The 'Channel 1 integration' dialog box contains the following settings:

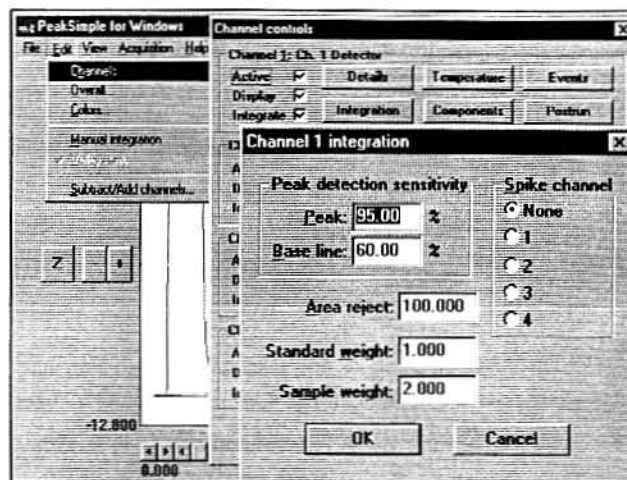
- Peak detection sensitivity: Peak: 95.00 %, Base line: 60.00 %
- Spike channel: ☒ None, ☐ 1, ☐ 2, ☐ 3, ☐ 4
- Area reject: 100.000
- Standard weight: 1.000
- Sample weight: 1.000
- Buttons: OK, Cancel



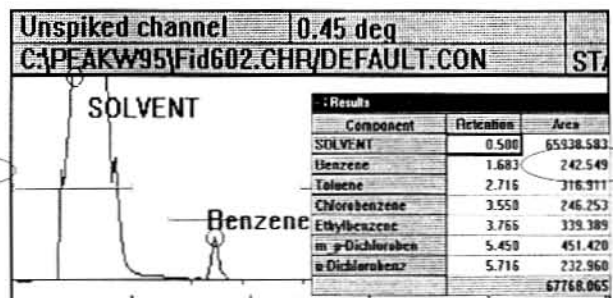
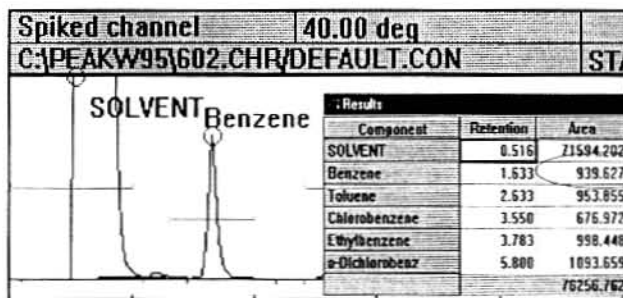
The EDIT-CHANNELS- INTEGRATION Screen (continued)

Spike Channel

Another feature of PeakSimple for Windows allows you to display the results of a matrix **Spike Channel** subtraction. The example shown below demonstrates the peak area counts of a unspiked channel being subtracted from the area counts of a spiked channel.

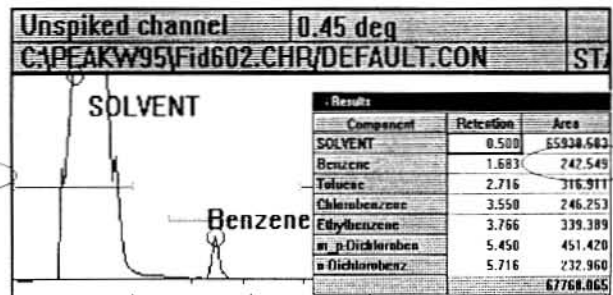
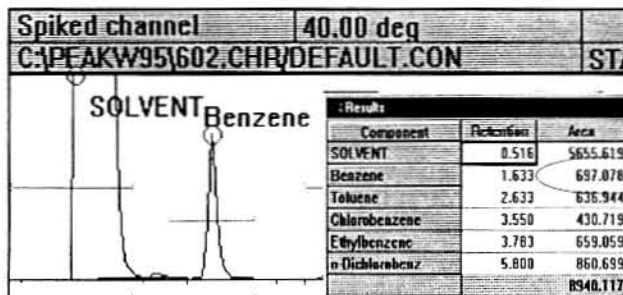


Before Spike Channel Subtraction



Notice that the area counts for Benzene are 939 on the spiked channel, and 242 area counts on the unspiked channel.

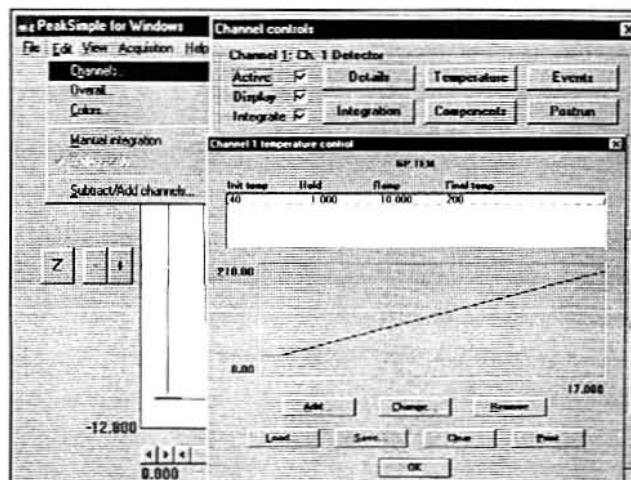
After Spike Channel Subtraction



After selecting channel 2 as the **Spike Channel**, the area counts for channel 2 are subtracted from channel 1 to equal 697, ($939 - 242 = 697$). The difference of 697 indicates the area counts of the amount of sample spiked into channel 1.

The EDIT-CHANNELS-TEMPERATURE Screen

PeakSimple for Windows features temperature-programming of the G.C 's column oven(s). Access the **Edit-Channel 1-Temperature** screen to specify the temperature parameters to be used during the analytical run. The temperature program is capable of executing an unlimited number of temperature ramp and hold periods during the analysis as well as maintaining a single temperature throughout the run for isothermal operation.



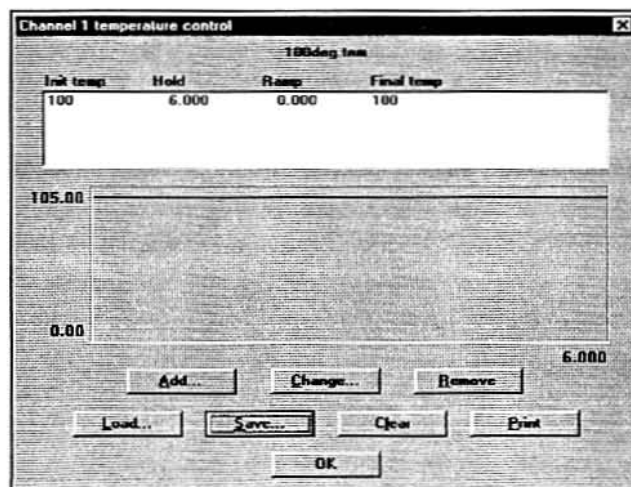
The Temperature Segment Details Screen

The Add Button

Click on the **Add** button from a blank **Channel 1 temperature control** window to create a new temperature program for Column oven #1. (Use the **Edit-Channel 2-Temperature** screen for controlling column oven #2). Type in the required data in the following fields; **Initial temperature**, the **Hold** period in minutes, the **Ramp** rate in °C / min, and the final **Temperature**, or the duration of the Ramp.

The length of the run is automatically calculated by PeakSimple based on the information provided in these fields, and is also displayed in the **Edit-Channels-Details End Time** field. Additional ramp segments may be added by clicking the **Add** button again.

In isothermal operation, the **Initial** and the final temperature are the same, so a **Ramp** rate of 0.000 is entered. The **Hold** period determines the length of the analytical run.



The EDIT-CHANNELS-TEMPERATURE Screen (continued)

The Change Button

Click on an existing temperature program segment to select it. Click on the **Change** button to change the parameters of the segment.

The Remove Button

Click on the **Remove** button to remove the segment from the current program.

The Load Button

Click on the **Load** button to load an existing temperature control file, designated with the **.TEM** file extension.

The Save Button

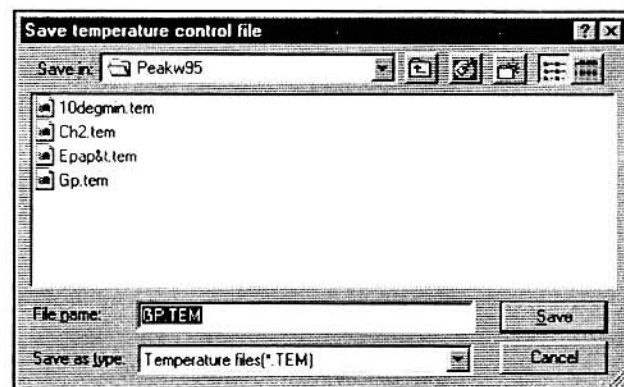
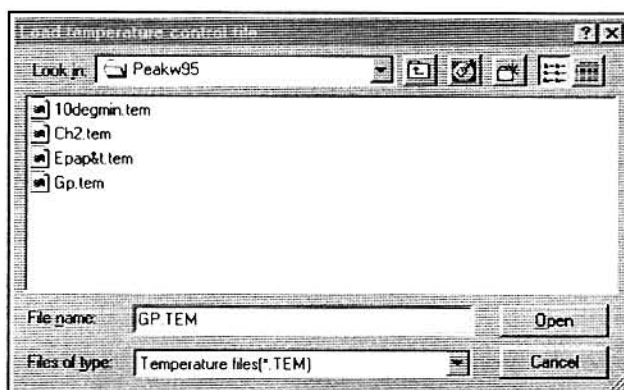
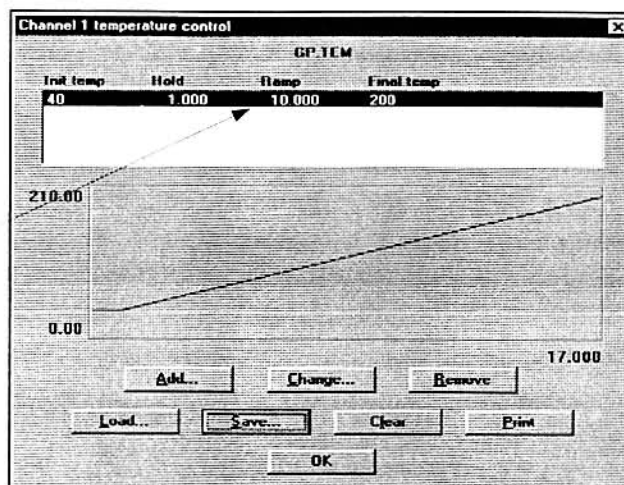
Click on the **Save** button to save a new temperature control file, or to update an existing one. Remember to use the **.TEM** extension when naming the temperature control file. The saved file name appears at the top of the temperature control window indicating the file in use.

The Clear Button

Clicking on the **Clear** button deletes all temperature data from the temperature control window. The temperature program name is also removed.

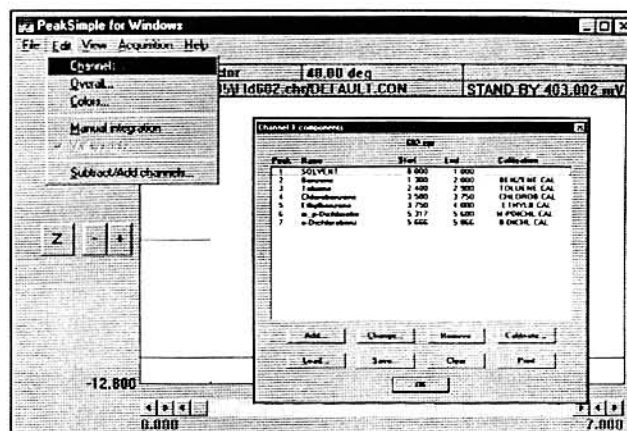
The Print Button

Clicking on the **Print** button sends the file data and temperature program profile to the printer.

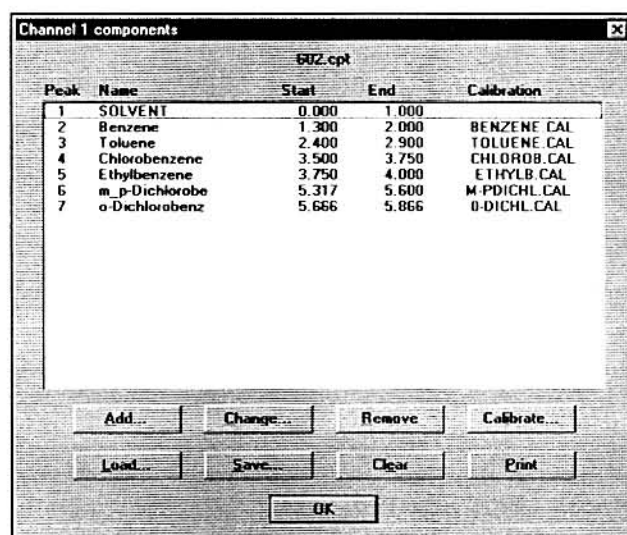


The EDIT-CHANNELS- COMPONENTS Screen

PeakSimple for Windows can identify and quantify sample components through the use of a component table. The component table enables PeakSimple to recognize each peak by its retention time and compare the area counts against the calibration curve to produce actual concentration data. The user can edit the component table for each channel by accessing the **Edit-Channels-Components** screen.

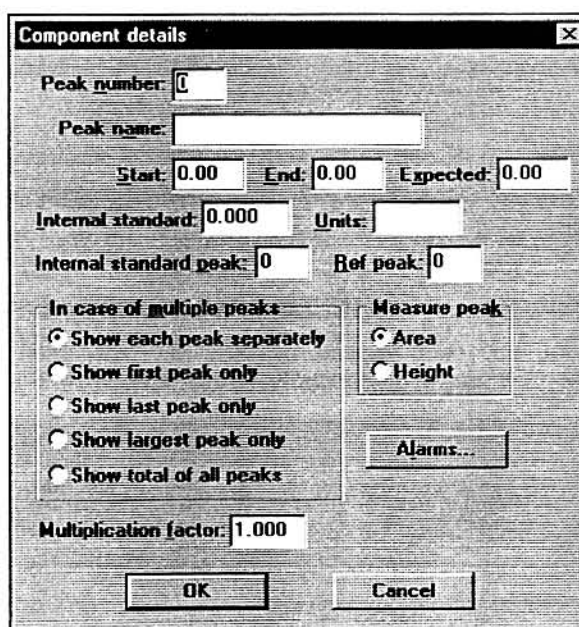


When a component table is loaded, the table will show each component by its peak number, peak name, the start time for the retention window, the stop time for the retention window, and the associated calibration file name. Different component tables may be used for each active channel and any component table can be saved as a component file for future use. Component files are designated with a .CPT extension. The component file-name appears at the top of the **Components** screen.



COMPONENT DETAILS

Select **Add** to add a new component to a blank or existing component table. The Component Details screen will open allowing the user to input specific peak parameters. As a minimum, enter the **Peak Number**, **Peak Name**, **Start** time and **End** time. Other optional parameters are the **Expected** peak time, the concentration **Units** to be reported, any **Internal Standard** or **Reference** peak information, peaks measured by **Area** or **Height**, handling of **Multiple Peaks**, the **Multiplication Factor** and **Alarm** parameters.



The EDIT-CHANNELS- COMPONENTS-DETAILS Screen (continued)

Peak Number, Peak Name, Start and End

A blank **Component Details** screen is opened by selecting the **Add** button. Enter a unique **Peak Number** for each component, typically starting with 1 and incrementing for each additional peak. Then enter a unique **Peak Name** for each component. **Start** and **End** define the beginning and ending of the retention windows, which is used to identify the peak. The width of the retention window should be set wide enough so that small fluctuations in the peak's retention time will still allow for proper integration.

Internal Standard and Units

Internal Standard calculations are used to correct for injection size variations, or to compensate for changes in detector sensitivity. An internal standard peak is added to the sample prior to injection at a known concentration. The internal standard peak is calculated just like any other peak using a calibration curve, typically a single point calibration. The known concentration of the internal standard peak is entered into the **Internal Standard** dialog box of the **Component Details** screen. In the example shown below, Benzene has been chosen as the internal standard peak. The known concentration of Benzene is entered as **100**, and **ppm** is entered in the **Units** dialog box. When a chromatogram is integrated and a report is produced, the external calculation yields a result which is the **peak area x calibration factor** (slope of the calibration curve) = **external standard result**.

The **internal standard** calculation yields a result which is the **external result times the ratio of the known concentration of the internal standard peak divided by the external result for the internal standard peak**. As shown in the example to the right, note that while the external result for Benzene yields 104.95, the internal result yields exactly 100 (the known concentration) as a result of the calculation $104.95 \times 100 / 104.95$. In the same way, the internal result for every analyte peak which is referenced to Benzene is calculated as **external result x 100 / 104.95 = internal standard result**.

Component	Retention	Area	External	Internal	Units
SOLVENT	0.516	71594.202	0.00	0.0000	%
Benzene	1.633	939.627	104.95	100.0000	ppm
Toluene	2.633	953.855	106.73	106.7319	ppm
Chlorobenzene	3.550	676.972	72.12	72.1215	ppm
Ethylbenzene	3.783	998.448	112.31	112.3059	ppm
m,p-Dichloroben	5.800	1093.559	124.21	124.2074	ppm
o-Dichlorobenz	6.150	536.767	54.60	54.5959	ppm
		76793.529	574.92	569.9626	

The EDIT-CHANNELS-COMPONENTS-DETAILS Screen (continued)

Internal Standard Peak

PeakSimple allows any peak to be referenced to any other peak for internal standard calculations. Typically all analyte peaks will be referenced against a single **Internal Standard Peak** (Benzene [peak #2] in the example shown below). To reference other peaks to Benzene, the number **2** must be entered in the **Component Details** screen dialog box labeled **Internal Standard Peak** for each analyte peak. Notice that the **Results** screen, (**View-Results**), will reflect the new value for all the peaks' internal results.

Component details

Peak number: 3
 Peak name: Toluene
 Start: 2.40 End: 2.90 Expected: 0.00
 Internal standard: 0.000 Units: ppm
 Internal standard peak: 0 Ref peak: 0

In case of multiple peaks
☒ Show each peak separately
☐ Measure peak
☒ Area

Component	Retention	Area	External	Internal	Units
SOLVENT	0.516	71594.202	0.00	0.0000 %	
Benzene	1.633	939.627	104.95	100.0000 ppm	
Toluene	2.633	953.855	106.73	106.7319 ppm	
Chlorobenzene	3.550	676.972	72.12	72.1215 ppm	
Ethylbenzene	3.783	998.448	112.31	112.3059 ppm	
m,p-Dichloroben	5.800	1093.659	124.21	124.2074 ppm	
o-Dichlorobenz	6.150	536.767	54.60	54.5959 ppm	
		76793.529	574.92	569.9626	

OK Cancel

Component details

Peak number: 3
 Peak name: Toluene
 Start: 2.40 End: 2.90 Expected: 0.00
 Internal standard: 0.000 Units: ppm
 Internal standard peak: 2 Ref peak: 0

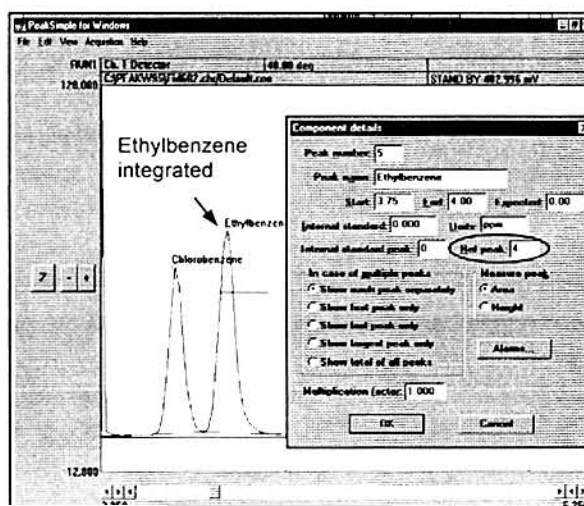
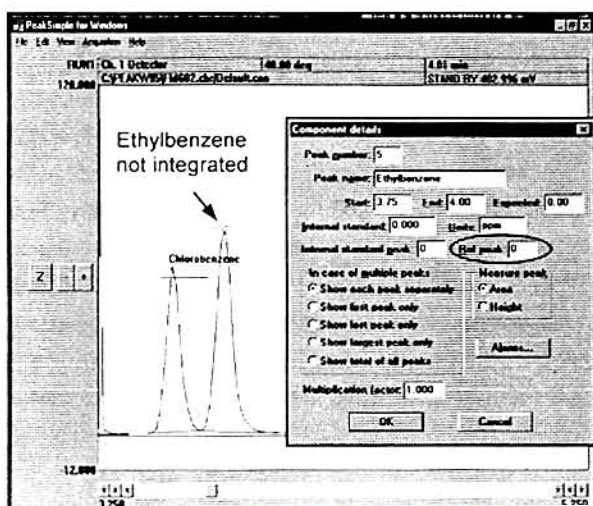
In case of multiple peaks
☒ Show each peak separately
☐ Measure peak
☒ Area

Component	Retention	Area	External	Internal	Units
SOLVENT	0.516	71594.202	0.00	0.0000 %	
Benzene	1.633	939.627	104.95	100.0000 ppm	
Toluene	2.633	953.855	106.73	101.6946 ppm	
Chlorobenzene	3.550	676.972	72.12	72.1215 ppm	
Ethylbenzene	3.783	998.448	112.31	112.3059 ppm	
m,p-Dichloroben	5.800	1093.659	124.21	124.2074 ppm	
o-Dichlorobenz	6.150	536.767	54.60	54.5959 ppm	
		76793.529	574.92	564.9252	

OK Cancel

Reference Peak

A **Reference Peak** is used to shift the retention windows of other peaks. In the example below, ethylbenzene eluted prior to its retention window so therefore it was not integrated. By entering a value of **4** in the **Reference Peak** box, ethylbenzene's retention windows are referenced to chlorobenzene, [peak #4]. Ethylbenzene's retention window is then shifted by a percentage equivalent to chlorobenzene's distance from the middle of its retention window. This shift in the ethylbenzene retention window allows ethylbenzene to be integrated.



The EDIT-CHANNELS- COMPONENTS Screen (continued)

The **Change** Button

Click on an existing component to select it. Click on the **Change** button to change the parameters of the component.

The **Remove** Button

Click on the **Remove** button to remove the component from the component table.

The **Load** Button

Click on the **Load** button to load an existing component file, designated with the .CPT file extension.

The **Save** Button

Click on the **Save** button to save a new component file, or to update an existing one. Remember to always use the .CPT extension when naming the component file. The saved file name appears at the top of the components window indicating the file in use.

The **Clear** Button

Clicking on the **Clear** button deletes all component data from the component window. The component file name is also removed.

The **Print** Button

Clicking on the **Print** button sends the file data and the component table information to the printer.

Channel 1 components

602.cpt

Peak	Name	Start	End	Calibration
1	SOLVENT	0.000	1.000	
2	Benzene	1.800	2.000	BENZENE.CAL
3	Toluene	2.400	2.900	TOLUENE.CAL
4	Chlorobenzene	3.500	3.750	CHLOROB.CAL
5	Ethylbenzene	3.750	4.000	ETHYLB.CAL
6	m,p-Dichlorobenz	5.317	5.600	M-PDICHL.CAL
7	o-Dichlorobenz	5.666	5.866	O-DICHL.CAL

Buttons: Add... Change... Remove Calibrate... Load... Save... Clear Print OK

Load component file

Look in: Peakw95

- 601.cpt
- 602.cpt
- Queue.cpt

File name: 602.cpt Open

Files of type: Component files (*.CPT) Cancel

Save component file

Save in: Peakw95

- 601.cpt
- 602.cpt
- Queue.cpt

File name: 602.cpt Save

Save as type: Component files (*.CPT) Cancel

The EDIT-CHANNELS-COMPONENTS Screen (continued)

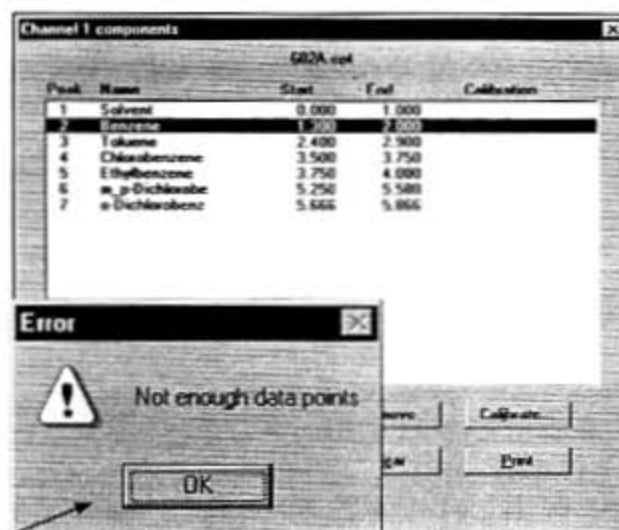
The Calibrate Button

After creating a component table, each component in the table will need to be calibrated. This allows PeakSimple for Windows to not only identify each analyte peak, but also to quantify each peak using a calibration curve. The calibration curve is calculated from user-generated results obtained at several different concentrations that span the expected range to be encountered in actual samples.

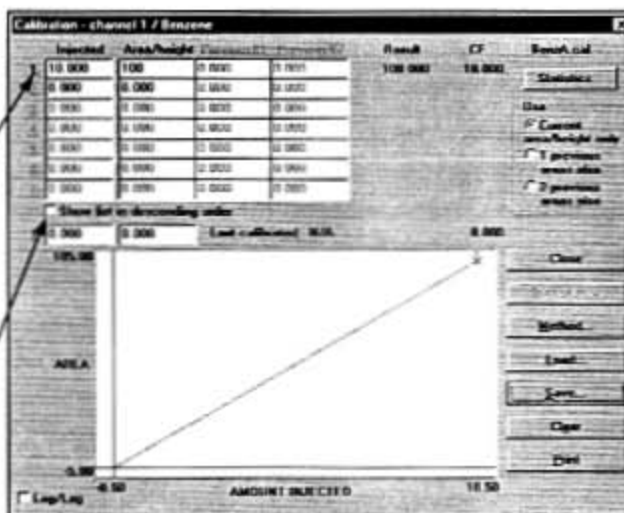
Inject a standard containing a known concentration of the component you wish to calibrate. Use a concentration higher than what you would expect to encounter in your analyses. Another few samples should be run at lower levels, using precise dilutions of your standard. Make note of the area counts or peak height at each concentration or use the shortcut method described in the next section.

The Calibration Window

In the **Edit-Channels-Components** screen, highlight the component to be calibrated and select **Calibrate**. If this is the first time calibrating a component, an error message will appear which says "Not enough data points". This is simply a warning to inform you that PeakSimple currently does not have enough data points for the calibration method in use. Once enough data is entered for the calibration curve, this message will no longer appear. Click **OK** to bypass the error message and continue to the calibration window.

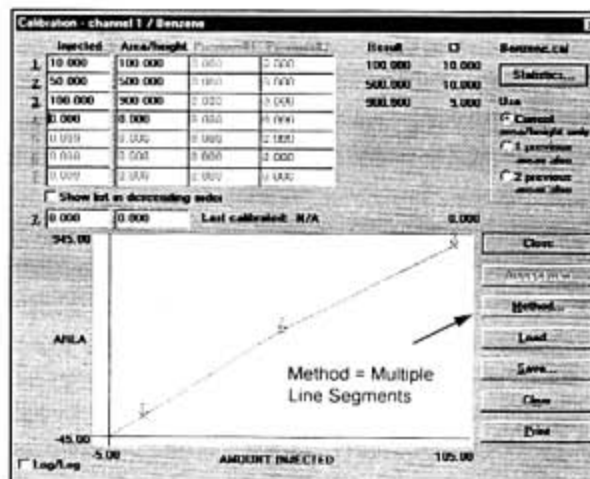


The **Calibration** window will open and allow you to enter the raw data that you previously obtained. In the example shown, data is entered into the table in the upper left corner of the calibration screen, beginning with the lowest concentration and ending with the highest concentration. If you wish to enter the data in descending order, check the **Show list in descending order** box. When entering data into the table, first enter the concentration injected, then the area count or peak height obtained.



The Calibrate Button (continued)

As data is entered for each concentration, a data point will be added to the calibration curve displayed in the lower section of the window. You may use as many as seven concentration levels for your calibration curve. In the fictitious example to the right, a Benzene standard was injected in concentrations of 10 ppm, 50 ppm, and 100 ppm. The area counts from the FID detector were 100, 500 and 900, respectively. Notice the three corresponding data points on the newly created calibration curve.



When calibration for each component has been completed, click on the **Save** button to save and name the component's calibration file. Then click on the **Close** button to close the calibration window. In our example, a unique file named **BenzFID.cal** was created. The **BenzFID.cal** file name will now appear in the **Components** window next to Benzene.

WARNING:

Do not use the same calibration curve file name for two different channels or detectors since each detector requires its own calibration curve. (ie **BenzFID.cal**; **BenzPID.cal**; etc)

Calibration is required for each component you expect to be present in your sample, and for each detector you will be using in your analysis. Once calibration curves have been completed, and calibration files saved, every component in the component table should show an associated calibration file. PeakSimple will now be able to quantify each component when actual samples are injected.

Peak	Name	Start	End	Calibration
1	Solvent	0.000	1.000	
2	Benzene	1.500	2.000	BenzFID.cal
3	Toluene	2.400	2.900	
4	Chlorobenzene	3.500	4.000	
5	Ethylbenzene	3.750	4.000	
6	m,p-Dichlorobenz	5.250	5.580	
7	o-Dichlorobenz	5.666	5.866	

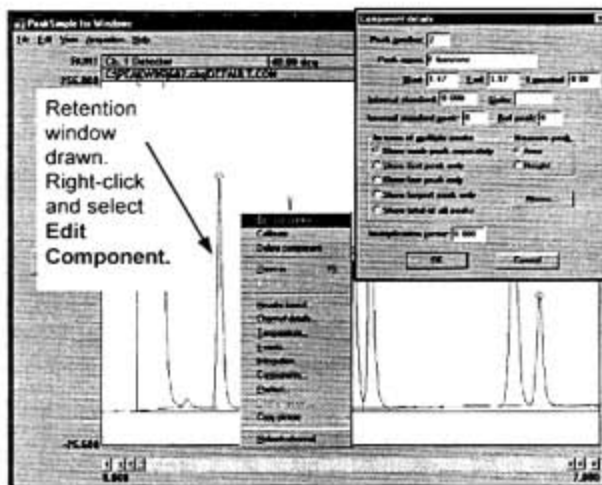
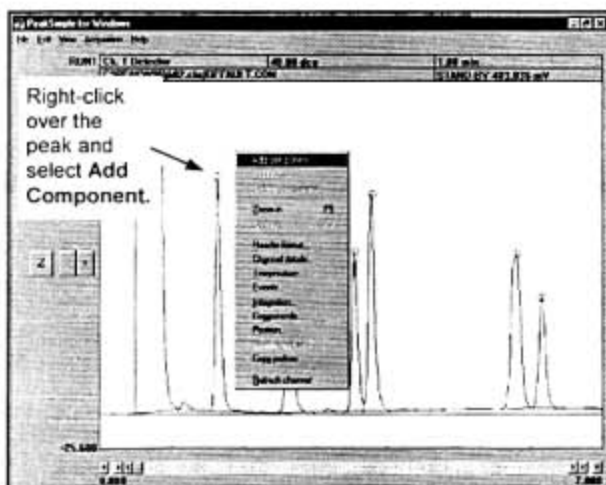
Peak	Name	Start	End	Calibration
1	SOLVENT	0.000	1.000	
2	Benzene	1.300	2.000	BENZENE.CAL
3	Toluene	2.400	2.900	TOLUENE.CAL
4	Chlorobenzene	3.500	3.750	CHLOROB.CAL
5	Ethylbenzene	3.750	4.000	ETHYLB.CAL
6	m,p-Dichlorobenz	5.317	5.600	M-PDICHL.CAL
7	o-Dichlorobenz	5.666	5.866	O-DICHL.CAL

Calibration Screen Shortcuts

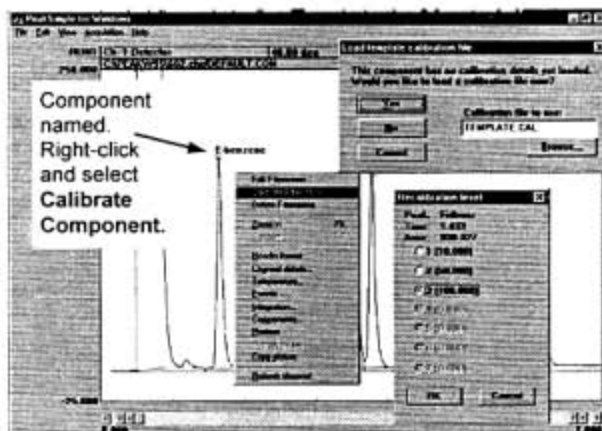
As an added convenience, PeakSimple for Windows offers shortcuts to commonly used screens. These shortcuts may be accessed by pointing to the desired channel and **clicking once on the right mouse button**. The following pages describe the shortcuts available to set up calibration tables and calibrate components.

After a known standard has been run and the peaks have been identified, a new component table may be constructed by simply positioning the mouse pointer over a peak and clicking once on the right mouse button, ("right-clicking"). The shortcut menu will appear. Select **Add component** from the menu. A retention window will be drawn horizontally across the peak. Right-click again over the peak and select **Edit component**. The **Component Details** screen will open allowing the peak to be named and numbered. The example below shows Benzene as peak #2. The component has been named F-benzene to avoid confusion with a benzene peak from another detector such as a PID.

Note: It is important that you choose the component name carefully since the calibration file name is derived from the first eight letters of the component name. The F-benzene calibration file would be named F-benzen.cal.



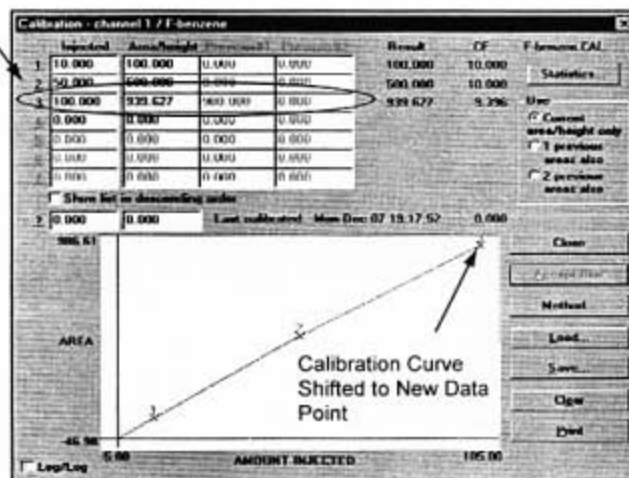
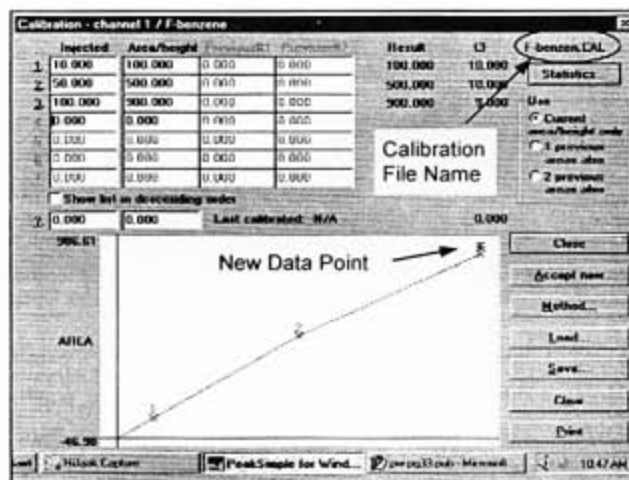
Right-click over the peak again and select **Calibrate**. If no calibration curve exists for the peak, a window will open asking if you would like to use a calibration file. PeakSimple offers a template calibration file aptly named TEMPLATE.CAL. Click yes to use the default TEMPLATE calibration file or select your own by clicking **Browse**. This example uses the template calibration file. Another window will open asking you to select the **Recalibration Level**. Select **100** for 100 ppm standards, **50** for 50 ppm, etc.



Calibration Screen Shortcuts (continued)

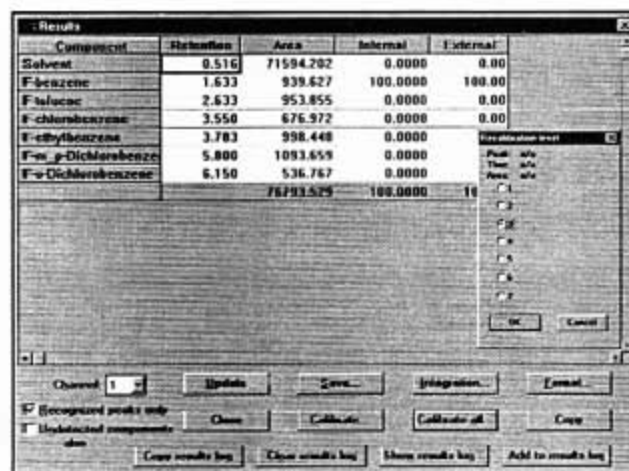
Click **OK** to accept the **Recalibration Level**. The Calibration screen will open and a flashing asterisk (*) will appear along the existing calibration curve depicting the new data point. Notice that the calibration curve has been named **F-benzen.CAL**. If the new calibration data point is acceptable, click **Accept New** to update the calibration curve data.

In the example to the right, the updated **F-benzen.CAL** calibration table reflects the new area count of **939.627** at the concentration level of **100 ppm**. (The previous calibration data of 900 area counts at 100 ppm is shown in the **Previous #1** column which is 'grayed out'). Notice also that the third data point (100 ppm) in the calibration curve has been shifted up slightly to incorporate the new data, (939.627 area counts). At this point, if the new calibration curve data is deemed to be acceptable, click on **Close** to automatically save the new calibration file, and close the **Calibration** window.



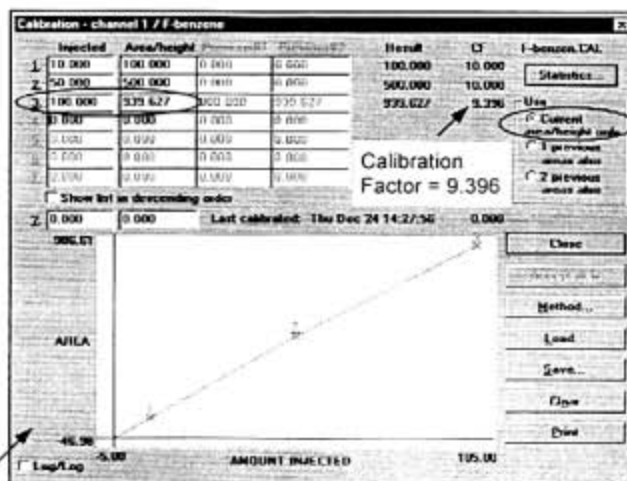
Calibrate All

PeakSimple offers a time-saving feature for **recalibrating all peaks** with just one mouse click. After a calibration curve has been created for each component, click on **View-Results** to bring up the results window. Select **Calibrate All** and choose an appropriate **Recalibration Level**, then click **OK**. PeakSimple will automatically recalibrate all components at the selected level and save each component's updated calibration file.

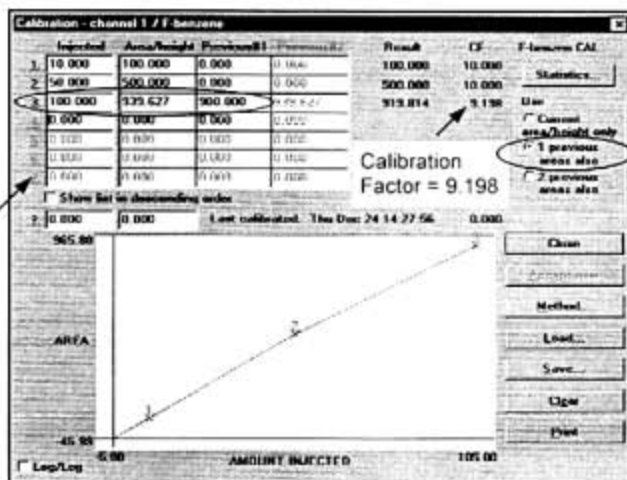


Calibration Screen – Use and Statistics Radial Buttons

To improve the calibration accuracy, chromatographers may prefer to average the areas of 1, 2 or 3 replicate injections. The **Use** radio button allows the user to select how many injections are used in the calculation of calibration factors, (CF). Calibration Factors are used to construct the calibration curve using the formula: $CF = \text{area count} / \text{divided by the amount injected}$. The example to the right shows the calibration data at the 100 ppm concentration level, (circled), with the **Use** button set to the default setting of **Current Area / Height Only**. This setting uses only the latest calibration data to calculate the calibration factor for the #3 data point. ($CF = 939.627 / 100 = 9.396$)

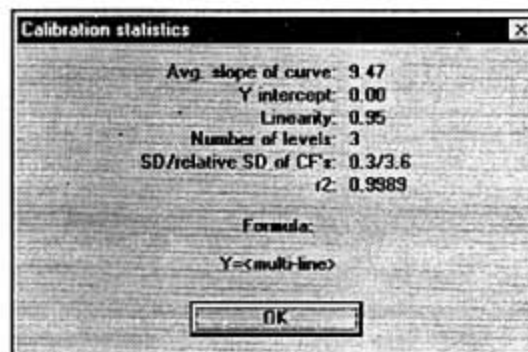


This next example shows how the calibration curve is changed when the **Use** button is set to **1 Previous Areas Also**. This setting averages the last two areas to derive the average calibration factor. Notice that the calibration factor is now 9.198 when the two area counts are averaged together. ($939.627 + 900.000 / 2 = 919.814$ average area counts. The CF is calculated as: $CF = 919.814 / 100 = 9.198$)



Setting **Use** to **2 Previous Areas Also** will average the last three areas to derive the calibration factor.

The **Calibration Statistics** screen shows calibration curve details such as the **Average Slope of the Curve**, the **Y Intercept**, the **Linearity** of the curve, the **Number of (calibration) Levels**, the **Standard Deviation and Relative Standard Deviation of Calibration Factors**, the **R2** and the **Formula** used which is based on the **Method** selected.



Calibration Window– Methods

The **Method** button opens the **Recalibration Type** window which allows the selection of one of six formulas used to draw the calibration curve. The algorithms are described below and corresponding calibration statistics are shown.

In the following:
 X is the sum of the external measures over the calibration levels
 Y is the sum of the corresponding areas at those calibration levels
 n is the number of active calibration levels
 Several other sums are used, for instance:
 X2 is the sum of the squares of the external measures
 Y4 is the sum of the (area to the 4th power)
 XY is the sum of the (external measure * area)
 X2Y is the sum of (external measure squared * area)
 Y|X is the sum of the (area / external measure) etc.

Single line through origin:

The resulting calibration curve is defined as

$$y = Ax$$

where:

x is external measure
 y is area
 $A = (Y|X)/n$

Notes:

The resulting factor is therefore the average of the calibration factors at the calibration levels. Note: any explicit calibration level point at $x=0$ is ignored (and n is reduced by 1). There must be at least one calibration level, not including any level at $x=0$.

Single line:

The resulting calibration curve is defined as

$$y = Ax + B$$

where:

x is external measure
 y is area
 $A = (XY * n) - (X * Y) / D$
 $B = (X * Y^2) - (XY * X) / D$
 $D = (X^2 * n) - (X * X)$

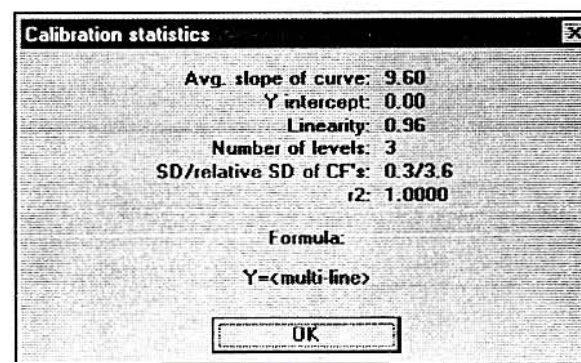
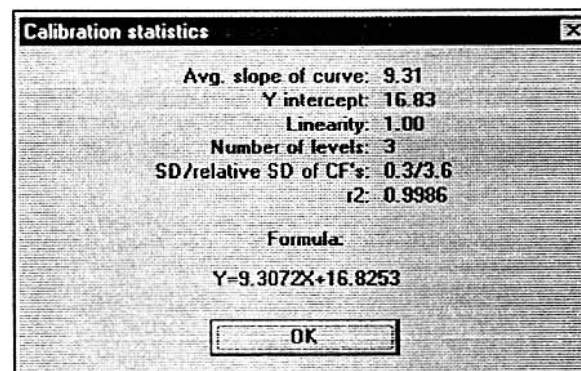
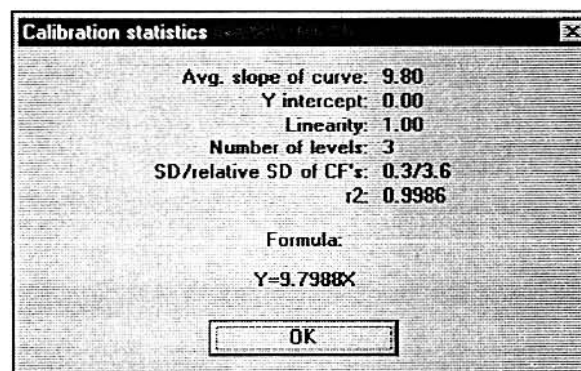
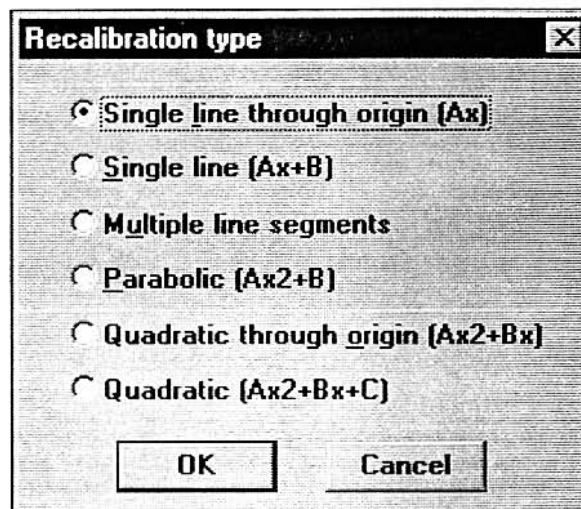
Notes:

This is a least squares fit algorithm over the calibration levels. A point at (0,0) is also assumed (by incrementing n) unless there is already a value at $x=0$, or if [Statistics]R2IncludeZero is set to 0 in the PEAKWIN.INI file. There must be at least 2 calibration levels.

EPA rules allow the use of Single Line Fit provided that the standard deviation of calibration factors is <20%.

Multiple line segments:

There is no resulting formula here, just interpolation between the levels, and the origin. There must be at least one calibration level.



Calibration Window– Methods (continued)

Parabolic:

The resulting calibration curve is defined as

$$y = Ax^2 + Bx$$

where:

x is external measure

y is area

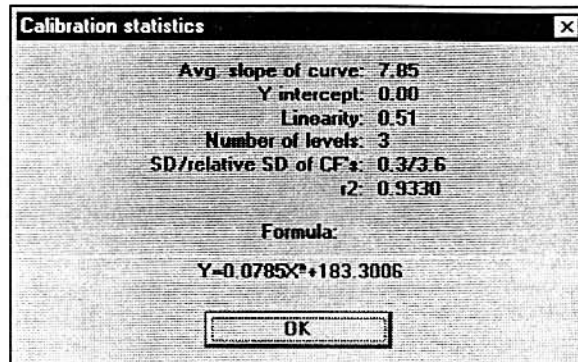
$$A = (X^2Y \cdot n) - (Y \cdot X^2) / D$$

$$B = (Y \cdot X^4) - (X^2Y \cdot X^2) / D$$

$$D = (X^4 \cdot n) - (X^2 \cdot X^2)$$

Notes:

This is a least squares fit algorithm over the calibration levels. A point at (0,0) is also assumed (by incrementing n) unless there is already a value at x=0, or if [Statistics]R2IncludeZero is set to 0 in the PEAKWIN.INI file. There must be at least 2 calibration levels (3 if the origin is not assumed).



Quadratic through origin:

The resulting calibration curve is defined as

$$y = Ax^2 + Bx$$

where:

x is external measure

y is area

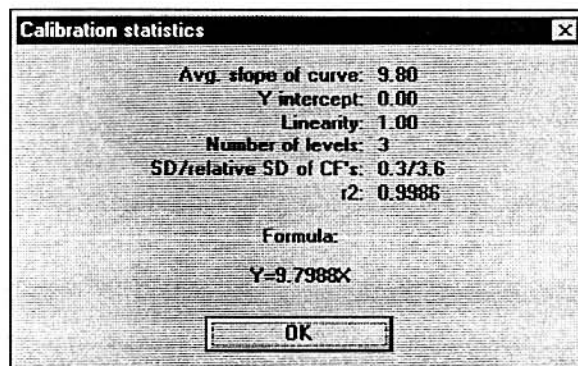
$$A = (XY \cdot X^3) - (X^2Y \cdot X^2) / D$$

$$B = (XY \cdot X^4) - (X^2Y \cdot X^3) / D$$

$$D = (X^3 \cdot X^3) - (X^4 \cdot X^2)$$

Notes:

This is a least squares fit algorithm over the calibration levels. There must be at least 2 calibration levels.



Quadratic:

The resulting calibration curve is defined as

$$y = Ax^2 + Bx + C$$

where:

x is external measure

y is area

$$A = ((XY \cdot X - Y \cdot X^2) \cdot (X^2 \cdot X^2 - X \cdot X^3) - (X^2Y \cdot X^2 - XY \cdot X^3) \cdot (X \cdot X - X^2 \cdot n)) / D$$

$$B = ((XY \cdot X^2 - Y \cdot X^3) \cdot (X^2 \cdot X^3 - X \cdot X^4) - (X^2Y \cdot X^3 - XY \cdot X^4) \cdot (X^2 \cdot X^2 - X \cdot X^3)) / E$$

$$C = ((XY \cdot X^2 - Y \cdot X^3) \cdot (X^3 \cdot X^3 - X^2 \cdot X^4) - (X^2Y \cdot X^3 - XY \cdot X^4) \cdot (X^2 \cdot X^2 - X \cdot X^3)) / F$$

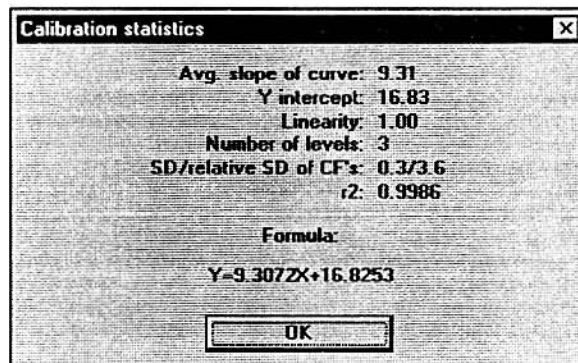
$$D = (X^3 \cdot X - X^2 \cdot X^2) \cdot (X^2 \cdot X^2 - X \cdot X^3) - (X^4 \cdot X^2 - X^3 \cdot X^3) \cdot (X \cdot X - X^2 \cdot n)$$

$$E = (X^2 \cdot X^2 - X \cdot X^3) \cdot (X^2 \cdot X^3 - X \cdot X^4) - (X^3 \cdot X^3 - X^2 \cdot X^4) \cdot (X \cdot X^2 - X^3 \cdot n)$$

$$F = (X \cdot X^2 - X^3 \cdot n) \cdot (X^3 \cdot X^3 - X^2 \cdot X^4) - (X^2 \cdot X^3 - X \cdot X^4) \cdot (X^2 \cdot X^2 - X \cdot X^3)$$

Notes:

This is a least squares fit algorithm over the calibration levels. A point at (0,0) is also assumed (by incrementing n) unless there is already a value at x=0, or if [Statistics]R2IncludeZero is set to 0 in the PEAKWIN.INI file. There must be at least 2 calibration levels (3 if the origin is not assumed).



The Calibration Window (continued)

The **Accept New** Button

If the new calibration data is acceptable, Click **Accept New** to update the calibration curve data.

The **Close** Button

Automatically saves the new calibration file and closes the Calibration window.

The **Load** Button

Click on the **Load** button to load an existing calibration file, designated with the **.CAL** file extension.

The **Save** Button

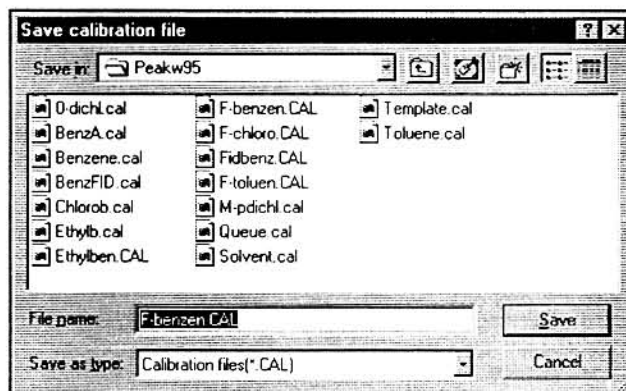
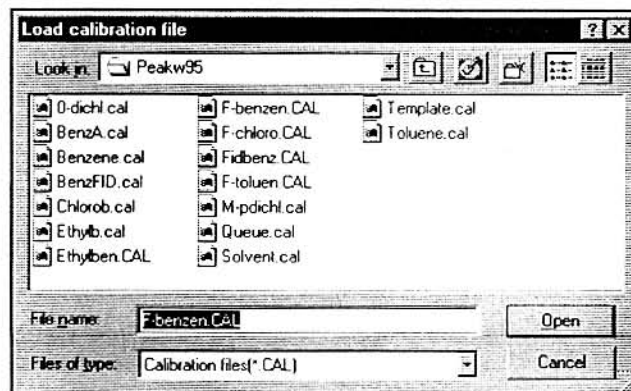
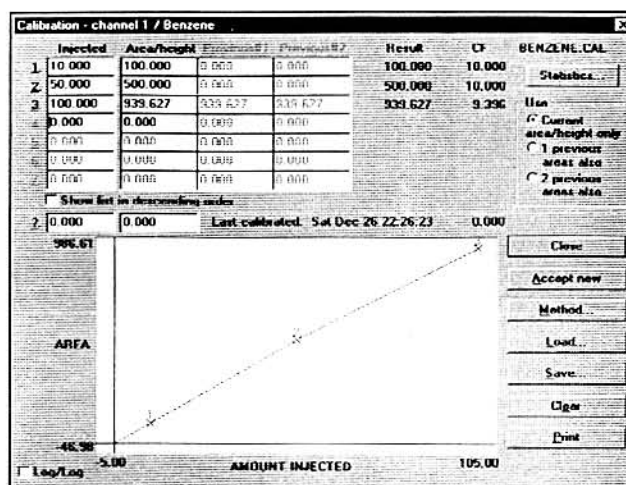
Click on the **Save** button to save a new calibration file, or to update an existing one. Remember to always use the **.CAL** extension when naming the calibration file. The saved file name appears at the top of the calibration window indicating the file in use.

The **Clear** Button

Clicking on the **Clear** button deletes all calibration data from the calibration window. The calibration file name is also removed.

The **Print** Button

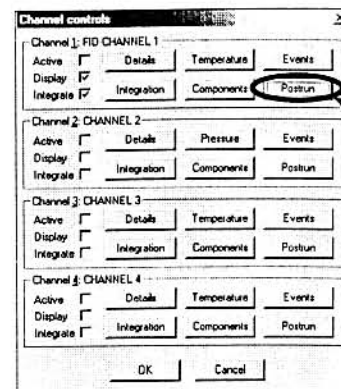
Clicking on the **Print** button sends the file data and the calibration curve information to the printer.



The Edit-Channels-Postrun Window

The Postrun Screen is used to determine all the actions that are to be done in PeakSimple after a chromatogram run. Clicking on the **Postrun** box for channel 1 in the Channel controls window will open up the Channel 1 post-run actions window.

Postrun

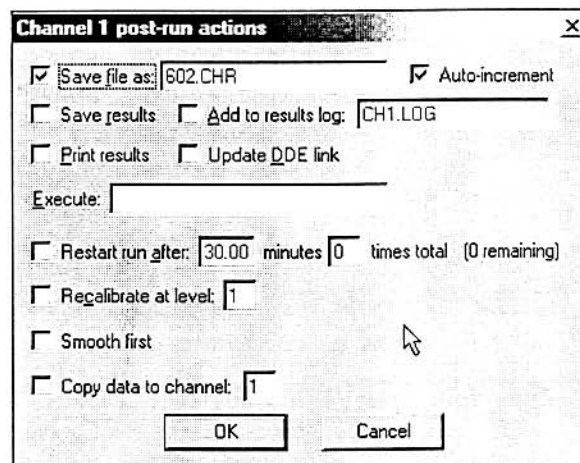


Save file as "X"

The Save file as checkbox, when selected, automatically saves a chromatogram file to disk after a run is completed. The file will be saved under the file name and path entered in the information field to the right of the checkbox.

Auto-increment

When selected, the Auto-increment checkbox will incrementally add a numerical digit to the entered filename after each run. For example, a chromatogram run saved as RUN.CHR would be saved as RUN1.CHR after the second run and RUN2.CHR after the third run.



The **Save results** checkbox when selected will save the data in the results screen to disk after a chromatogram run (*Note: This is not the raw data but instead is the ASCII results*). The **Add to results log "X"** checkbox adds the results of a run to the results log specified in the information field to its right. It will be saved under the same filename as the raw data but with the extension .RES, for example 602.RES. The **Print results** checkbox will print whatever is specified to be printed in the Print format window, this might include the chromatogram and its results data. The **Update DDE link** checkbox when selected will automatically update the Dynamic Data Exchange link once the run is completed.

Execute "X"

The Execute information field opens any executable file (.exe, .bat, .bas) after the chromatogram run is completed. *Note: Be sure to include the full filename and path for the executable file.* Control is returned to PeakSimple when the called application closes.

Restart run after "X"

The Restart run after "X" checkbox and information field restarts a chromatogram run after an inputted delay time. The delay time is inputted in minutes and can be repeated as many times as is entered into the times total information field. *Note: If 0 is entered into the times total information field then the run will be restarted an infinite number of times.*

The Edit-Channels-Postrun Window (continued)

Recalibrate at level "X"

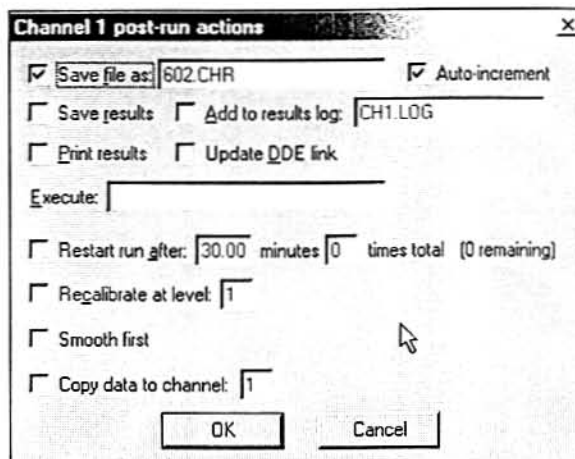
The Recalibrate at level "X" checkbox and information field recalibrates all identified peaks at the end of a run at a given level from 1 to 7. This feature is normally implemented as part of an autosampler queue. Detailed instructions are given in the Autosampler queue documentation section.

Smooth first

The Smooth first checkbox runs the smoothing algorithm as it was last applied to the chromatogram before the final integration is done. If the box is left unchecked no smoothing will be done to the chromatogram run.

Copy data to channel "X"

The Copy data to channel "X" checkbox and information field inputs the chromatogram run into whatever channel is selected in the information field. Only the values 1 to 4 can be inputted into the information field as there are four chromatogram channels in PeakSimple.



The Edit-Overall Window

The Overall controls window is used to define and control many of the options in PeakSimple. Clicking on **Edit** in the PeakSimple menu bar and then **Overall** from the drop down menu will open up the Overall controls window.

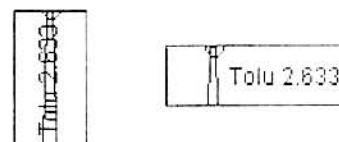
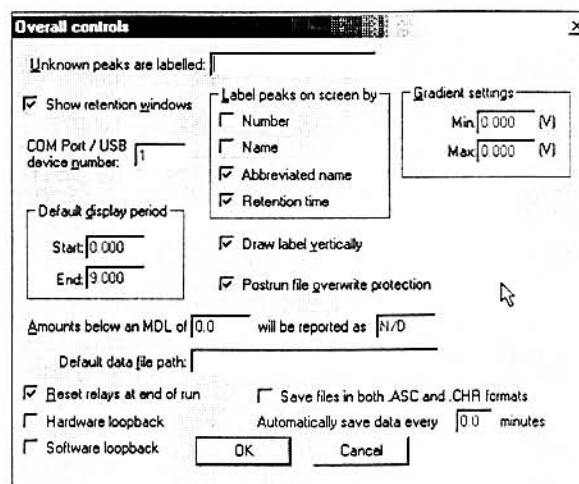
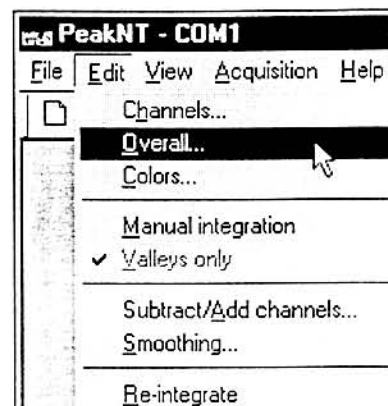
Unknown peaks are labeled "X"

The Unknown peaks are labeled information field, when filled out, labels all unknown peaks the value that is in the information field. If the word Peak was entered into the information field then all unknown peaks would be labeled Peak.

The **Show retention windows** checkbox is checked by default and thus retention windows are visible in PeakSimple; unchecking the Show retention windows checkbox removes the retention windows from sight. The **COM Port / USB device number "X"** information field specifies the COM port or USB device number that is to be used for the connection between PeakSimple and hardware. The COM port number is typically 1 or 2 while the USB device number is typically between 5000 and 9999.

Label peaks onscreen by

The Label peaks onscreen by options box enables a peak to be labeled by as many as four options. The **Number** checkbox labels all peaks with their peak number. The **Name** checkbox labels all peaks with their full name. The **Abbreviated name** checkbox labels all peaks with a shorter, four character abbreviated name while the **Retention time** checkbox labels peaks with their retention times. The **Draw label vertically** checkbox specifies whether peaks should be labeled horizontally or vertically on the chromatogram screen. When the box is checked the peaks labels will be drawn vertically when it is deselected they will be drawn horizontally.



Gradient settings

Gradient settings are only used when PeakSimple is controlling an SRI HPLC Pump. The **Min** and **Max** voltage settings are used to calibrate the Pump.

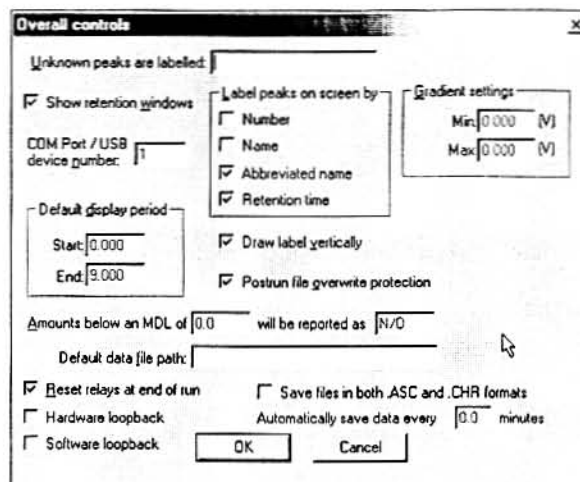
The Edit-Overall Window (continued)

Default display period

The default display period options box is used to define the default display limits for a PeakSimple chromatogram. The **Start** information field is used to specify the default beginning limits while the **End** field is used to specify the end to the default display limits. The start and end display limits can also be adjusted by the left and right arrows below the chromatogram in the main display window.

Postrun file overwrite protection

Postrun file overwrite protection protects a saved file from being written over when the auto-increment feature is selected in the Postrun window. Instead of writing over a used filename an auto-incremented run will select the next unused number in the sequence to save the file to disk. For example, if file TEST02.CHR already exists on disk PeakSimple will save the file as TEST03.CHR.



Amounts below an MDL of "X" will be reported as "Y"

Peaks with a value below a specified Minimum Detection Level or MDL will be reported as whatever is specified in the second information field, typically N/D or not detected. The number that is below the MDL will not be reported, only the entry in the second information field will be seen.

Default data file path

Typically all PeakSimple files are saved to the PeakSimple directory but by entering a full directory path into the Default data file path information field another directory can be selected to save files to. *Note: It is recommended that users save all PeakSimple files to the PeakSimple directory. If necessary export files to a different directory after saving them to the PeakSimple directory.*

Reset relays at end of run

The Reset relays at end of run checkbox when selected turns off all relays (A-H) at the end of a chromatogram run. If the box is left unselected the relays will not be shut off after a chromatogram run.

Hardware loopback and **Software loopback** are used for system validation and will be discussed in further detail in the Loopback test section.

The Edit-Overall Window (continued)

Save files in both .ASC and .CHR formats

The Save files in both .ASC and .CHR formats checkbox when selected saves files in the .ASC format (ASCII) and the .CHR format (chromatogram). If the checkbox is not selected files will be saved only in the .CHR format.

Automatically save data every "X" minutes

The Automatically save data every "X" minutes checkbox and information field when selected saves the data during a chromatogram run at intervals specified by the information in the information field. This feature is useful for runs where power outages are frequent and data cannot be lost.

Overall controls

Unknown peaks are labelled: ☐

☒ Show retention windows

COM Port / USB device number:

Default display period:

Start: 0.000

End: 9.000

Label peaks on screen by:

☐ Number

☐ Name

☒ Abbreviated name

☒ Retention time

Gradient settings:

Min: 0.000 (M)

Max: 0.000 (M)

☒ Draw label vertically

☒ Postrun file overwrite protection

Amounts below an MDL of 0.0 will be reported as N/D

Default data file path:

☒ Reset relays at end of run

☐ Save files in both .ASC and .CHR formats

☐ Hardware loopback

☐ Software loopback

Automatically save data every 0.0 minutes

OK Cancel

The Edit-Colors Window

The Colors window determines the color schemes that are to be used throughout PeakSimple. Open the Colors window by selecting **Edit** from the PeakSimple menu bar and then **Colors** from the list of options.

Selecting the **Background** button with the mouse cursor opens up the Background color window. The background color can be chosen from a set of 48 colors by selecting a color and then affirming the choice by clicking on the OK button.

The Graph background window is opened up by selecting the **Graph background** button in the Colors window. The graph background color is changed by selecting a color and then clicking on the OK button to make the color change.

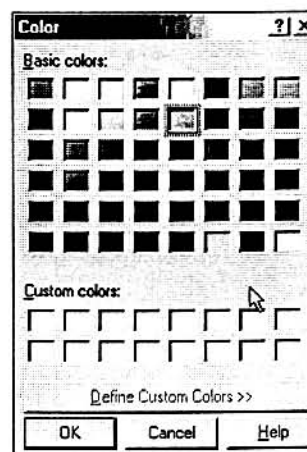
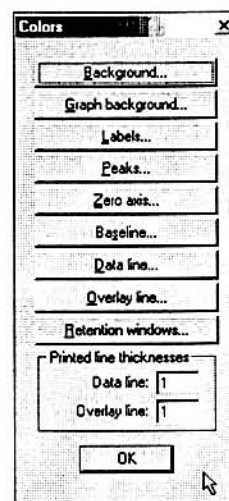
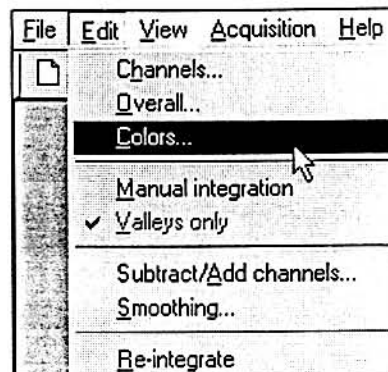
The color of the labels controls the color of the words that belong to the peaks. The color of the labels is changed by selecting the **Labels** button to open up the Labels color window. In the Labels color window select a color and then press on the OK button to make the change to the labels color.

The peak color is the color of the circle at the top of each identified peak and is determined by the Peak color window which is opened up by selecting the **Peak** button in the Color window. Select the desired peak color and then click on the OK button to close the window and affirm the change.

The color of the zero axis is chosen by clicking on the **Zero axis** button and then selecting a color from the Zero axis color window. Clicking on the OK button closes the window and makes the change to the color of the zero axis. Don't set the Zero axis color to the same color as the Graph background because they won't be distinguishable from each other.

The baseline is the line that runs along the bottom of the peaks and its color is changed by selecting the **Baseline** button and then choosing a color from the Baseline color window. The change is made once the OK button is selected and the window is closed.

The data line is the signal line that makes up the peaks in PeakSimple and its color is defined by selecting the **Data line** button in the Colors window and then selecting a color from the Data line colors window. Once the desired color is selected apply the color change by clicking on the OK button to close the window.



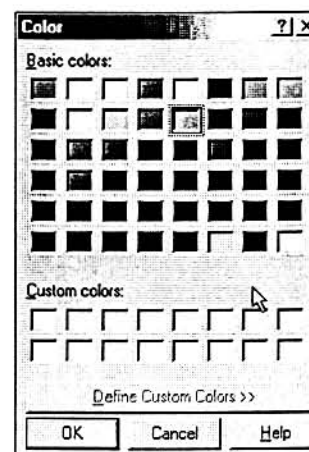
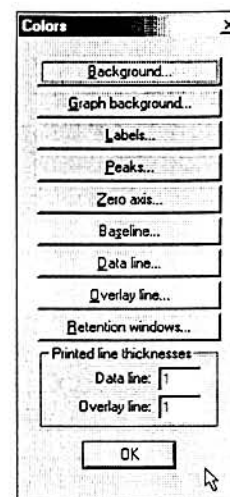
The Edit-Colors Window (continued)

The overlay line is a data line from a chromatogram that has been overlaid on top of an existing chromatogram and its color is changed by selecting the **Overlay line** button in the Colors window and then selecting a color with the mouse cursor in the Overlay line colors window. The color changes are made once the OK button is selected and the window closes.

Retention windows are the horizontal bars that appear onscreen and their color can be changed by clicking on the **Retention windows** button in the Colors window and then selecting the desired color in the Retention windows colors window. To apply the color changes click on the OK button to close the window.

Printed line thickness

The thickness of the Data line and the Overlay line when a chromatogram is printed is determined by the **Data line** information field and the **Overlay line** information field. The thickness of the Data line is determined by the numerical value in the Data line information field, larger numerical values will result in thicker lines. The thickness of the Overlay line is also determined by the numerical value in its information field. Larger numbers in the information field will result in a thicker overlay line.



Manual Integration

The manual integration tools are used to manually draw in the baseline in a PeakSimple chromatogram. The manual integration toolbar is opened up by selecting **Edit** from the PeakSimple menu bar and then clicking on the **Manual integration** option. The manual integration toolbar appears to the right of the PeakSimple toolbar in the upper right hand corner of the screen.

Off Integration Tool



The Off integration tool or the mouse cursor is used to end a manual integration mode once it has been selected. When the mouse cursor icon is selected no more changes to the baseline of a chromatogram can be performed until another manual integration tool is selected.

None Integration Tool



The None integration tool adds the area of one peak to the area of an adjacent peak. Once the None integration tool is selected click on a valley between two peaks with the mouse cursor to change the baseline.

Drop Integration Tool

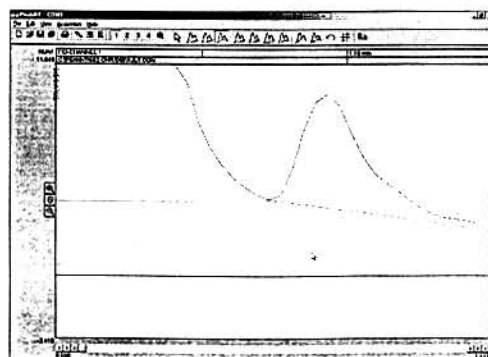
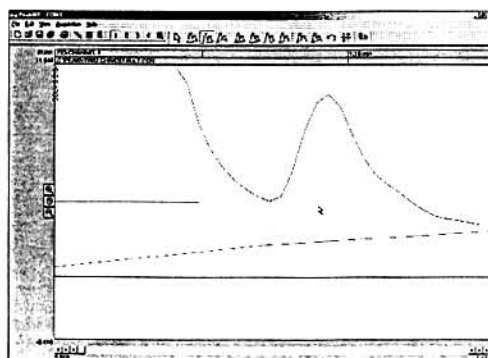
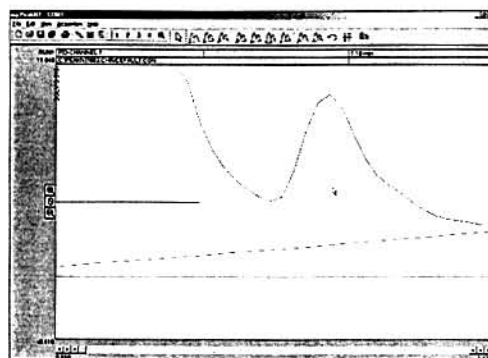
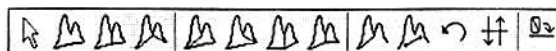
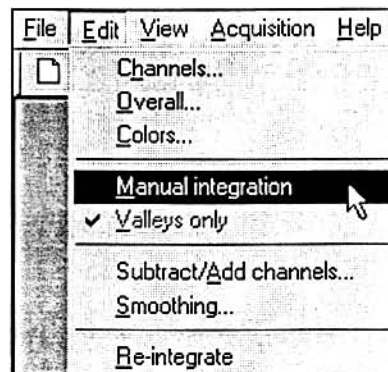


The Drop integration tool drops the baseline between two peaks straight down onto an existing baseline. The Drop integration tool is used by selecting the Drop tool in the manual integration toolbar and then clicking on a valley between two peaks to change the baseline.

Based Integration Tool



The Based integration tool raises the baseline to a valley between two specified peaks. To change the baseline select the Based tool and click on a peak with the mouse cursor to raise the baseline up to the valley.

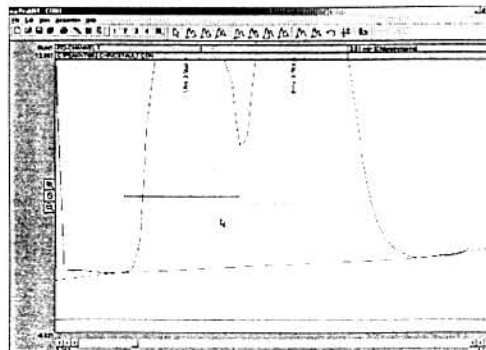


Manual Integration (continued)

Lead Skim Integration Tool



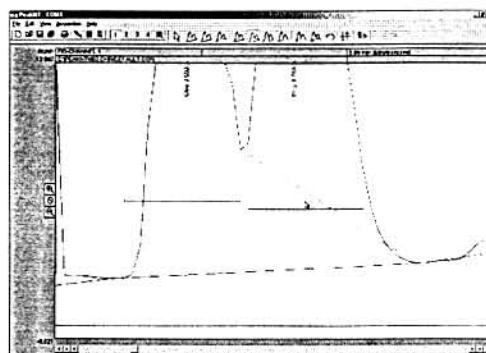
The Lead skim integration tool skims a peak's area off of the leading edge of an adjacent peak. To skim a peak off of the leading edge of another peak select the Lead skim tool from the manual integration toolbar and then click on the valley between the two specified peaks with the mouse cursor.



Trail Skim Integration Tool



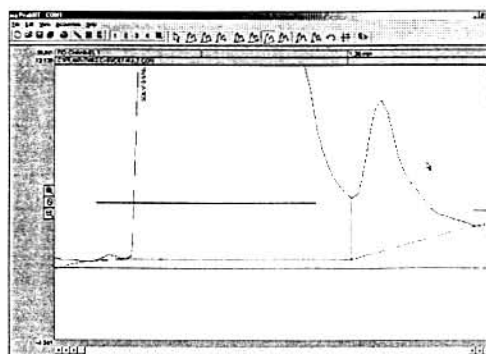
The Trail skim integration tool skims a peak's area off of the trailing edge of another, adjacent peak. To skim a peak off of the trailing edge of another peak select the Trail skim tool and click on a valley between two peaks with the mouse cursor to make the change.



Lead Horizontal Integration Tool



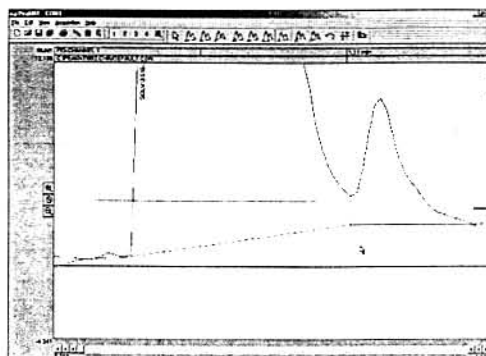
The Lead horizontal integration tool draws the baseline horizontally for the leading peak while the trailing peak's baseline stretches from the horizontal line to the next valley. The Lead horizontal tool is selected in the manual integration toolbar and once a valley is selected the change to the baseline is made.



Trail Horizontal Integration Tool



The Trail horizontal integration tool draws the baseline horizontally for the trailing peak while the leading peak's baseline stretches from the horizontal line to the previous valley in the chromatogram. The Trail horizontal tool is used by selecting the Trail horizontal tool in the manual integration toolbar and then clicking on a valley with the mouse cursor to make the change.

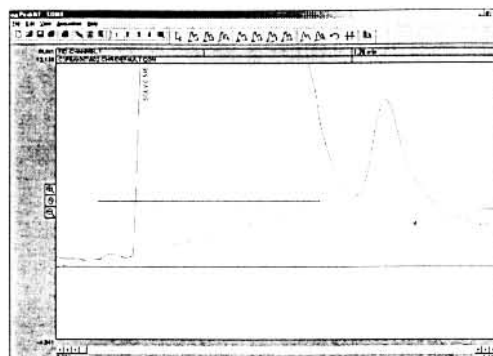


Manual Integration (continued)

Inhibit Integration Tool



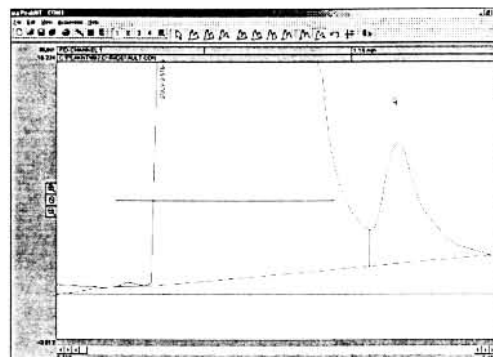
The Inhibit integration tool ends a baseline after a valley thereby stopping the peak's area from being counted along with the rest of the chromatogram. To use the Inhibit tool select the tool in the manual integration toolbar and then click on the valley between two peaks to end the baseline.



Rubber Band Integration Tool



The Rubber band integration tool is used to manually draw the baseline in a chromatogram. The Rubber band tool is selected in the manual integration toolbar and is clicked and dragged on the chromatogram to draw in the baseline.

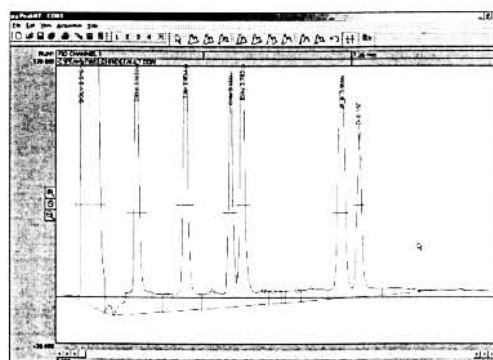


Undo Integration Tool



The Undo integration tool removes all changes done to the baseline of a chromatogram with the manual integration tools. To use the Undo tool click on the tool in the manual integration toolbar and all changes will be undone.

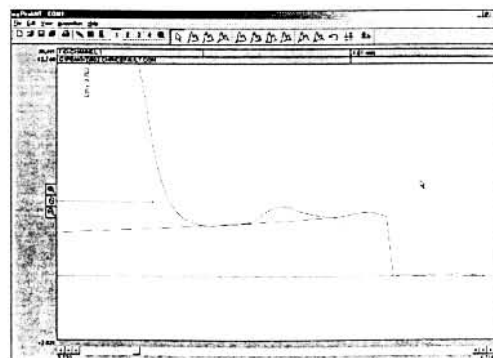
Note: Changes made to a chromatogram with the Reverse and Zero integration tools cannot be undone with the Undo tool.



Reverse Integration Tool



The Reverse integration tool inverts a selected peak or a selected group of peaks in a chromatogram. A peak is inverted by selecting the Reverse tool in the manual integration toolbar and then clicking and dragging the mouse cursor over the peak.



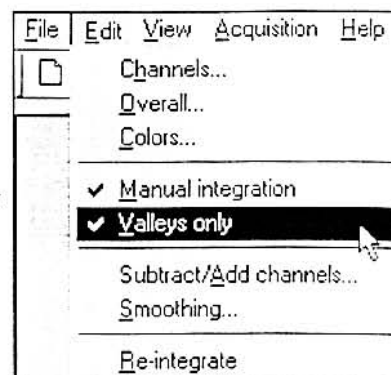
Zero Integration Tool



The Zero integration tool sets the value of the data line at zero starting at a selected point. To zero the data line at a given point select the Zero tool from the manual integration toolbar and click on the data line with the mouse cursor.

The Edit-Valleys Only Option

The Valleys only option is available only when the Manual integration toolbar is open in PeakSimple. The Valleys only option can be selected by opening up the Manual integration toolbar in the Edit menu and then selecting the Valleys only option immediately below Manual integration in the drop down menu. When the Valleys only option is selected all changes made to the baseline of a chromatogram will snap only to the valleys of the chromatogram. When the Valleys only option is turned off changes made to the baseline of a chromatogram will go to wherever the mouse cursor was clicked.

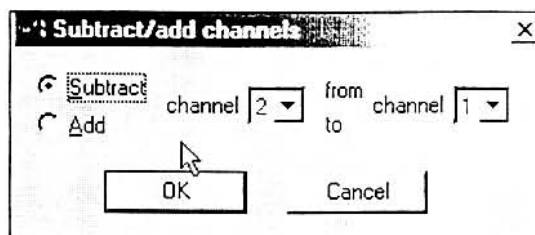
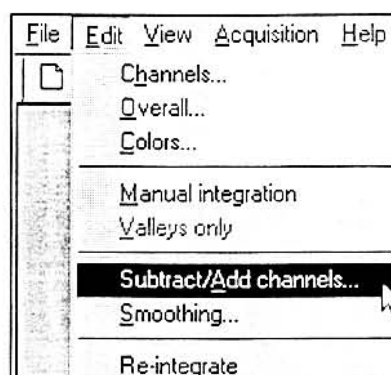


The Edit-Subtract/Add Channels Menu

The Subtract/Add channels menu removes or adds the analog data signal from/to one channel in PeakSimple from/to another channel. The Subtract/Add channels menu is opened by selecting the Edit menu and then by clicking on Subtract/Add channel in the drop down menu.

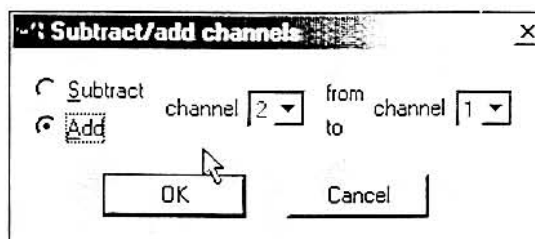
Subtracting a Channel

To subtract one channel from another channel click on the Subtract radio button with the mouse cursor and select the channel that is to be taken away in the first dialogue box. In the second dialogue box select the channel that is to have the first selection taken away from. Click on OK with the mouse cursor to effect the changes.



Adding a Channel

To add one channel to another channel select the Add radio button in the Subtract/Add channels menu. Select the channel that is to be added by selecting a number in the first dialogue box and then choose the channel that it is to be added to by selecting a number in the second dialogue box. All changes are made once the OK button is selected.



The Edit-Smoothing Window

The Data smoothing window determines all the smoothing options that are to be performed on a data line. The Data smoothing window is opened up by selecting Edit from the PeakSimple menu bar and then selecting Smoothing from the list of options.

The **Source channel** dialogue box specifies which channel the data line that is to be smoothed is in. The **Destination channel** is the channel that the smoothed data line from the source channel will be displayed in.

Method

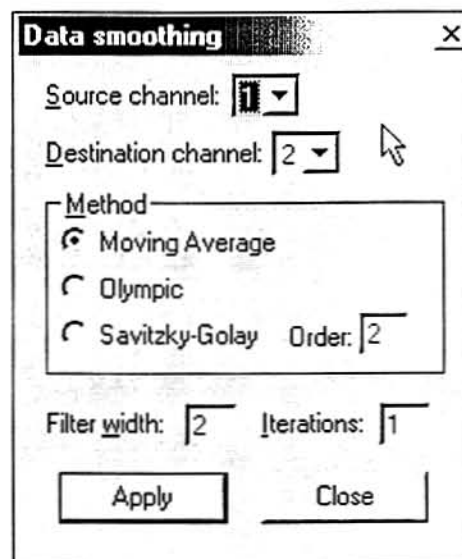
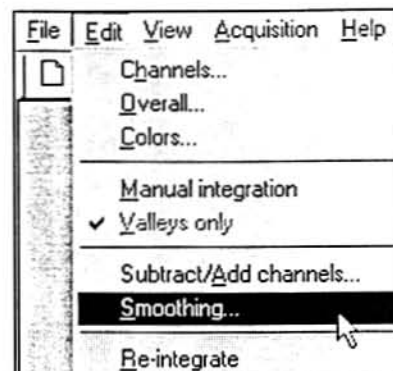
The method of smoothing is determined by the smoothing algorithm selected in the Method box. The **Moving Average** algorithm sets each sample to the average of the samples around it including itself. The number of samples taken into account depends on the Filter width. The **Olympic** algorithm is similar to the Moving Average but the highest and lowest values in the set of samples are discarded before the average is taken. The **Savitsky-Golay** algorithm is similar to the Moving Average but each of the samples is weighted according to a set of weighting factors. Increasing the number in the **Order** dialogue box gives more weight to the central samples when using the Savitsky-Golay method.

Filter Width

The Filter width dialogue box controls the number of samples that are to be taken into account when using the Moving Average smoothing method. A filter width of 2 means that 2+1+2 samples are taken while a filter width of 5 means that 5+1+5 samples are taken.

Iterations

The Iterations dialogue box controls the number of times a smoothing method is to be applied to a chromatogram peak. Every iteration smooths the data line more than the previous iteration eventually making the data line flat.



The Re-Integrate Option

The Re-integrate option is used to fully re-integrate a baseline in PeakSimple. When changes are made to a baseline often a partial integration will occur, selecting Re-integrate will perform a full integration on the baseline. The Re-integrate option can be selected by clicking on Edit in the PeakSimple menu bar and then Re-integrate from the list of options.

The View-Results Window

The Results window displays the results of the chromatogram runs performed in PeakSimple. The Results window is opened up by clicking on View in the PeakSimple menu bar and then selecting Results from the list of options.

The **Channel** option scrollbar specifies which of the four channels the results data should be displayed for. When the **Recognized peaks only** checkbox is selected only the results for named peaks will be displayed. The **Undetected components also** checkbox displays the results for the undetected components as well as the detected components in the chromatogram run when the option is selected.

Update

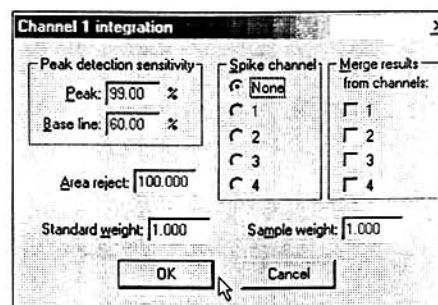
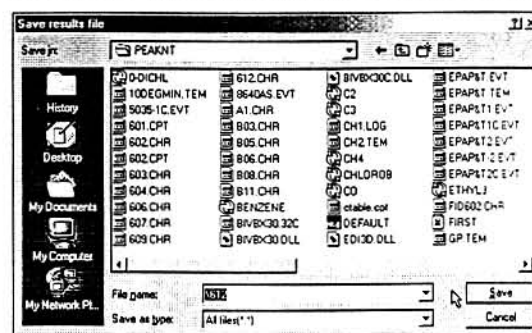
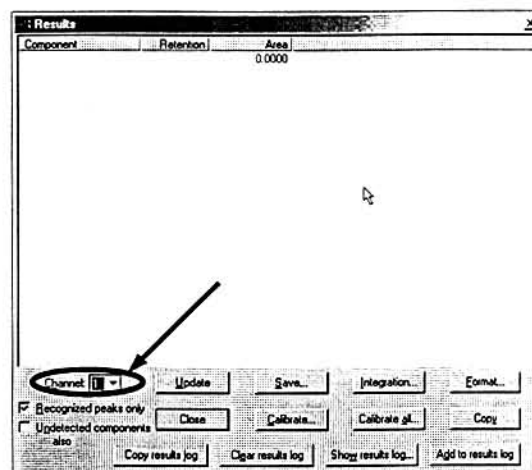
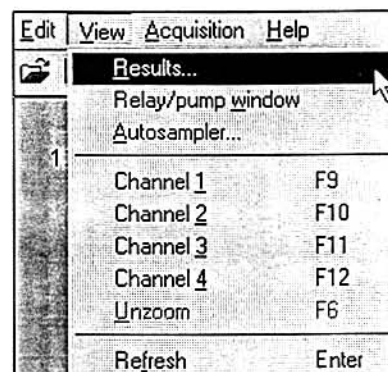
The Update button in the Results window updates the DDE link between the Results data and the DDE host program (typically Excel).

Save

Selecting the Save button in the results window opens up the Save results file window. In the Save results file window the results file is saved with a .res extension. The file is an ASCII file and not the raw chromatogram data.

Integration

As a convenience the integration button in the results window opens up the same Integration window that can be accessed in the Channels window. For more information on the Integration window consult the Channels-Integration portion of this manual.



The View-Results Window (cont.)

Format

Selecting the Format button in the Results window opens up the Edit format window. The Edit format window allows the user to specify the information that is to be included in the Results table.

The **Available** options box in the Edit format window displays all the available options that can be included in the results but that aren't selected. An option is added to the **Selected** options box by highlighting the item in the Available box and clicking on the right facing arrow button. To deselect an option from the Selected box highlight the item and click on the left facing arrow button. The **Dec. places** dialogue box specifies how many decimal places a highlighted unit will display in the Results table.

Close

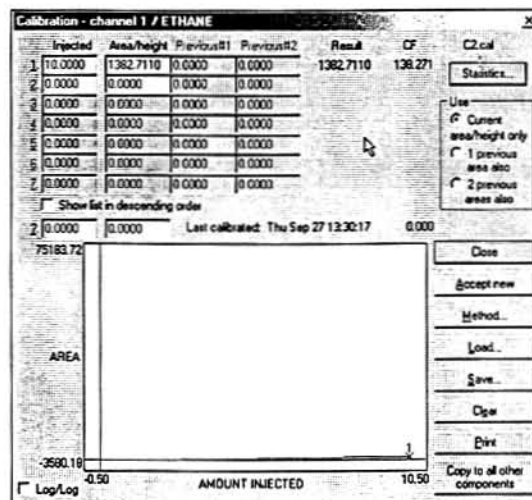
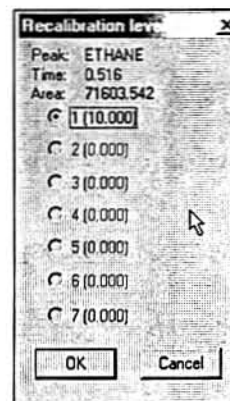
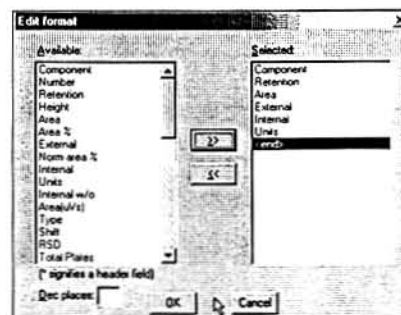
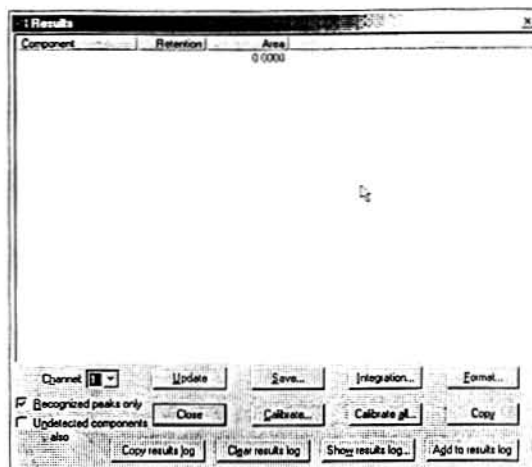
The Close button exits the Results window and returns the user to the main screen.

Calibrate

The Calibrate button recalibrates a recognized peak in the Results table. Highlighting a peak name and selecting the Calibrate button opens up the Recalibration Level window. The window specifies which peak level should be calibrated. Following the Recalibration level window is the Calibration window which is discussed at further length in the Calibration section of this document.

Calibrate All

The Calibrate all button recalibrates all the recognized peaks at once. The Calibrate all button calibrates all peaks with existing calibration curves on a particular calibration level. If named peaks are in the results table without calibration curves an error message, (NOT ENOUGH DATA POINTS), will be displayed. The calibration will



The View-Results Window (cont.)

Copy

The Copy button in the results window copies the results report to the Clipboard. Once the report is copied it can be pasted into other programs i.e. Excel.

Copy Results Log

The Copy results log button copies the .log file for the results to the Clipboard. This log file can be pasted into any Windows program. A certain number of lines in the results log will always be copied, by default the number is 20. If more than 20 lines are needed for an application the user must modify the peakwin.ini file located in the Windows folder. The default entry in the file is (SpareLines=20), delete the number 20 and insert the number of lines that are needed (up to a maximum of 100).

Clear Results Log

Clicking on the Clear results log button erases the results log file.

Show Results Log

The Show results log button when selected opens up Windows Notepad to view the results log.

Add to Results Log

To add the current report to the results log click the Add to results log button. The report can automatically be added to the results log at the end of each chromatogram run by checking the Add to results log checkbox in the Postrun window.



The screenshot shows a window titled 'CHN LOG - Notepad'. It contains a table with the following data:

File	Date	Time	ETNAME	RT	Area	Height
002.CHN	12/14/1999	14:29:01	"ETNAME"	8.519	71042.5428	517.8489
002.CHN	12/14/1999	14:29:01	"ETNAME"	8.519	71042.5428	517.8489
002.CHN	12/14/1999	14:29:01	"ETNAME"	8.519	71042.5428	517.8489

The View-Relay/Pump Window

The Relay/pump window manually controls the actions of the relays in PeakSimple. The Relay/pump window is opened up by opening the View menu and then selecting Relay/pump window from the list of options.

Selecting/Deselecting a Relay

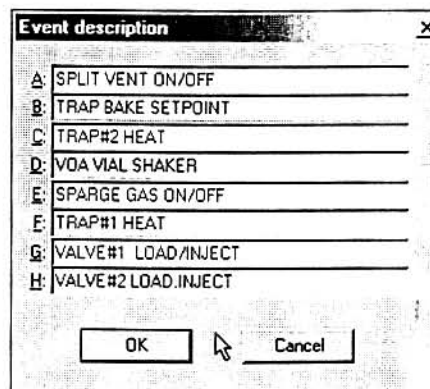
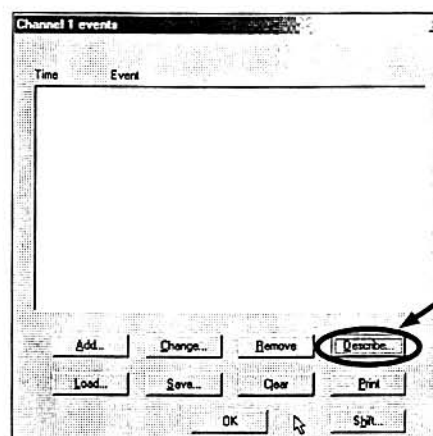
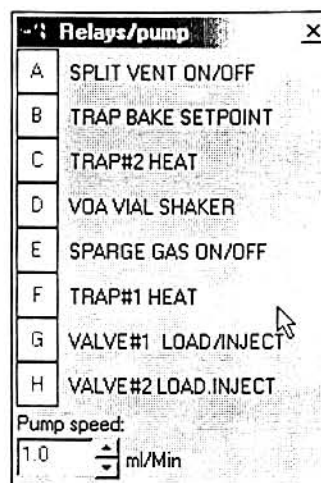
To manually activate a relay click on the letter next to the relay label to make the button dark. To deactivate a relay select the specified lettered button to turn it black. Pressing the control button and the letter corresponding to the relay together also selects/deselects the relay.

Pump Speed

When an SRI HPLC pump is connected to the data system the pump speed can be controlled in the Relay/pump window. To change the pump speed click on the arrow icons to increase or decrease the pump speed. The pump speed can also be entered by highlighting the value in the box and typing in the new number.

Describing a Relay

To label a relay in the Relay/pump window right click on the main screen and select Events from the list of options. Once the Events window is opened up clicking on the Describe button opens up the Event description window. To enter a relay description click on the specified relay's dialogue box and type in the information. The description of the relays has no effect on the relay function and will not affect hardware.

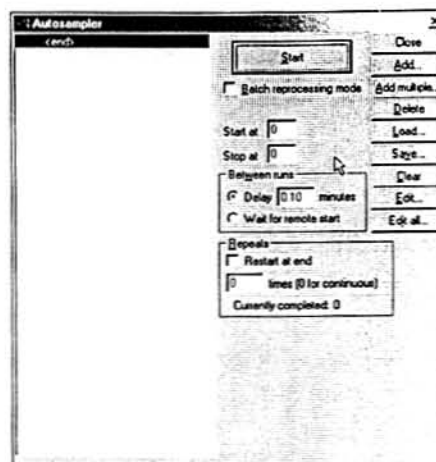


The View-Autosampler Window

The Autosampler window allows a list of control files to be run automatically. Control files are the master files which specify all parameters including temperature programming, component, and event files. These control files run tasks in PeakSimple. To open up the Autosampler window click on the View menu in the menu bar and then select Autosampler from the available options.

Start/Stop

The Start button when pressed begins the operation of the autosampler queue or reprocessing queue. A queue must be created or loaded before the control files can run. Once the autosampler is in operation the Start button changes into the Stop button. The Stop button ceases the autosampler operations that were previously running.



Batch Reprocessing Mode

To select Batch reprocessing mode click on the check box to the options left. While using the Batch reprocessing mode the user loads a list of previously stored chromatogram files in the list box to the left and then selects a control file which will reprocess the data files. When the operation begins PeakSimple will load each data file in the list into channel 1, perform the specified functions, and then increment to the next data file in the list.

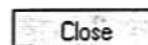
The **Start at** dialogue box specifies which control file number to begin operation at first. If no number is entered the autosampler will begin at the first control file. The **Stop at** dialogue box specifies the last control file to be run before operations of the autosampler cease. If no number is entered in the dialogue box the autosampler will end after the last control file in the list is run.

The **Delay "x" minutes** radio button when selected specifies how many minutes PeakSimple will wait before running the next control file in the list box. The **Wait for remote start** radio button when selected instructs the autosampler to wait for a remote start signal before advancing to the next control file.

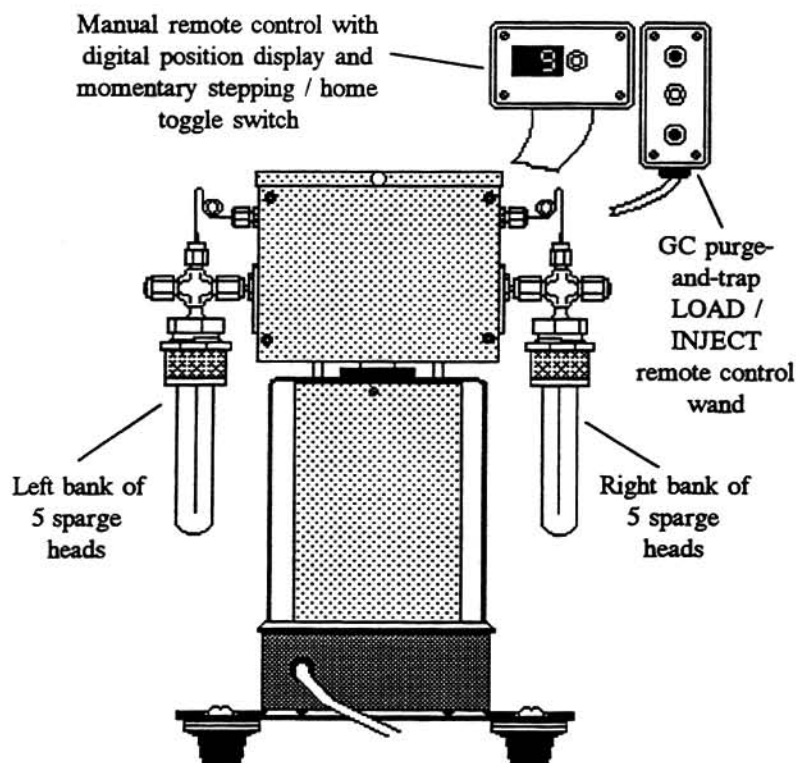
The **Restart at end** checkbox restarts the queue after getting to the end of the control files in the list box. In the **"x" times** the user enters the number of times the control files in the list box should be cycled if the Restart at end checkbox is selected. If the value 0 is selected the queue will be cycled continuously.

Close

The Close button closes the Autosampler window when it is selected.



The SRI 10 Station Purge-and-Trap Autosampler permits the unattended sparge, concentration, and analysis of up to 10 separate water and / or soil-in-water samples, when used in conjunction with the SRI EPA-Style Automated Purge-and-Trap Sample Concentrator option available as a built-in option for all SRI 8610C gas chromatographs. The PeakSimple data system (or other data system offering timed event control of external events via relays) is required for automated operation of this



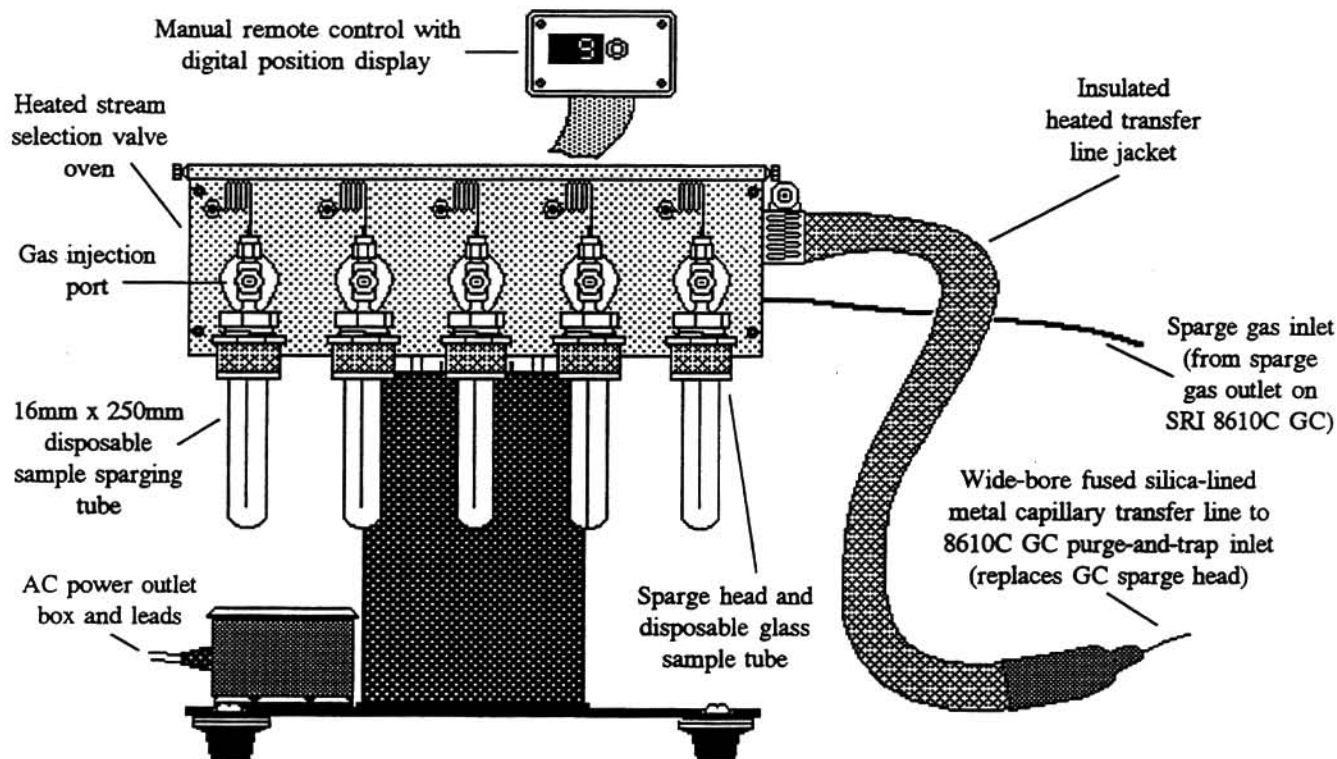
10 STATION PURGE-AND-TRAP AUTOSAMPLER SIDE VIEW

system. Manual controls are also provided for direct, manual control by the analyst, including a remote cabled sample position stepping control that features a digital LED display of the sample position in use. The toggle switch on this control has two momentary-on positions. Pushing the toggle switch to the up momentary-on position causes the automated sample stream selection valve to step to the next sample in order. Pushing the toggle switch down causes the sampling valve to return to the home (sample sparge head 1) position. The sample sparge heads are numbered according to their sampling order. A stainless steel knurled fitting holds the disposable glass sample tubes in place. Teflon ferrules in the knurled fitting seal the sample tubes in place, preventing gas leaks.

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

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

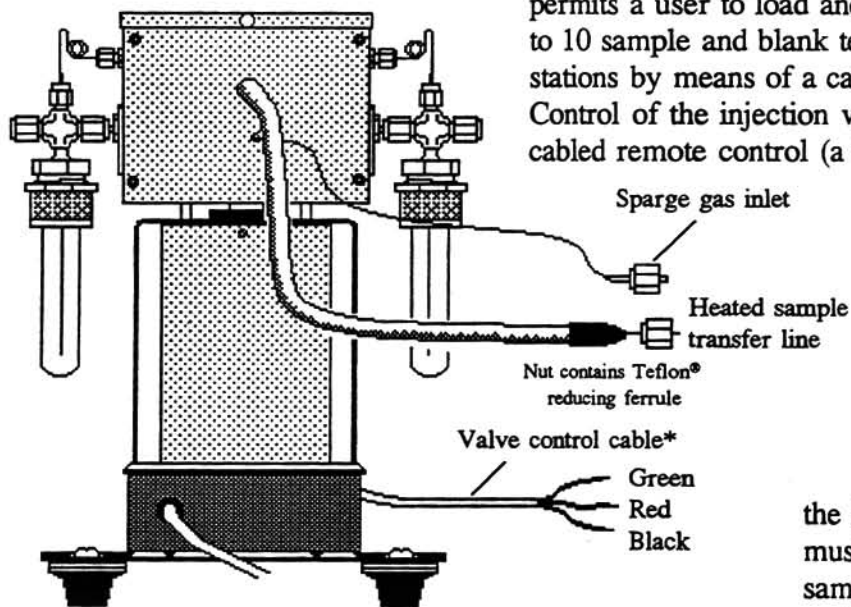
As illustrated below, the SRI 10 Station Purge-and-Trap Autosampler is configured in a symmetrical, space-saving bilateral design. Located along each side of the unit are 5 sparge heads with respective headspace injection ports. The autosampler should be located to the left side of the SRI 8610C GC for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



SRI 10 STATION PURGE-AND-TRAP AUTOSAMPLER FRONT VIEW

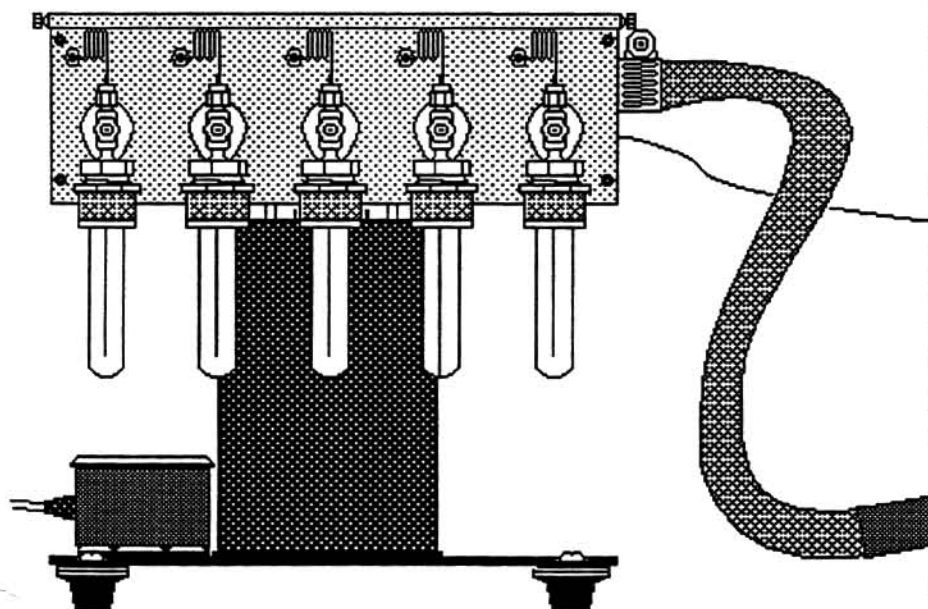
allows the analyst to have access to each sample sparging head and injection port, and to move the autosampler around on the lab bench as needed, while maintaining all connections and operability. As the autosampler is a stand-alone unit, it does not require a hard attachment or bracket for GC mounting.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

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



SIDE VIEW SHOWING CONNECTIONS

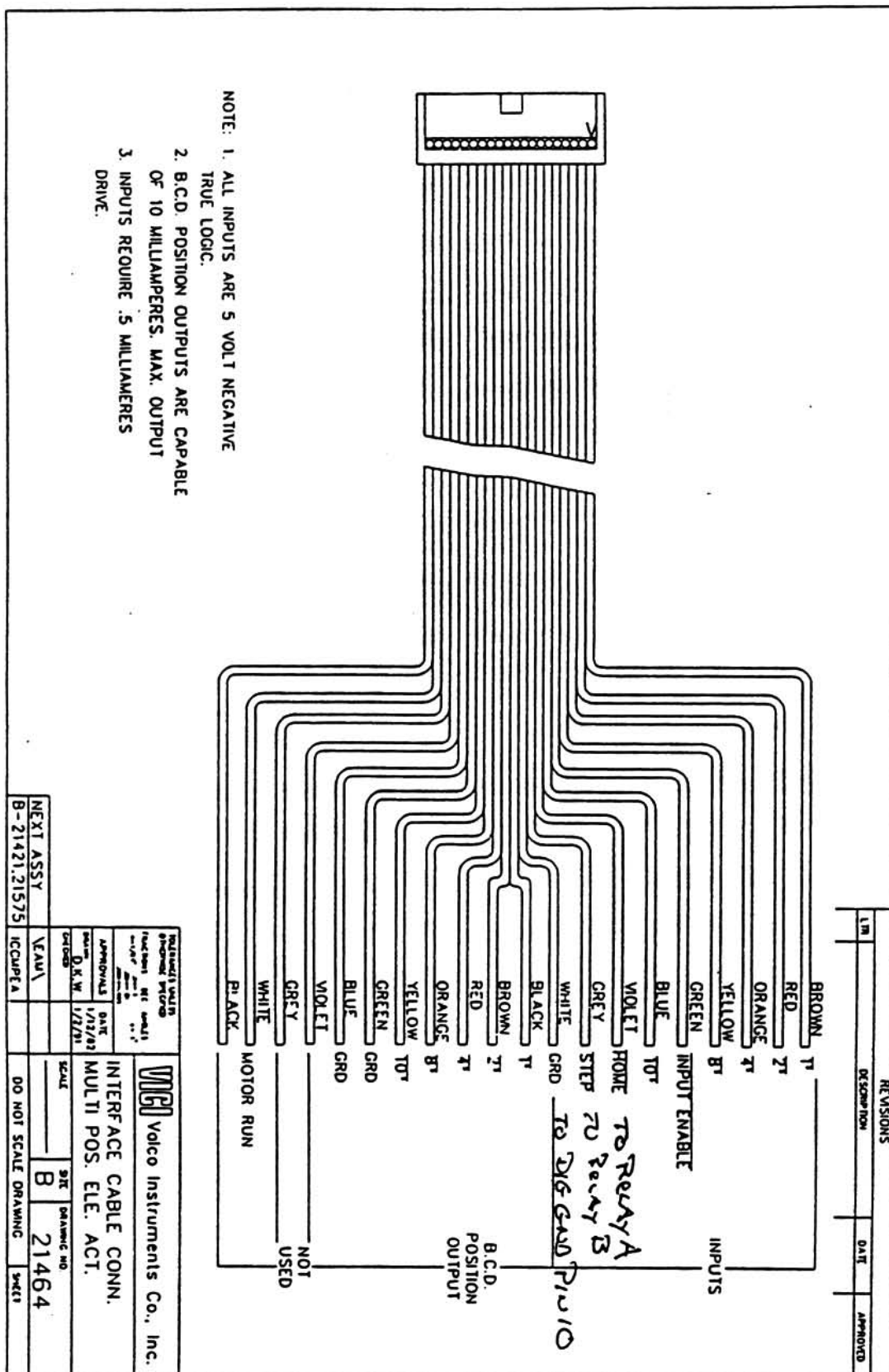
* for information regarding rainbow ribbon cable used for remote control station selection, see INJECTOR & GAS VALVE section of this manual.



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

The SRI 10-station purge and trap autosampler option for the SRI gas chromatograph is a free-standing purge and trap system that may be connected to any purge-and-trap concentrator-equipped brand of gas chromatograph in a matter of minutes. The system permits a user to load and sparge any combination of up to 10 sample and blank test tubes and select sampling stations by means of a cabled digital remote control. Control of the injection valve is performed by another cabled remote control (a three-wire cable is also provided

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



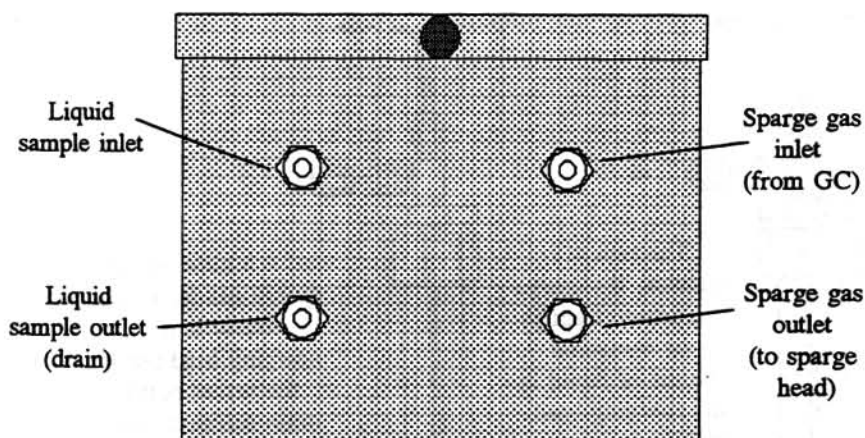
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The on-line liquid sampler accessory for the SRI purge-and-trap sampling system is an external unit designed to deliver, purge, and evacuate a liquid sample from the SRI purge-and-trap sample concentrator, on a repetitive basis, under data system automation.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

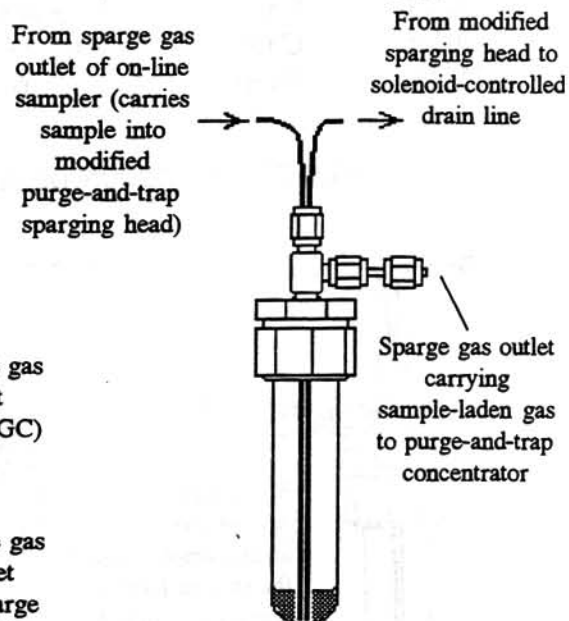
1. Locate the three cables exiting the rear of the on-line liquid sampler. Find and connect the AC supply cable to an available AC wall outlet. A second cable terminates in a remote control wand for the optional manual operation of the liquid sampling valve. Operation of this control is by means of the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

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

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



FRONT VIEW OF ON-LINE
LIQUID SAMPLER ACCESSORY



MODIFIED SPARGE
HEAD FOR
PURGE-AND-TRAP
SAMPLER

Chapter: PURGE-AND-TRAP

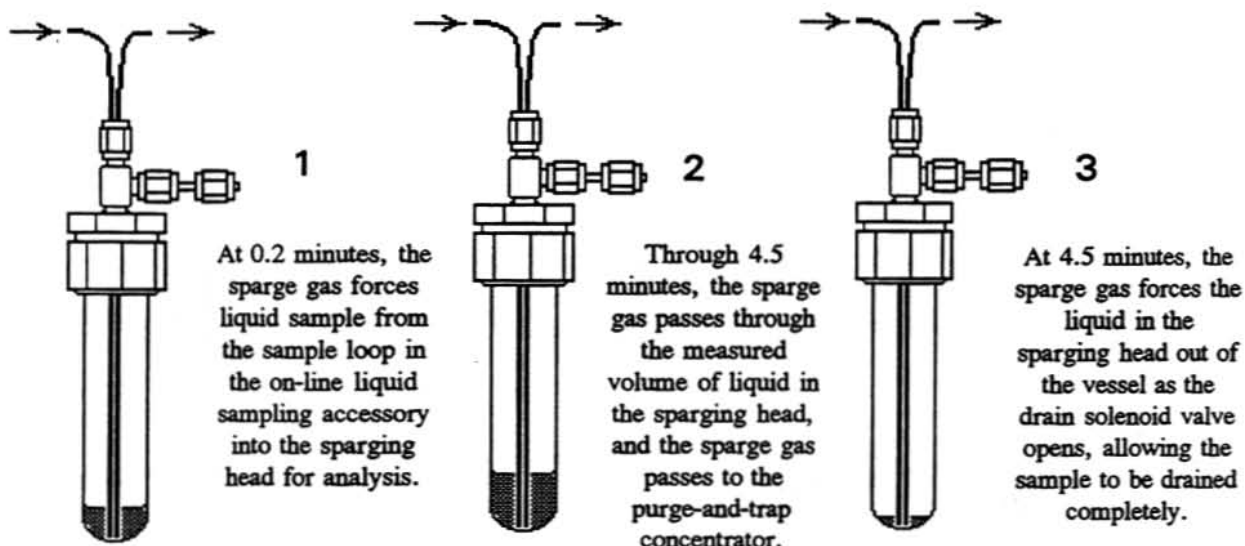
Topic: Connecting The On-Line Liquid Sampler To An Existing SRI P-&-T (con't.)

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

TIMED EVENT TABLE FOR ON-LINE LIQUID SAMPLING ACCESSORY

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

SIMPLIFIED PROCESSION OF OPERATION - ON-LINE LIQUID SAMPLING ACCESSORY



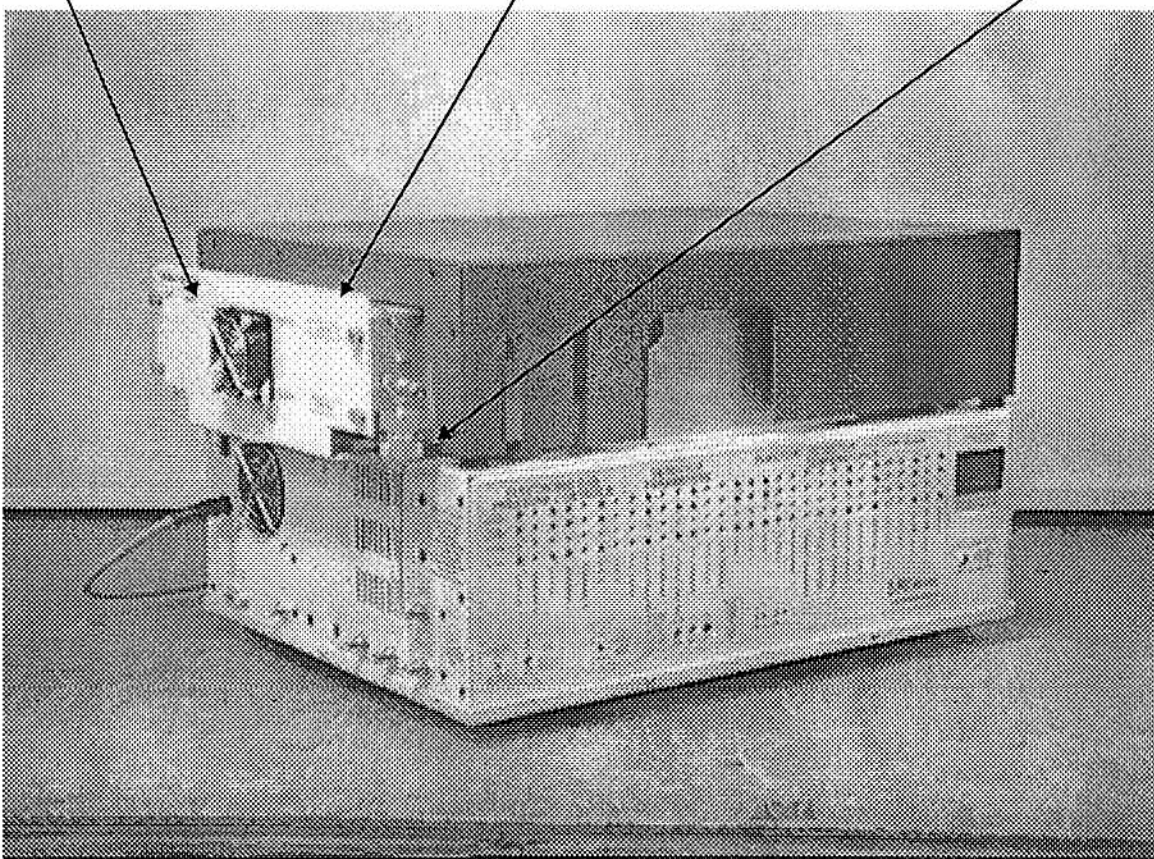
Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

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

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

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



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

Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

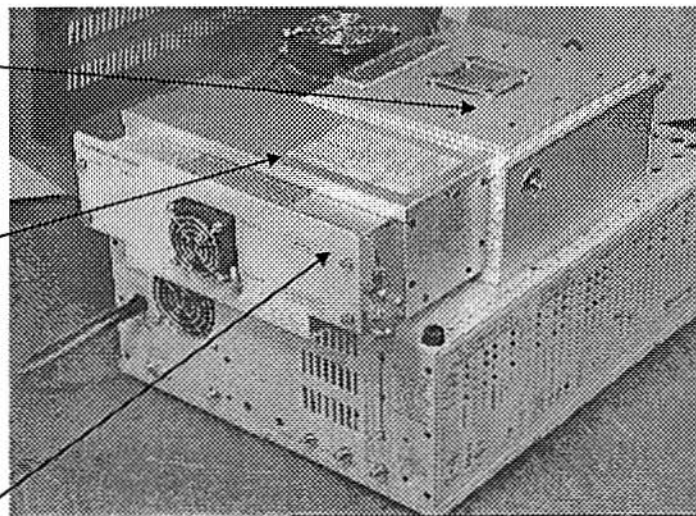
Column oven

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

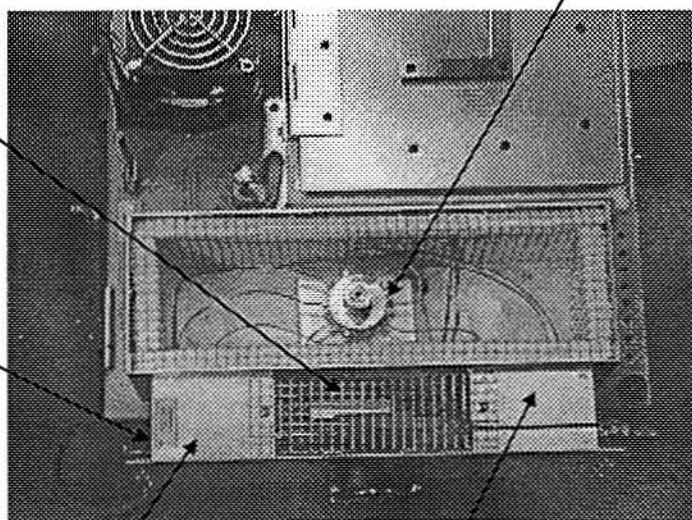
To change traps this cover plate must be removed by loosening the four brass thumbscrews located at the corners.

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

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



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



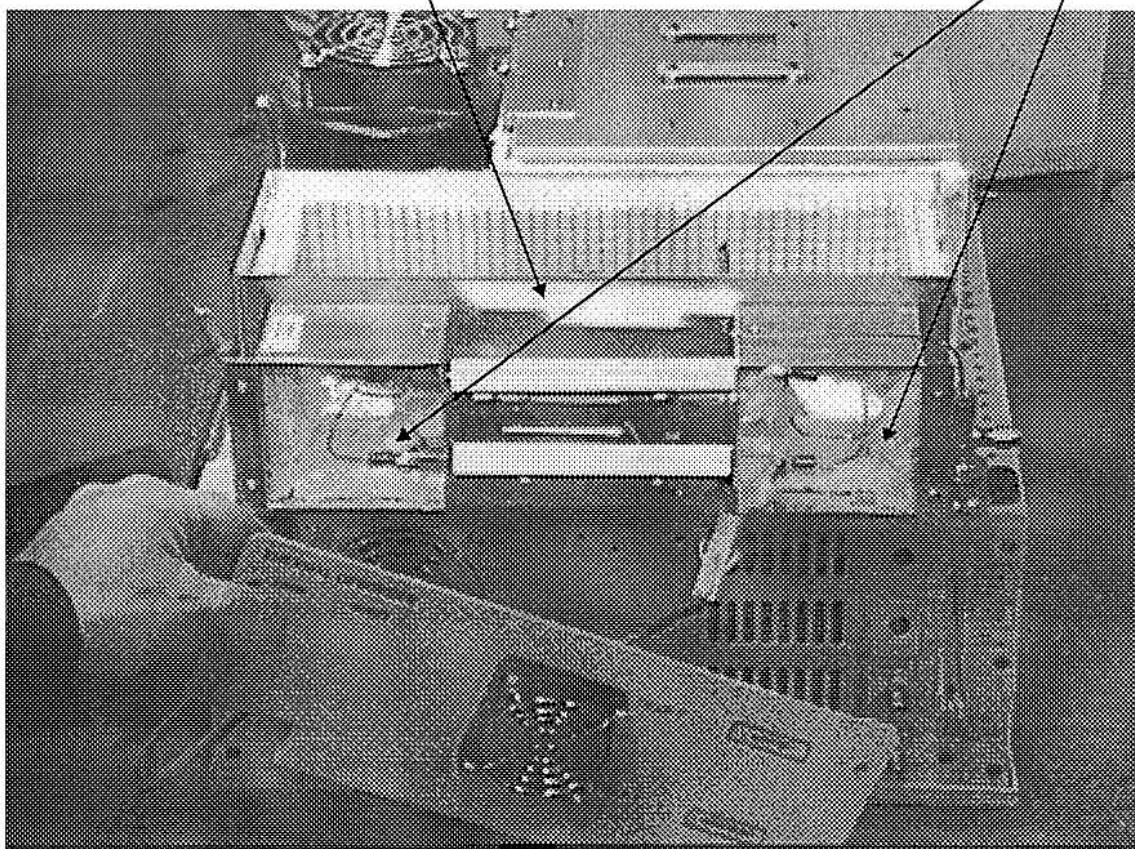
Ducts enclose ends of traps within heated valve oven to prevent cold spots.

Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

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

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



To access the traps for maintenance or replacement:

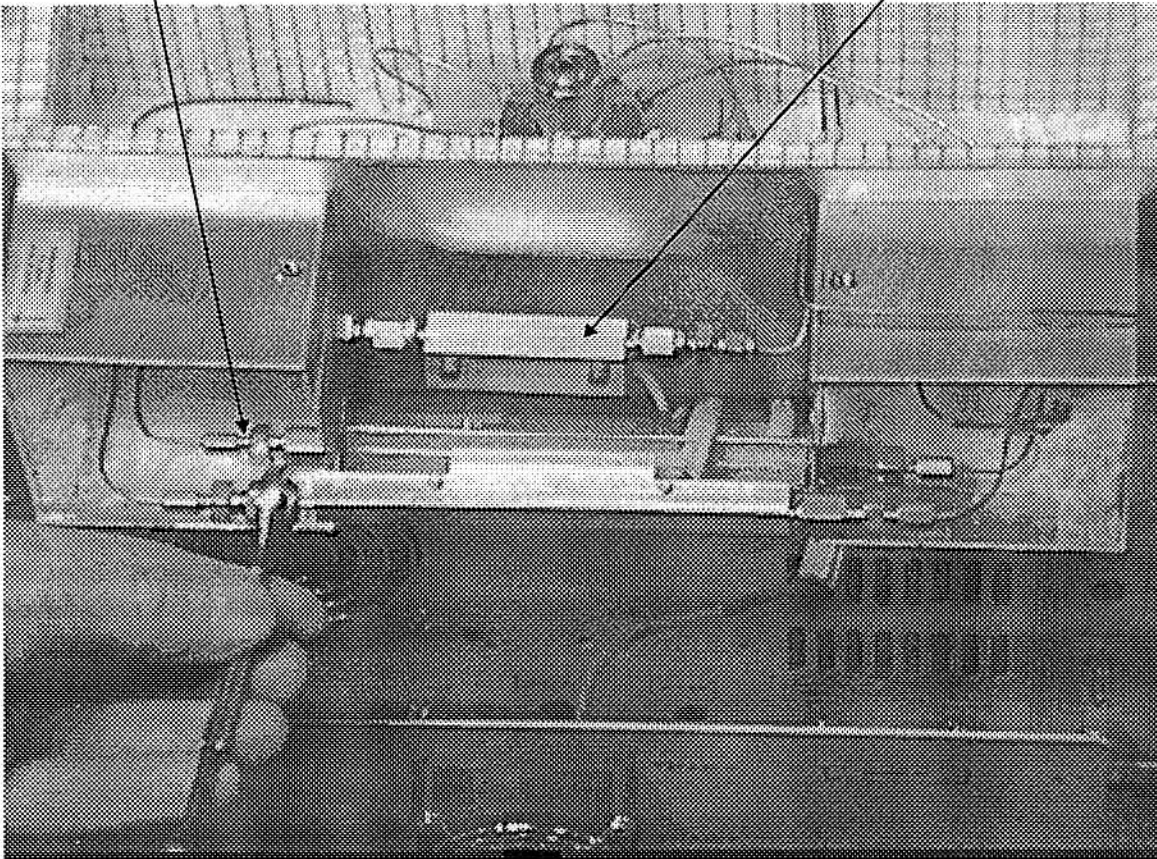
- 1) Remove the left side plate from the purge and trap valve oven by loosening the 4 brass thumbscrews at the corners.
- 2) Remove the protective grill from the top of the valve oven by loosening the two screws.
- 3) Carefully remove the two squares of white insulation from the ducts at the ends of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

Chapter: PURGE AND TRAP CONCENTRATOR

Topic: HARDWARE ORIENTATION

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

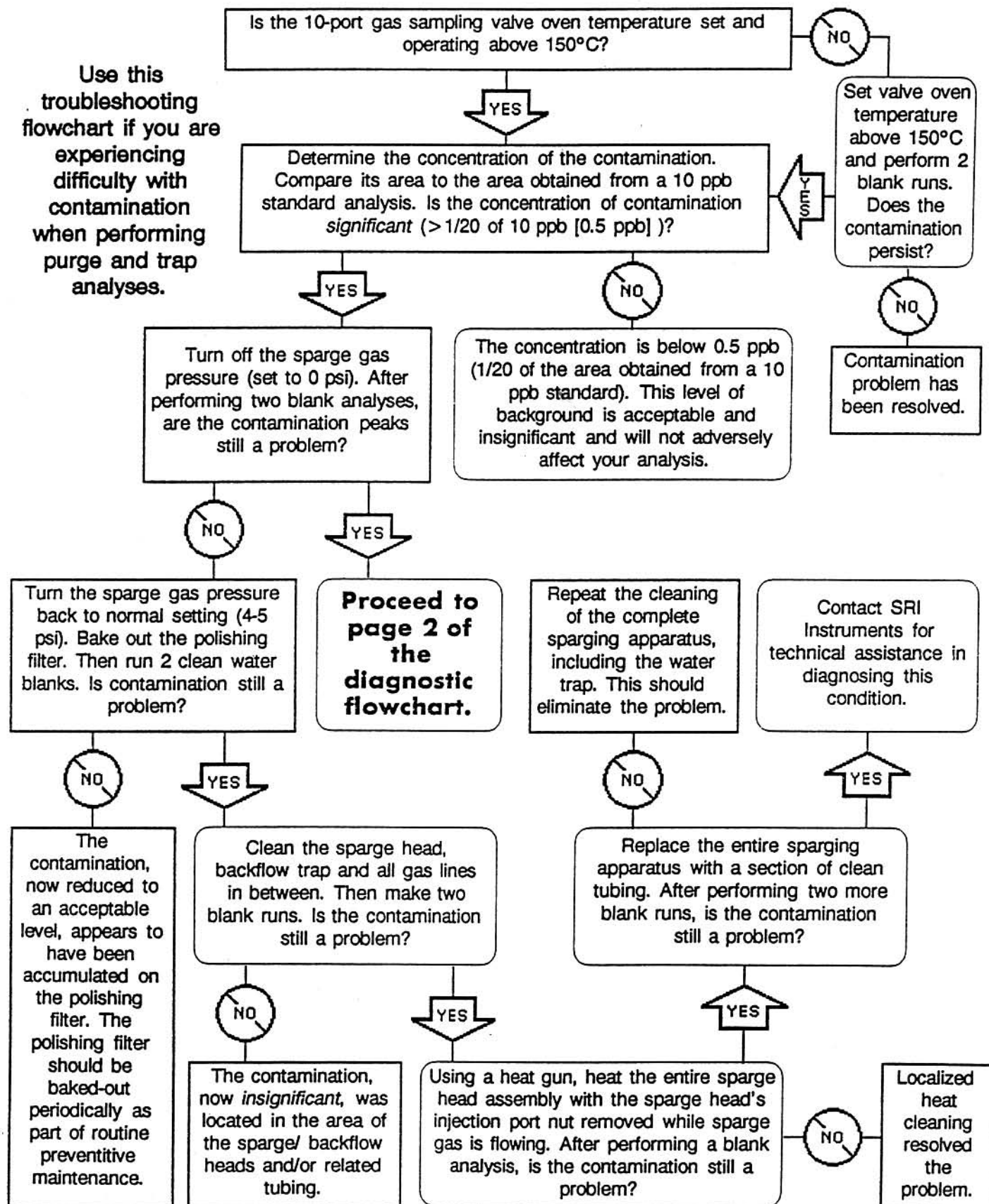
Clamshell type trap heater halves can be
separated by loosening two hex head type
screws.



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

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

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



From page 1

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

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

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

YES

NO

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

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

YES

NO

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

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

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

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

NO

YES

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

YES

NO

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

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

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

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

NO

YES

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

YES

NO

Call SRI for further assistance.

Dirty tubing. New tubing was the fix.

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

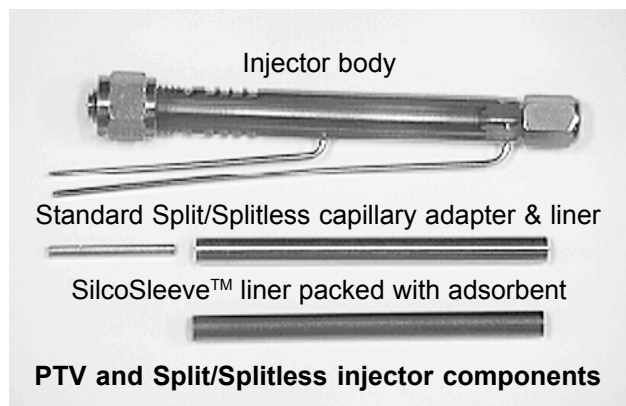
Call SRI for tech. support

Dirty tubing. New tubing was the fix.

INJECTORS

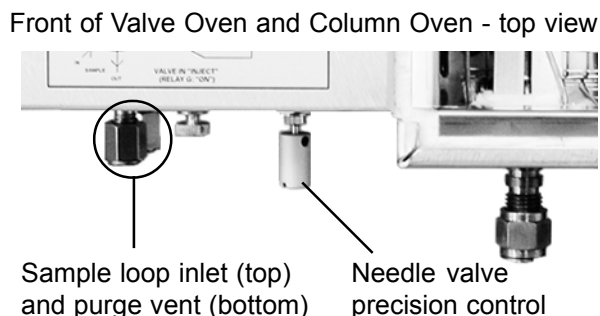
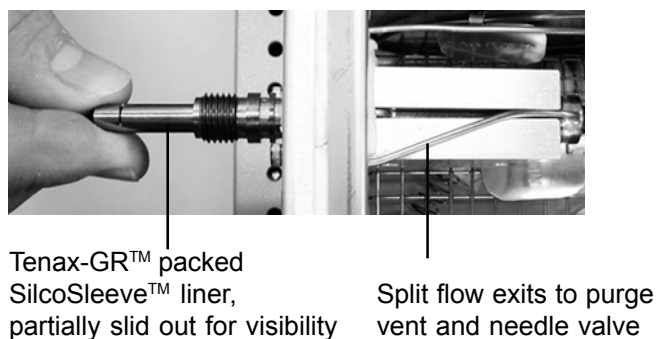
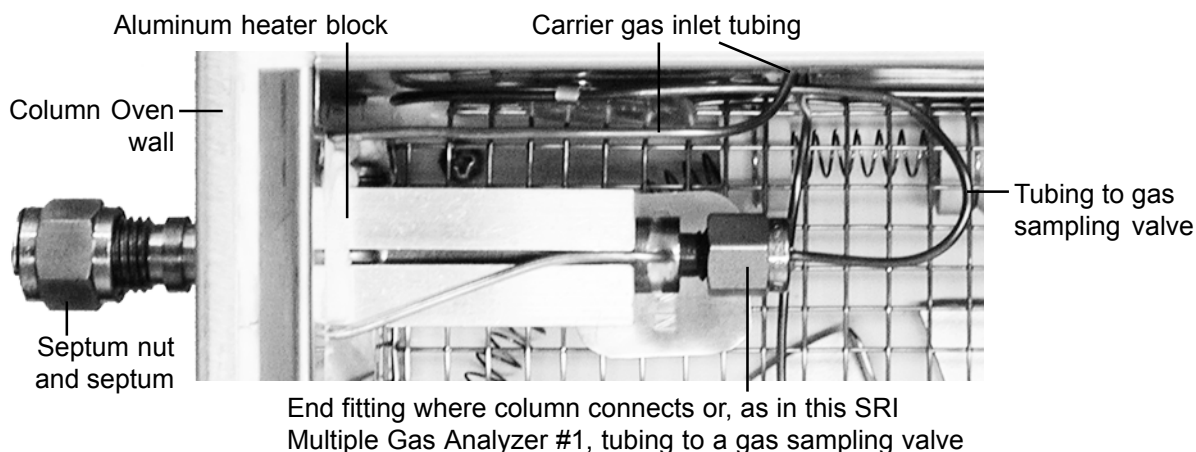
PTV - Programmed Temperature Vaporization Injector

Overview



The Programmed Temperature Vaporization (PTV) injector is composed of the same parts as the Heated Split/Splitless injector: the injector body, a SilcoSleeve™ liner, an injector purge restrictor, a precision needle valve for adjustment of split flow rate, a split flow solenoid that turns on & off from the PeakSimple data system, and an aluminum heater block containing a heater cartridge and Type K thermocouple. Contrasted with the Split/Splitless injector, the PTV injector has a removable insulating sleeve, a larger (250 watts) heater cartridge with

ballistic heating capability, and carrier flow ON/OFF control. The SilcoSleeve™ liner can be packed with a variety of optional adsorbents, depending on the application. The SRI PTV injector has three modes of operation: **1)** large volume liquid injector, **2)** an offline thermal desorber, or **3)** an online thermal desorber in conjunction with a gas sampling valve.



INJECTORS

PTV - Programmed Temperature Vaporization Injector

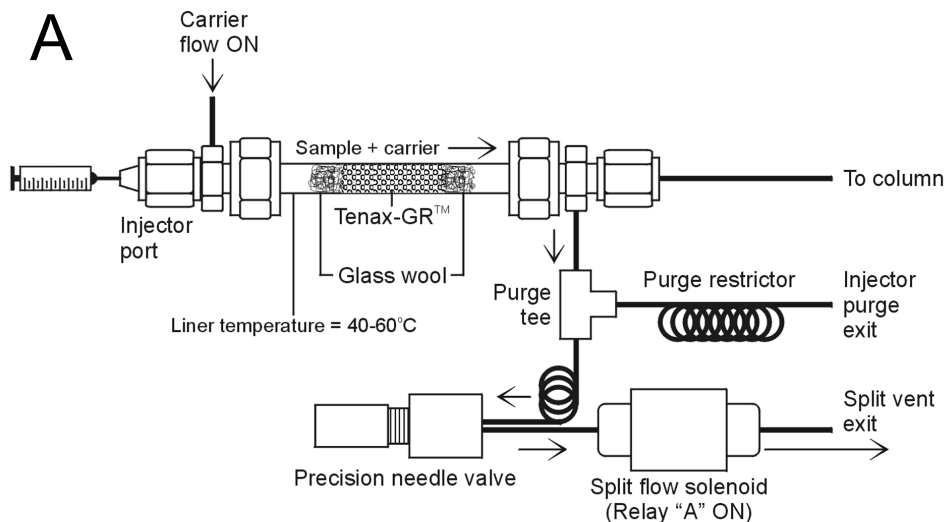
Theory of Operation

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

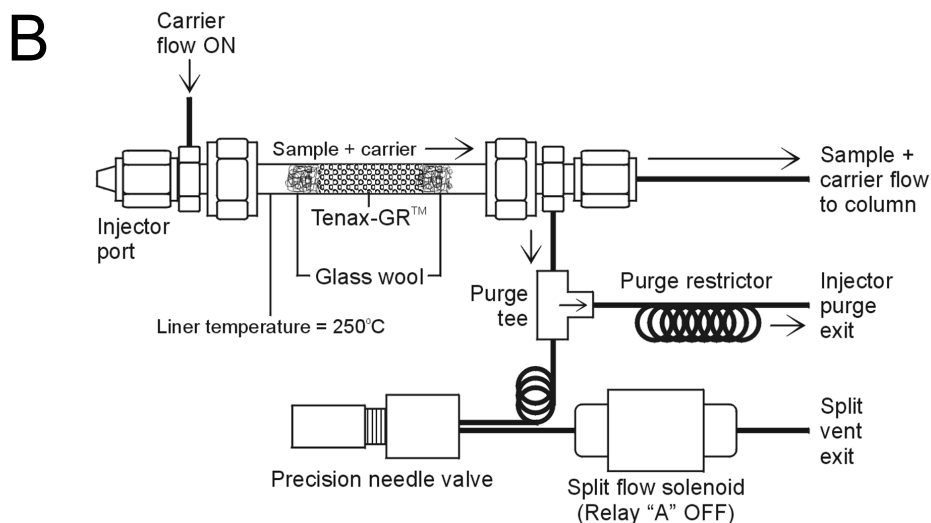
1) Large Volume Liquid Injector

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

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



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



INJECTORS

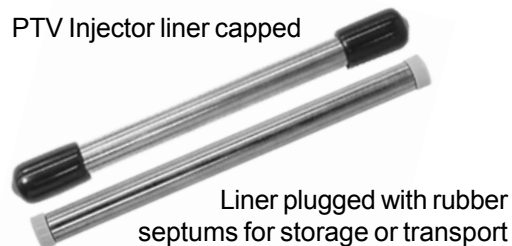
PTV - Programmed Temperature Vaporization Injector

Theory of Operation continued

2) Offline Thermal Desorber

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

PTV Injector liner capped



Liner plugged with rubber septums for storage or transport

Channel 1 events	
ZERO.evt	
Time	Event
0.000	ZERO
0.000	B ON (CARRIER FLOW-STOP)
0.500	C ON (PTV HEAT)
3.500	B OFF (CARRIER FLOW-STOP)

Buttons: Add... Change... Remove... Describe... Load... Save... Clear... Print... OK... Shift...

Example PTV as offline thermal desorber event table

3) Online Thermal Desorber

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

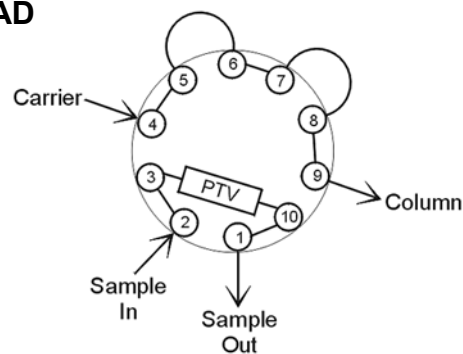
LOAD Position:
(Relay "G" OFF)

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

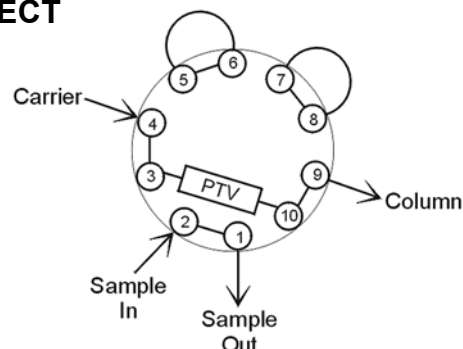
INJECT Position:
(Relay "G" ON)

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

LOAD



INJECT

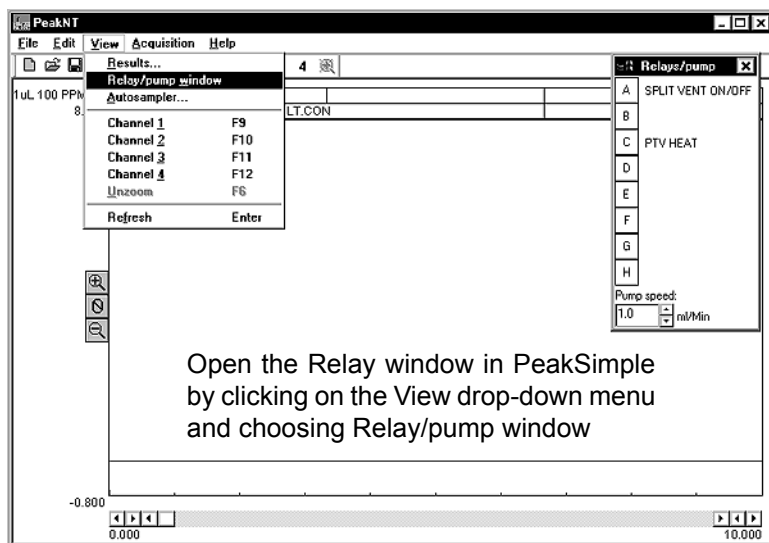


INJECTORS

PTV - Programmed Temperature Vaporization Injector

General Operating Procedure Large Volume Liquid Injection Steps

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



Channel 1 events	
ZERO.evt	
Time	Event
0.000	ZERO
3.000	A OFF (SPLIT VENT OPEN/CLOSED)
3.100	B ON (CARRIER FLOW ON/OFF)
3.200	C ON (PTV HEAT)
5.000	B OFF (CARRIER FLOW ON/OFF)

Example PeakSimple PTV event table

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

2. Type in an event program as follows:

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

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

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

INJECTORS

PTV - Programmed Temperature Vaporization Injector

Expected Performance

The following three chromatograms are from the FID in a SRI GC with a PTV injector upgrade. The liner was packed with 0.1 grams of Tenax-GR™ adsorbent. All three 25 minute runs utilized the same temperature and event programs. In the first one, a 1µL 2000ppm C₁₀-C₂₈ sample was injected through the PTV injector. In the second chromatogram, the same sample was diluted 1:100, then 100µl injected, achieving results consistent with the first run, and demonstrating the high volume liquid injection capability of the PTV injector. In the third chromatogram, 100µL of methanol was injected as a blank, resulting in a small hump between the 4 and 7 minute marks and miniscule peaks which correspond to contaminants in the methanol blank and bleed from the Tenax-GR™.

Chromatogram 1 Results:

Component	Retention	Area
Solvent	0.866	84953.1370
C10	5.366	5299.9150
C12	7.300	5034.0980
C14	10.233	4814.2000
C16	12.450	4600.0300
C18	14.216	4436.1780
C20	15.750	4528.2890
C22	17.150	4570.0975
C24	18.483	4778.9380
C26	20.033	4863.4290
C28	22.216	4135.4760
Total		132013.7875

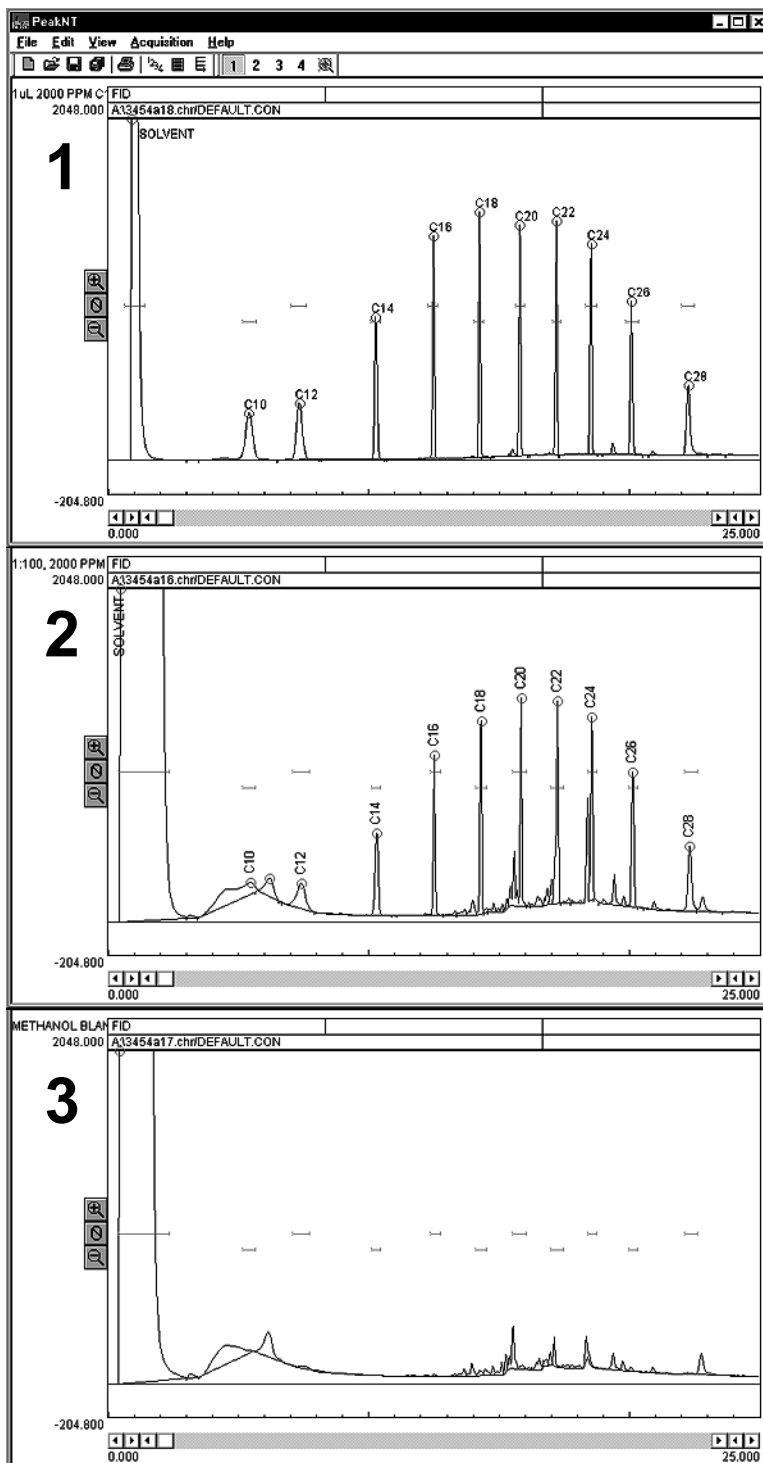
Chromatogram 2 Results:

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

Temperature programs & events for all 3 runs:

PTV = 110°C (3min) to 275°C		Events: (A = split vent)
		Time Event
		0.00 ZERO
		3.00 A OFF
		3.10 B ON
		3.20 C ON
Temperature program:		5.00 B OFF

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



8610C Power Consumption

7/16/2002

Basal Power

With no zones heating, Power Usage = 50W

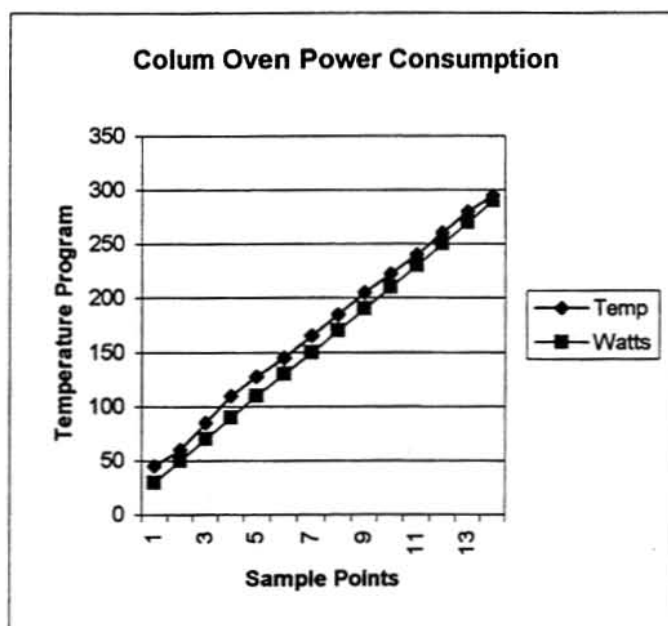
With 2 Detector zones heating = 150W

With Detector Zones Stabilized, Total Basal Power = 100W

Column Oven

Temperature Program 40C to 300C @ 5C/min

Average Temp	Watts	Temp Range
45	30	40-50
60	50	50-70
85	70	70-100
110	90	100-120
127.5	110	120-135
145	130	135-155
165	150	155-175
185	170	175-195
205	190	195-215
222.5	210	215-230
240	230	230-250
260	250	250-270
280	270	270-290
295	290	290-300



Maximum Power Usage

Ballistic Heating to 300C = 675W

Total Power = (Basal + Detector + Column Oven) = 825W

Isothermal Power Usage

Column Oven Stabilized @ 300C

2 detectors @ 150C

Total Power = (Basal + Detectors + Column Oven) = 400W

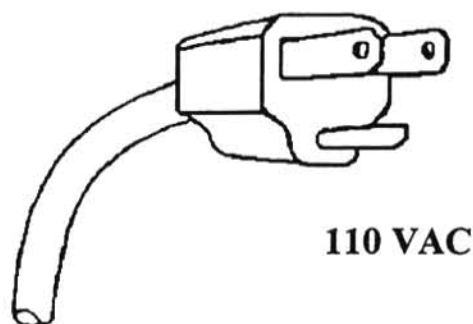
Chapter: INSTALLATION

Topic: Power Supplies and Space Requirements

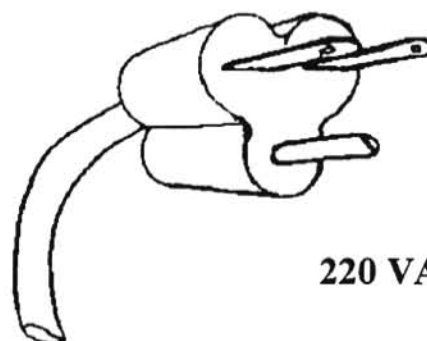
Once the equipment has been removed from all the packing material, check the contents of the container against the packing slip and make sure everything listed is included. If any item(s) have been omitted or are missing, contact SRI Instruments for location and or replacement of the item(s).

The SRI model 8610C gas chromatograph requires AC power at either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, depending on the AC power ordered. Both AC power supplies are equipped with a three prong grounded outlet (see diagrams to the right). Proper grounding is required for safe operation. Do not disable the ground prong under any circumstance. These plug configurations are for EIA standard U.S. outlets. It may be necessary to replace the plug provided with a local standard plug.

A standard SRI 8610C GC measures 18.5" X 14.5" X 12.5" and requires a minimum counter space of 28" X 22" X 23.5" for proper operation (see diagram to the right). Roughly 8" of clearance beside the left side control panel is needed for data cable, gas line and power switch access. 6" of clearance to the rear of the GC and 11" of clearance above the GC is required. This will provide adequate access to the column oven for maintenance and provide space for proper GC ventillation. To the front and right side, 1.5" of clearance should be adequate to prevent the GC from coming into contact with surrounding objects or falling off the counter. The right side of the GC does contain general information on your instrument and some operators may want additional clearance for easy reference. The front control panel of the GC should be easily accessible in order to properly monitor digital display and control operating conditions, as well as providing access to the injection port for sample injections.

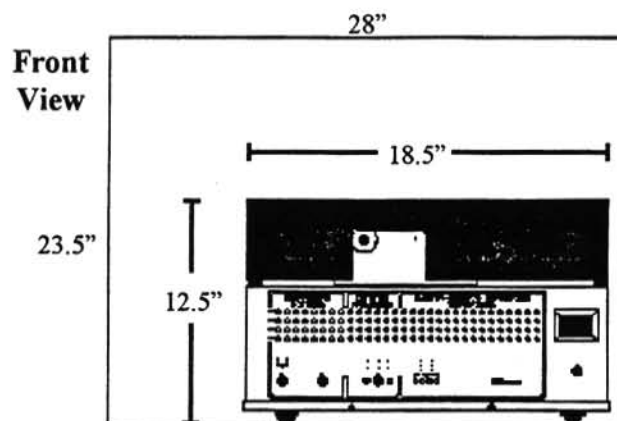
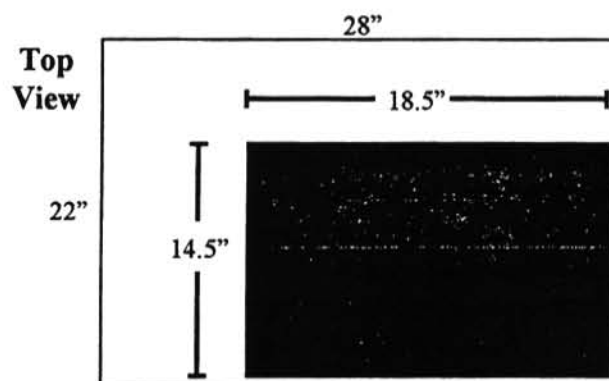


110 VAC



220 VAC

Required Operating Counter Space



Invited Paper

Recent Advances in Thermionic Ionization Detection for Gas Chromatography

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Abstract

Thermionic ionization detectors are most widely used for the specific detection of nitrogen-phosphorus compounds in gas chromatography. The operating mechanism of these detectors is a surface ionization process in which the key parameters are the work function of the thermionic emission surface, the temperature of the thermionic surface, and the composition of the gas environment in the immediate vicinity of the thermionic surface. By systematic variations of each of these three key parameters, the technique of thermionic ionization detection has been greatly expanded to encompass a number of different modes of response, all of which use similar detector hardware and electronic components.

process was operative, it was possible to identify three key operating parameters which control the ionization produced. These parameters are: the electronic work function of the thermionic emission surface which is determined by the chemical composition of the surface; the temperature of the thermionic surface; and the chemical composition of the gas environment immediately surrounding the thermionic surface.

The identification of these parameters has led to a clearer understanding of the complex chemistry active in NP detection, and has provided an important guide for expanding the applications of thermionic ionization techniques. Through systematic variations in each of the key parameters, many different modes of detector response have been achieved (3-6). Hence, the technique of thermionic ionization detection now correctly refers to a number of GC detector responses which are related through the use of many common hardware and electronic components. This article reviews the present state of development of the members of this unique group of detectors.

Introduction

Thermionic ionization detectors (TID) are best known in gas chromatography (GC) for their application to the specific detection of nitrogen (N) or phosphorus (P) compounds. All modern TIDs are essentially derivations of a basic design first described by Kolb and Bischoff (1) in 1974. The main component in this type of detector is an electrically-heated thermionic emission source in the form of a bead or cylinder which is usually composed of an alkali-metal compound impregnating a glass or ceramic matrix. In the TID, the thermionic source is positioned so that sample compounds may impinge upon its surface, and any ionization produced is measured by an adjacent collector electrode. Kolb and Bischoff were the first to report that a thermionic source comprised of a Rb-silicate glass bead produced very specific NP responses when the bead was operated at high temperatures in a gas environment of dilute H₂ in air.

Since the original work of Kolb and Bischoff, there have been continuing developments in NP detectors, with much emphasis on improved methods of construction and composition of the thermionic emission sources. The most important development, however, has been the recognition in recent years that the operation mechanism of a TID is a surface ionization process (2) rather than the gas phase ionization process originally proposed by Kolb et al. (1,3). Once it was clear that a surface ionization

Types of Thermionic Emission Sources

All commercially available TIDs use thermionic emission sources formed according to one of the following four general methods:

- (A) homogeneous alkali-glass bead formed on a loop of bare platinum wire (1);
- (B) alkali salt activator coated on a ceramic cylinder core containing an embedded heater coil (7);
- (C) homogeneous alkali-ceramic bead formed on a coil of nichrome heater wire (2,8,9);
- (D) multiple layers of cylindrically-shaped ceramic coatings, with a non-corrosive, electrically-conducting sub-layer of Ni-ceramic completely covering a loop of nichrome wire, and a surface layer comprised of alkali and/or other additives in a ceramic matrix (5,10).

Thermionic sources representing all four categories cited above have been used in NP detectors available from different manufacturers. Generally, those sources formed from ceramic materials provide greater flexibility for varying the chemical composition of the source. This is because the ceramic compositions are formulated and coated from a slurry at room temperature (9), whereas the glass compositions are formed in

a process that proceeds through a molten glass state (11).

The detailed chemical compositions of thermionic emission sources are usually regarded as confidential proprietary information by the manufacturer. Since the first alkali-glass bead reported by Kolb and Bischoff used Rb as the alkali compound, there existed for many years a belief that Rb was an essential component for optimum NP responses. However, in recent years, NP detectors with state-of-the-art performance specifications have been reported in which Cs rather than Rb is used as the alkali component (5). Also, another recent report (12) has described an NP detector which uses a $\text{LaB}_6/\text{SiO}_2$ bead and no alkali additive. In accordance with a mechanism of surface ionization prevailing in the TID, the most important characteristic of the thermionic emission surface is its electronic work function (i.e., the amount of energy required to emit a unit of electrical charge from the surface). Alkali-metal compounds have been especially successful additives because they lower the work function of the glass or ceramic matrix, thereby facilitating the emission of charged particles from the heated thermionic surface. The mathematical relationships between work function, surface temperature, and thermionic emission current have been discussed (2,12).

The development of multiple-layered, ceramic-coated thermionic emission sources has allowed examination of coatings of many different chemical compositions without the risk of materials in the surface layer corroding the heater wire. In the search for expanded applications for thermionic ionization techniques, the basic task is to define a specific match of a thermionic source type with an operating gas environment and a range of operating source temperatures. To date, three different chemical compositions of thermionic sources have been shown (5,6) to have useful applications in differing modes of thermionic detection. These source compositions are shown in Table I. Data obtained using these three types of thermionic emission sources are presented in the following sections.

Modes of Response

Schematic illustrations of four different versions of thermionic ionization detection equipment are shown in Figures 1 through 4. Common components in each version are as follows:

- an electrically-heated, thermionic/catalytic source constructed of multiple layers of ceramic coatings;
- a cylindrical collector electrode surrounding the cylindrically shaped thermionic source;
- a source power supply that provides heating current to heat the source to typical temperatures of 400° to 800°C, and a bias voltage to polarize the source structure at a negative voltage with respect to the collector;

Table I. Thermionic Source Surface Layers

Source type*	Additive	Work function
TID-1	High concentration Cs	Low
TID-2	Low concentration Cs/Sr	Medium
CFID	Nickel	High

*Nomenclature adopted from Patterson (5).

(D) an electrometer that measures negative ionization currents arriving at the collector electrode.

The TID hardware usually mounts onto an FID-type detector base that is resident on a GC, so that two different detector gases may be supplied in addition to the GC effluent. Therefore, changes in the modes of detector response that correspond to the schematics of Figures 1 through 4 are accomplished by changes in the type of thermionic source, changes in the composition of gases supplied to the detector, or by changes in the operating temperature of the thermionic surface.

Most of the TIDs available commercially function by the collection of negative ionization rather than positive ionization. In the discussion that follows, it will be shown that the concepts of negative ion chemistry provide a logical pattern for correlating the responses of the different modes of thermionic detection.

TID-1-N₂: Nitro/electronegative specific response

The simplest mode of thermionic detection is represented by the schematic in Figure 1. In this mode, the low work function thermionic source designated by the TID-1 nomenclature is operated in a detector gas environment of N₂. Because the detector gases are inert, sample compounds interact directly with the TID-1 surface, which is typically heated to temperatures in the range of 400° to 600°C. The ionization process in this case is direct transfer of negative charge from the TID-1 surface to

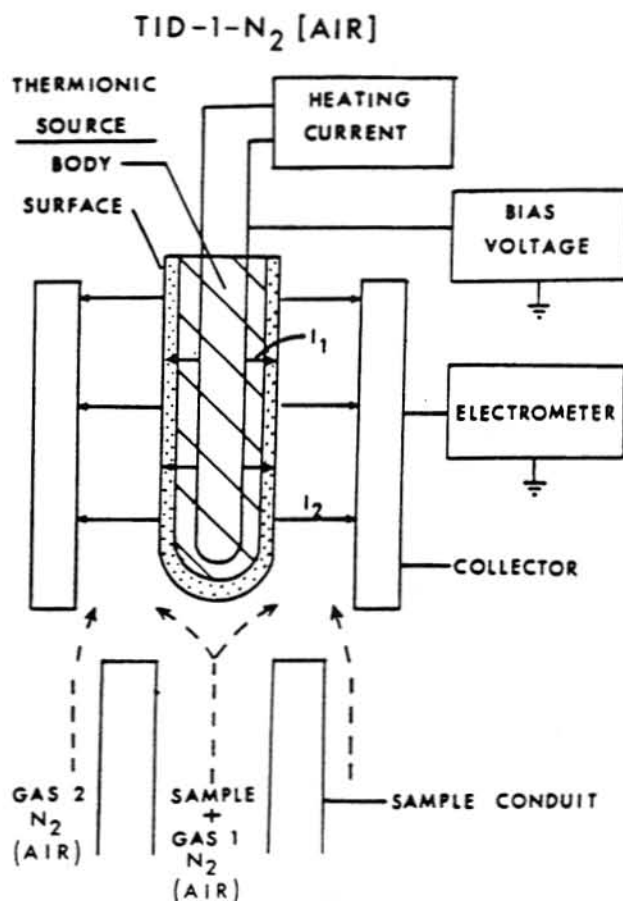


Figure 1. Schematic illustration of the detection configuration for the thermionic ionization modes TID-1-N₂ and TID-1-air. Detector gas 1=FID-H₂ inlet line; detector gas 2=FID-air inlet line.

the sample molecule. Consequently, this mode of response provides exceptionally high specificity and sensitivity to many compounds containing the nitro (NO_2) functional group (5,13), as well as to certain other electronegative compounds (e.g., pentachlorophenol, diazepam, and methaqualone). This mode of detection is very sensitive to the detailed electronegativity of the sample's molecular structure, as has been illustrated by the observation that a larger signal is obtained for the 2,4-isomer of dinitrotoluene in comparison to the 2,6-isomer (5). The TID-1- N_2 mode is superior to a conventional NP detector or an electron capture detector (ECD) for detection of trace level nitro-compounds such as nitro-PAH, nitro-explosives, nitro-pesticides (e.g., parathion, methyl parathion), nitro-drugs, nitro-derivatives. For many nitro-compounds, the specificity vs. hydrocarbons is an astonishing 10^4 , and detectivity is in the 0.1 to 1.0-pg range (5).

TID-1-air: Halogen/nitro specific response

When the TID-1 thermionic source is operated in an oxygen-containing gas environment rather than one of N_2 , specific responses to halogenated compounds are enhanced while responses to nitro-compounds are decreased somewhat (14). The TID-1-air mode of detection is generally not as sensitive as an ECD or Hall detector for chlorinated compounds, but it provides halogen specificity at higher concentrations where ECD and Hall are saturated. Typical specificity is 10^4 and detectivity

is 0.1 to 1.0 ng. This is an especially simple mode of detection for ethylene dibromide (EDB) in the headspace vapors of food products.

TID-2- H_2 /air: Nitrogen/phosphorus specific response

The schematic illustration of Figure 2 represents the situation that prevails in an NP detection mode. For this mode, H_2 and air gases are supplied to the detector, and a thermionic source of moderate work function (i.e., TID-2) is operated hot enough (600° to 800°C) to cause thermal/chemical decomposition of the H_2 and O_2 gases. A critical parameter in this NP mode is the restriction of the H_2 to low flows (e.g., 3 to 6 ml/min) which are not sufficient to maintain a self-sustaining flame at the sample conduit (i.e., jet structure) depicted in Figure 2. Instead, a flame-like gaseous boundary layer is created in the immediate vicinity of the hot thermionic source. Since this boundary layer is very reactive chemically, sample compounds are decomposed by the active gas phase chemistry, and electronegative products of decomposition are selectively ionized by surface ionization on the thermionic source. N or P compounds are ionized with especially high specificity by this process. An essential condition for the onset of NP detection is that the thermionic source must be hot enough to "ignite" the boundary layer chemistry. Under these conditions, a thermionic source of moderate work function provides the optimum compromise of sample response signal vs. detector background signal. A low work function therm-

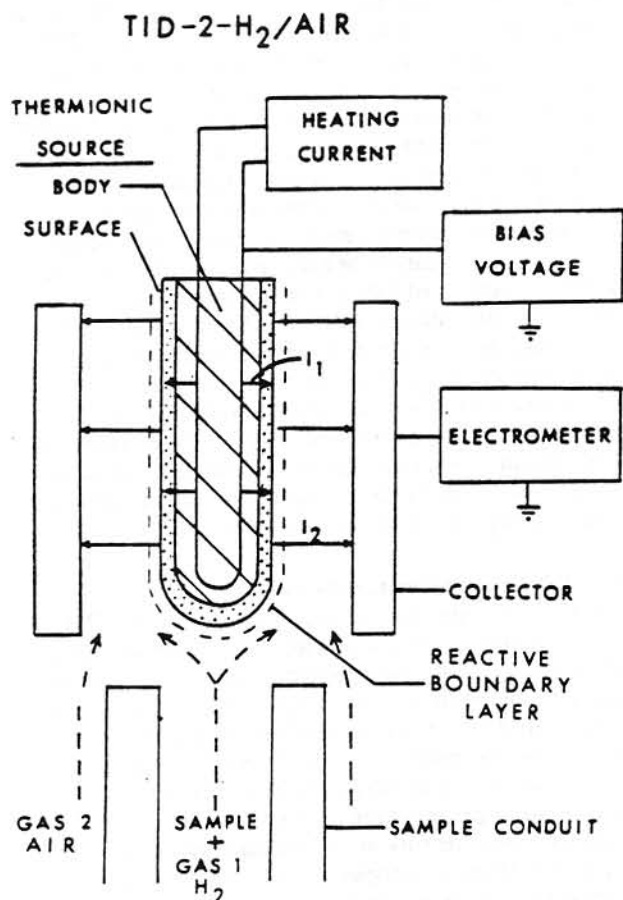


Figure 2. Schematic illustration of the detection configuration for the TID-2- H_2 /air or NP mode of thermionic ionization.

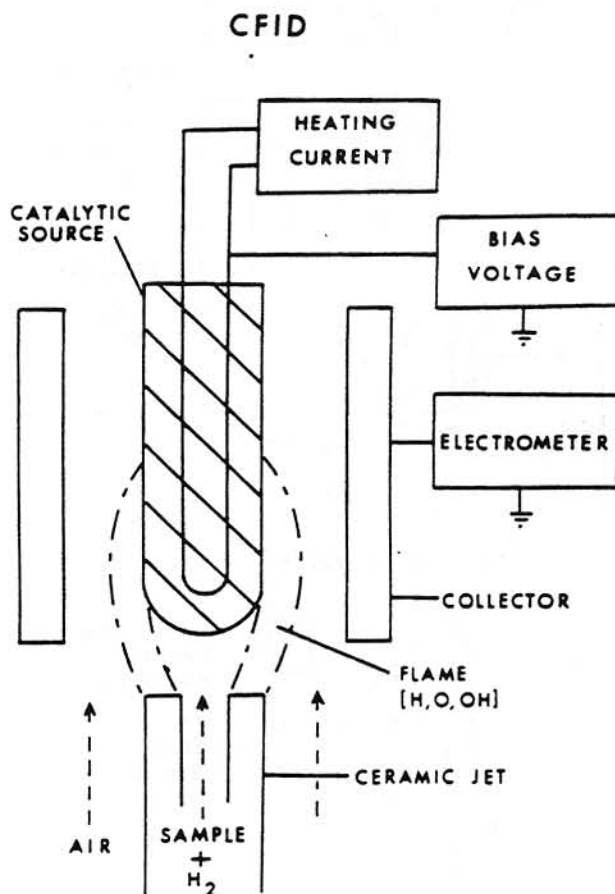


Figure 3. Schematic illustration of the detection configuration for the catalytic flame ionization detector (CFID) mode of response.

ionic source (e.g., TID-1) operated under these conditions would produce an overwhelmingly large background signal. Conversely, a high work function thermionic source (e.g., catalytic flame ionization detection) operated under these conditions would produce smaller NP signals and less specificity than the moderate work function thermionic source. Typical performance specifications for NP detectors are detectivities in the range of 1 to 10 pg, and specificity with respect to hydrocarbons in the range of 10^4 to 10^5 .

CFID: Universal response to all organics

Figure 3 depicts a mode of response achieved when the H_2 flow to the detector is sufficient to produce a true self-sustaining flame burning at the jet structure. This mode of operation has been designated (5) catalytic flame ionization detection (CFID) because of its close similarity to a conventional FID. The CFID is essentially an FID which has been modified by inserting an electrically-heated catalytic source comprised of a Ni-impregnated ceramic into the center of the active flame region. In this detection mode, the catalytic source structure serves the three-fold function of flame ignitor, flame polarizer, and catalytic combustion modifier/thermionic surface ionizer. In the CFID, two types of ionization processes are active: gas phase ioniza-

tion processes identical to those which occur in a conventional FID, and surface ionization processes at the catalytic source structure which especially enhance the ionization efficiency of many heteroatom compounds (especially halogenated and phosphorus compounds).

Like a conventional FID, the magnitude of the gas phase ionization is determined primarily by the magnitudes of H_2 and air flows and the size of the jet orifice, so that additional electrical heating of the CFID source has little effect on the gas phase ionization. However, the magnitudes of ionization produced by surface processes at the CFID source is strongly dependent on the electrical heating of the source. Hence, in many cases, response factors for heteroatom compounds can be enhanced to be comparable to hydrocarbons by a judicious selection of source heating current. For this CFID mode of detection, the thermionic/catalytic source of highest work function is most suitable because the flame heat would otherwise cause an excessive thermionic emission background signal. The CFID provides detectivities in the 10- to 100-pg range for most organic compounds.

FTID: Nitrogen/halogen specific response

Figure 4 depicts a further detection mode in which the thermionic source and collector electrode structure are positioned well downstream of the active region of a self-sustaining flame. The basic concept (6) of this flame thermionic ionization detection (FTID) mode is to burn sample compounds in a self-sustaining H_2 /air flame at a flame jet, and to selectively re-ionize electronegative combustion products by means of the thermionic ionization components located downstream. In the FTID, a large physical separation between the flame and the thermionic source/collector electrode provides minimal collection of ionization produced in the flame, but excellent collection of ionization produced at the heated surface of the thermionic source. An auxiliary ion-suppress voltage can be applied to further prevent ionization produced in the flame from reaching the ionization collector. This mode of detection provides specific responses to compounds containing N or halogen atoms with a specificity of 10^3 and detectivity of 1.0 ng. The precombustion of samples in the flame minimizes interferences from sample matrices and provides more uniform responses independent of the original molecular structure of the sample compound. Both the low work function (TID-1) and moderate work function (TID-2) thermionic sources have been used in this FTID configuration. FTID-1 provides good responses to both nitrogen and halogen compounds, whereas FTID-2 responds best for halogen compounds with suppressed nitrogen response.

Remote FID: Organo-lead specific response

This mode of detection is a simple variation of the FTID in which no electrical heating is supplied to the thermionic source, and the ion-suppress voltage depicted in Figure 4 is not applied. In this case, the thermionic source serves merely as a polarizer to drive negative ions in the flame effluent to the TID collector electrode. The ionization sensed in this mode corresponds to long-lived negative ions originally produced in the flame. Because of the large separation between the flame and the TID collector electrode, the bulk of the hydrocarbon ionization produced by the flame is dissipated (i.e., positive-negative ion recombination or neutralization at a wall surface) before reaching the TID collector. However, certain heteroatom compounds appear to combust to negative ion products which are especially stable and long-lived. The outstanding demonstration

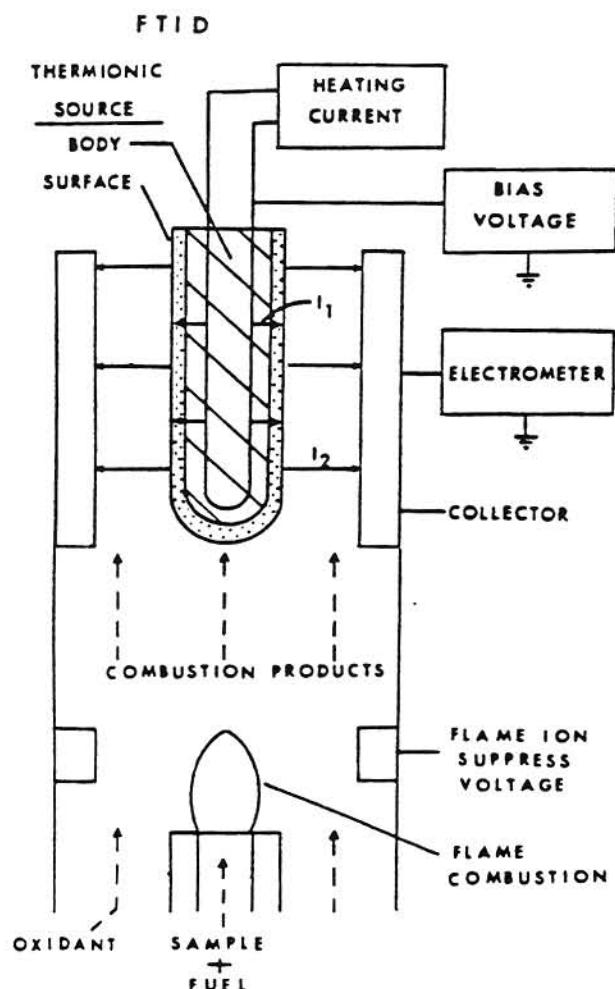


Figure 4. Schematic illustration of the detection configuration for the flame thermionic ionization detector (FTID) mode of response.

of this effect is the specific detection of lead compounds in gasoline. Since the thermionic source is not electrically-heated and is not used as a source of surface ionization, any type of thermionic source may be employed in this mode.

Experimental

The applications data presented in this report were obtained with equipment previously described (5,6,14). All the data were obtained using a Model 3740 gas chromatograph (Varian Associates) equipped with either a TID/CFID detector assembly (TID-1-N₂, TID-1-air, TID-2-H₂/air, and CFID modes) or an FTID/TID detector assembly (FTID and remote FID modes) (Detector Engineering and Technology). The TID-1, TID-2, and CFID thermionic emission sources were also manufactured by Detector Engineering and Technology, as was the Model 4000 detector current supply. Negative ionization signals from the detector were measured using the differential electrometer (Varian) in a negative polarity configuration.

The detector assemblies mounted onto the FID-base on the GC, so that different detector gas environments were implemented by plumbing in the appropriate gas through the two gas lines that normally supply H₂ and air to an FID. Typical gas flows supplied for different modes of detection are detailed in Table II.

All the data presented in this report were obtained using glass columns, 6 ft x 1/8 in. x 2 mm i.d., packed with either 3% SP-2250, 3% SP-2100, or 1% SP-1240 DA on 100/120 Supelcoport (Supelco) or 80/100 Chromosorb 102. The GC carrier gas in all cases was N₂ at a flow rate of 30 ml/min. For complex samples like gasoline, the chromatographic separation was intentionally very poor in order to produce a challenging detector environment to demonstrate specificity of response in the simultaneous presence of many overlapping compounds.

Sample mixtures that were analyzed included a TSD test sample (ng levels of azobenzene, methyl parathion, and malathion) (Varian); Base-Neutral 1 sample (Supelco); phenol mix (Supelco); DCMA PCB mixture (Supelco); and nitroaromatic mixture (Supelco) diluted in reagent-grade benzene. Gasoline, cologne, and diesel fuel samples were analyzed by direct injection of 0.5 to 1.0 µl amounts onto the column. Other samples chromatographed were a 1% v/v each mixture of acetone and carbon tetrachloride in water; and an 11% v/v each mixture of methylene chloride, n-C₄, benzene, i-C₄, toluene, n-C₆, p-Xylene, n-C₁₀, and n-C₁₂.

Table II. Typical Gas Flows Supplied for Each Mode of Detection

Mode	Gas 1	Gas 2
TID-1-N ₂	10 ml/min N ₂	60 ml/min N ₂
TID-1-air	10 ml/min air	60 ml/min air
TID-2-H ₂ /air	3 ml/min H ₂	60 ml/min air
CFID	25 ml/min H ₂	200 ml/min air
FTID	20 ml/min H ₂	200 ml/min air
Remote FID	20 ml/min H ₂	200 ml/min air

Applications*

Specially formulated test samples are often employed to demonstrate the specificity and sensitivity of NP detectors. The data in Figure 5 correspond to such a test sample comprised of 2.2 ng each of azobenzene (N) and methyl parathion (N,P), 4.4 ng of malathion (P), and 4400 ng of n-C₁₇ in a solvent of iso-octane. The data illustrate clearly the substantial differences in response between two different modes of thermionic detection, and the very high specificity of the TID-1-N₂ mode for sensing the NO₂ group in methyl parathion. The ionization signals in both chromatograms are very large, indicating detectivities in the pg and sub-pg range.

Figure 6 shows another comparison of the TID-1-N₂ mode vs. the NP mode of detection for a sample consisting of 15 pg amounts of the 2,4- and 2,6-isomers of dinitrotoluene in a relatively impure, reagent grade benzene solvent. For many nitro-compounds, the TID-1-N₂ mode provides substantial improvements in specificity and sensitivity in comparison to an NP mode. However, the NP mode (i.e., TID-2-H₂/air) provides more universal detection for all N-compounds. In Figure 6, the differing magnitudes of TID-1-N₂ signals for the two dinitrotoluene isomers also demonstrate that the TID-1-N₂ signals are very sensitive to the detailed electronegative character of the sample's molecular structure.

Figure 7 shows the differing responses of six modes of thermionic detection in the analysis of base neutral compounds of concern as water pollutants. The CFID provides universal response to all compounds in this sample with a relatively uniform sensitivity of 0.01 coul/gC. The TID-1-N₂ mode of opera-

*Data presented are from References 6 and 14.

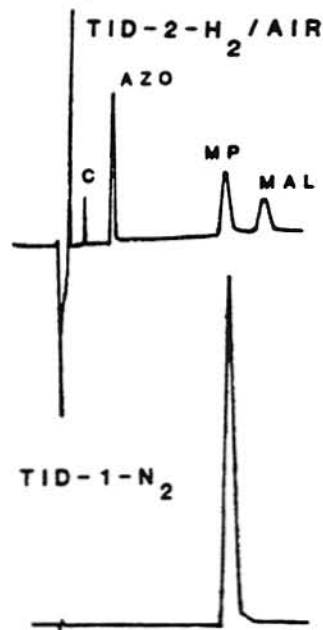


Figure 5. Chromatograms showing two modes of response to a test sample containing a large concentration of a hydrocarbon (C) and trace levels of azobenzene (AZO), methyl parathion (MP), and malathion (MAL). Both chromatograms were recorded at the same sensitivity of 128×10^{-11} amps/mV. Column: SP-2250, isothermal at 210°C.

tion provides very specific responses only to a nitro-compound (2,6-dinitrotoluene) and a chlorinated compound (3,3'-dichlorobenzidine). The lack of significant TID-1-N₂ response to nitrobenzene, bis(2-chloroethyl)ether, bis(2-chloroisopropyl)ether, or 4-bromophenyl-phenyl-ether demonstrates that the TID-1-N₂ response depends on how the electronegative functionalities are bound up in the molecular structure of the sample compound. When the detector gas environment of the TID-1 source is changed from N₂ to O₂, the TID-1-O₂ mode provides enhanced relative responses to the chlorinated compounds and a diminished relative response to the nitro-compound. The TID-1-O₂ mode continues to provide good discrimination in favor of chlorinated compounds with respect to hydrocarbons, while exhibiting some low level responses to phthalate compounds. The TID-2-H₂/air mode responds to all the N-compounds, with some small interferences from chlorinated compounds. The FTID-1 mode uses a TID-1 source and provides responses to all the nitrogen and halogen compounds in the sample. The FTID-2 mode uses a TID-2 source which produces responses to the halogenated compounds but suppressed responses to nitrogen compounds in comparison to FTID-1. This set of six chromatograms provides a good illustration of how the detector response can be varied through simple changes in the composition of the detector gas environment.

Figure 8 shows chromatograms of a sample mixture consisting of 75 ng each of 2-chlorophenol, 2-nitrophenol, phenol, 2,4-dimethylphenol, and 2,4-dichlorophenol; 225 ng each of 2,4,6-trichlorophenol, and 2,4-dinitrophenol; and 375 ng each of 4-chloro-*m*-cresol, 4,6-dinitro-*o*-cresol, pentachlorophenol, and 4-nitrophenol. The CFID provides a relatively uniform response of 0.004 coul/gC for all these compounds. (Note: The flame tip orifice for these CFID data was 0.062 in. instead of 0.031 in., which is normally used. The smaller orifice usually provides improved sensitivities of approximately 0.01 coul/gC.)

The CFID data provide a good illustration of a principal difference in the responses of a CFID vs. a conventional FID. It is well known that conventional FIDs provide relatively uniform

response to many hydrocarbon compounds. However, when heteroatoms, such as O, Cl, or P, are present in the sample compounds, the FID response is frequently significantly lower than its response to hydrocarbons. In contrast, the CFID appears to yield more uniform response to all organic compounds ir-

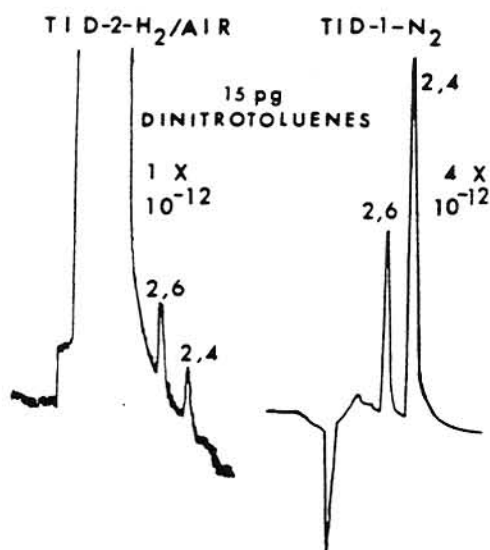


Figure 6. Chromatograms comparing the responses of the TID-1-N₂ mode and the TID-2-H₂/air (NP) mode to traces of dinitrotoluenes. Column: SP-2100, 160° to 200°C at 10°/min.

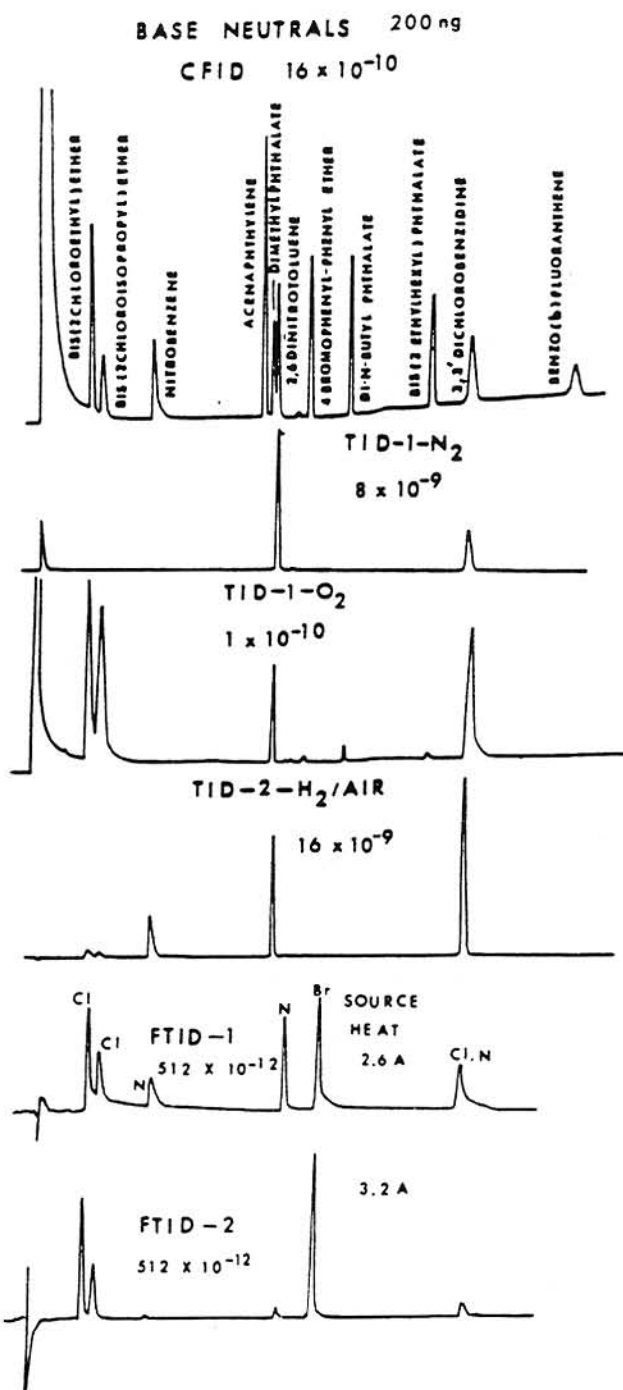


Figure 7. Chromatograms showing six different modes of detector response to a sample of base neutral compounds. In increasing order of retention time, the components of the sample are bis(2-chloroethyl)ether; bis(2-chloroisopropyl)ether; nitrobenzene; acenaphthylene; dimethylphthalate; 2,6-dinitrotoluene; 4-bromophenyl-phenyl ether; di-*n*-butylphthalate; bis(2-ethylhexyl)phthalate; 3,3'-dichlorobenzidine; and benzo(b)fluoranthene. Column: SP-2250; 100°C, held for 4 min, then 100° to 270°C at 16°/min.

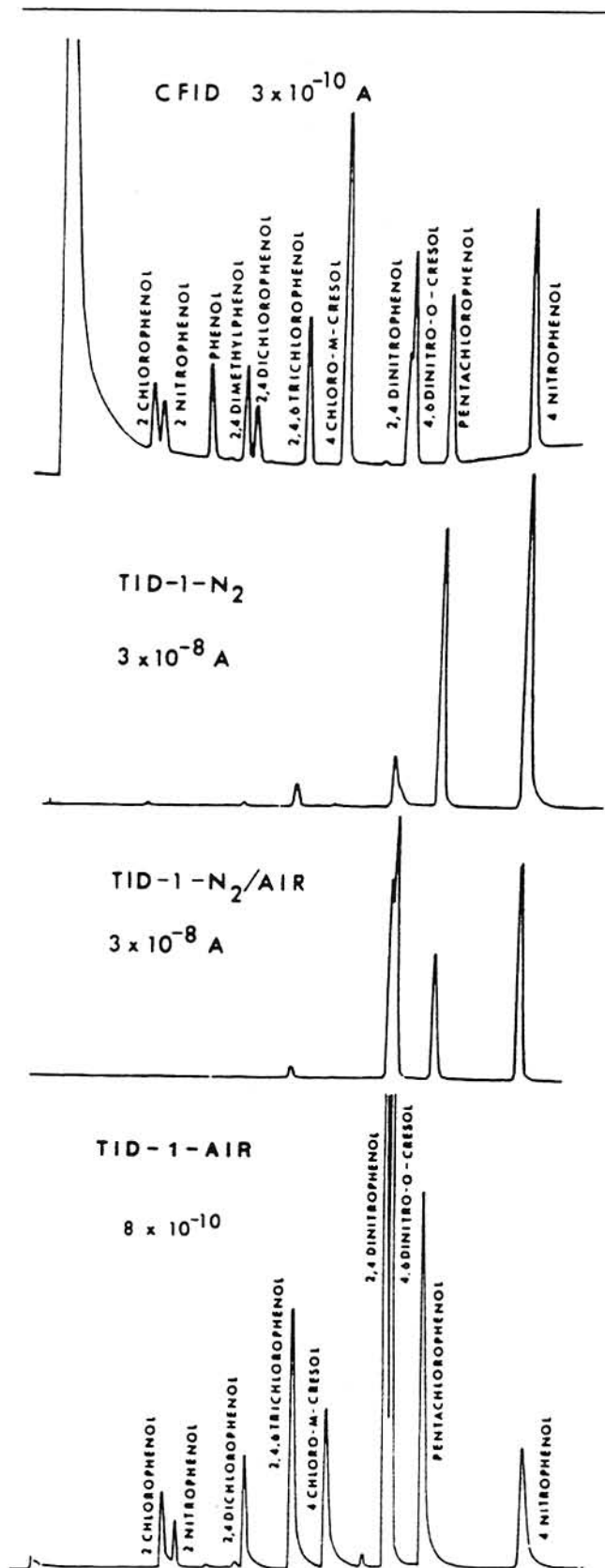


Figure 8. Chromatograms showing different modes of detector response to phenol sample containing 2-chlorophenol; 2-nitrophenol; phenol; 2,4-dimethylphenol; 2,4-dichlorophenol; 2,4,6-trichlorophenol; 4-chloro-*m*-cresol; 2,4-dinitrophenol; 4,6-dinitro-*o*-cresol; pentachlorophenol; and 4-nitrophenol in order of increasing retention time. Column: SP-1240, 100° to 210°C at 8°/min.

respective of whether they are hydrocarbon or heteroatom compounds. This is the result of the additional ionization process that occurs at the surface of the catalytic CFID source.

As anticipated, the TID-1-N₂ mode in Figure 8 provides high specificity and sensitivity to certain nitro- and polychlorinated-phenols. The responses to pentachlorophenol and 4-nitrophenol are more than 100 times larger than the CFID responses to these compounds. Comparing the responses of 4-nitrophenol and 2-nitrophenol again illustrates a significantly greater TID-1-N₂ response for the isomer with the nitro group located at the 4-position in the molecule.

In the third chromatogram in Figure 8, the low work function thermionic source (TID-1) was operated in a detector gas environment comprised of approximately equal flows of N₂ and air. This illustrates that the composition of the gas environment is an additional parameter which can be used to suppress the response to certain compounds while enhancing the response to others.

The bottom chromatogram in Figure 8 shows the analysis of the phenol sample for the case where both detector gases 1 and 2 are air. Responses are now obtained for all the chlorinated and nitro-phenols, with the dinitro-compounds continuing to give the dominant responses. The responses to 2-nitrophenol and 4-nitrophenol are now comparable, in contrast to the TID-1-N₂ response in which there was significant preference for the nitro group in the 4-location vs. the 2-location.

Figure 9 demonstrates the high specificity for lead alkyls in

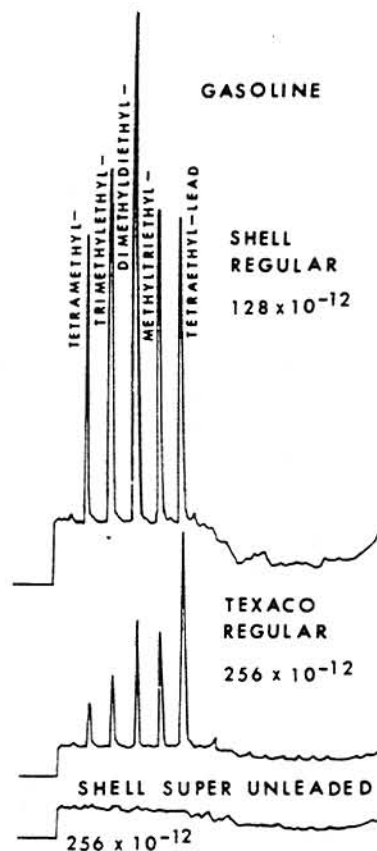


Figure 9. Chromatograms illustrating the specific detection of lead alkyls in gasoline using the remote FID. Data correspond to samples randomly obtained from local gasoline stations. Column: SP-2100, 50° to 230°C at 15°/min.

gasoline provided by the remote FID mode of detection. Previously, GC/atomic absorption spectroscopy (AAS) instrumentation has been described for the specific detection of lead alkyls in gasoline or other samples (15). By comparison with AAS, the remote FID provides excellent sensitivity and specificity, and is considerably simpler and less expensive to operate. The data shown in Figure 9 were obtained by direct injections of 0.8- μ l amounts of the gasolines onto a temperature-programmed, packed column.

The remote FID mode of detection has some configuration similarities to a hydrogen atmosphere flame ionization detector (HAFID), which has also been reported to provide specific detection of lead alkyls in gasoline (16). Both the remote FID and HAFID use a collector electrode well removed from the flame jet structure. However, the signals in the remote FID are largest with an oxygen-rich flame, whereas the HAFID requires a hydrogen-rich atmosphere doped with silane.

Gasoline is a readily available, complex mixture of organic compounds which is especially well suited to demonstrating the different responses obtained in the family of thermionic detection modes. Figure 10 shows multiple modes of analysis of a sample of regular gasoline. The chromatographic separation was performed on a packed column to purposely create the demanding situation in which many overlapping compounds are present in the detector volume at the same time. This situation is shown to be the case by the CFID response. When the same gasoline sample is chromatographed using the remote FID mode, selective responses are obtained only for the five lead alkyls. The remote FID data in Figure 10 were obtained with an FTID detector assembly and a TID-2 source mounted on a Model 3700 GC (Varian). The detector conditions were $H_2 = 30$, air = 200 ml/min; zero heating current to the TID-2 source; and flame ion-suppress voltage disconnected. The FTID-2 data in Figure 10 correspond to the following changed detector conditions; $H_2 = 30$, air = 80 ml/min; source heating current = 3.2 A; and ion-suppress voltage on. This change in conditions produced an FTID-2 response which was selective for the halogenated lead scavengers, ethylene dichloride (EDC) and ethylene dibromide (EDB). Figure 10 shows that selective responses to EDB and tetraethyllead (TEL) are also obtained in the TID-1-air mode, while the TID-1- N_2 mode provides responses to all the lead alkyls and lead scavengers. Note the large TID-1- N_2 response to TEL in comparison to tetramethyllead (TML), thereby illustrating a greater electronegative character for the TEL molecular structure vs. TML. Note also that precombustion of all samples in the flame of the remote FID mode provides a more uniform response for all lead alkyls irrespective of their original molecular structures.

Figure 11 illustrates six different detector responses in the analysis of diesel fuel containing a trace nitro-compound additive. It is clear from the CFID chromatogram that there are many unresolved component peaks in this packed column analysis. A primary objective of this particular set of analyses was to define the best method of measuring the amount of the nitro-compound added to such samples. The TID-1- N_2 mode clearly gives the best specificity and excellent sensitivity to the nitro-additive. The other specific modes of detection illustrate selective enhancement of other segments of this complex sample in addition to the nitro-additive. For example, the cluster of peaks at late retention times in the TID-2- H_2 /air chromatogram undoubtedly corresponds to other N-compounds in the sample. The two FTID-1 chromatograms illustrate that the H_2 -air mixture ratio is a further means of significantly altering the FTID re-

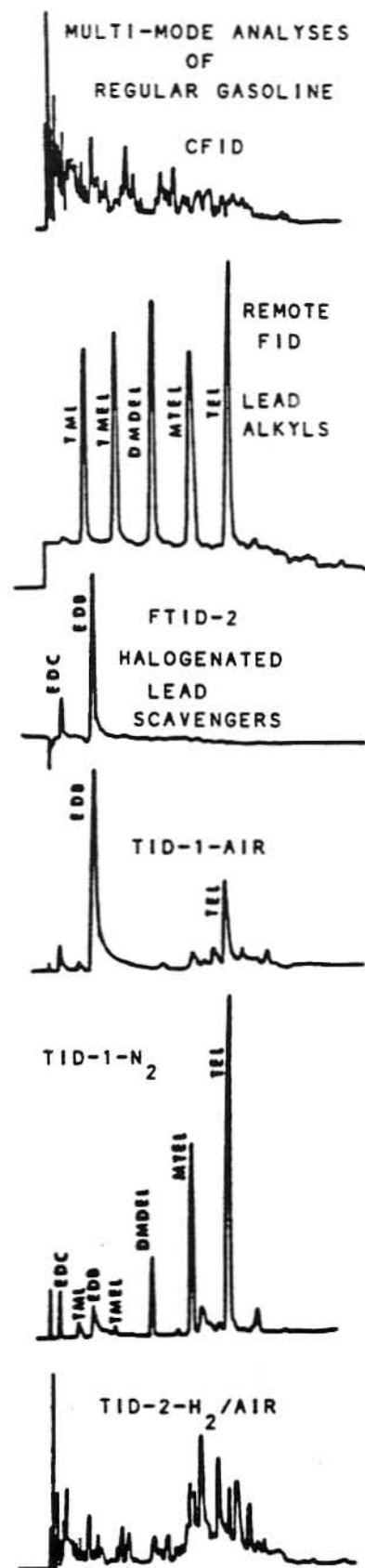


Figure 10. Chromatograms showing multiple modes of detector response to a sample of regular leaded gasoline. Column and program same as in Figure 9.

sponse. The mixture of 20 ml/min H_2 , 100 ml/min air represents an oxygen-rich flame, while the mixture of 35 ml/min H_2 , 70 ml/min air is a hydrogen-rich flame. Generally, stoichiometric or oxygen-rich flames are the most useful for the FTID.

Figure 12 shows multiple mode analyses of a commercial brand of cologne. These sets of chromatograms illustrate the advantageous use of the specific detection modes to enhance responses for trace fragrance components in colognes. The TID-1-N₂ mode is especially useful because it is non-destructive. Therefore, TID-1-N₂ emits exhaust gases that are characterized

by distinctly different fragrances that change with time as the various segments of the chromatogram elute. For the cologne shown, as well as for other brands that have been examined, the set of chromatograms obtained from the different modes of thermionic detection provide a characteristic fingerprint that distinguishes one brand from another.

All the modes of detection described in this article measure negative ionization currents. The magnitudes of these currents are very dependent on the electronegative character of the chemical species adjacent to the heated thermionic source.

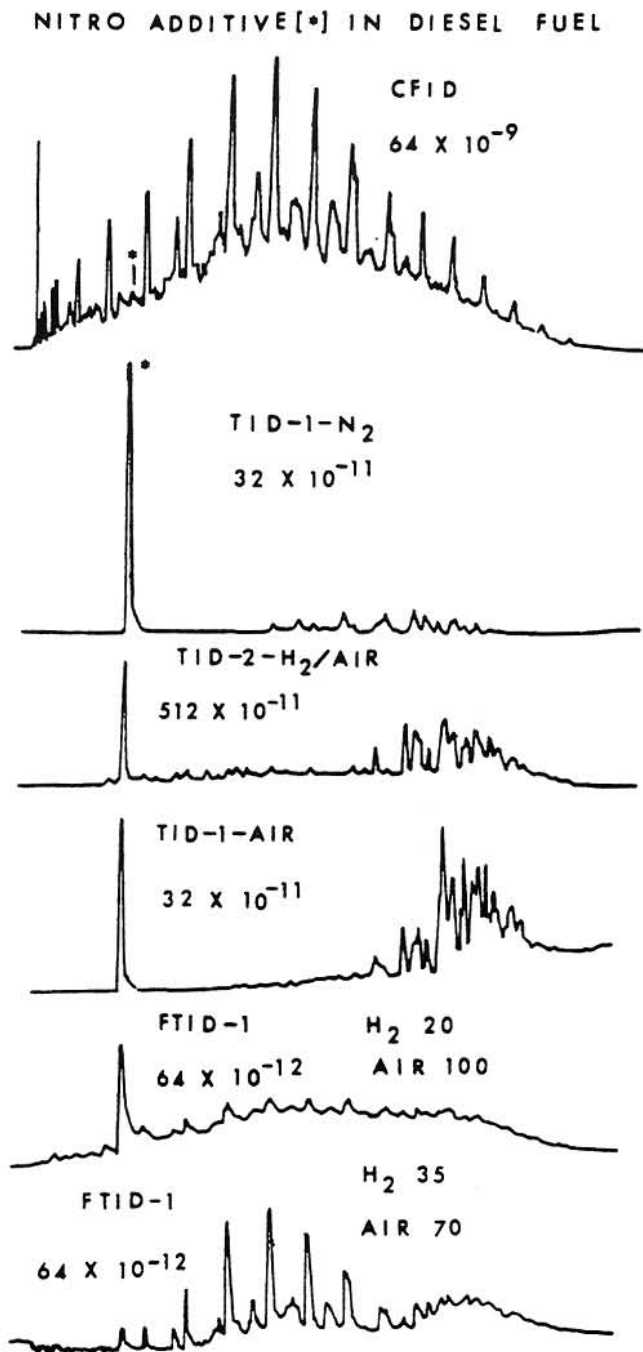


Figure 11. Chromatograms showing different detector responses to a sample of diesel fuel containing a trace nitro-compound additive identified by the astensck. Column SP-2100, 90° to 270°C at 10°/min.

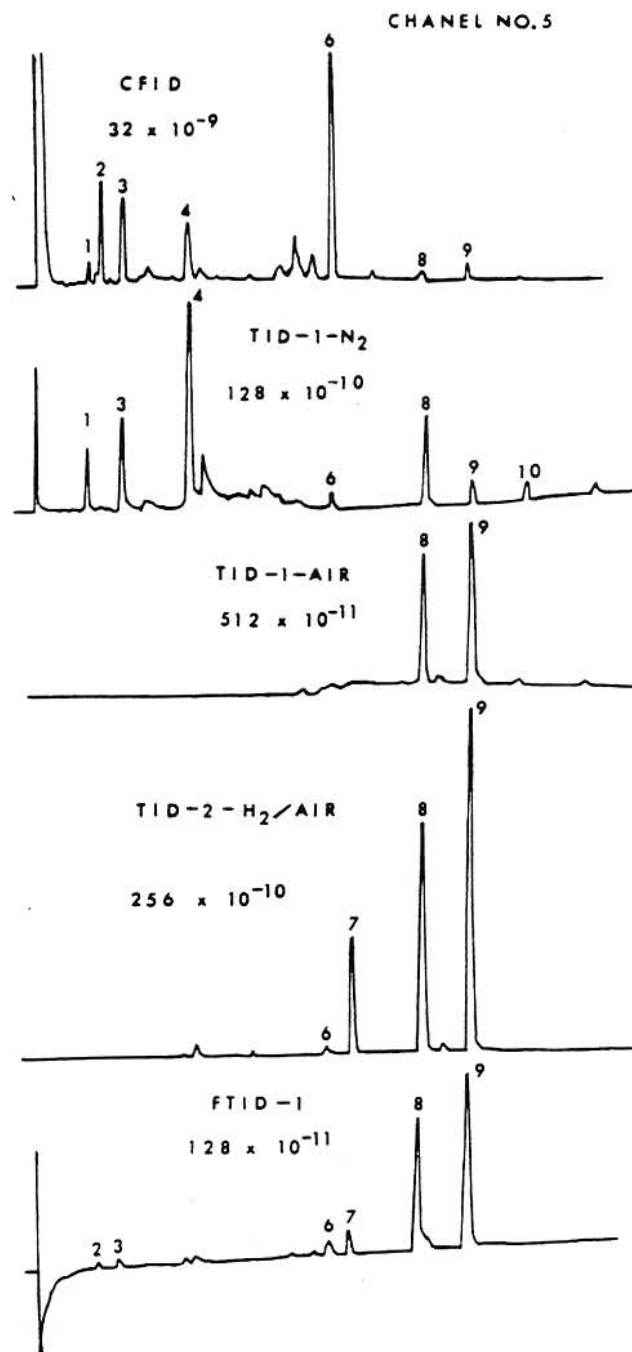


Figure 12. Chromatograms showing multiple modes of detector response to a brand of cologne randomly obtained. Column: SP-2100, 100° to 270°C at 16°/min.

Figure 13 illustrates how the response to a group of polychlorinated biphenyl (PCB) compounds changes with precombustion of the compounds. The data obtained with a TID-2 source in an air environment exhibit large differences in response between the different PCB compounds, similar to the known characteristics of an ECD. The FTID-2 data show that the precombustion of the PCBs yield negative ionization currents, which are larger in magnitude as well as more uniform per Cl atom. In this case of PCBs, precombustion improves the detectability by producing chemical species more electronegative than the original compounds. For other compound types, precombustion sometimes causes the opposite effect of producing combustion products which are not as electronegative as the original compound.

The data in Figure 14 provide an illustration of the relative electronegativity of phenol compounds before (TID-1-air) and after (FTID-1) combustion. The FTID-1 mode of operation can

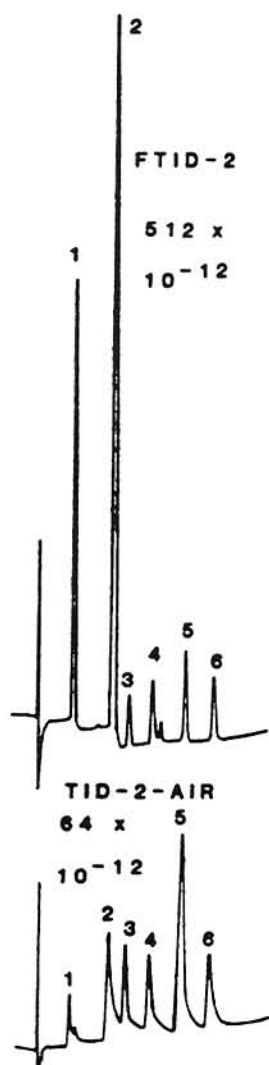


Figure 13. Chromatograms illustrating detector responses obtained before (TID-2-air) and after (FTID-2) combustion of polychlorinated biphenyl compounds. PCB peak identifications: 1=1000 ng 2-chloro-, 2=1000 ng 3,3'-dichloro-, 3=100 ng 2,4,5-trichloro-, 4=100 ng 2,2',4,4'-tetrachloro-, 5=100 ng 2,3',4,5', 6-pentachloro-, 6=100 ng 2,2',3,3',6,6'-hexachlorobiphenyl. Column: SP-2250, 200° to 270°C at 10°/min.

be easily converted to the TID-1-air mode by simply turning off the H_2 fuel to the FTID flame. Figure 14 shows that the FTID-1 mode produces a much more uniform response for all the chloro- and nitrophenols of this sample, but the absolute magnitudes of FTID response for the dinitrophenols, pentachlorophenol, and 4-nitrophenol are substantially lower than the TID-1-air response to these compounds.

Figure 15 illustrates the use of the TID-1-air mode to detect EDB in food products. A simple headspace technique was used for the data. One of the advantages of the TID-1-air mode is that it is insensitive to the large air component in the injection headspace vapors. Hence, all chromatographic peaks that are obtained may be attributed to vapors emanating from the food product itself. In addition to EDB, this cake mix sample provided other large TID-1-air signals, thereby suggesting the presence of other halogenated or electronegative constituents that may be of interest in such products. With a better optimization of GC column for the EDB separation, this simple technique should be capable of EDB detection in the 1 to 10 ppb range of concentration in the food product.

Figure 16 illustrates that one difference between a CFID and an FID is that the CFID will provide significant responses to halogenated compounds. Figure 17 further shows that the CFID

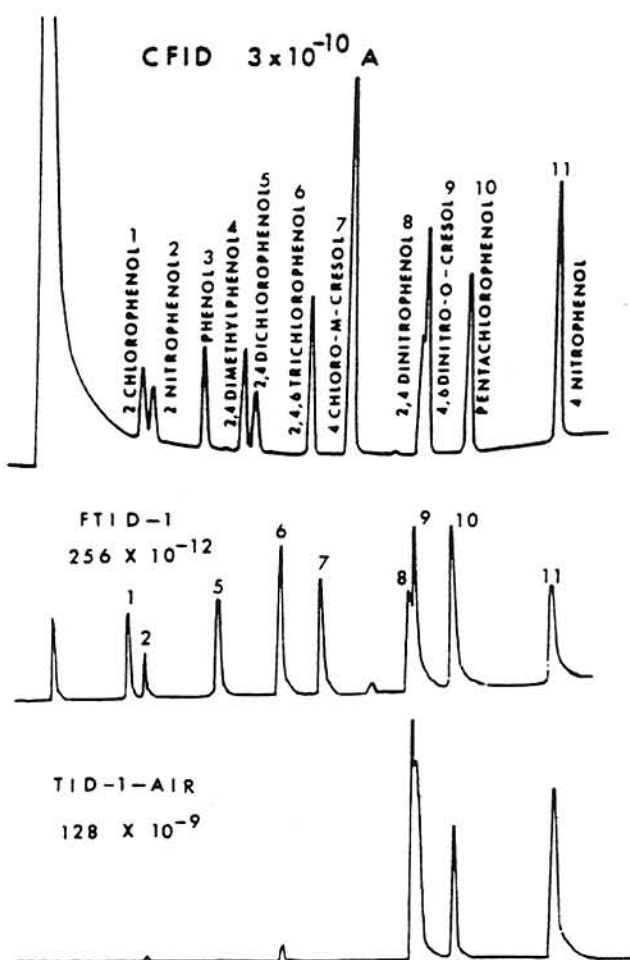


Figure 14. Chromatograms illustrating detector responses obtained before (TID-1-air) and after (FTID-1) combustion of phenol compounds. Sample, column, and program were the same as in Figure 8.

response to halogenated compounds can be selectively enhanced by increasing the source heating current, while the CFID responses to hydrocarbons remain unchanged. Consequently, by judiciously adjusting the source heating current, the CFID response to halogenated and some other heteroatom compounds can be tuned to yield about the same response factor as obtained for hydrocarbons.

Summary

The preceding data have demonstrated that thermionic ionization techniques and equipment have applications in gas chromatography that go well beyond the usual NP detection, such as that of TID-2-H₂/air. To achieve the best possible signal-to-noise ratio and specificity for each mode of detection, the specific chemical composition of the thermionic emission source needs to be matched with the temperature and gas phase environment in which the source is operated.

From their extensive use in NP detection, thermionic ionization detectors are known to often exhibit decreasing sensitivity with increasing operating time as a result of depletion of the thermionic source activity. Consequently, the thermionic source usually needs to be replaced at periodic intervals. Of the different modes of detection described in this report, the NP mode is the most demanding with regard to the operating life of the thermionic source. Generally, in modes of detection (e.g., TID-1-N₂) where the operating temperature is lower and the gas environment is less reactive, the thermionic sources maintain their responses over longer periods of time. For all modes of detection, a practical guideline for achieving the longest possible source lifetime is to operate the thermionic source just hot enough to achieve the response required.

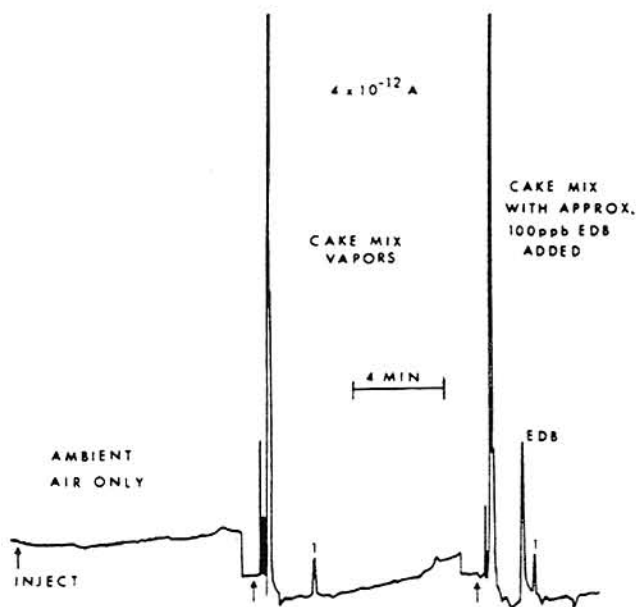


Figure 15. Example of TID-1-air application to determine EDB in cake mix. Sample: headspace vapors from 0.7 gm Duncan Hines Deluxe Yellow Cake Mix in a sealed 2-ml vial. Sampling procedure: heat sealed sample vial to 120°C; extract 200 μ l headspace vapor with gas-tight syringe; inject into GC and start column oven temperature program. Column: SP-2100, 40° to 110°C at 10°/min.

The developments of thermionic techniques in GC have also spawned applications of the technology in liquid chromatography detection (17), thin layer chromatography (18), and mass spectrometry ion sources (19). Since there remain to be studied many different combinations of thermionic source compositions, thermionic source temperatures, and gas environment compositions, it is probable that the technology will continue to evolve in coming years.

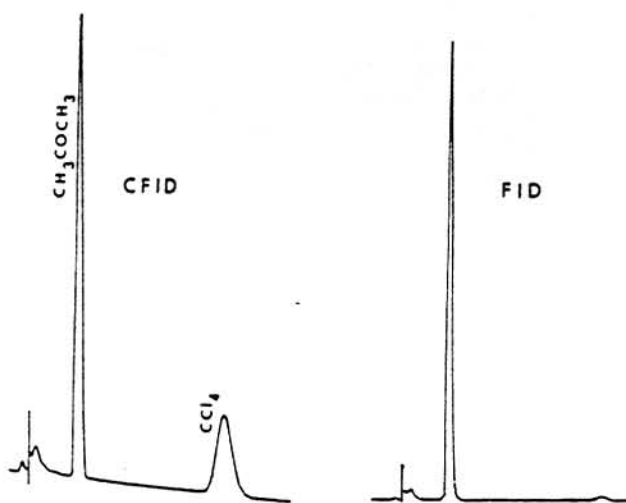


Figure 16. Comparison of CFID and FID responses to a water sample containing acetone and carbon tetrachloride. Column: Chromosorb 102, isothermal 120°C.

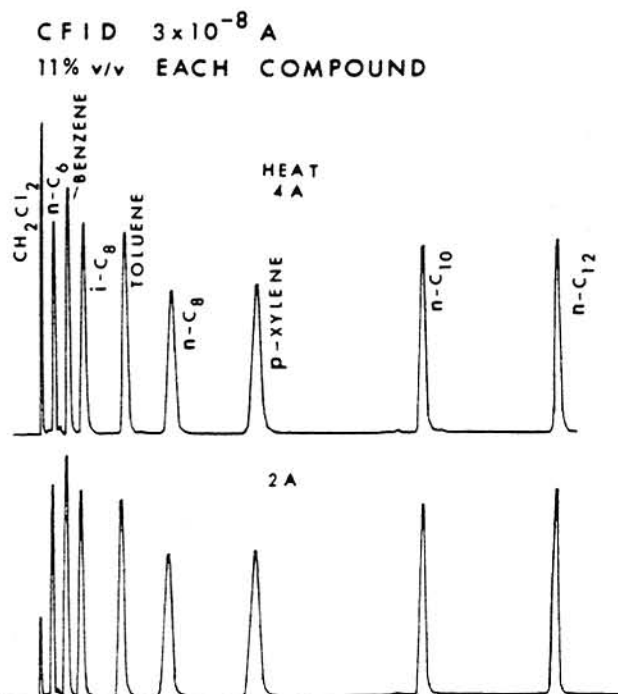


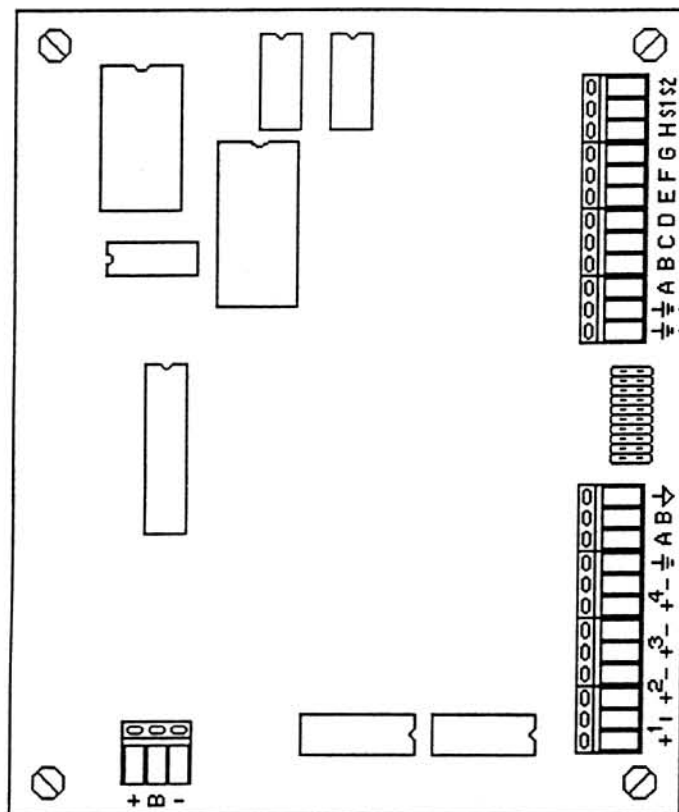
Figure 17. Comparison of CFID responses to sample of methylene chloride and various hydrocarbons at two different magnitudes of heating current to the catalytic source. Column: SP-2100, 40° to 180°C at 10°/min.

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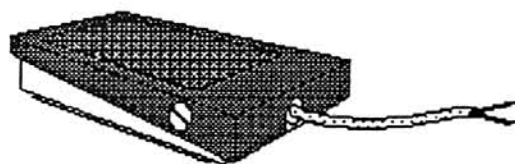
The SRI serial port data system offers a remote starting capability as a standard feature. This permits the user to start the data system (and SRI gas chromatograph, if attached) by means of a switch closure, such as a footswitch. In some applications, such as when a different brand of gas chromatograph is being used with the serial data system, the chromatograph offers a remote start signal output (or switch closure output) that permits starting an integrator or other device when the START button is pressed on the chromatograph's on-board control panel. Typically, this signal can be used to start the serial data system.



Serial port data system interface board (Lawson 202)

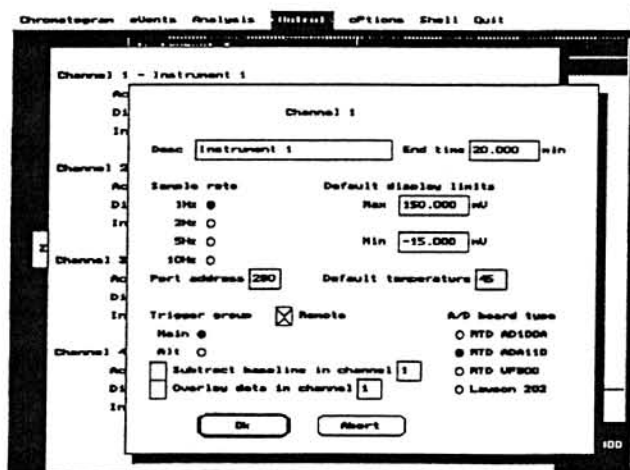
A switch closure between terminal S1 and GROUND remotely starts the channels assigned to the MAIN control group.

A switch closure between terminal S2 and GROUND remotely starts the channels assigned to the ALTERNATE control group.



A simple footswitch or pushbutton connected as shown in the diagram at left may be used to provide the switch closure required by the serial data system

Once the hardware has been configured for operation, the PeakSimple program must be instructed to seek a remote start signal for the appropriate control group. This is selected from the CONTROLS - CHANNELS - DETAILS screen for each channel in use.



PeakSimple DETAILS screen

The REMOTE box located in the lower central portion of the screen should be "X"ed on. As shown at left, the screen indicates the selection of the remote start feature for the MAIN control group. The factory defaulted MAIN group channels are Ch. 1 and Ch. 2. By default, Ch.3 and Ch. 4 are set to the ALTERNATE control

GC DETECTORS

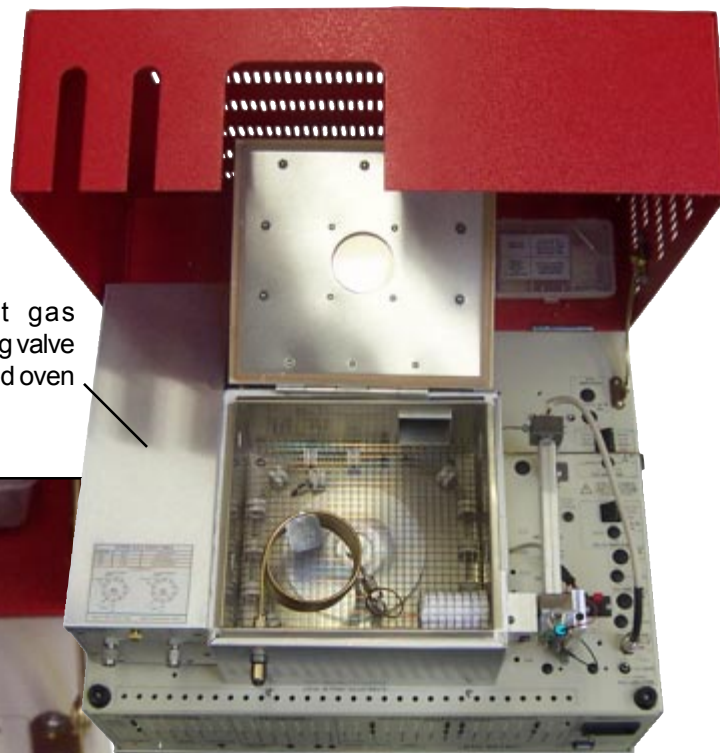
Reduction Gas Detector - RGD

Overview

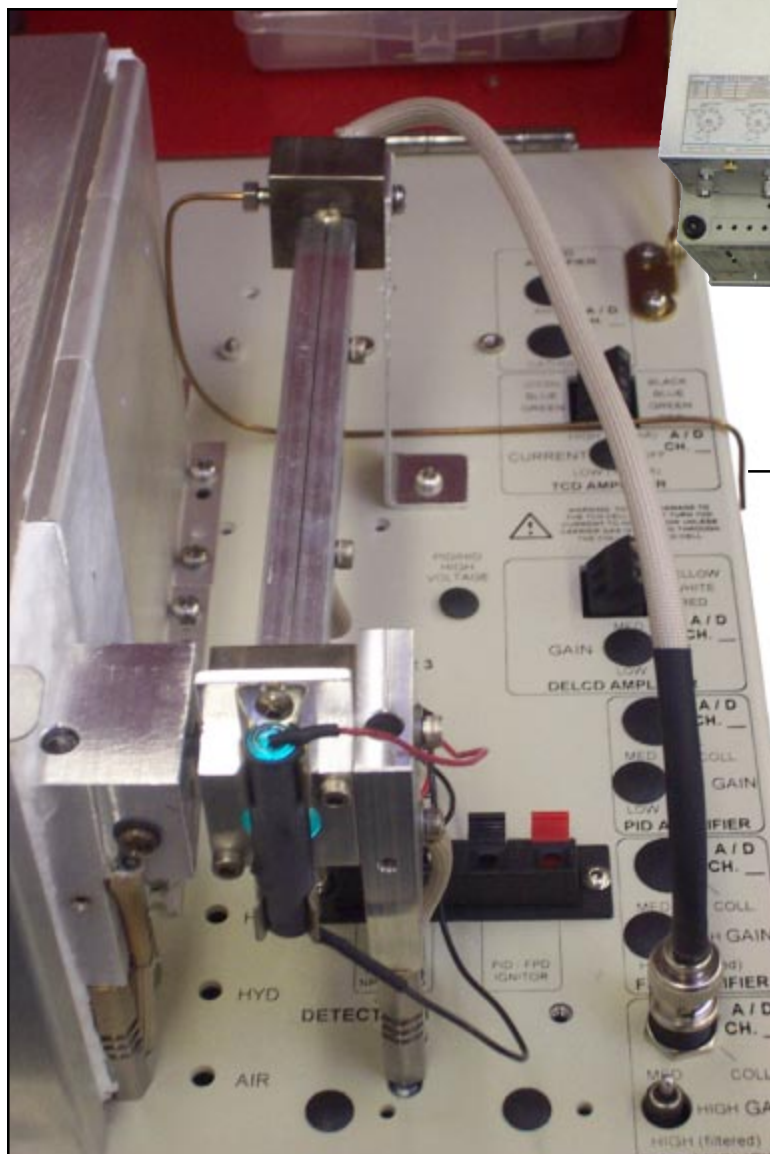
The SRI Reduction Gas Detector is the most sensitive detector available for hydrogen, carbon monoxide, and other reducing gases. Low ppb detection limits are achievable for hydrogen. Detection limits in the ppt range are achievable for carbon monoxide.

The RGD comes with a 10-port gas sampling valve and 1mL sample loop for injecting samples.

10-port gas sampling valve in heated oven



Exit tube



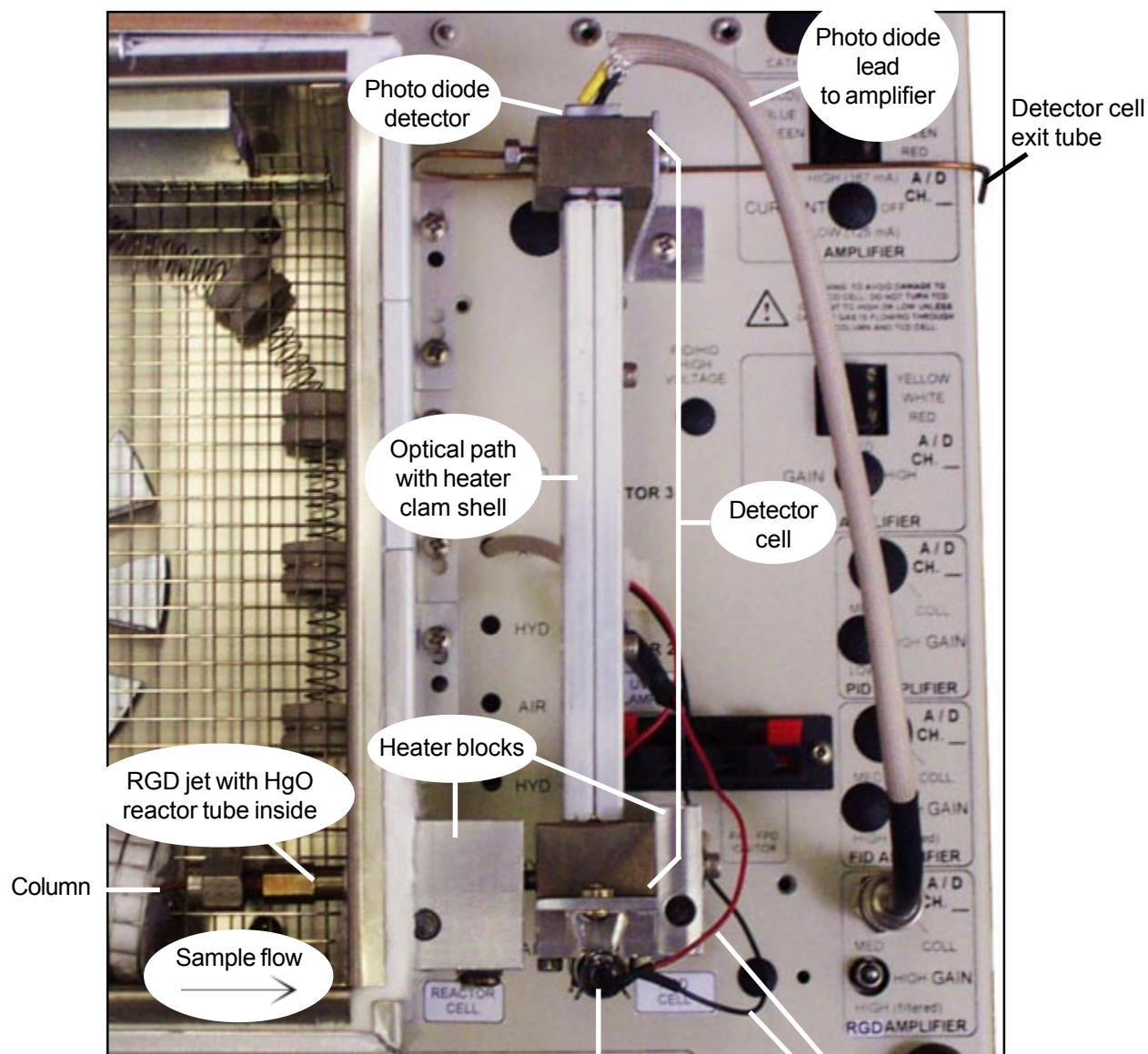
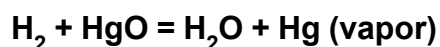
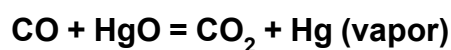
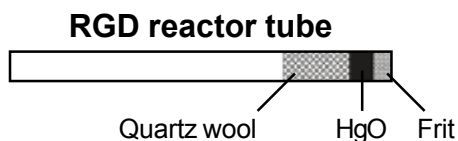
The RGD exit tube is not factory equipped with a mercury vapor filter, since it was designed for trace detection. Depending on the concentration of the reducing gases in the sample, you may want to add a filter for safety. The amount of mercury vapor produced is proportional to the concentration of reducing gases in the sample.

GC DETECTORS

Reduction Gas Detector - RGD

Theory of Operation

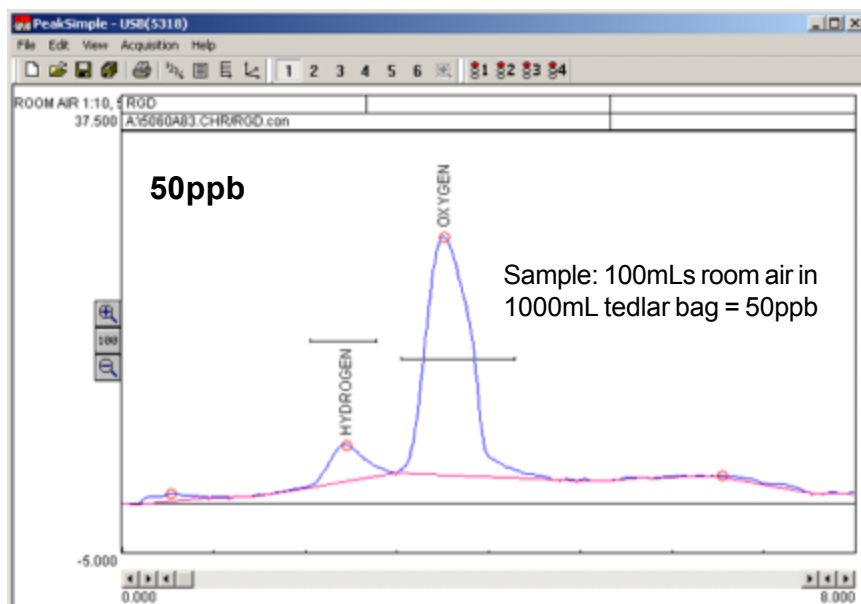
The SRI RGD detector uses a mercuric oxide (HgO) reaction tube and a mercury lamp in a heated UV detector cell. The reaction tube is heated to 260-300°C. Located immediately downstream of the reaction tube, the UV detector cell is heated to 170°C. The UV detector cell is equipped with a mercury lamp and a UV photodiode. When a reducing gas such as carbon monoxide elutes from the GC column, it reacts with the HgO to form gaseous mercury vapor, which is then swept into the UV cell. The gaseous mercury absorbs the UV light from the mercury lamp as it flows through the cell. The change in transmittance is converted by the data system into an absorbance output (1.00 volt per absorbance unit), which is linearly proportional to the amount of reducing gas.



GC DETECTORS

Reduction Gas Detector - RGD

Expected Performance



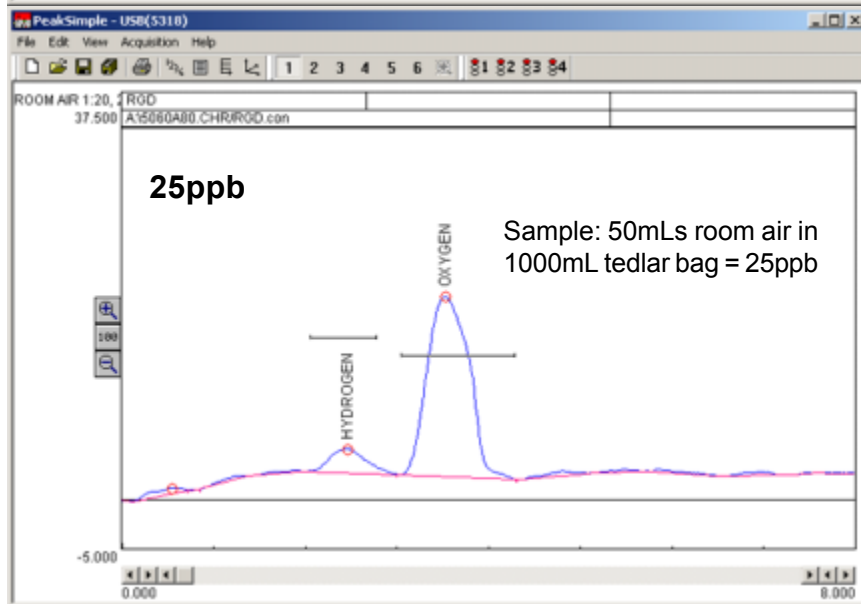
The following two chromatograms were produced with an SRI RGD equipped GC. Room air samples were diluted with nitrogen in tedlar bags, then injected by gas sampling valve onto the Molecular Sieve packed column.

Results:

Component	Ret.Time	Area
HYDROGEN	2.433	100.8250
OXYGEN	3.500	796.4090

Common Analytical Parameters:

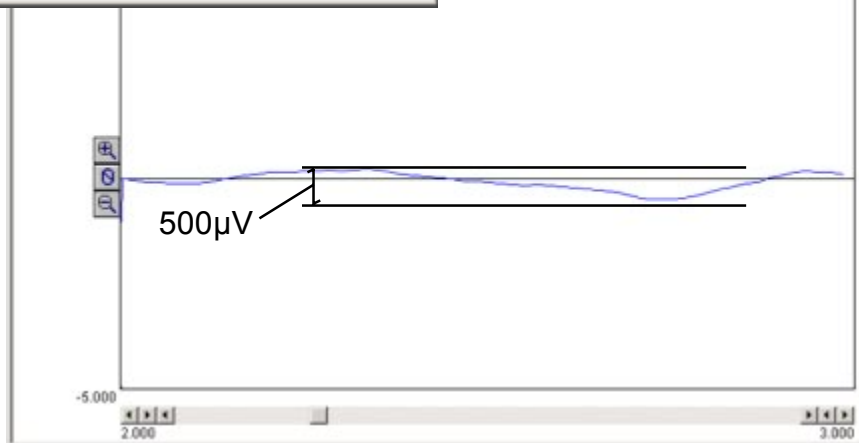
Valve injection; 1mL sample loop
 Column: 2-meter Molecular Sieve 13X
 Carrier: helium at 5mLs/minute
 RGD gain: HIGH
 Reactor temperature: 290°C
 Detector temperature: 170°C
 Valve temperature: 60°C



Results:

Component	Ret.Time	Area
HYDROGEN	2.450	55.2755
OXYGEN	3.516	571.6580

This chromatogram shows the noise level of the RGD detector, which is approximately 500µV from peak to peak.



GC DETECTORS

Reduction Gas Detector - RGD

General Operating Procedure

1. When your RGD GC arrives, the photo diode amplifier leads will have been disconnected for transport. Locate the insulated electrical lead with two protruding wires: 1 black, 1 yellow. These wires plug into the back end of the RGD assembly, where the photo diode is located. The black is the ground wire; plug it onto the upper prong. Plug the yellow wire onto the lower prong.

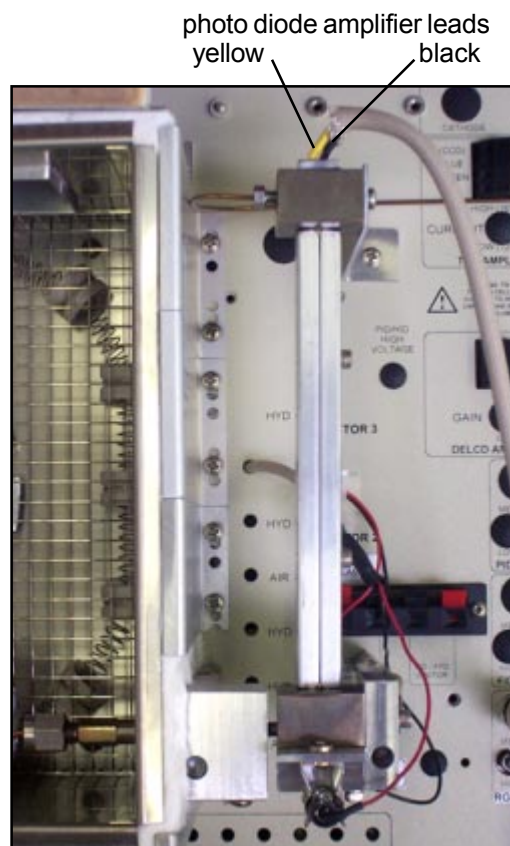
2. Turn ON the main GC power (the switch is located on the left-hand side of the GC).

3. Connect helium or nitrogen carrier gas to the fittings on the left-hand side of the GC. The pressure for the required flow is printed on the right-hand side of the GC. The required flow for the RGD is 5mLs/minute. This flow gives the best response.

4. Turn ON the detector lamp by flipping the switch on the GC's front control panel. Detector heat is factory set at 170°C, which is hot enough to keep the mercury vapor from condensing. The reactor is factory set at 290°C (a higher temperature gives greater sensitivity).

5. The mercuric oxide needs time to equilibrate when it is first installed, or when the reactor tube is changed; 24 hours works well. Leave the reactor and detector temperatures on the above settings continuously with the carrier gas flowing for day to day operation.

6. The first analytical run of the day should be discarded, because the purged reactor will generate unusually high area counts which will not be reproducible once the detector equilibrates. The reactor can also be poisoned by very high sample concentrations. Purge time with clean carrier gas is usually the best cure.

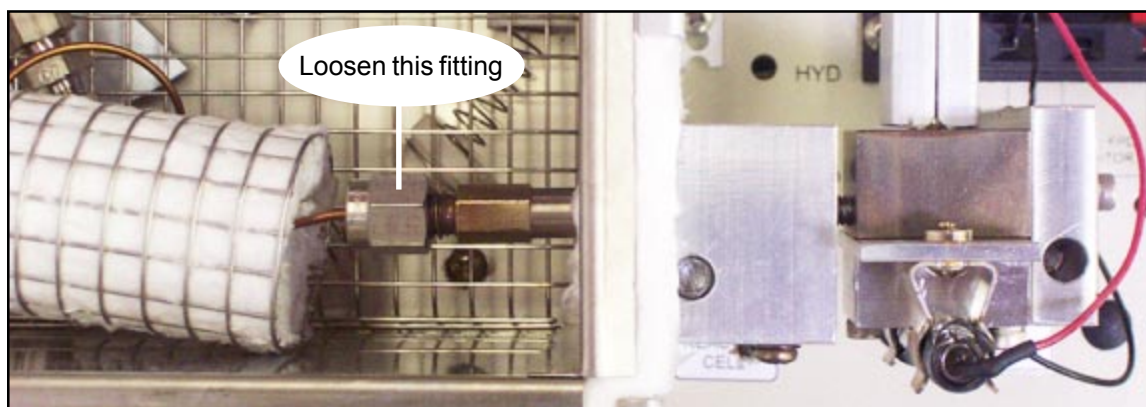


Maintenance

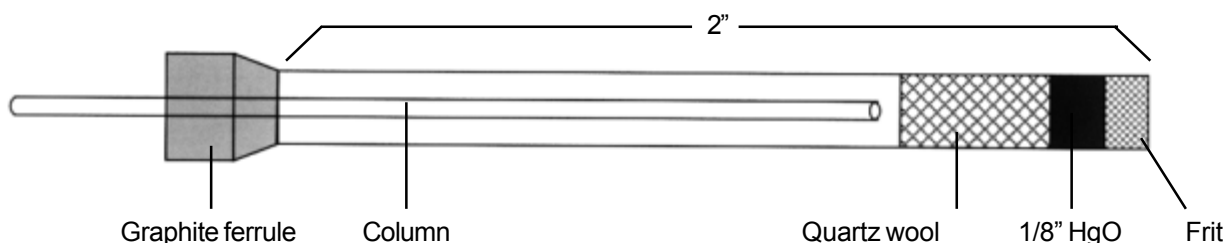
Reaction Tube Packing and Replacement

SRI provides you with one replacement reactor tube in your accessories kit. However, you may also remove the mercuric oxide (HgO) and re-pack the reactor tube, for which you will need fresh HgO and quartz wool. HgO powder is available from Sigma-Aldrich: part #203793-2G: www.sigmaaldrich.com, or call: 1-800-325-5832. Quartz wool is available from Restek: catalog #20999: www.restekcorp.com, or call 1-800-356-1688.

To remove the reactor tube, loosen the fitting that secures it to the column and RGD jet, and pull the reactor tube out.



Remove the quartz wool and HgO with a long sharp object, and dispose of them properly. Wipe any HgO off the sharp object with a disposable wipe or tissue and discard. Re-pack the reactor tube with 1/8" of HgO and add fresh quartz wool. The HgO must cover the face of the frit entirely to force the sample through the HgO and into the UV cell.

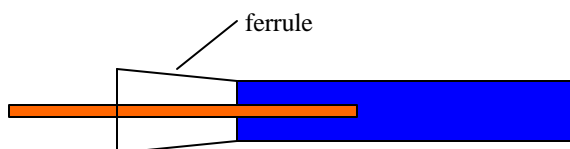


The graphite ferrule seals the end of the reaction tube around the column. Change the graphite ferrule when you change the reaction tube.

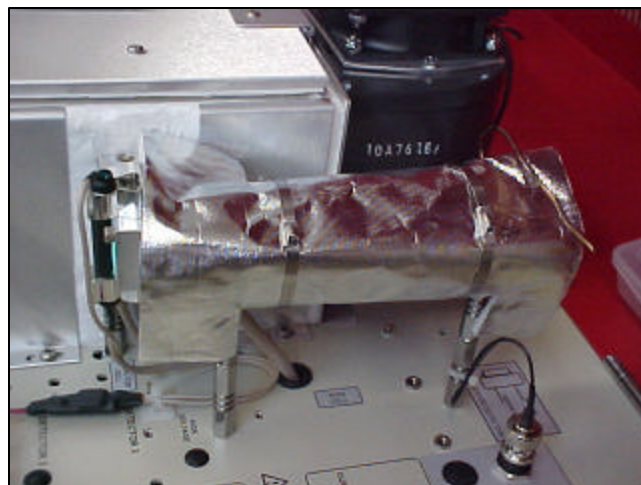
WARNING! Always avoid contact with the HgO. Wear protective gloves and eyecovers, and take care not to inhale the powder.

Packing the RGD reaction tube

- 1) The RGD detector is located at the right rear of the SRI 8610C GC's column oven.
- 2) A 1/16" od stainless tube and 1/8 to 1/16 soft graphite reducing ferrule connects the column effluent to the RGD's inlet bulkhead fitting inside the column oven. The soft graphite ferrule seals against the reaction tube to insure that column effluent actually passes through the reaction tube and not around it.



- 3 Withdraw the reaction tube from the detector inlet. Be careful, the tube may be HOT.

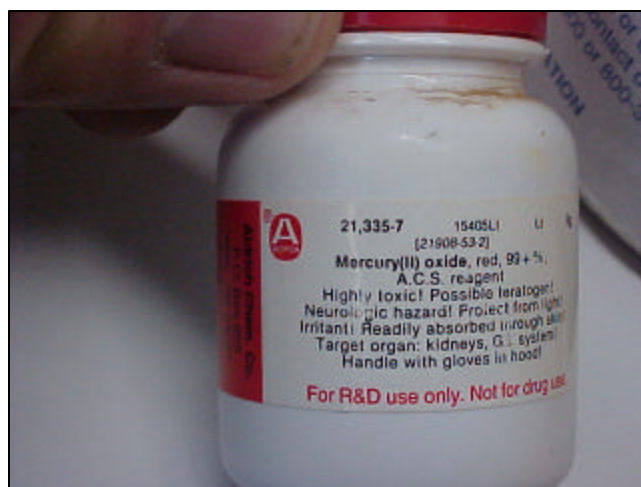


Packing the RGD reaction tube

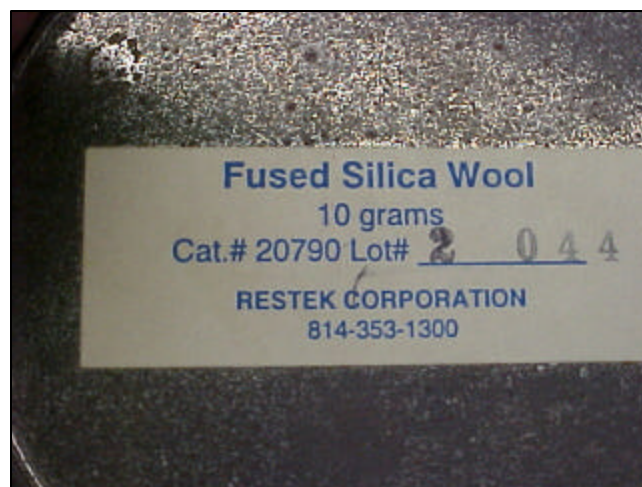
4 The reaction tube is open on one side and is closed with a stainless steel frit on the other. The tube is shipped empty. It is the customer's responsibility to pack the tube with mercuric oxide (HgO).



5 Obtain HgO power from Aldrich or other supplier.



6 Obtain quartz wool (fused silica wool) from restek or other supplier



Packing the RGD reaction tube

7 Transfer a small amount of HgO powder into a dish.



8 Roll a bit of the quartz wool into a cigar shaped plug and coat it with the HgO powder. Insert the plug into the blank reaction tube. Optionally insert another plug of un-coated quartz wool after.



9 Insert the packed reaction tube into the detector inlet. Set the reaction tube temperature to 260C. RGD cell temperature to 180C. Note the transmittance reading on the detector before heating the tube. When the tube is first heated some mercury vapor may be released lowering the transmittance. Condition the system until the background drops to a useable level.



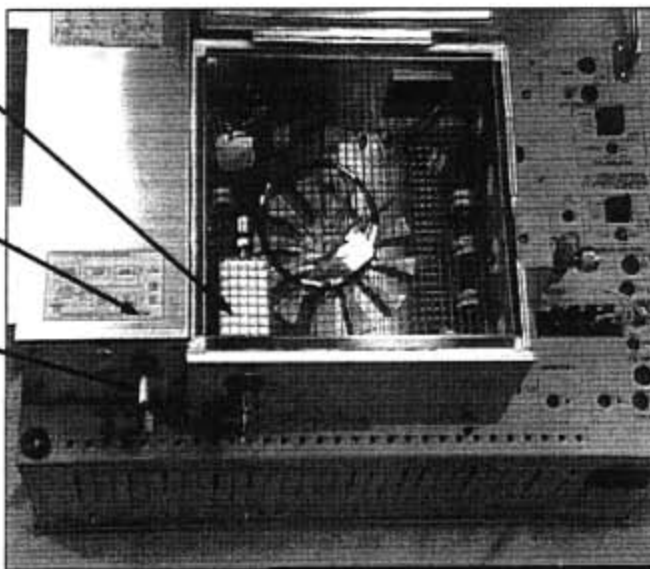
Chapter: Injectors

Topic: Heated Split/Splitless Injector

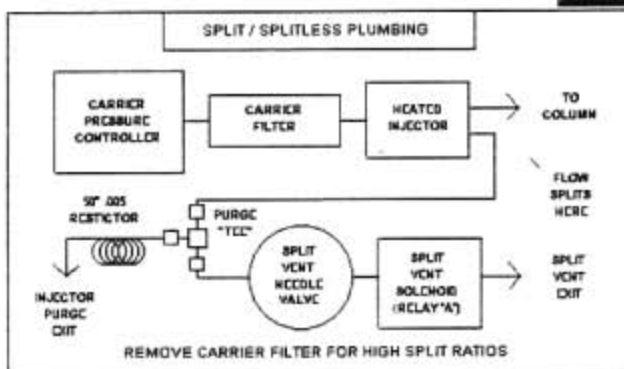
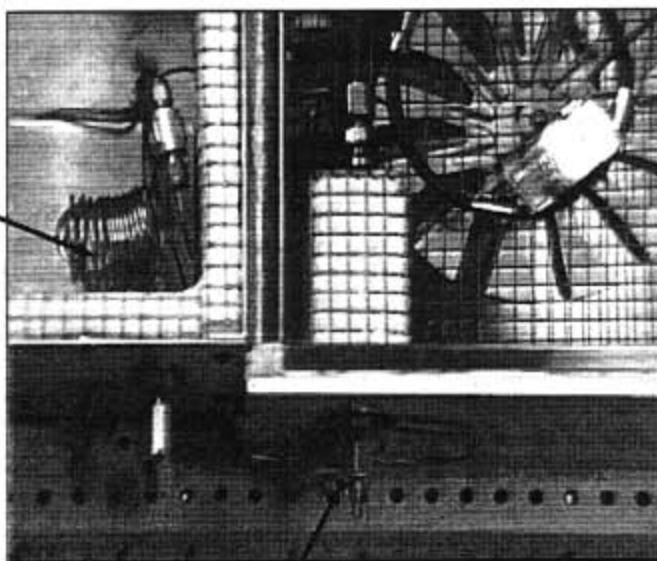
The Heated Split/Splitless Injector can be mounted on the 8610C or 310 GC. It is shown installed on the 8610C GC at right.

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

The split flow is adjusted by rotating this knob.



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

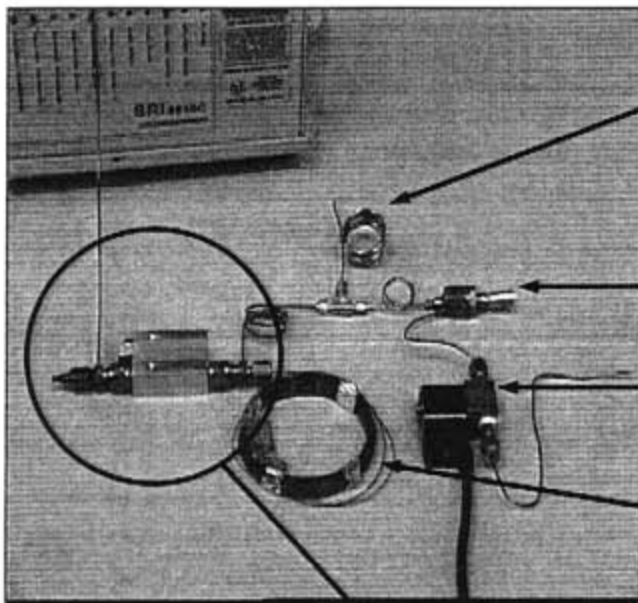


Septum nut mounted on front of Split/Splitless Injector.

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

Chapter: Injectors

Topic: Heated Split/Splitless Injector

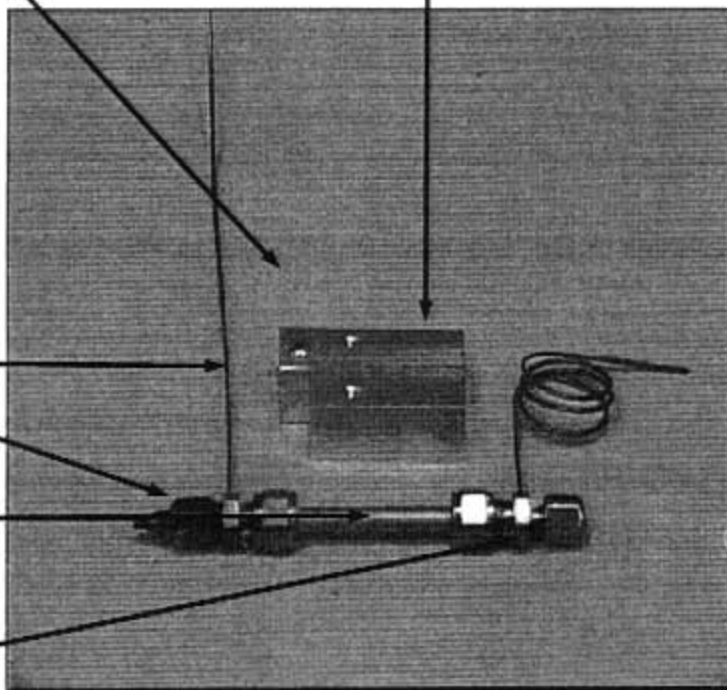


The Heated Split/Splitless Injector parts are shown at left removed from the GC for clarity.

- 1) Injector purge restrictor. A few ml/min of carrier gas continuously exit the injector through this restrictor tubing to prevent high boiling point analytes from diffusing back into the injector.
- 2) Precision needle valve for adjustment of split flow rate.
- 3) Split flow solenoid turns split on/off under control of the PeakSimple data system.
- 4) Column is secured into injector using nut and graphite ferrule.
- 5) Aluminum heater block contains heater cartridge and Type K thermocouple.

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

- 1) Carrier gas inlet tubing.
- 2) Septum nut and septum.
- 3) SRI stainless steel injector liner.
- 4) End fitting where column connects and split flow exits to purge vent and needle valve.

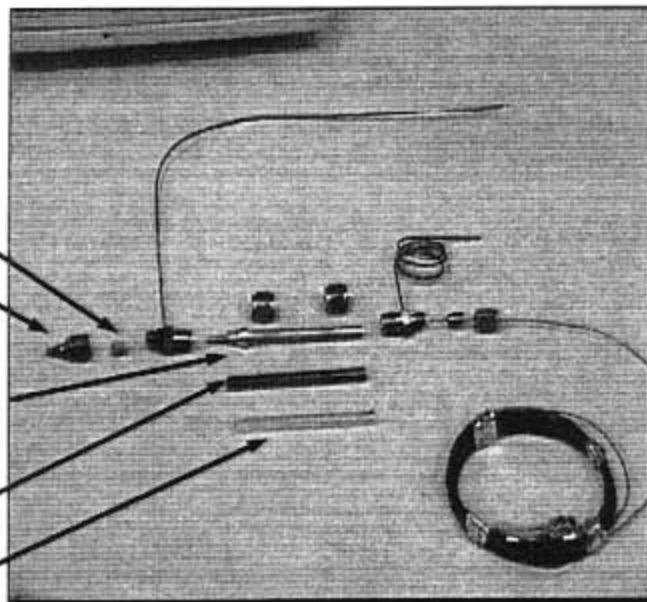


Chapter: Injectors

Topic: Heated Split/Splitless Injector

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

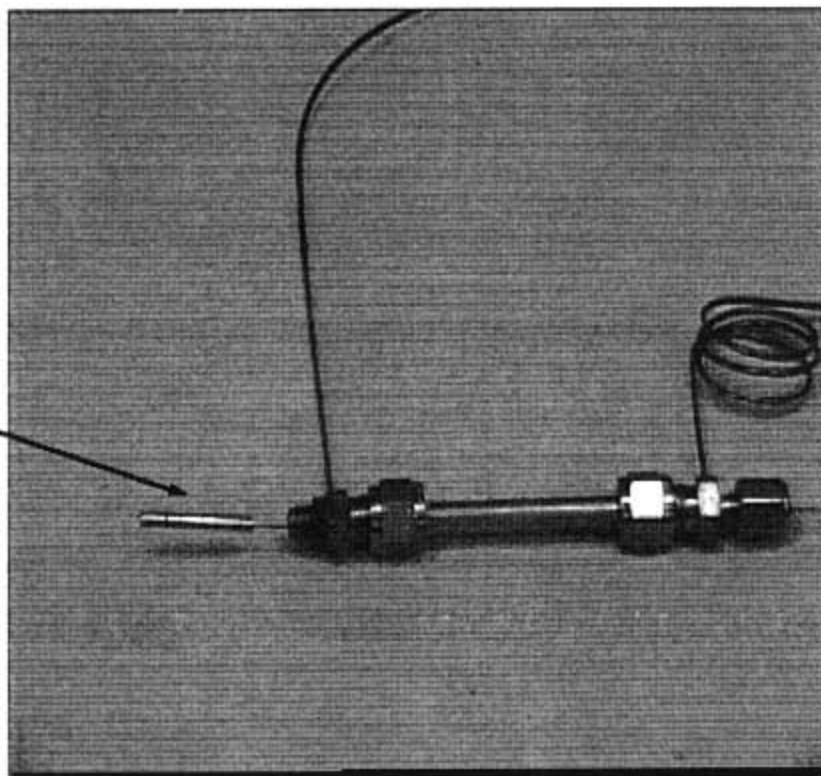
Septum
Septum nut.
SRI stainless liner with wide-bore column adapter.
Restek Silco-Steel liner.
Supelco glass liner



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

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

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

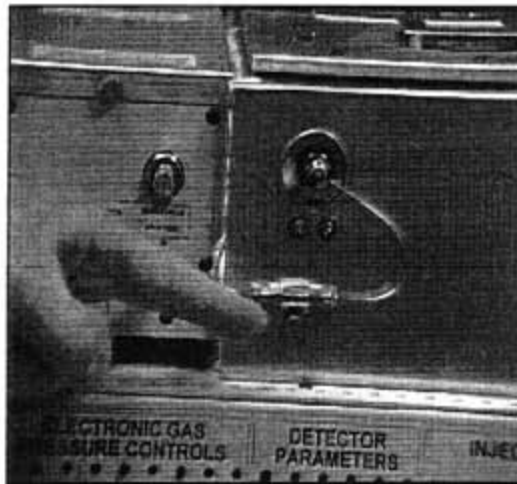


Chapter: Injectors

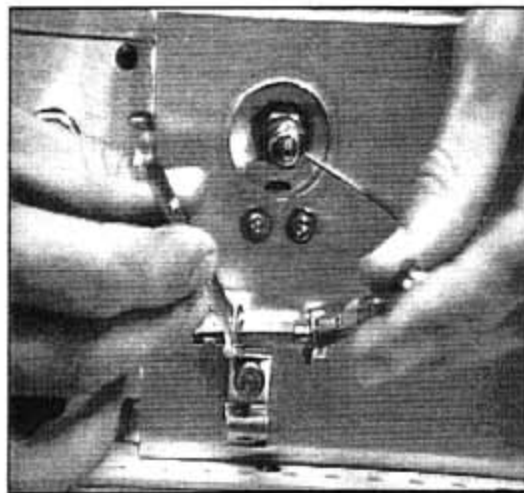
Topic: Heated Split/Splitless Injector

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

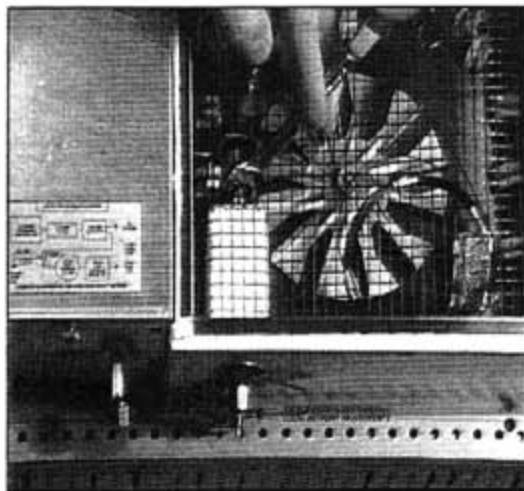
1) Loosen the brass thumbscrew holding the 1/16" stainless union in the carrier gas supply line.



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



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

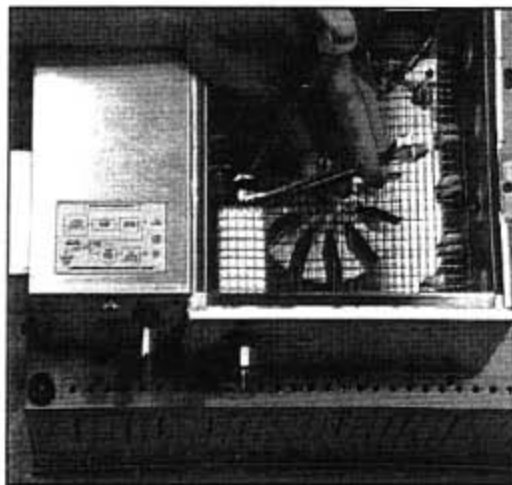


Chapter: Injectors

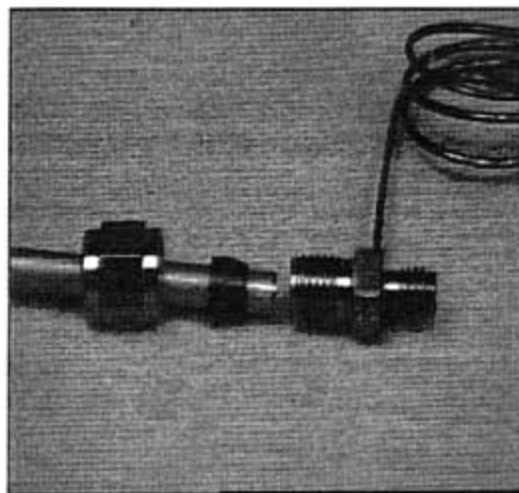
Topic: Heated Split/Splitless Injector

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

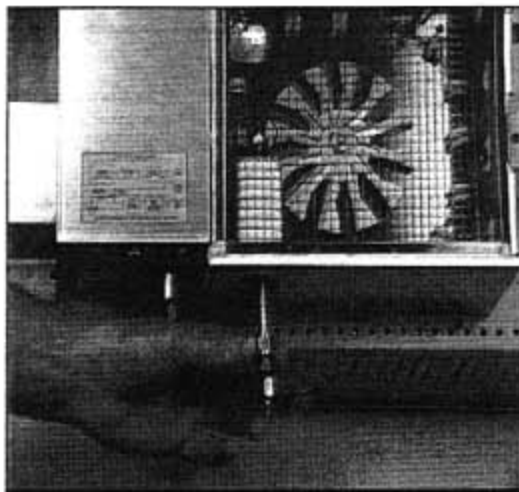
4) Using a 1/2" and 9/16" wrench remove the swagelok type nut securing the end fitting.



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



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

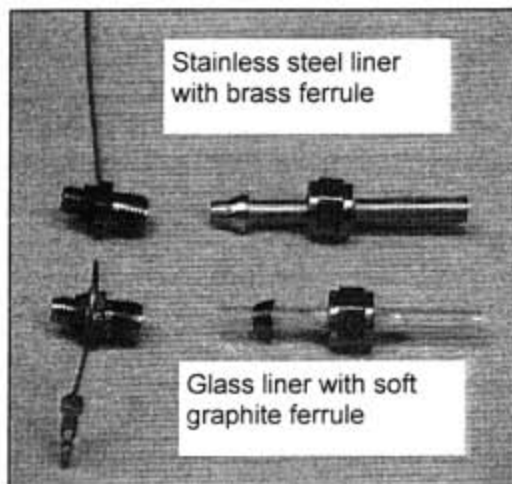


Chapter: Injectors

Topic: Heated Split/Splitless Injector

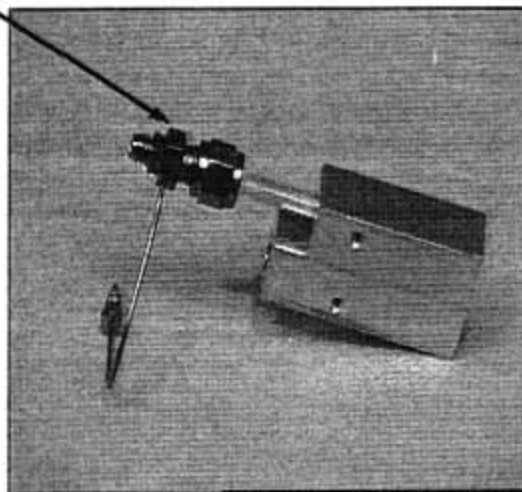
To replace the injection liner:

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

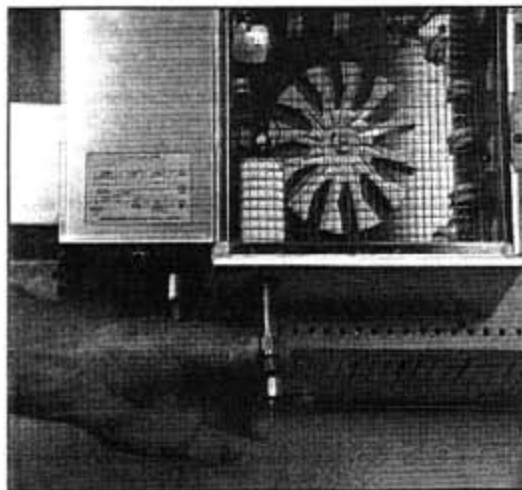


Align the flat surfaces of the nut and fitting

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



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

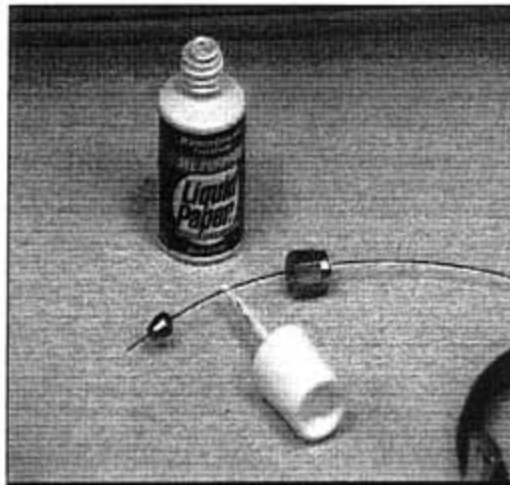


Chapter: Injectors

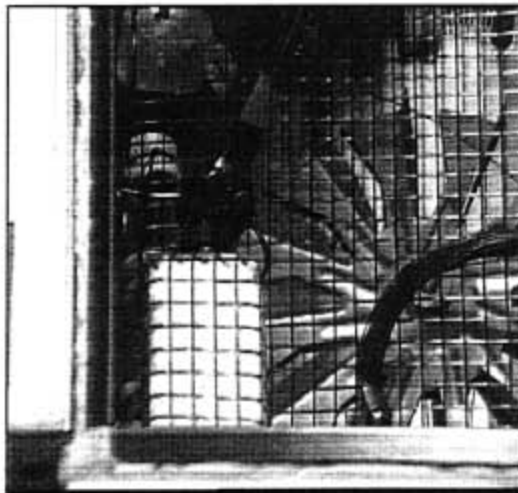
Topic: Heated Split/Splitless Injector

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

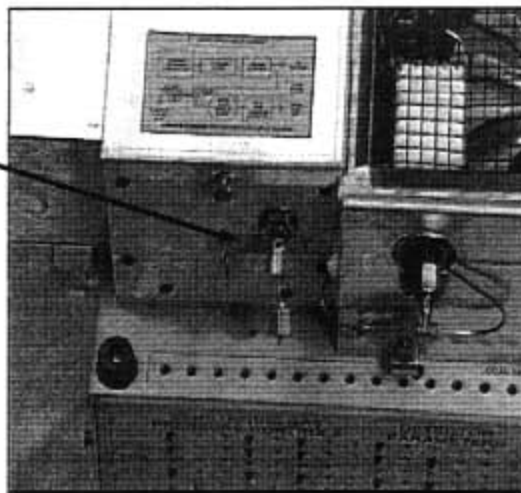
10) Use some white correction fluid to mark the column approximately 1.5" (4cm) from the end. Slip a 1/8" swagelok type nut and 1/8" to .5mm graphite reducing ferrule over the column. You can use soft or hard graphite ferrules.



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



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



Chapter: Injectors

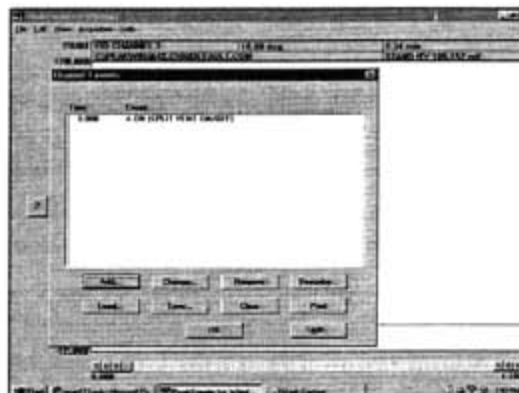
Topic: Heated Split/Splitless Injector

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

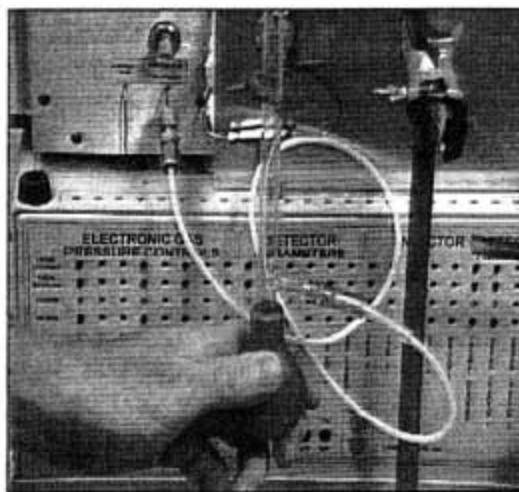
13) The split vent must be opened by activating one of the relay outputs from the PeakSimple data system. Typically Relay A is used to activate the split vent solenoid. If another relay has been allocated to this function, it will be annotated in the relay assignment chart located on the right hand side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



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

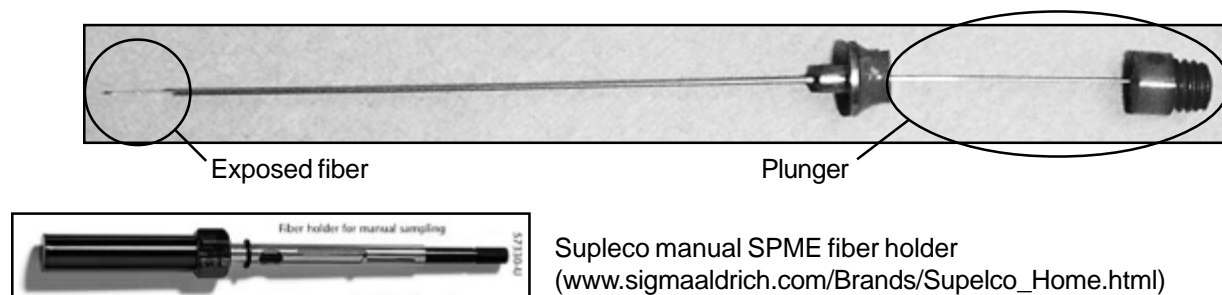


15) Carrier gas will only exit the split vent when Relay A is activated. Connect your bubble-meter or other flow measuring device to the split vent exit tube. Activate Relay A. Make sure the red lid of the GC is down (lid interlock disables solenoid function). Adjust the needle valve to obtain desired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



Solid Phase Microextraction

SPME injections may be made with the following SRI syringe injectors: split/splitless, PTV, and heated vaporization.



Pictured above is a typical SPME fiber assembly, and a Supleco SPME holder. SRI prefers to use the fiber without a holder, after removing the spring. Without the holder, the SPME assembly is lighter, and easier to leave in the injection port for desorption and conditioning. Whether you use a holder or not, be sure to handle the fragile fiber with care.

1. Load the SPME fiber by exposing it to a sample inside a vial. The length of exposure time depends upon the sample and the fiber coating.

2. Pull the fiber back into the barrel. Inject into a hot injector by inserting the syringe, pushing the plunger to expose the fiber in the megabore column, and letting it remain there long enough to release all the analytes. Pull the plunger back in so the fiber doesn't break, and withdraw the syringe.

3. Condition the SPME fiber between runs by leaving it in the hot injector port long enough to remove any analytes.



Carefully insert the SPME syringe into the hot injector port and push the plunger

4. SRI recommends that you do SPME injection directly onto a megabore (0.53mm) column, like any other syringe injection. If you want to use a narrowbore column, SRI can provide you with a simple adaptor (see next page).

GC INJECTORS

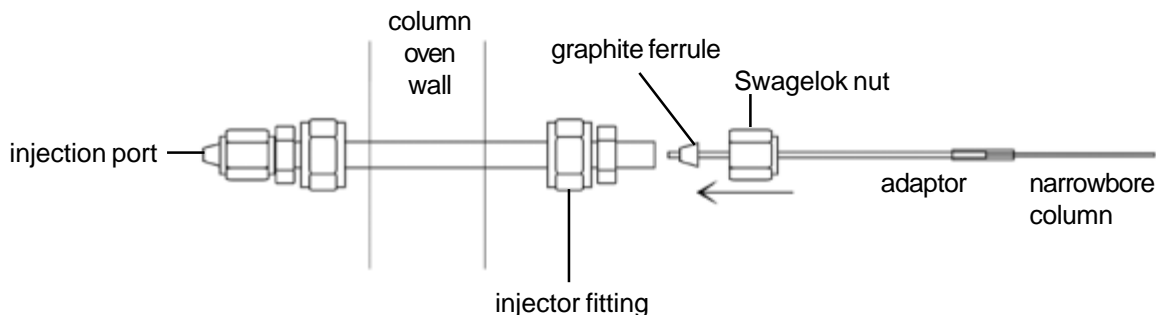
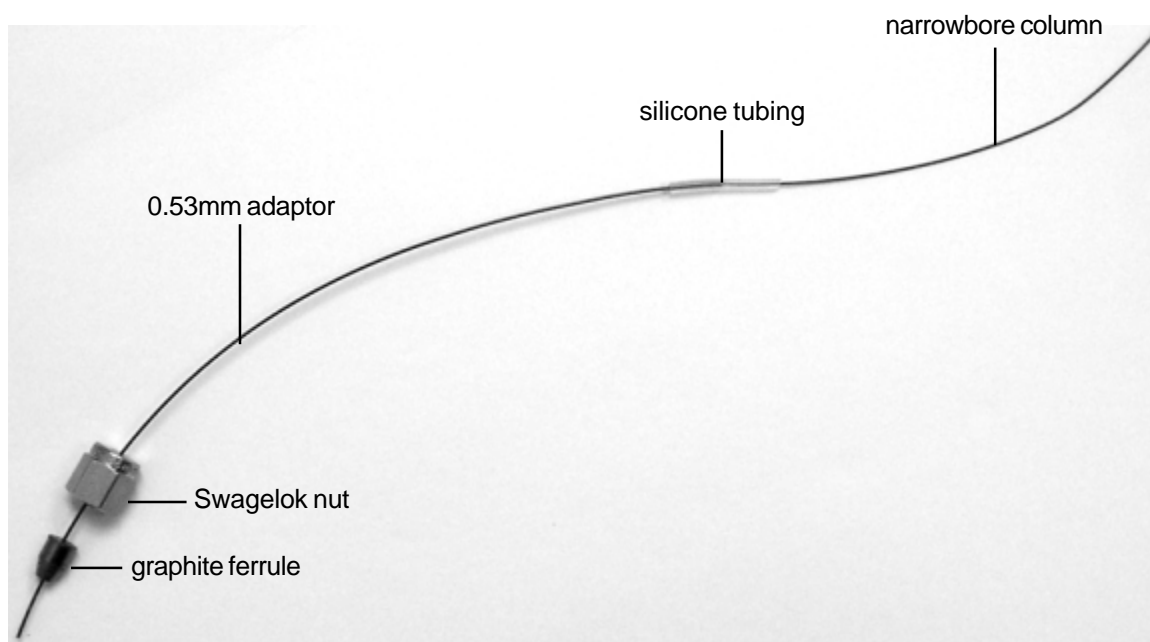
SPME Injection

Narrowbore Column Adaptor

Using this simple adaptor, you can perform SPME fiber injections into narrowbore columns with the following SRI heated syringe injectors: split/splitless, PTV, and heated vaporization.

The adaptor consists of a length of 0.53mm capillary column and a small piece of 0.50mm ID silicone tubing. Insert the narrowbore column into the 0.53mm section. Slide the silicone tubing to over the joint. Insert the end of the adaptor into the graphite ferrule and swagelok injector fitting.

The megabore injector liner provides high linear carrier gas velocity to sweep the desorbed analytes onto the column.

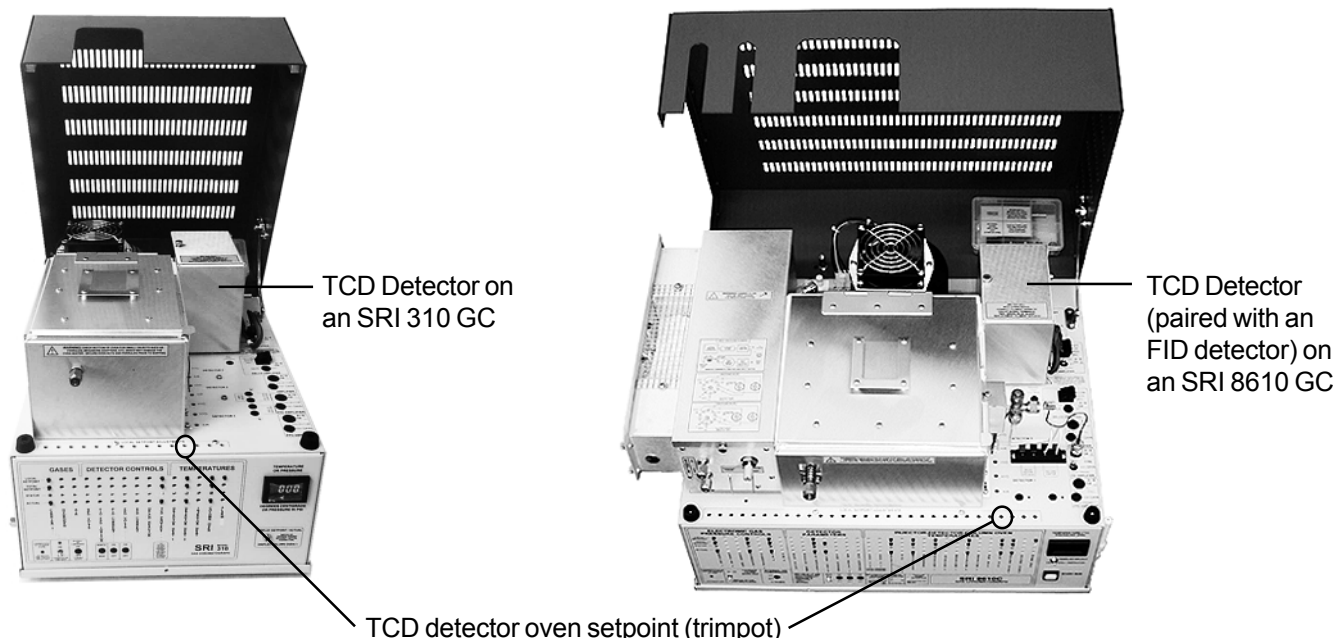


DETECTORS

Thermal Conductivity Detector - TCD

Overview

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



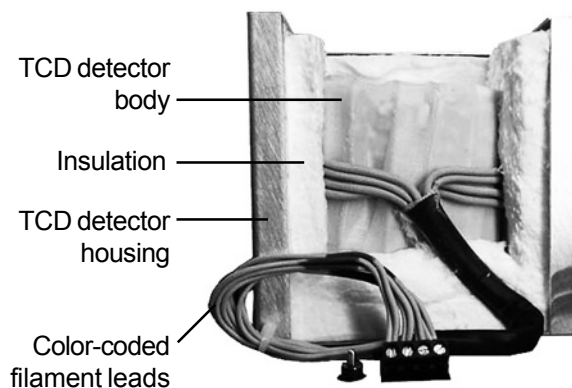
TCD Detector on an SRI GC



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

TCD filament current control switch

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

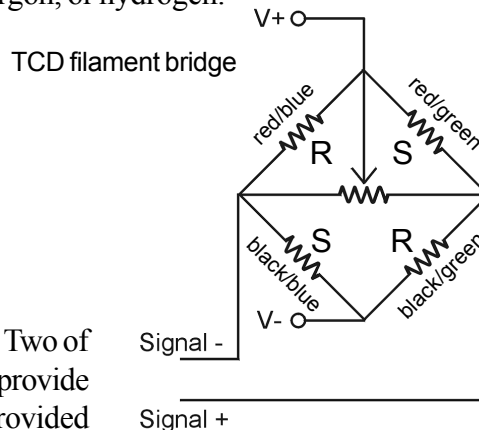
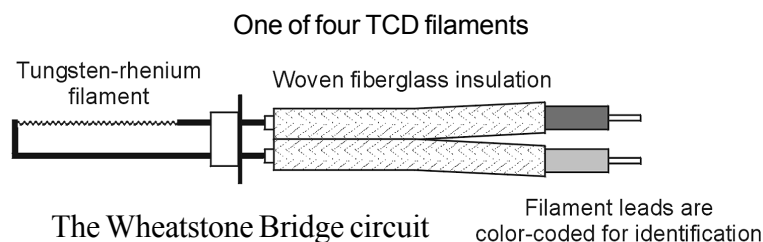


DETECTORS

Thermal Conductivity Detector - TCD

Theory of Operation

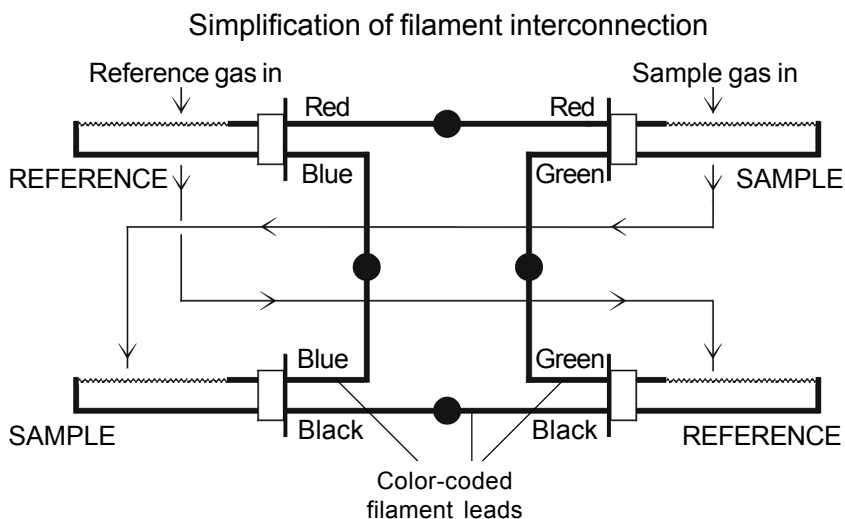
The TCD detector measures the difference in thermal conductivity in the carrier gas flow and the analyte peaks. Every compound possesses some degree of thermal conductivity, and may therefore be measured with a TCD detector. Due to its high thermal conductivity and safety, helium carrier is most often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



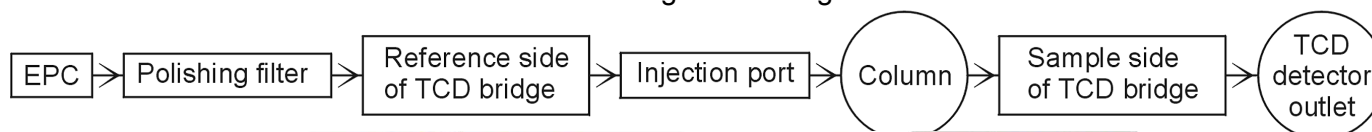
The Wheatstone Bridge circuit design in the SRI TCD uses four general-purpose tungsten-rhenium filaments for sample analysis. Two of the filaments are exposed to the sample-laden carrier gas flow and provide the actual chromatographic signal. The other two filaments are provided with clean carrier flow,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

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 it 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



DETECTORS

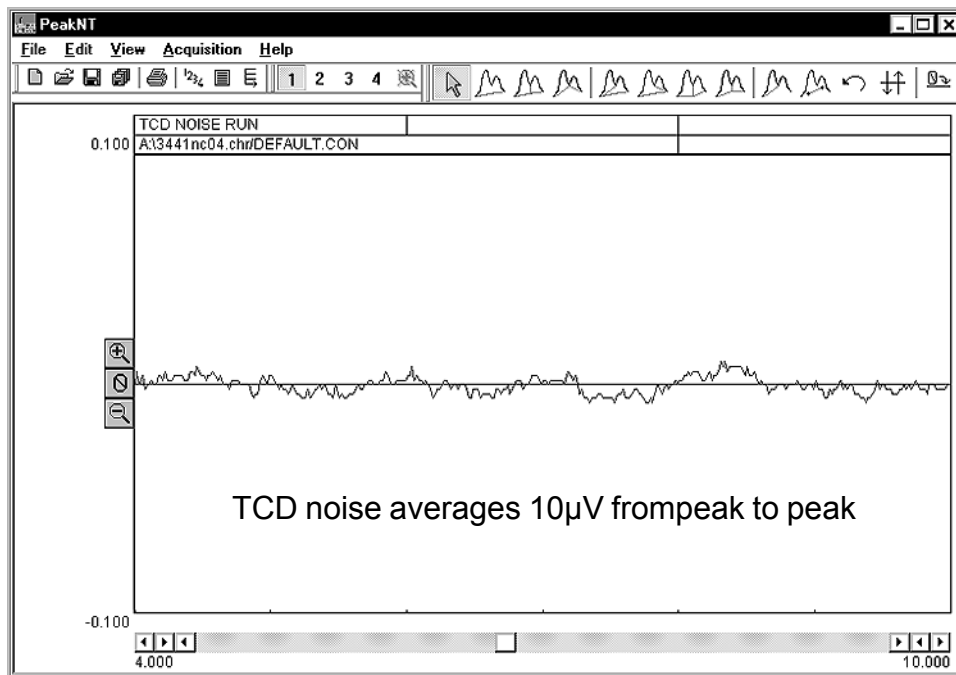
Thermal Conductivity Detector - TCD

Expected Performance

TCD Noise Run

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

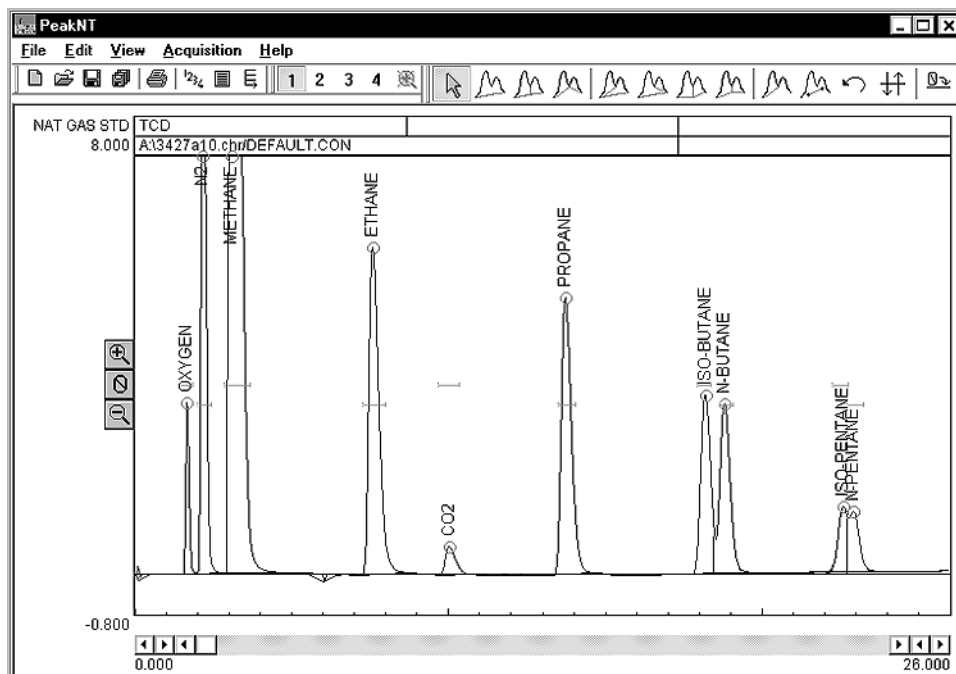
Temperature program:
Initial Hold Ramp Final
80°C 15.00 0.00 80°C



Factory Test Run of a TCD-equipped SRI GC

Sample: natural gas standard, 1mL sample loop

Columns: 1m Molecular Sieve, 2m Silica Gel



Events:

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

Temperature program:

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

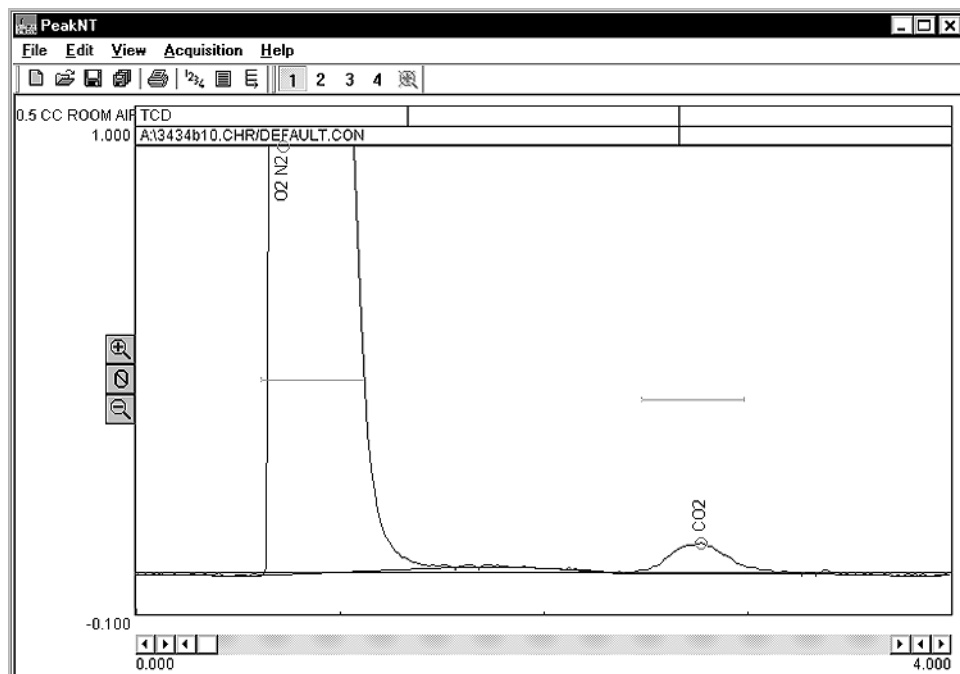
Results:

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

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 CO₂ content of the room air analyzed is approximately 350ppm.

Results:

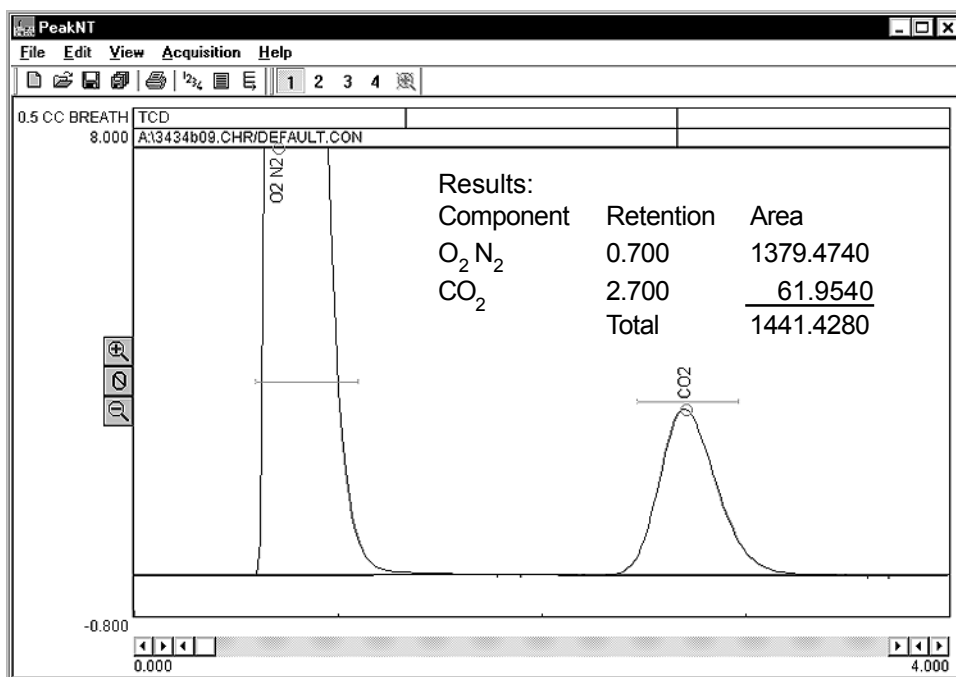
Component	Retention	Area
O ₂ N ₂	0.716	1021.3830
CO ₂	2.766	1.5060
Total		1022.8890

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



Results:

Component	Retention	Area
O ₂ N ₂	0.700	1379.4740
CO ₂	2.700	61.9540
Total		1441.4280

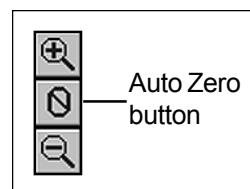
DETECTORS

Thermal Conductivity Detector - TCD

General Operating Procedure

1. Check to make sure that the TCD filament current is switched OFF. Plug in and turn on your GC. Allow the TCD detector oven to reach temperature (100°C) and stabilize. With the “Display Select” switch in the UP position, press on the TCD Temperature Actual button on the front control panel to read the TCD cell temperature. The TCD oven block is set to 100°C at the factory, but is adjustable by turning the trimpot with a small blade screwdriver while observing the TCD BLOCK setpoint temperature on the digital display. The trimpot is located on the top edge of the GC’s front control panel, under the red lid.
2. All TCD-equipped SRI GCs are tested with a 1m, 1/8” stainless steel silica gel-packed column. The carrier gas head pressure is preset at the factory to 10mL/min for this type and size column. Look on the right side of the GC for the carrier pressure that correlates to a flow of 10mL/min. Because different columns require different flow rates, the carrier head pressure may be adjusted by the user with the trimpot above the “CARRIER 1” buttons.
3. Make sure that the setpoint and actual pressures are within 1psi.
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column Oven on the same side as the detector. Place the end of the tube in liquid and observe (a little spit on a finger can suffice). If there are no bubbles exiting the tube, there is a flow problem. DO NOT turn on the TCD current if carrier gas flow is not detectable. A filament protection circuit prevents filament damage if carrier gas pressure is not detected at the GC, but it cannot prevent filament damage under all circumstances. Any lack of carrier gas flow should be corrected before proceeding.
5. With the TCD filaments switched OFF, zero the data system signal. Switch the filaments to LOW. The signal’s deflection should not be more than 5-10mV from zero for a brand-new TCD detector. Any more than a 5-10mV deflection indicates partial or complete oxidation of the TCD filaments; more deflection means more oxidation. Therefore, it is a good habit to use the data system signal to check the working order of the TCD filaments.
6. In PeakSimple, set an isothermal column oven temperature ramp program as follows:

Initial Temp.	Hold	Ramp	Final Temp.
80°C	7.00	0.00	80°C
7. Zero the data system signal (clicking on the Auto Zero button at the left edge of the chromatogram window is one way to do it), then start the run (hit the computer keyboard spacebar or hit the “RUN” button on the GC).
8. Inject sample. Injection volumes of 0.5mL for gas and 1 µL for liquid is recommended to prolong TCD filament life.



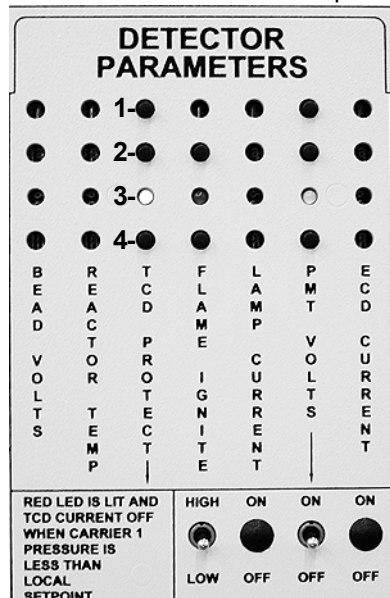
DETECTORS

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 SRI TCD filament protection circuit is a current “cut-out” circuit that monitors the column head pressure during GC operation. Under normal circumstances, there is no reason for the column head pressure to drop below 3psi, with most columns operating at 8psi or above. When the head pressure sensor located in the carrier gas flow path drops below 3psi, the protection circuit is activated, and the current to the TCD filaments is interrupted immediately. A red LED on the GC’s front control panel under “DETECTOR PARAMETERS” will light to indicate that the protection circuit has detected a gas pressure loss and shut down the filament current. The cause of the protection circuit activation should be immediately investigated and corrected. As an additional caution, use HIGH current only with helium or hydrogen carrier gases. With nitrogen carrier, use LOW current only, or the filaments may be damaged. The pressure at which the protection circuit activates is user adjustable with the trimpot on the top edge of the front control panel, above the label reading “TCD PROTECT.”

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



- 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.

DETECTORS

Thermal Conductivity Detector - TCD

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 ohmmeter function of a multimeter or volt-ohmmeter (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 drifts. 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 ohmmeter, making sure the correct pair of colored wires is tested together for each filament. An infinite reading is an indication that the filament is open, or burned out. If any of the filaments has a significantly different resistance than the others (which should be in the ranges mentioned above), it should be replaced. Replacement filaments, o-rings, and TCD blocks with four new filaments are available from SRI. In addition to the standard filaments, optional gold-plated filaments for improved corrosion resistance are also available.



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

SRI TCD detector replacement parts

Standard TCD filament with rubber O-ring gasket
High temperature TCD filament with copper gasket

8670-9120
8690-9123

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

DETECTORS

Thermal Conductivity Detector - TCD

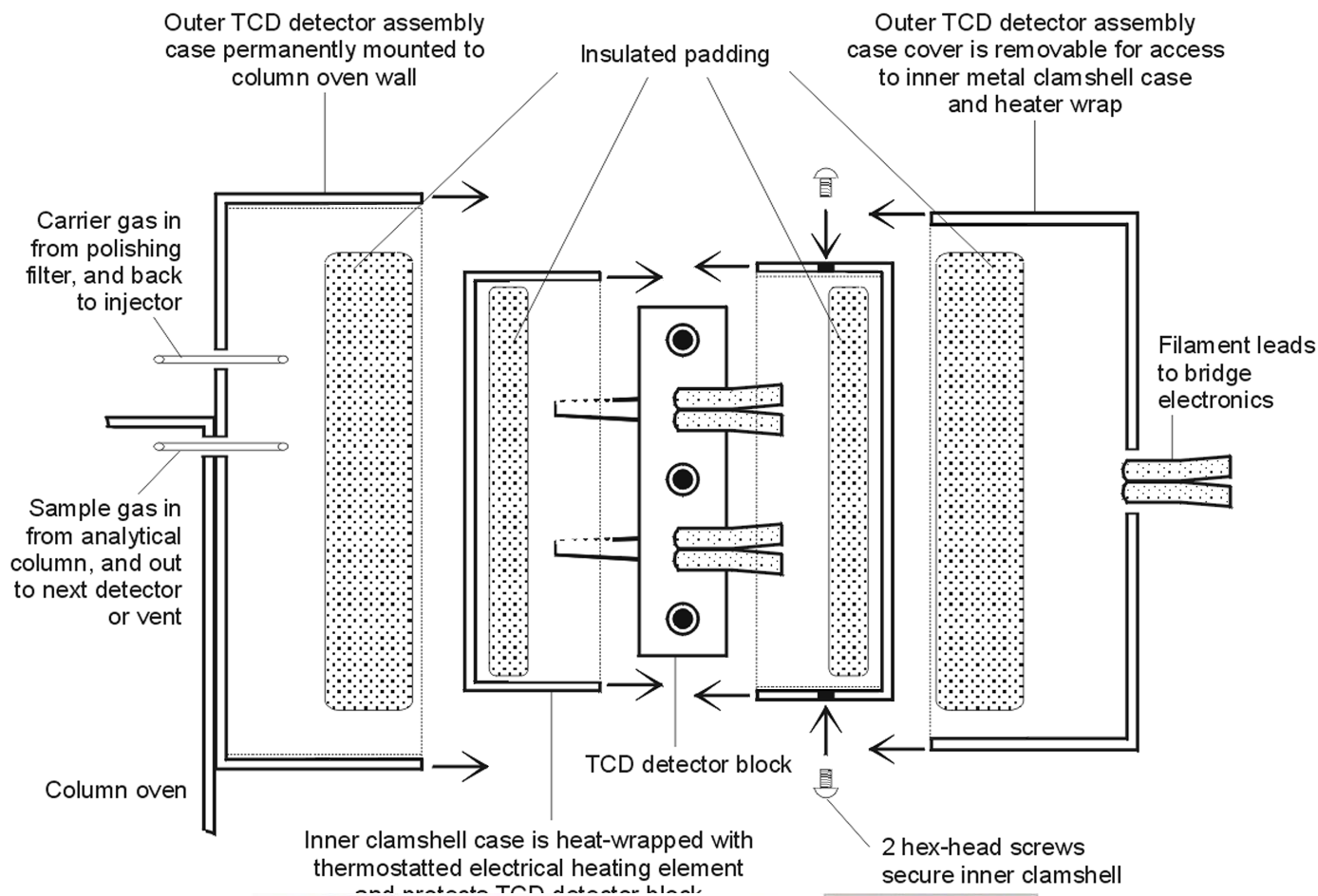
Replacing the TCD Filaments

SRI TCD detectors are made to last a long time without ever replacing the filaments. However, any TCD filaments that fail the diagnostic ohmmeter 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



DETECTORS

Thermal Conductivity Detector - TCD

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.

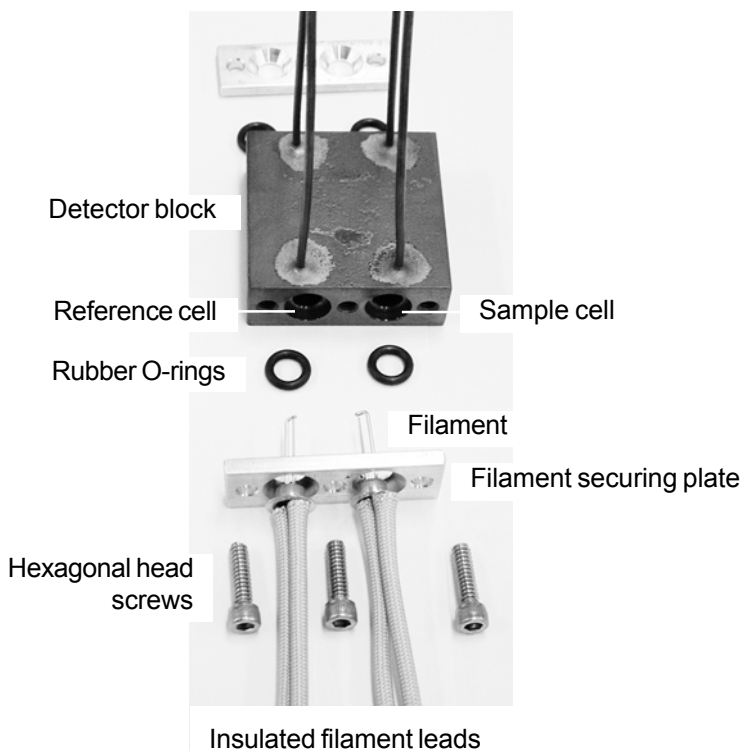
6. The TCD filaments are secured in the detector block by two plates, each of which is held in place with three hexagonal head screws. Holding the detector block with one hand, use an Allen wrench to unscrew and remove the hexagonal head screws from one of the filament securing plates. Then, slide the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



DETECTORS

Thermal Conductivity Detector - TCD

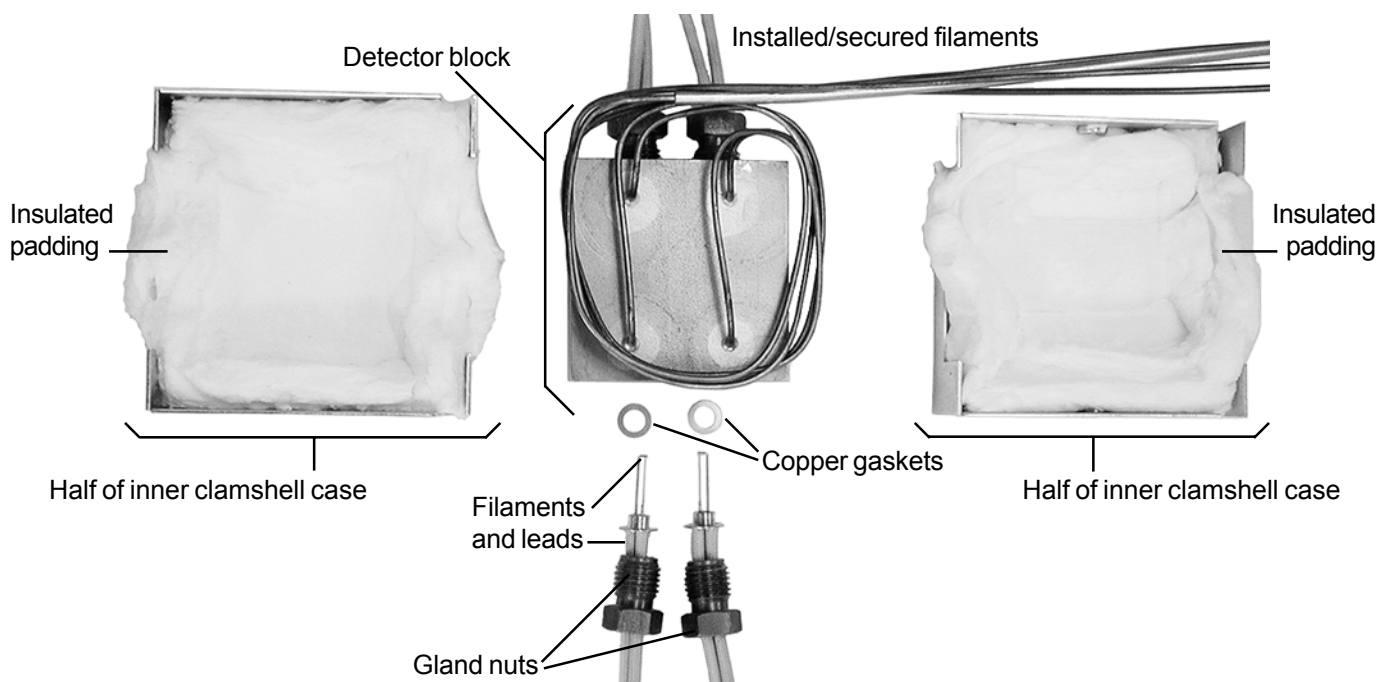
Replacing the TCD Filaments continued

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



DETECTORS

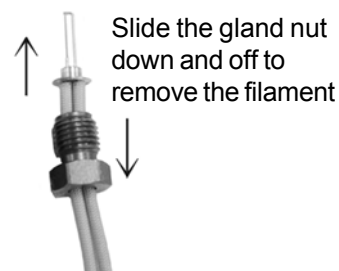
Thermal Conductivity Detector - TCD

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.

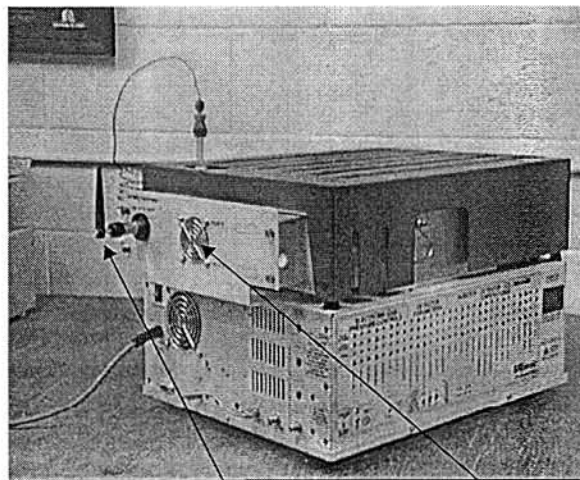
10. Reconnect the filament leads to the bridge current terminal block. Use the color guide labels on the terminal block to insert the color-coded leads into the appropriate terminal.

Chapter: INJECTORS

Topic: THERMAL DESORBER OPERATION

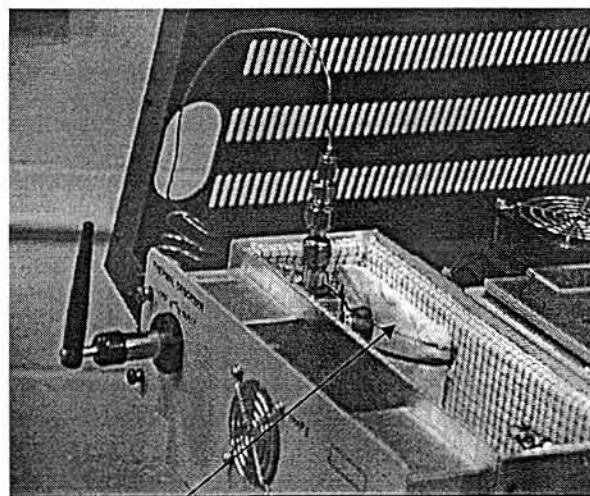
The SRI Thermal Soil Desorber accessory is useful for the analysis of volatile and especially semi-volatile compounds in soil or other granular solids. Because the analyte can be extracted from the soil by heat alone, with little or no sample preparation, field analyses can be performed without liquid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

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



Valco Valve handle
rotates to inject
sample

Heated Valve
Oven contains
Thermal Desorber



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

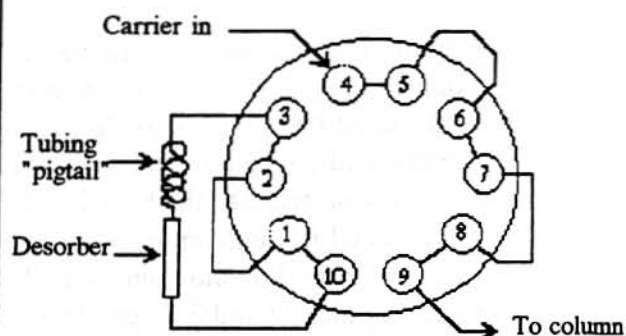
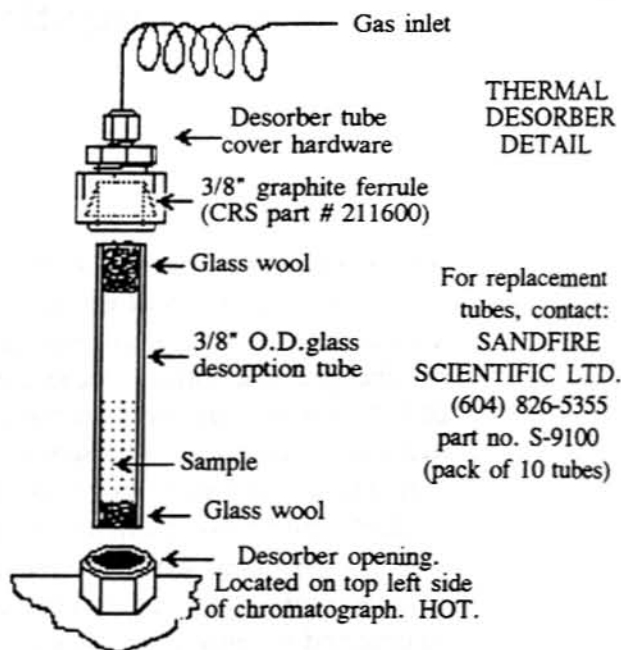
H007.doc

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

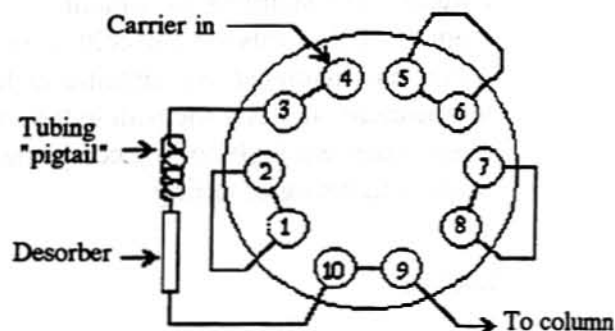
Historically, samples in soil have required solvent extraction with methylene chloride, hexane, carbon disulfide or others prior to injection into a gas chromatograph. Unfortunately, solvent extraction often dilutes the sample and adversely affects detection limits. The detection limit for diesel fuel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

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

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



10 PORT VALVE DIAGRAM
"LOAD" POSITION



10 PORT VALVE DIAGRAM
"INJECT" POSITION

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

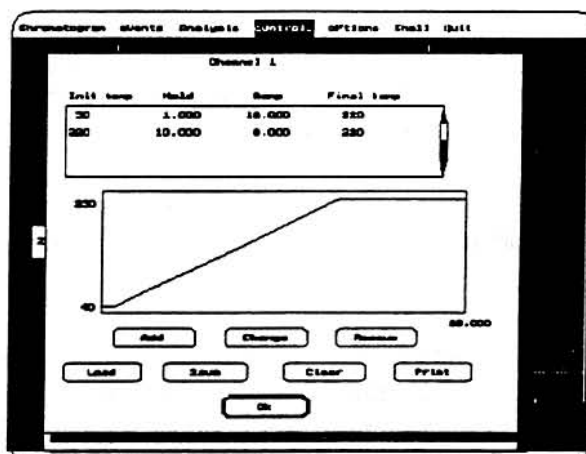
1. Place a clean desorption tube with a glass wool plug at one end on a scale of known accuracy. The tare weight is obtained. This is done by either weighing the clean, empty tube and recording the weight, or by placing the tube on the weighing platform and zeroing the balance.
2. Load the sample into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

Users may make their own tubes if so desired.

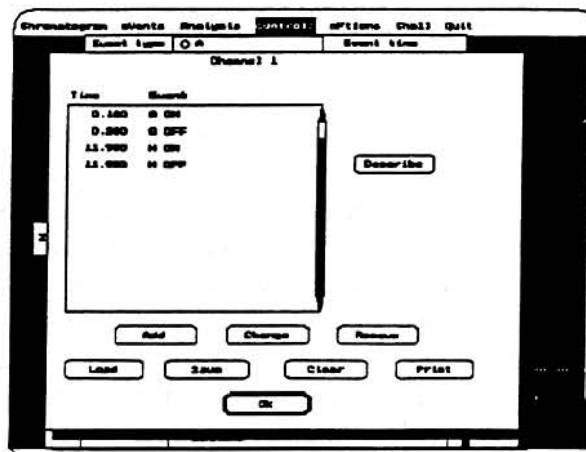
The column is connected to the injection valve inside the valve oven using a 1/16" to 1/8" adapter and 0.040" I.D. stainless steel tubing (1/16" O.D.). This ensures a uniform sample temperature while en route to the column and eliminates any possible cold spots.

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

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



EXAMPLE OF TEMPERATURE PROGRAM FOR DESORPTION



EXAMPLE OF TIMED EVENT TABLE FOR CONTROL OF AUTOMATED INJECTION

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

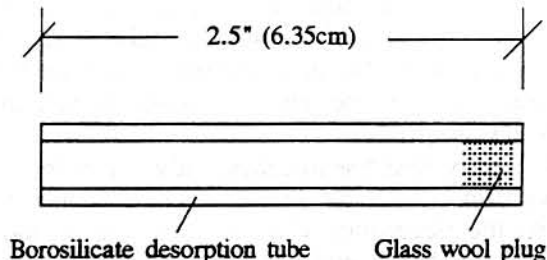
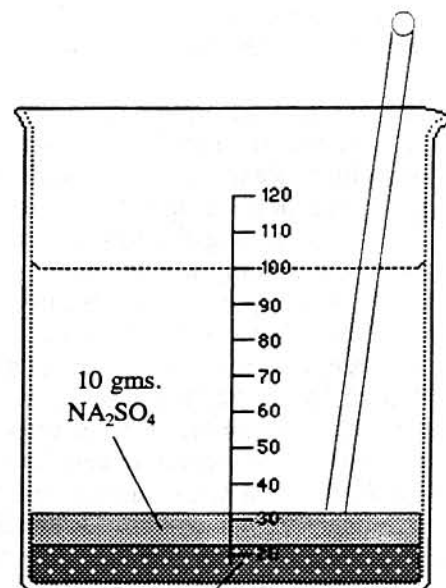
2) Add 10 grams of granular sodium sulfate (Na_2SO_4) to the beaker and mix with a stirring rod or spatula. The granular sodium sulfate, when mixed with the soil, absorbs most of the moisture from the soil, allowing clay soils to be ground into smaller particles. This is important because dense clay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

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

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

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

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



Placement of glass wool in desorption tube prior to sample insertion

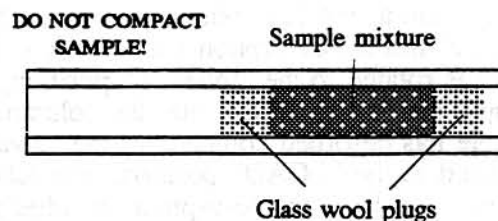


Diagram of assembled sample desorption tube containing 0.5 gms of soil - Na_2SO_4 sample mixture

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

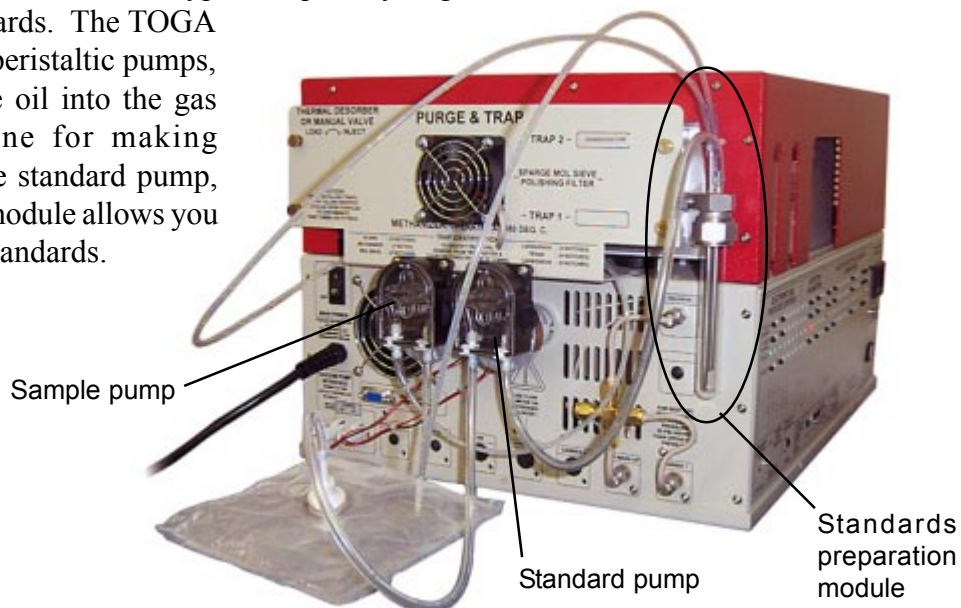
THERMAL CONDUCTIVITIES OF SOME COMMON GASES

Air	58
Argon	39
CO	53
CO ₂	34
H ₂	419
HE	343
N ₂	57
O ₂	58
Neon	109
Methane	73
Ethane	43
Propane	36
Butane	32

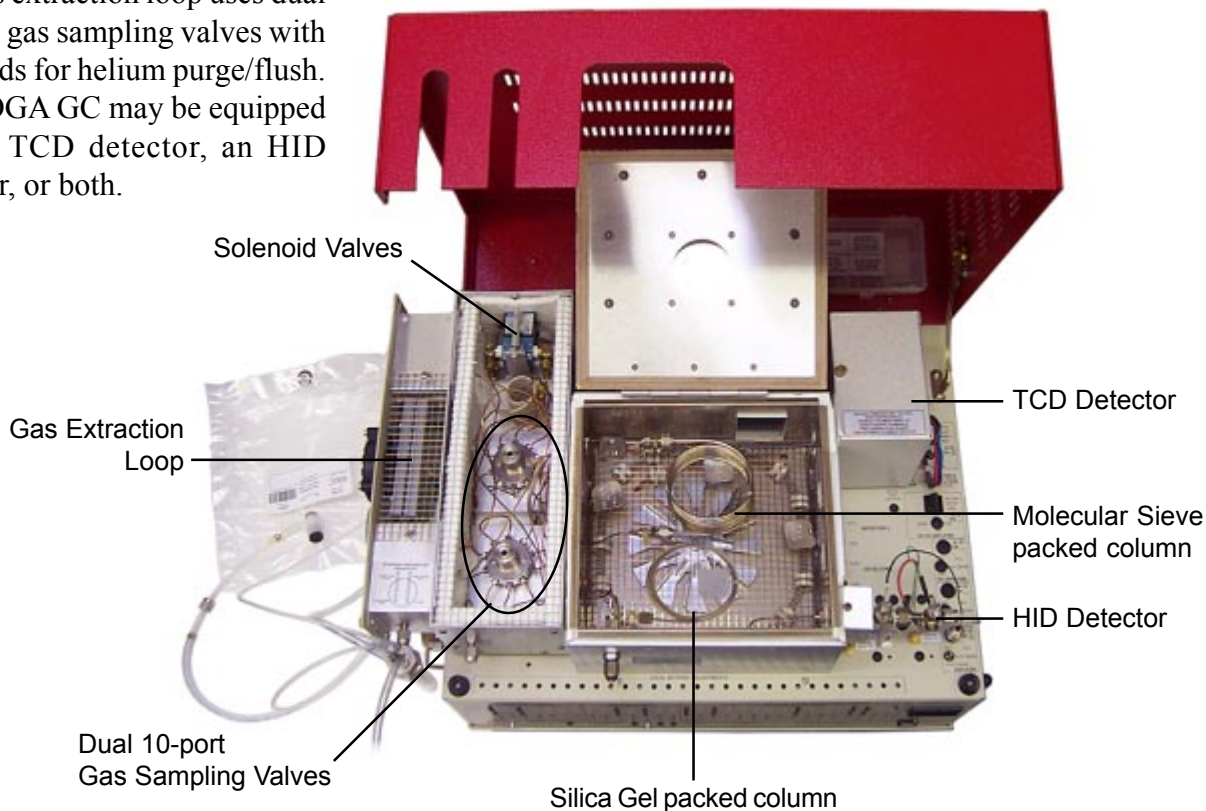
POPULAR CONFIGURATION GCs Transformer Oil Gas Analyzer (TOGA) GC

System Overview

Use the SRI TOGA GC to determine the type and quantity of gases dissolved in transformer oil, and to create dissolved gas standards. The TOGA GC is equipped with two peristaltic pumps, one to convey the sample oil into the gas extraction loop, and one for making standards. Along with the standard pump, the standards preparation module allows you to prepare dissolved gas standards.



The gas extraction loop uses dual 10-port gas sampling valves with solenoids for helium purge/flush. The TOGA GC may be equipped with a TCD detector, an HID detector, or both.

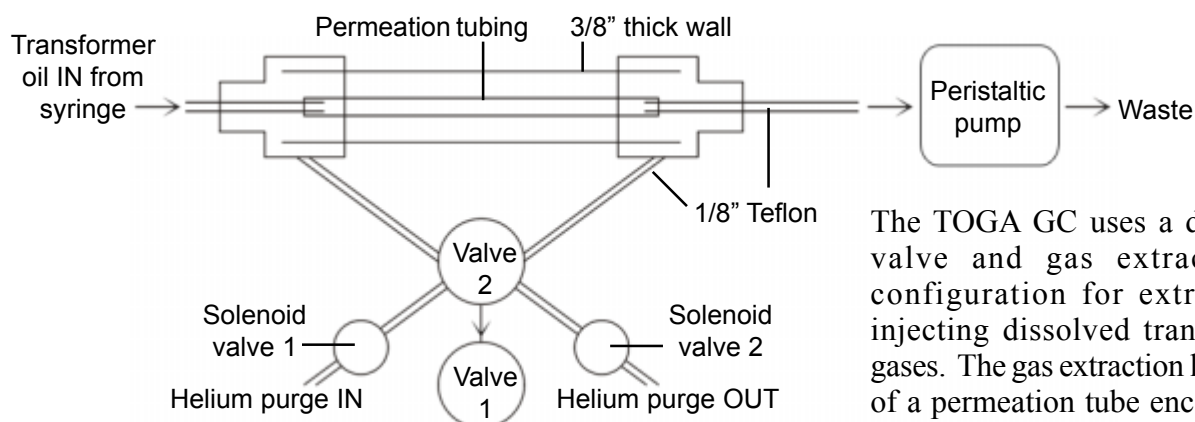


The TOGA GC may also be used to perform more general dissolved gas analyses (DGA) with sample liquids like water and soft drinks.

POPULAR CONFIGURATION GCs

Transformer Oil Gas Analyzer (TOGA) GC

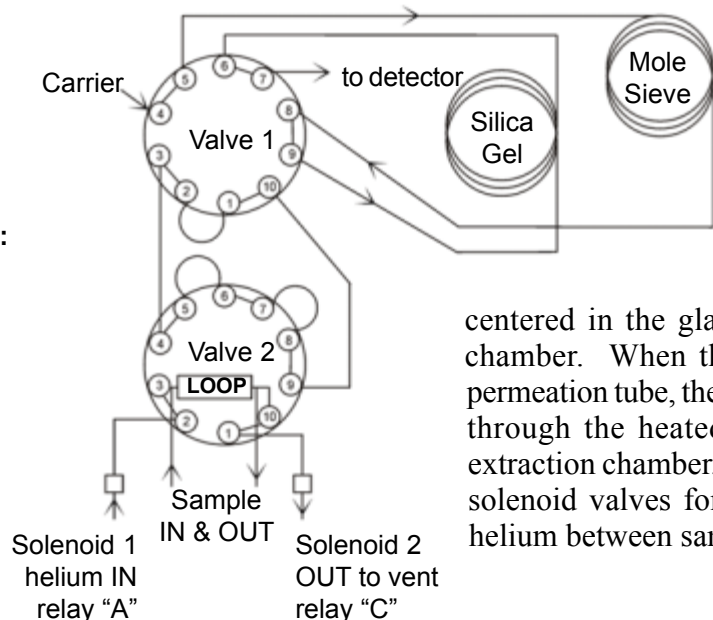
Theory of Operation



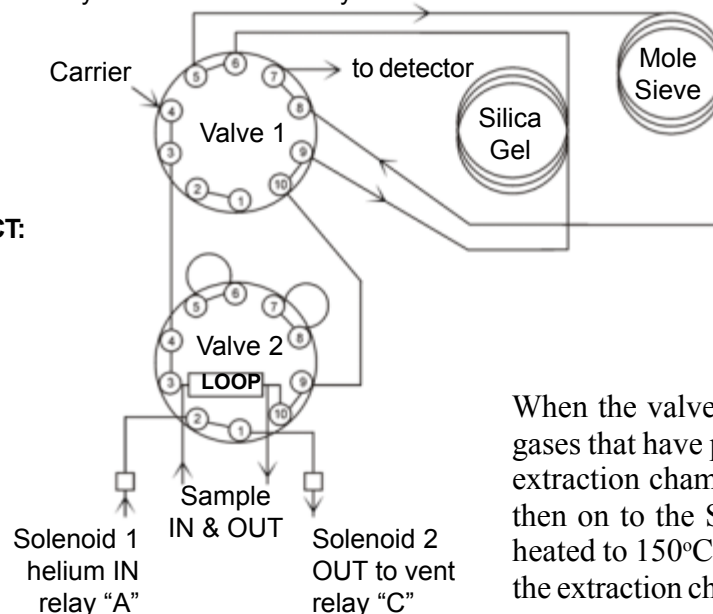
The TOGA GC uses a dual 10-port valve and gas extraction loop configuration for extracting and injecting dissolved transformer oil gases. The gas extraction loop consists of a permeation tube encapsulated in a glass tube equipped with a heated trap, and replaces the sample loop in the valve circuit. Teflon tubing is used to convey the sample oil from the syringe to the permeation tube, and from the permeation tube out to waste. The Teflon tubing and the permeation tube are secured in the gas extraction loop, with the permeation tube

centered in the glass tube which functions as the extraction chamber. When the transformer oil is pumped through the permeation tube, the dissolved gases therein selectively permeate through the heated (70°C) membrane into the surrounding extraction chamber. Plumbed to the dual 10-port valves are two solenoid valves for purging the gas extraction chamber with helium between sample injections, to prevent carryover.

LOAD:



INJECT:



Valve 1 is plumbed to the Molecular Sieve 13X column, then to the Silica Gel column. Valve 2 is plumbed to the gas extraction loop. While the transformer oil is being pumped through the gas extraction loop, both valves are in the LOAD position. During this time, the gases dissolved in the transformer oil are extracted by selective permeation through the membrane.

When the valves are rotated into the INJECT position, the gases that have permeated the membrane are swept from the extraction chamber onto the Molecular Sieve 13X column, then on to the Silica Gel column. The permeation tube is heated to 150°C while the solenoids are turned ON to sweep the extraction chamber clean with helium for the next sample.

POPULAR CONFIGURATION GCs

Transformer Oil Gas Analyzer (TOGA) GC

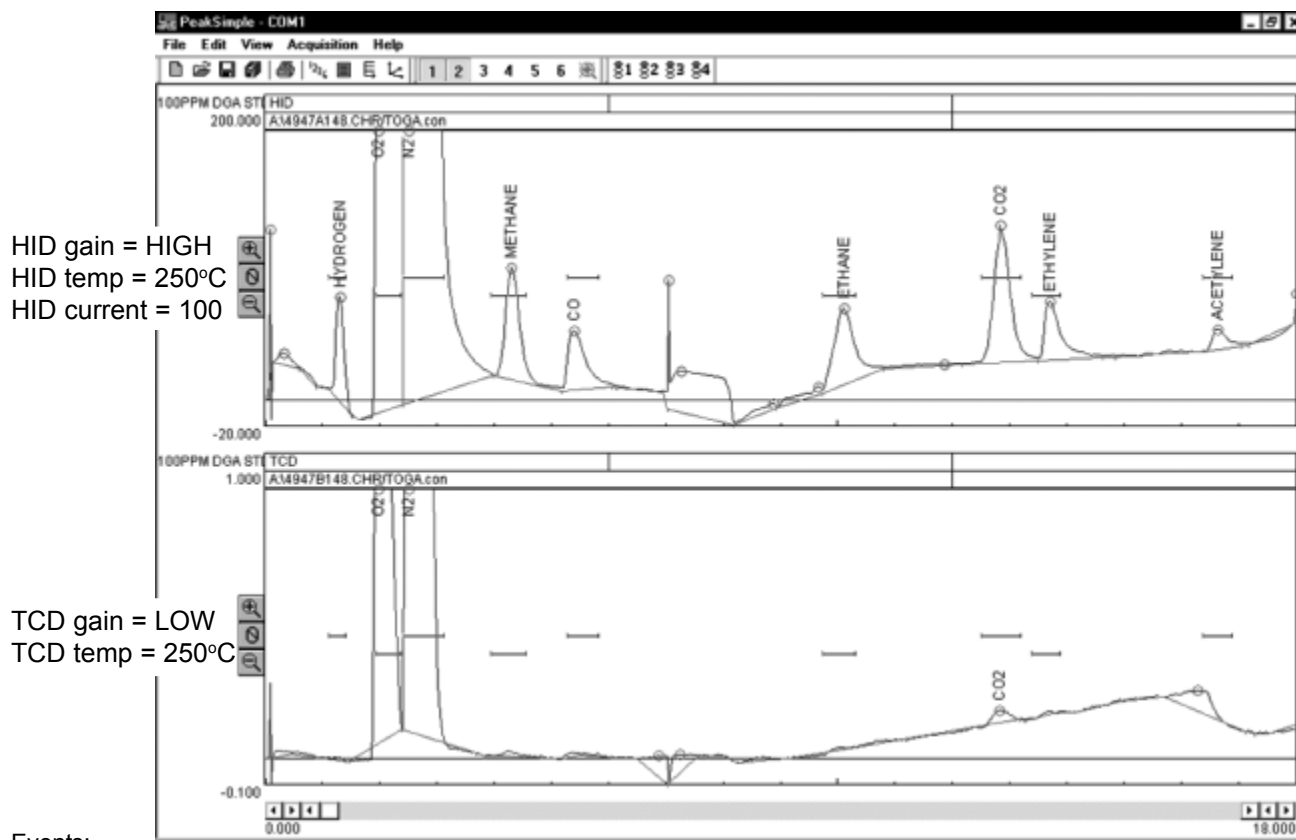
Expected Performance

These two chromatograms show a 100ppm DGA standard as separated by the TOGA GC. The permeation chamber was baked out at 150°C between runs.

Sample: 10mL 100ppm DGA standard
Columns: 1-meter Molecular Sieve, 2-meter Silica Gel
Carrier: Helium @ 20mL/minute
Pump rate = 2.0mL/minute
Valve oven temp = 90°C
Permeation tube temp = 70°C

Temperature program:

Initial	Hold	Ramp	Final
40.00	7.000	10.000	100.00
100.00	0.000	20.000	250.00
250.00	5.000	0.000	250.00



Events:

Time	Event
0.000	C ON (OUTLET SOLENOID)
0.050	A ON (INLET SOLENOID)
1.000	A OFF
1.050	C OFF
1.100	E ON (SAMPLE PUMP)
7.100	E OFF
7.150	ZERO
7.200	G ON (VALVE 1 INJECT)
7.250	H ON (VALVE 2 INJECT)
7.750	H OFF
7.850	C ON (OUTLET SOLENOID)
7.900	A ON (INLET SOLENOID)
7.950	F ON (TRAP HEAT)
16.500	F OFF
17.500	A OFF
17.700	C OFF

Results:

Component	Retention	Area
Hydrogen	1.283	732.8160
O2	1.950	76505.4300
N2	2.483	118654.2520
Methane	4.283	1388.4300
CO	5.366	1083.2865
Ethane	10.083	1247.8820
CO2	12.816	1941.6180
Ethylene	13.683	798.1490
Acetylene	16.616	251.2315

POPULAR CONFIGURATION GCs

Transformer Oil Gas Analyzer (TOGA) GC

General Operating Procedure

1. Connect your helium source to the carrier and detector make-up gas inlets on the lower left-hand side of the GC. The pressures correlating with the proper flow rate for your instrument are printed on the right hand side of the GC, in a table under the heading GAS FLOW RATES. For best EPC performance, set the incoming helium pressure 15-20psi higher than the pressure listed in the table.

2. Turn the GC ON and let the system warm up and stabilize. Once you have ensured proper carrier gas flow, turn the TCD gain switch to LOW (this turns the current ON). The TCD temperature is factory set at 250°C. You may adjust this temperature if required. Turn ON the HID current. The HID temperature is also factory set at 250°C. Set the HID gain switch to HIGH. See each separate detector's manual section for more operating details.

3. Type in this column oven temperature program for channel 1: Temperature program:

Initial	Hold	Ramp	Final
40.00	7.000	10.000	100.00
100.00	0.000	20.000	250.00
250.00	5.000	0.000	250.00

4. Type in the following event table for channel 1:

What this Event Table will do:

- Pre-purge the gas extraction loop.
- Turn ON the sample pump for 6 minutes (or until the waste line is full; your sample may take more or less time).
- Zero the data system signal.
- Actuate Valves 1 and 2 to the INJECT position.
- Leave Valve 1 in INJECT and return Valve 2 to the LOAD position.
- Turn ON the two solenoids.
- Heat the gas extraction loop to 150°C for several minutes (again, your sample may take more or less time).
- Turn OFF the two solenoids.

Events:

Time	Event
0.000	C ON (OUTLET SOLENOID)
0.050	A ON (INLET SOLENOID)
1.000	A OFF
1.050	C OFF
1.100	E ON (SAMPLE PUMP)
7.100	E OFF
7.150	ZERO
7.200	G ON (VALVE 1 INJECT)
7.250	H ON (VALVE 2 INJECT)
7.750	H OFF
7.850	C ON (OUTLET SOLENOID)
7.900	A ON (INLET SOLENOID)
7.950	F ON (TRAP HEAT)
16.500	F OFF
17.500	A OFF
17.700	C OFF

NOTE: Turning ON the two solenoids for 30 seconds before the run pre-purges the extraction chamber. As shown in this event table, open solenoid C first, then A. Close A first after 30 seconds, then close C. Maintain this order to avoid collapsing the permeation tubing under excessive gas pressure.

6. Connect the sample syringe outlet to the gas extraction loop inlet through the SAMPLE IN line.

7. Hit RUN on the front of the GC or hit the spacebar on your computer keyboard.

POPULAR CONFIGURATION GCs Transformer Oil Gas Analyzer (TOGA) GC

Changing the Permeation Tube

1. Remove the plate covering the gas extraction loop by unscrewing the four brass thumbscrews that hold it in place, and unplugging the fan power cord. Remove the two squares of white insulation to reveal the permeation tube assembly. Gently slide the assembly out of the valve oven ducts.

2. Loosen the glass tube's two stainless steel nuts and Teflon ferrules with a wrench to free the Teflon line. The fittings with the attached gas line are stationary.

3. Slide the Teflon tubing out of the glass tube until you can see the permeation tube.



4. Pull the old permeation tube off the Teflon tubing and discard. Wipe any oil off the Teflon tubing with a KimWipe or other lint-free wipe.

5. Slide the permeation tube over one end of the Teflon tubing. It is a tight squeeze to get the Teflon tubing into the permeation tube. To facilitate this, the Teflon tubing is cut at a 45° angle or sharper. Slide it on about 3/4" on each side. The permeation tube should be 7 inches long. It will be stretched slightly inside the glass tube.



6. Slide the permeation tube and Teflon tubing back into the glass tube and center it.

7. Re-secure the two stainless steel nuts and Teflon ferrules onto the Teflon tubing. Make sure that the permeation tubing protrudes beyond the stainless steel nut, so that the nut and the ferrule are securing it to the Teflon tubing. You may have to stretch the permeation tubing a bit to fasten it with the nuts and ferrules. While you are tightening the nuts and ferrules, firmly grip the Teflon tubing where it protrudes about 2-3" from the end of the trap to avoid twisting or kinking the permeation tube. When you are finished, check the flow through the permeation tube to ensure there is no constriction (it should be the same as it was before you replaced the permeation tube). Once the transformer oil starts flowing through the permeation tube, it will stretch, resulting in a corkscrew appearance, which is normal for operating mode.

8. Slide the entire assembly back into the valve oven ducts. Replace the two squares of white insulation. Replace the cover plate and secure its four thumbscrews.

POPULAR CONFIGURATION GCs

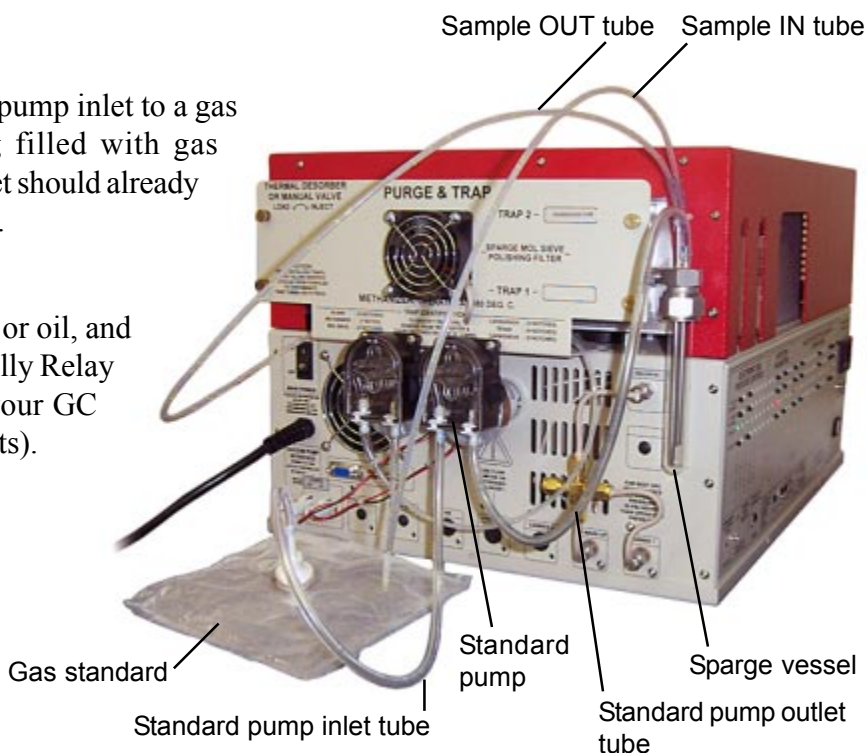
Transformer Oil Gas Analyzer (TOGA) GC

Standards Preparation with the TOGA GC

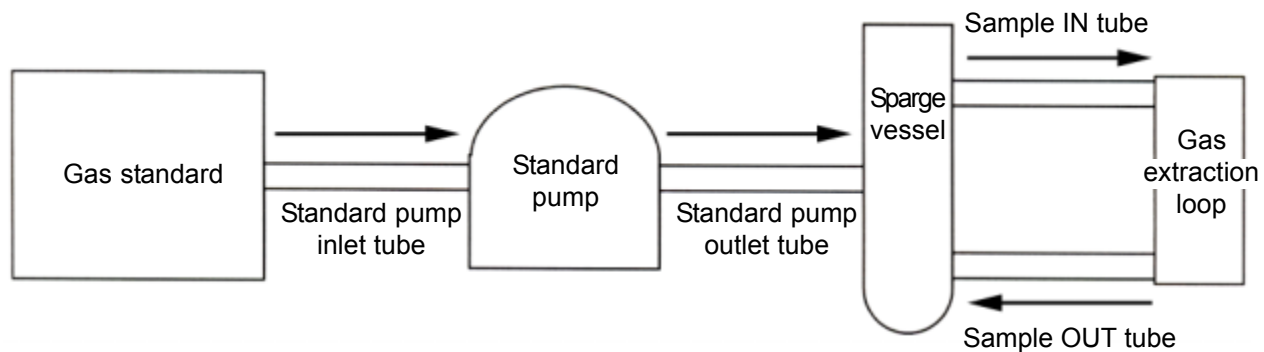
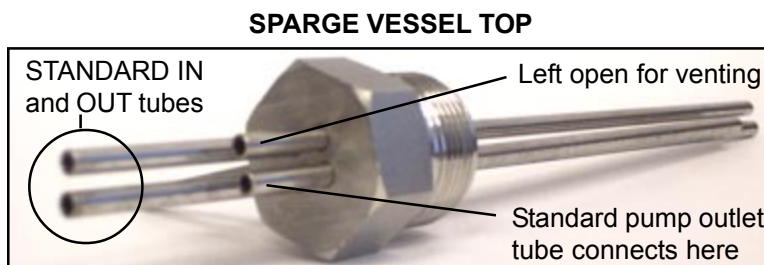
1. Attach the standard peristaltic pump inlet to a gas standard; we used a tedlar bag filled with gas standard. The standard pump outlet should already be connected to the sparge vessel.

2. Fill the sparge tube with water or oil, and turn ON the standards pump (usually Relay D; check the right-hand side of your GC for your specific relay assignments).

3. Continuously pump gas standard through the sparge tube. Over time, it will equilibrate; this could take up to two hours.



4. To sample the standard you've created with the tedlar bag and liquid-filled sparge tube: attach the SAMPLE IN and OUT tubes to the STANDARD IN and OUT tubes on the top of the sparge vessel.



5. The extracted standard will return to the sparge tube to be regenerated for subsequent analysis.

6. Re-attach the SAMPLE OUT tube to the waste connection, and the SAMPLE IN tube to the next syringe.

POPULAR CONFIGURATION GCs Transformer Oil Gas Analyzer (TOGA) GC

General Information

Many factors determine the solubility of gases in a given liquid, such as temperature and pressure and type of liquid. The following tables were downloaded from the internet and are provided here for general information only. Table 1 lists the saturation solubilities for dissolved gases in transformer oil (percent by volume). The saturation solubility for a gas is the maximum amount of gas a liquid can hold when 100% of that gas is bubbled through the liquid and fills the headspace above the liquid.

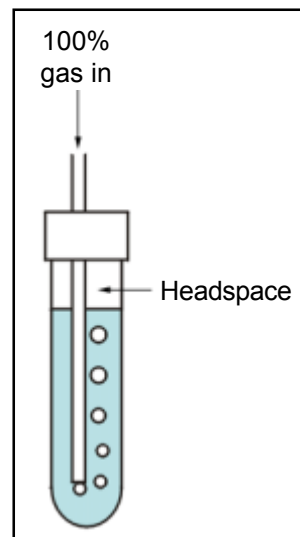


Table 1 Solubility of Gases in Transformer Oil:

Gas	% by volume
Hydrogen	7
Nitrogen	8.6
Carbon monoxide	9
Oxygen	16
Methane	30
Carbon dioxide	120
Ethane	280
Ethylene	280
Acetylene	400

“The majority of gases that are indicative of faults are also those that are in general the more soluble in the oil.”

—Table and quote from “Dissolved Gas Analysis of Mineral Oil Insulating Fluids,” by Joseph B. DiGiorgio, Ph.D., for Northern Technology and Testing.

<http://www.nttworldwide.com/tech2102.htm>

The following equation can help you convert the percent by volume numbers to ppm by weight (within about 15%):

hydrogen (H₂):

$$\frac{1\text{L oil}}{0.910\text{kg}} \times \frac{0.07\text{L H}_2}{1\text{L oil}} \times \frac{1\text{ mole H}_2}{24\text{L H}_2} \times \frac{2\text{g H}_2}{2\text{ moles H}_2} = 0.0032 \frac{\text{g}}{\text{kg}} = \frac{3.2\text{g}}{1000\text{kg}} = 3.2\text{ppm H}_2$$

(24L is a constant representing the volume occupied by 1 mole of an ideal gas at room temperature and pressure.)

Table 2 shows the solubility of gases in water (ppm by weight) for general DGA.

PPM by weight = the weight of the gas divided by the weight of one liter of water (2.205 pounds).

Table 2

Gas	Solubility (ppm by weight)
Acetylene	117ppm
Ammonia	5290ppm
Bromine	1490ppm
Carbon dioxide	169ppm
Carbon monoxide	28ppm
Chlorine	7290ppm
Ethane	62ppm
Ethylene	149ppm
Hydrogen	1.6ppm
Hydrogen sulfide	3850ppm
Methane	23ppm
Nitrogen	19ppm
Oxygen	43ppm
Sulfur dioxide	1128ppm

Table 2 is from The Wired Chemist:

http://wulfenite.fandm.edu/data%20/Table_16.html

TUBE VOLUME SELECTION GUIDE

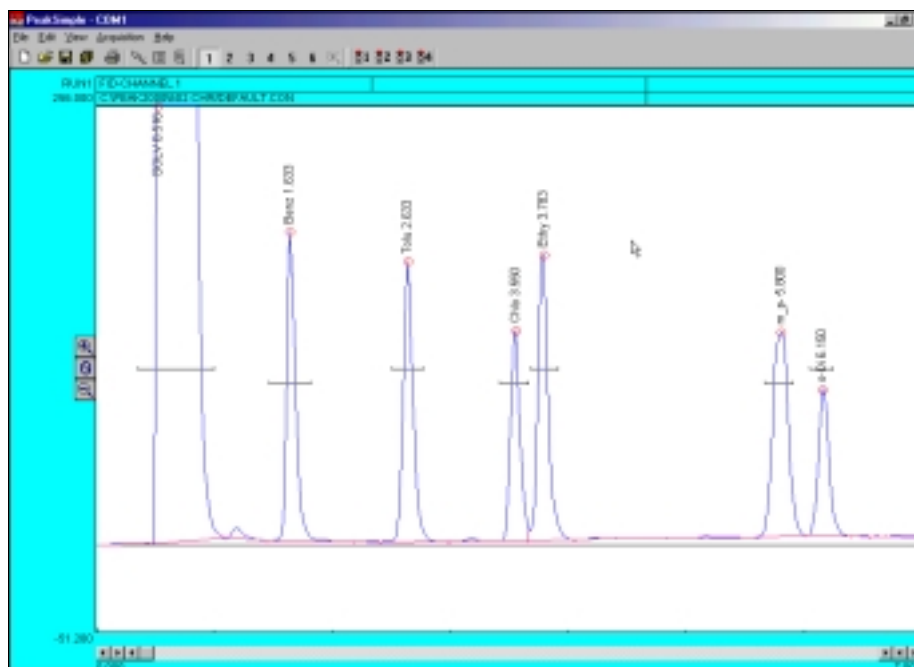
INTERNAL DIAMETER	MICROLITERS PER INCH	INCHES PER MICROLITER	INTERNAL DIAMETER	MICROLITERS PER INCH	INCHES PER MICROLITER
0.001	0.0129	77.6979	0.051	33.4757	0.0299
0.002	0.0515	19.4245	0.052	34.8014	0.0287
0.003	0.1158	8.6331	0.053	36.1527	0.0277
0.004	0.2059	4.8561	0.054	37.5299	0.0266
0.005	0.3218	3.1079	0.055	38.9327	0.0257
0.006	0.4633	2.1583	0.056	40.3613	0.0248
0.007	0.6306	1.5857	0.057	41.8157	0.0239
0.008	0.8237	1.2140	0.058	43.2958	0.0231
0.009	1.0425	0.9592	0.059	44.8016	0.0223
0.010	1.2870	0.7770	0.060	46.3332	0.0216
0.011	1.5573	0.6421	0.061	47.8905	0.0209
0.012	1.8533	0.5396	0.062	49.4735	0.0202
0.013	2.1751	0.4598	0.063	51.0822	0.0196
0.014	2.5226	0.3964	0.064	52.7167	0.0190
0.015	2.8958	0.3453	0.065	54.3770	0.0184
0.016	3.2948	0.3035	0.066	56.0630	0.0178
0.017	3.7195	0.2689	0.067	57.7747	0.0173
0.018	4.1700	0.2398	0.068	59.5122	0.0168
0.019	4.6462	0.2152	0.069	61.2754	0.0163
0.020	5.1481	0.1942	0.070	63.0643	0.0159
0.021	5.6758	0.1762	0.071	64.8790	0.0154
0.022	6.2292	0.1605	0.072	66.7195	0.0150
0.023	6.8084	0.1469	0.073	68.5856	0.0146
0.024	7.4133	0.1349	0.074	70.4775	0.0142
0.025	8.0440	0.1243	0.075	72.3952	0.0138
0.026	8.7003	0.1149	0.076	74.3386	0.0135
0.027	9.3825	0.1066	0.077	76.3077	0.0131
0.028	10.0903	0.0991	0.078	78.3026	0.0128
0.029	10.8239	0.0924	0.079	80.3232	0.0124
0.030	11.5833	0.0863	0.080	82.3696	0.0121
0.031	12.3684	0.0809	0.081	84.4417	0.0118
0.032	13.1792	0.0759	0.082	86.5395	0.0116
0.033	14.0158	0.0713	0.083	88.6631	0.0113
0.034	14.8781	0.0672	0.084	90.8124	0.0110
0.035	15.7662	0.0634	0.085	92.9875	0.0108
0.036	16.6799	0.0600	0.086	95.1882	0.0105
0.037	17.6195	0.0568	0.087	97.4148	0.0103
0.038	18.5847	0.0538	0.088	99.6670	0.0100
0.039	19.5758	0.0511	0.089	101.9450	0.0098
0.040	20.5925	0.0486	0.090	104.2488	0.0096
0.041	21.6350	0.0462	0.091	106.5783	0.0094
0.042	22.7032	0.0440	0.092	108.9335	0.0092
0.043	23.7972	0.0420	0.093	111.3145	0.0090
0.044	24.9169	0.0401	0.094	113.7212	0.0088
0.045	26.0624	0.0384	0.095	116.1537	0.0086
0.046	27.2336	0.0367	0.096	118.6119	0.0084
0.047	28.4306	0.0352	0.097	121.0958	0.0083
0.048	29.6532	0.0337	0.098	123.6055	0.0081
0.049	30.9017	0.0324	0.099	126.1409	0.0079
0.050	32.1758	0.0311	0.100	128.7020	0.0078

SRI Instruments

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.

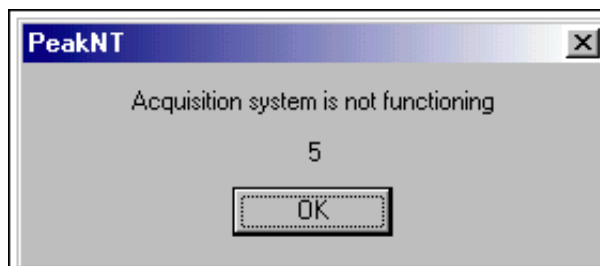
Installing PeakSimple 2000 from software download

- 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.

SRI Instruments 20720 Earl Street Torrance, CA 90503 U.S.A
 Telephone: (310) 214-5092 Fax: (310) 214-5097 sales@srigc.com www.srigc.com

Launching PeakSimple 2000

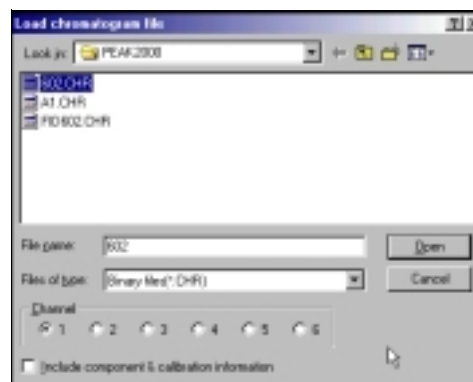
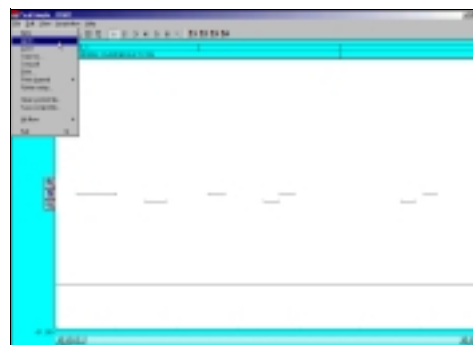
1. Click on the windows **Start** button in the bottom left-hand corner of the screen. Select **Programs** and then **PeakSimple** from the list of program groups on the screen and then click on **PeakSimple**.
2. This will launch PeakSimple and initialize the data acquisition system.
3. If PeakSimple 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.



Click this button to jump to the integration parameters screen. This is sometimes useful when reviewing the results data. For example, if the area reject caused some peaks to be skipped, you can jump right to the integration parameters and adjust the area reject number.

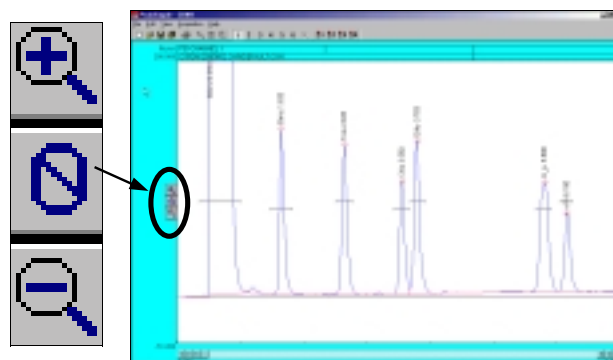
Opening a PeakSimple Data File

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.



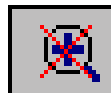
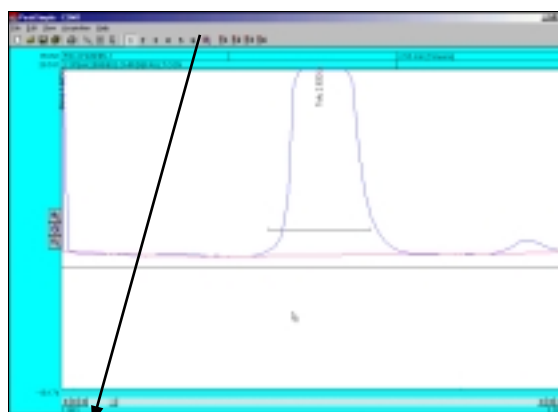
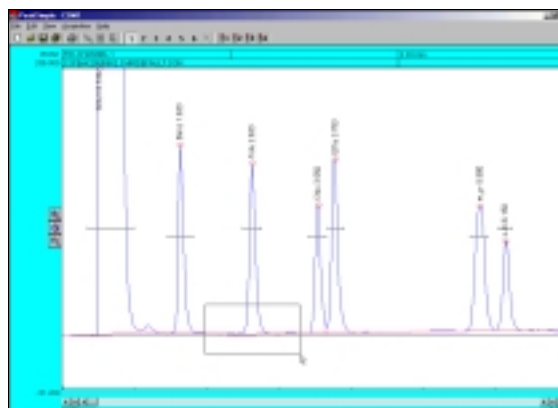
Adjusting Display Limits

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.



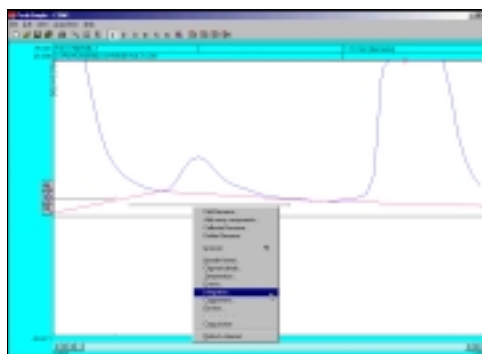
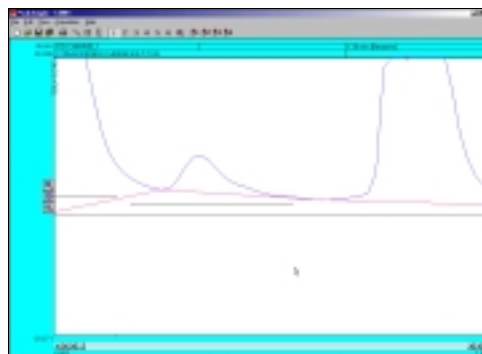
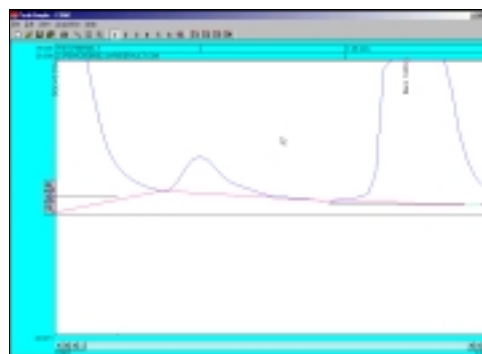
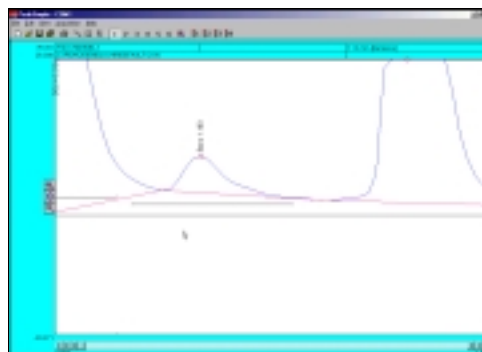
Zooming

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.



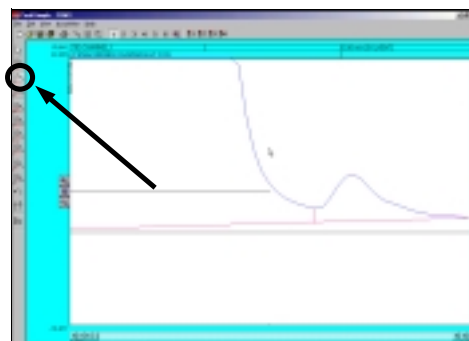
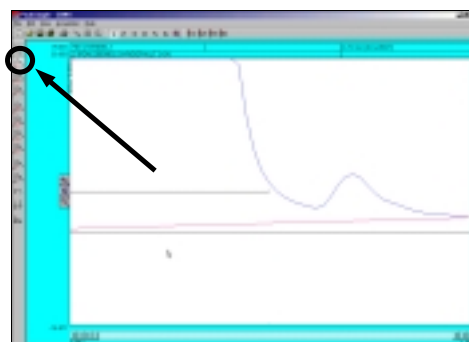
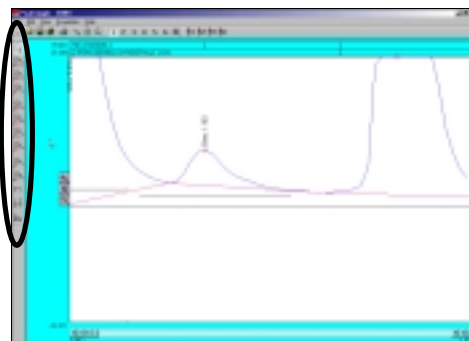
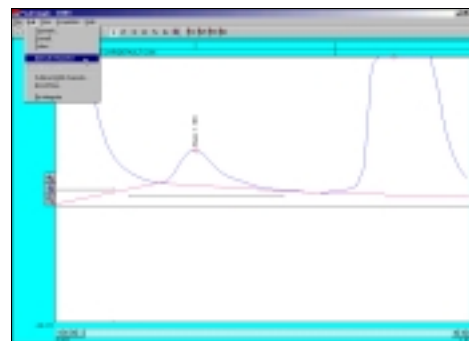
Dragging Retention Windows

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.

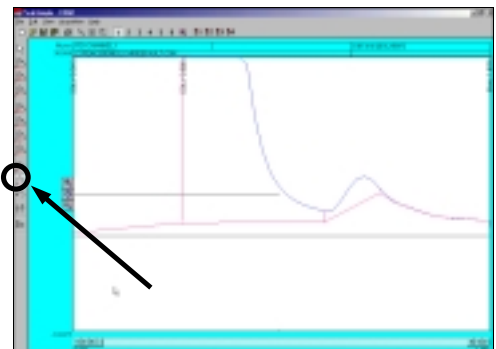
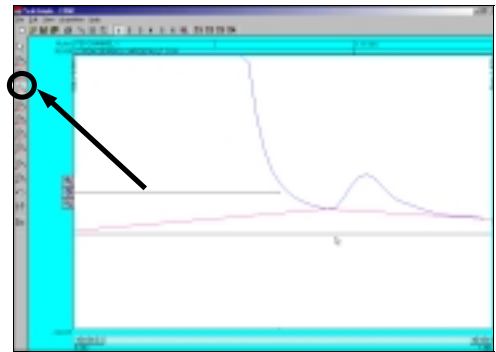



Manual Integration

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.

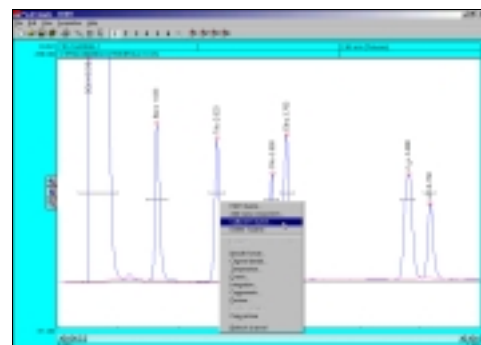


6. After the manual integration between the two peaks is dropped use the **Based** integration tool to raise the baseline to the valley between the peaks. Once the Based integration tool is selected, click on the valley between the solvent peak and the smaller peak to its right with the mouse cursor.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



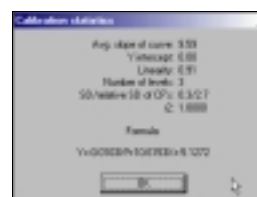
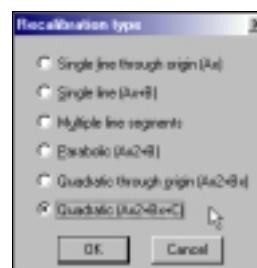
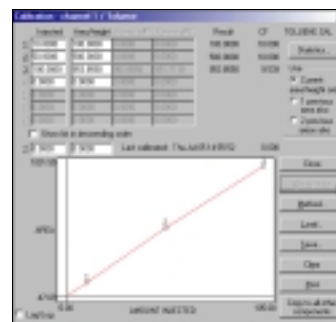
Recalibration level

Peak: Toluene
Time: 2.633
Area: 953.855

☐ 1 (10.000)
☐ 2 (50.000)
☒ 3 (100.000)
☐ 4 (20.000)
☐ 5 (2.000)
☐ 6 (2.000)
☐ 7 (2.000)

OK Cancel

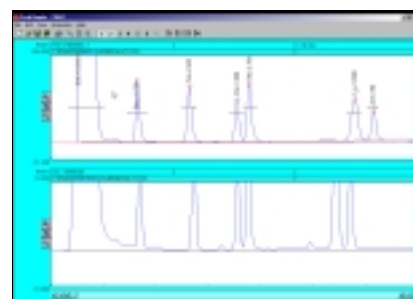
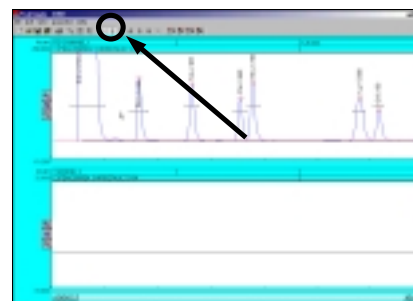
- After selecting OK from the Recalibration level menu the Calibration menu for Toluene will pop up. Check to make sure the flashing asterisk on the calibration curve is on level 3 and then click on the **Accept New** button to the right of the window.
- Once the new data is accepted, click on the **Method** 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 (Ax^2+Bx+C)** radio button and then click on **OK** with the mouse cursor.
- 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.



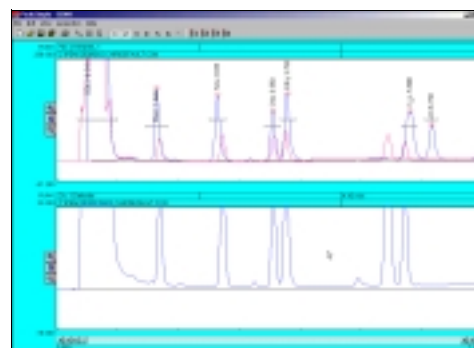
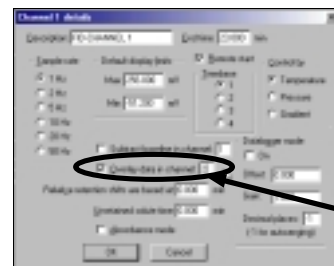
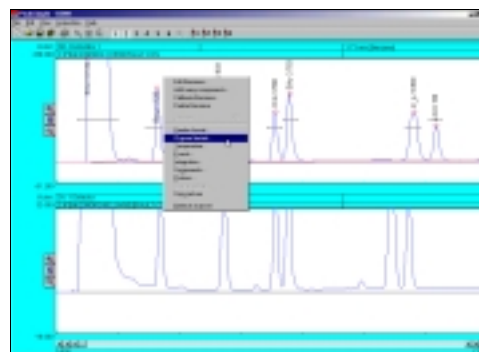
Overlay

- 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.
- 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.

2

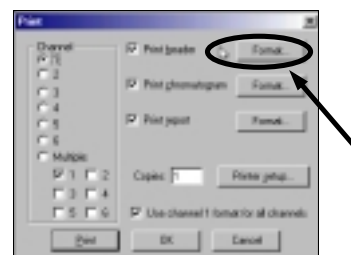
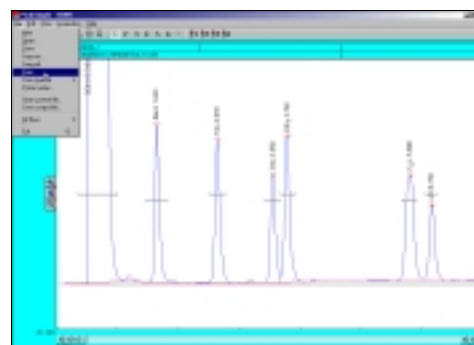


- Once FID602.CHR is open in the second channel right click using the mouse on the chromatogram in the first channel and select **Channel Details** from the list of options.
- After the Channel 1 details window appears on the screen locate the **Overlay data in channel** check box and select it. Look 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.
- 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

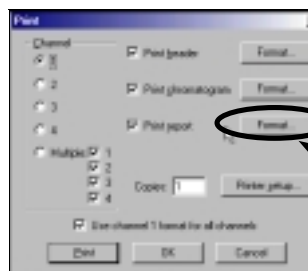
- To print a chromatogram first open chromatogram 602.CHR. Once the chromatogram is open select **File** from the PeakSimple menu bar and then select **Print** from the drop-down menu.
- The Print window will open and will allow the user to customize the printing of a chromatogram. Click on the **Format** 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.



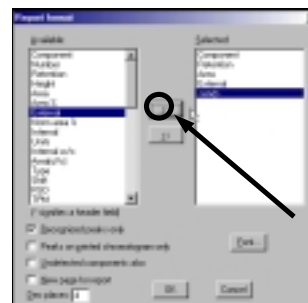
3. In the Print window click on the **Format** button for Print chromatogram to open up the Chromatogram format window. Locate the **Chart speed** dialogue box and insert the number of inches each minute on the chromatogram will take up when printed (for a nine minute run try **0.50** inches per minute).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



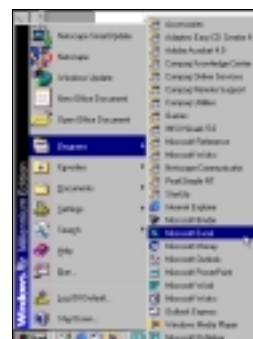
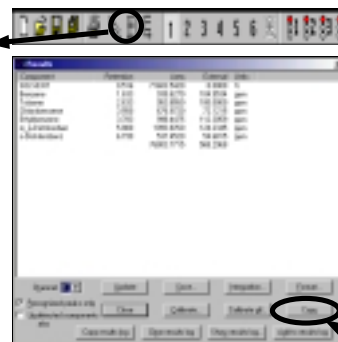
5. Once the Report format window is open click on **External** in the Available dialogue menu (on the left) and then click with the mouse cursor on the right facing arrow button to add External to the Selected dialogue box (on the right). After External is added to the Selected dialogue box click on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



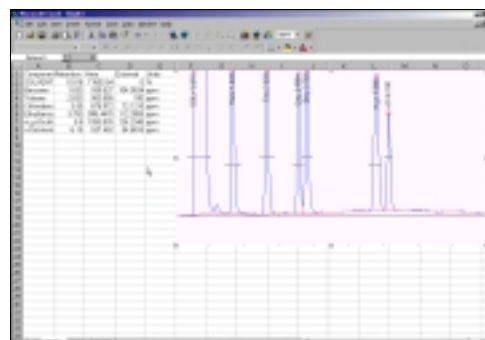
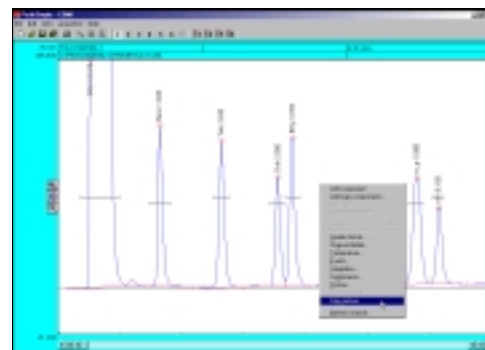
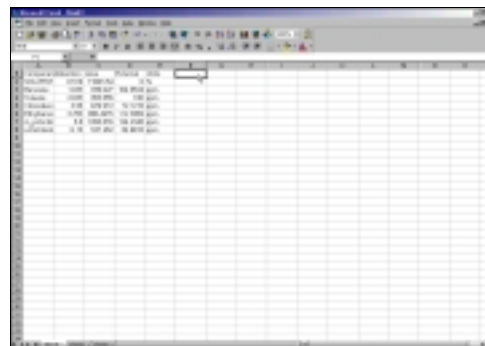
6. Select **Print** in the Print window to print the chromatogram or click on **OK** in the Print window to exit the window.

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

An Advanced Tutorial can be obtained by going to:
www.srigc.com online

If you have questions or would like to place an order call:
(310) 214-5092

Model 302

Six Channel USB PeakSimple Data System

7. Connect Power to the Model 302

The Model 302 is provided with a power cord which plugs into a standard 110 (or 220) volt outlet. Plug the Model 302 into the wall outlet. Turn ON the power switch and verify that the POWER LED on the front of the Model 302 is lit.



The power LED is lit when the Model 302 is connected to a power source & switched ON.

8. Install PeakSimple Chromatography Software

8-1. Locate your copy of PeakSimple, which is shipped inside the front cover of your manual. Insert the CD or floppy disk(s) into your computer's appropriate drive.



8-2. Open the appropriate drive through My Computer, then double click on "Setup.exe" and follow the instructions. By default, the setup program places the PeakSimple application directory on the hard drive: c:\peak2000. If you put the application directory elsewhere, take note of the path as you may have to enter it in a dialog box during the USB driver installation procedure.

9. Install the USB Drivers

There are three important files saved to the PeakSimple application directory at the conclusion of the software installation: LL_USB.inf, LL_USB.sys, and LL_USB2K.sys. These files are required for Windows to recognize the A/D board connected to the computer's USB port.

9-1. Double-click on the My Computer icon on your desktop, then on Control Panel, then on Add New Hardware, which should open the Add New Hardware Wizard.

9-2. Click the Next button twice, until you get to the screen that gives you a choice between letting Windows find the new hardware, or selecting it yourself from a list. Click the radio button to choose the hardware from a list and click the Next button.



Model 302

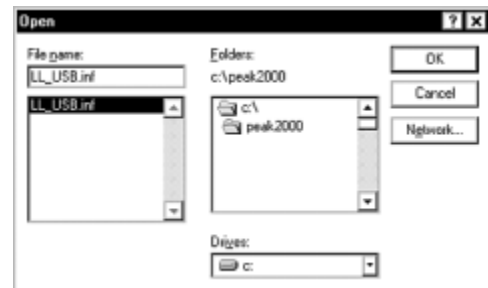
Six Channel USB PeakSimple Data System



9-3. Scroll down the hardware list, click on Universal Serial Bus controllers, then click Next. From the following screen click the Have Disk button.



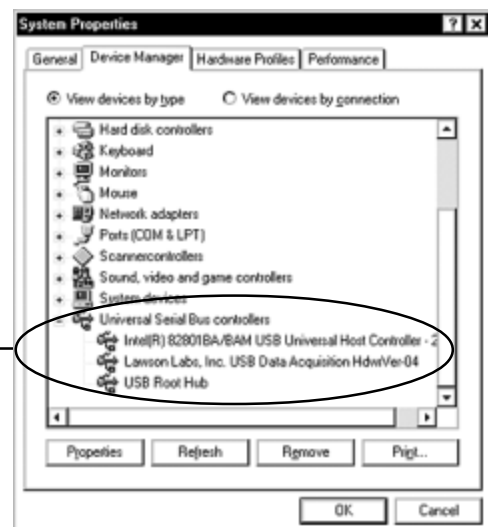
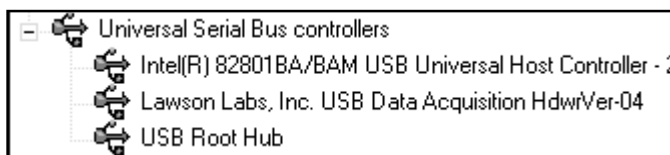
9-4. Click Browse and navigate to the PeakSimple application directory, or type in the path ("c:\peak2000" or the name you have chosen). The Wizard should find the LL_USB.inf file. When you click OK, the Wizard will verify that you want to copy files from the PeakSimple directory ("Copy manufacturer's files from: c:\peak2000").



9-5. When you click OK again, the Wizard will confirm that the drivers are for Lawson Labs. Click Next on this screen and the following screen, and Windows will finish installing the software for the Model 302. Click Finish.



9-6. Restart your computer (you MUST restart your computer before the drivers will work). Open the Control Panel again, then System, then click on the Device Manager tab. If the USB drivers have been successfully installed, the Universal Serial Bus controllers section will list "Lawson Labs, Inc. USB Data Acquisition HdwrVer-04."




Model 302

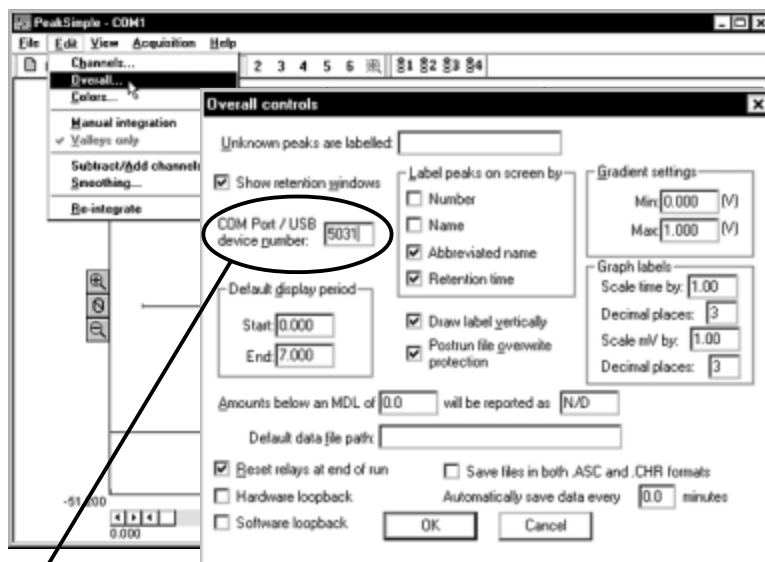
Six Channel USB PeakSimple Data System

10. Launch PeakSimple

10-1. Double-click on the PeakSimple icon to launch the program. Verify that communication has been established between your computer and the Model 302. An error message will appear if communication is not established. This is normal until you complete the following step.

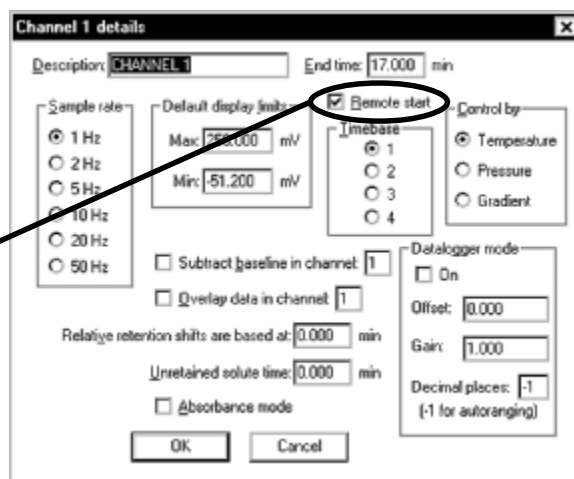


10-2. Each SRI USB data system has a unique 4-digit USB device number beginning with "5" (5031, 5032, etc.). This I.D. number is printed on the back of your Model 302, and on your PeakSimple disk. Open the PeakSimple Edit menu and choose Overall. Enter your Model 302 I.D. number in the box labeled "Com port / USB device number." Click OK, and PeakSimple will attempt to "wake-up" the data system. Click the Save All  icon so you don't have to re-enter the USB device number.



Enter the 4-digit USB device number here

10-3. For the remote start option:
Open the Edit menu and choose Channels. Click on the Details button for channel 1. Verify that Remote start is enabled (the box should be checked). Repeat this step for channels 2-6 if necessary.



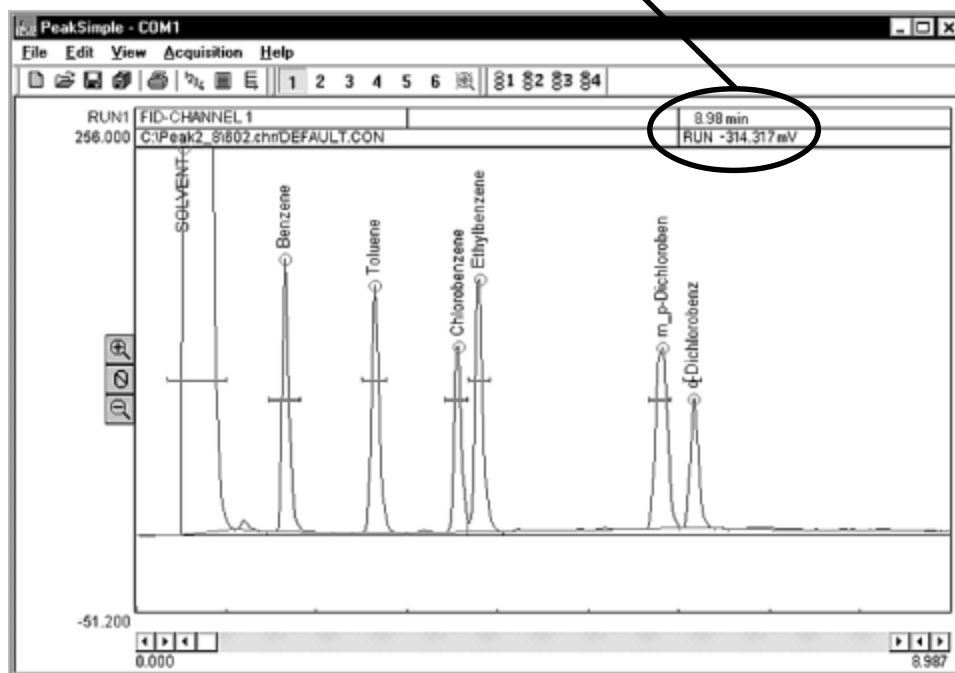
10-4. For information about using Event tables, manual Relay activation, etc., see the "PeakSimple Tutorials" and the "PeakSimple Software" sections in the manual (and online at www.srigc.com—click on the "Download Our Documents" button on the homepage).

Model 302

Six Channel USB PeakSimple Data System

11. Starting an Analysis

10-1. The upper right corner of the PeakSimple chromatogram window contains real-time information pertinent to your analysis in progress. The status of the run (STAND BY, RUN) is displayed in capital letters next to the millivolt (mV) reading, underneath the amount of time into the run.



11-2. Hit your computer keyboard spacebar to begin the run, and the data is plotted onscreen in the chromatogram window.



Press the spacebar to begin the run

Press the End key to stop the run

11-3. Hit the End key on your computer keyboard to stop the run.

Technical Support:

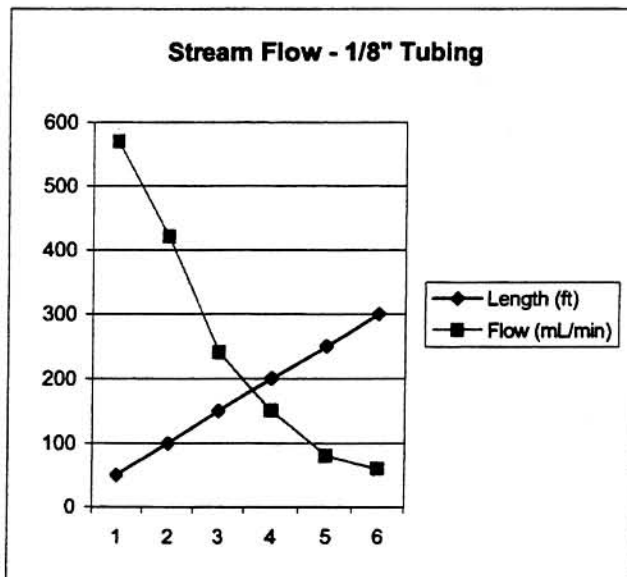
If you have questions or problems, call SRI for free technical support at 310-214-5092, 8am - 5pm California time.

Stream Flow using Standard Vacuum Pump

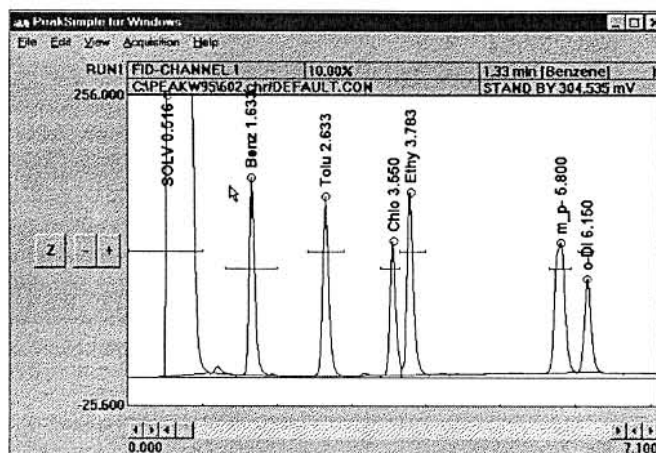
3/16/03

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

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



PeakSimple for Windows Software and Chromatography Data System Validation Statement



February 1, 1999

- 1) PeakSimple for Windows software and Chromatography Data System (PeakSimple) is written, manufactured and maintained by SRI Instruments, Inc. a Nevada Corporation.
- 2) PeakSimple for Windows software has been under continuous development since 1994. Periodic testing of the software is performed by SRI employees.
- 3) PeakSimple software is designed to be self-validating to enable quick verification by customers that PeakSimple is functioning consistently, reliably and according to specifications under actual operating conditions.
- 4) Self-validation is performed by configuring PeakSimple into the "Loopback mode" (see loopback instructions in manual). In this mode, an actual user generated chromatogram which is loaded into channel 4 is re-played (like a tape recorder) through the TP2 output channel, and then re-acquired and processed through any one of the remaining input channels. This is done 7 or more times to insure that data is being processed consistently and reliably.
The results from multiple loopback analyses are used to calculate the percent relative standard deviation (precision) of each peak in the chromatogram. Chromatographic data is highly variable, and the precision obtained is dependent on many factors including the peak shape, signal to noise ratio, interferences, co-eluting peaks, data acquisition rate and customer selected integration parameters. For this reason, self-validation is more valid than factory validation, since self-validation takes into account the exact chromatographic conditions and user specified parameters in effect for the particular application whereas factory validation can not.

Loopback Test: For Data Validation

A loopback test may be performed if you are required to validate the precision of the G.C. or Data System's analog to digital conversion. This test requires the user to install a jumper wire on the A/D board inside the G.C. or Data System.

Description of Test:

A jumper wire is installed on the A/D board between 'temperature program one', (TP1), and 'channel one signal input', (Sig. 1+). A data file is then loaded into channel four. When the 'loopback' mode is selected in PeakSimple, the data on channel four is routed out TP1 to the channel one signal input. When a chromatogram run is started, channel one will begin to reproduce the data loaded into channel four. After the run has completed, area counts from a specific data peak may be collected and the run repeated several times. After at least three runs, the user may then calculate the average area counts and the percent relative standard deviation, (%RSD) and thus the precision of the A/D converter. Less than 0.5% RSD is typical for the SRI Model 202 and 203 A/D boards.

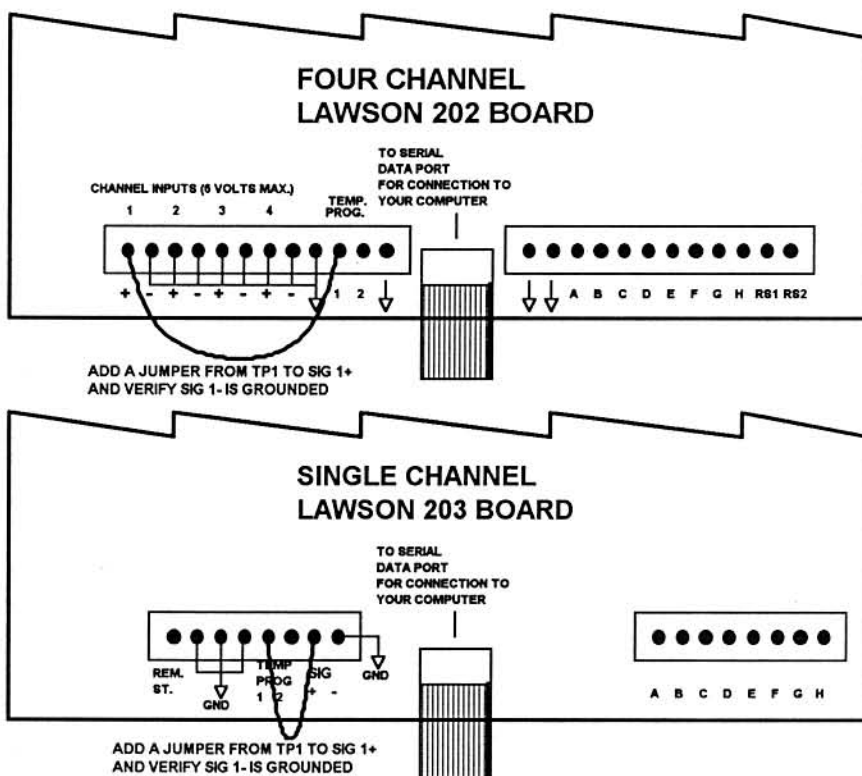
Setting Up The Hardware:

With the G.C. unplugged, remove the six screws securing the bottom cover. Flip the G.C. on its back and locate the A/D board on the right-hand side. Remove any wires from 'TP1' and 'SIG 1+' and add an insulated 22 AWG wire between TP1 and SIG 1. Refer to the diagram below for jumper placement.

Most systems will contain the Four Channel 202 Board. Also, verify that SIG 1- is grounded. Add another jumper if needed.

Some systems will contain the Single Channel 203 Board. Also, verify that SIG 1- is grounded. Add another jumper if needed.

You could also run the TP1 wire to SIG 1+ through a relay for automatic hardware setup.

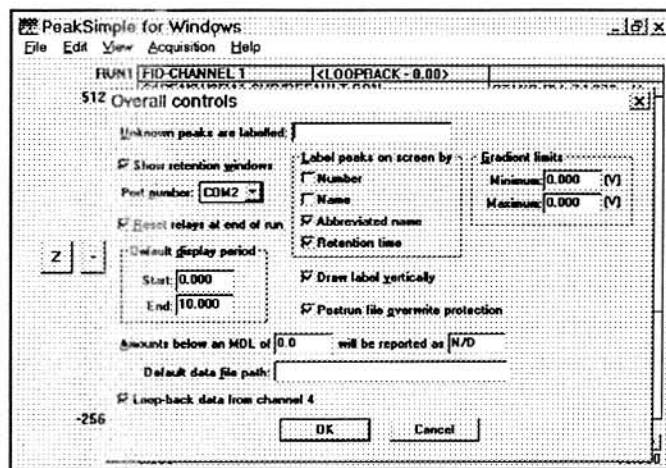


Loopback Test: (continued)

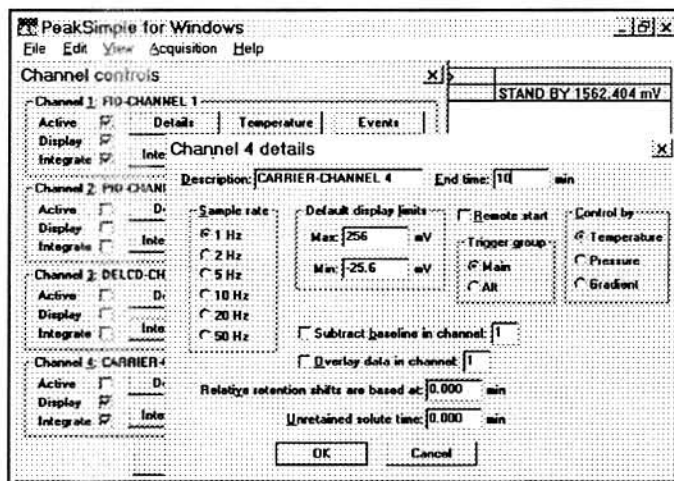
Setting Up The Software:

Re-attach the bottom cover and plug the G.C. back in. Turn the G.C. power on and start PeakSimple. Verify that the computer is communicating properly with the G.C..

In the **EDIT-OVERALL** screen, check the **LOOPBACK** box. Set the **START TIME** to 0 minutes and the **END TIME** to 10 minutes. Also verify that the **SHOW RETENTION WINDOWS** box is checked.

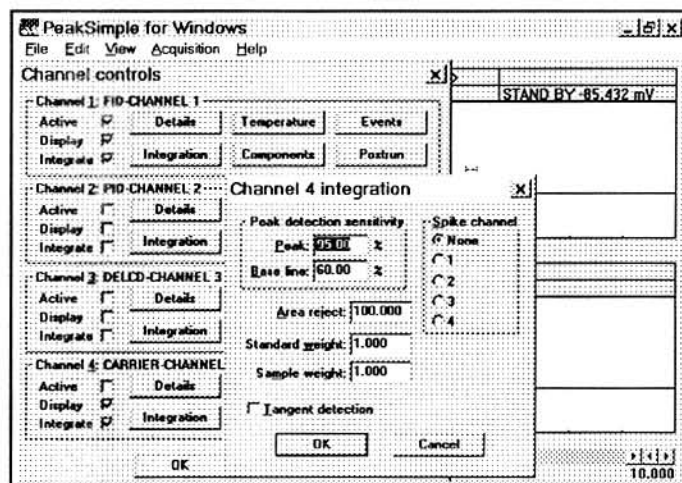


In the **EDIT-CHANNELS** screen, check the **ACTIVE**, **DISPLAY** and **INTEGRATE** boxes for channel 1. And check the **DISPLAY** and **INTEGRATE** boxes for channel 4.



In the **EDIT-CHANNEL 1-DETAILS** screen, set the **END TIME** to 10 min. Set the **SAMPLE RATE** to 1. Set the **DEFAULT DISPLAY LIMITS** to 256 MAX and -25.6 MIN. Set the **TRIGGER GROUP** to MAIN. Click OK to close the **DETAILS** screen. Then repeat for **CHANNEL 4-DETAILS**.

In the **EDIT-CHANNEL 1-INTEGRATION** screen, set the **AREA REJECT** to 100. Set the **PEAK DETECTION SENSITIVITY** to 'PEAK=95%, BASELINE= 60%'. Set the **SPIKE CHANNEL=NONE**, **STANDARD WEIGHT=1**, **SAMPLE WEIGHT=1**, and make sure the **TANGENT DETECTION BOX** is **UNCHECKED**. Click OK to close the **INTEGRATION** screen. Then repeat for **CHANNEL 4-INTEGRATION**.



Loopback Test: (continued)

Software Setup: (continued)

In the **EDIT-CHANNEL 1-COMPONENTS-LOAD** screen, highlight the **602.cpt** sample components file and click **OPEN**. Click **OK** again to close the **COMPONENTS** screen. Then repeat for **CHANNEL 4-COMPONENTS**.

In the **FILE-OPEN** screen, select **CHANNEL 4** at the bottom of the window and then highlight the **602.chr** sample chromatogram file and select **OPEN**.

The 602.chr sample chromatogram that is now displayed on channel four represents the data that will be fed back through the A/D converter.

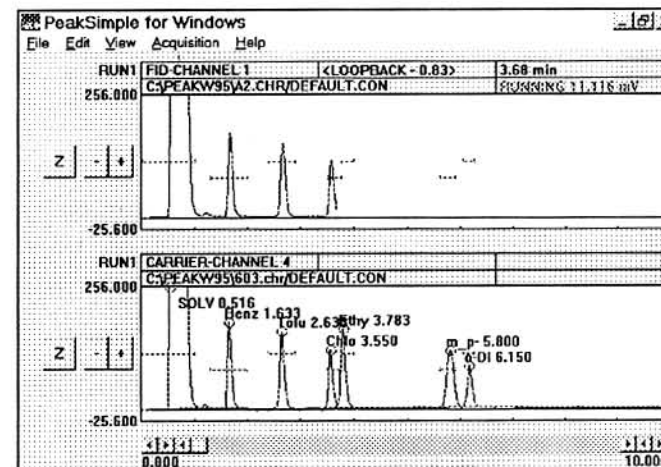
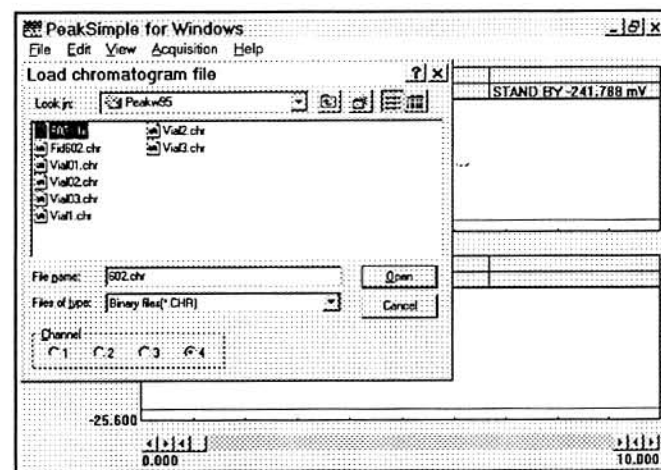
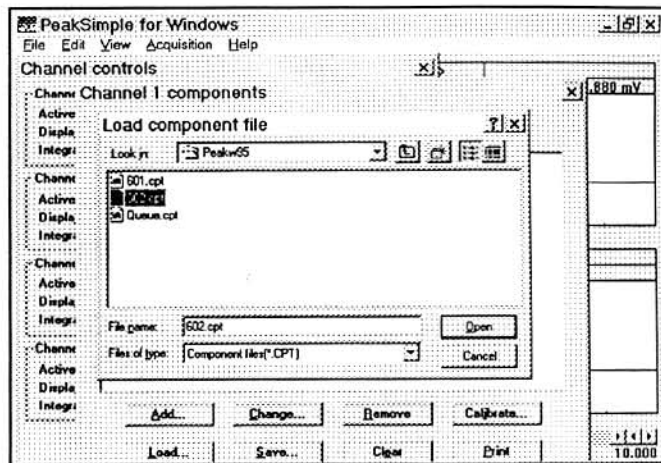
Starting the Run:

Auto-zero channel 1 by clicking the '**Z**' button. Depress the **SPACEBAR** and the chromatogram will start running. The data on **CHANNEL 1** should appear to be an exact replica of the data that was fed into **CHANNEL 4**.

Collecting the Data:

After the run has completed, make note of the area counts of one of the peaks by left-clicking on one of the peaks. Toluene, for example, may have an area count of 931.

Repeat the run three or more times; for each run, record the area counts of the same peak. Once the data has been collected from at least three runs; an average area count can be calculated as well as the percent relative standard deviation.



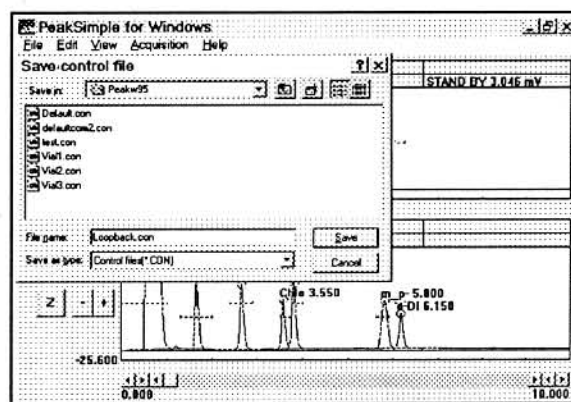
Loopback Test: (continued)

Calculate the Standard Deviation:

Using the data collected, calculate the average area counts for Toluene. Typically this value is around 950. Then calculate the %RSD which is usually less than 0.5%. You may notice that there is a small magnitude of error between the **CHANNEL 1** and **CHANNEL 4** area counts. This is due to the D/A converter and not the A/D converter. Since the loopback test measures the **PRECISION** of the A/D converter and not its **ACCURACY**, this minor discrepancy is insignificant.

Save Your Loopback Test as a CONTROL FILE:

If you wish to run this test again or if you continue with the next step and modify the **Peakwin.ini** file, you will need to save the loopback parameters as a **CONTROL FILE**. In the **FILE-SAVE CONTROL FILE** screen, type in **loopback.con** and click **OK**.



Modifying the Peakwin.ini File, (optional)

If desired, any inaccuracy of the D/A converter can be adjusted by attenuating the **LOOPBACK OUTPUT** to match the input signal. This adjustment can be made by entering the line "**LoopbackFactor=X**" in the [Lawson] section of the **PEAKWIN.INI** file located in the **WINDOWS** directory. The default value of '**X**' is **0.098**.

NOTE: Changes to the Peakwin.ini file will not be recognized unless the PeakSimple application is restarted. After you have obtained the average area count for Toluene on **CHANNEL 1**, exit PeakSimple by pressing '**Q**', then '**Y**'.

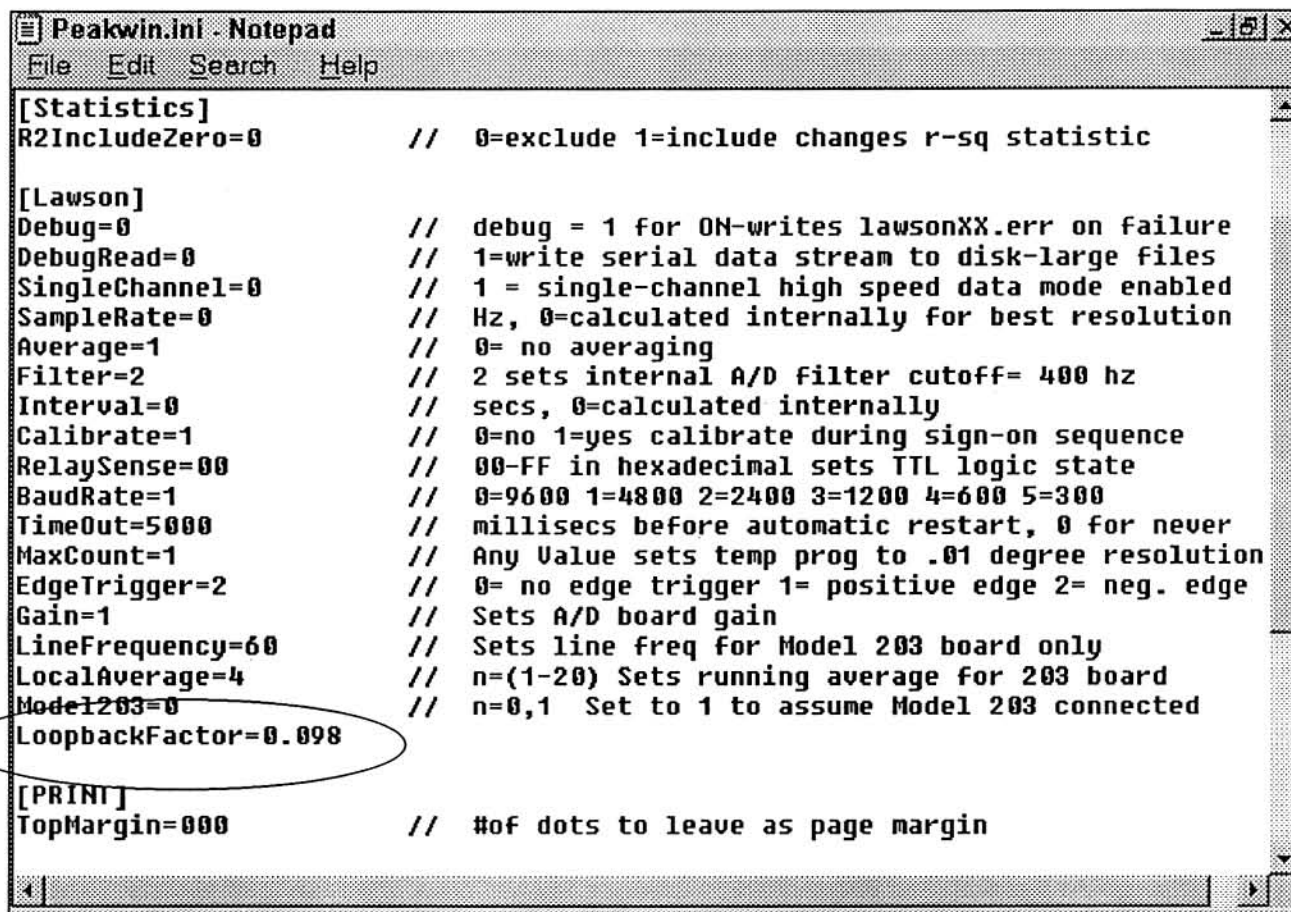
To calculate the new value for '**X**', first determine the average area counts of a specific peak for **CHANNEL 1** and also determine the area count of the corresponding peak on **CHANNEL 4**. Next, divide the **CHANNEL 4** area count by the **CHANNEL 1** area count. Multiply this ratio by 0.098. Substitute this new value for '**X**'.

For example, using 602.chr as the chromatogram file, the toluene area count for **CHANNEL 4** is **953**. If the average area count of toluene on **CHANNEL 1** is **931** then the ratio would be $953 / 931 = 1.0236$. Multiply 1.0236×0.098 , the answer is **0.1003128**. Round this new value for '**X**' to 0.1003.

Loopback Test: (continued)

Modifying the Peakwin.ini File, (continued)

Find the **PEAKWIN.INI** file in the **WINDOWS** sub-directory. Double-click to open it. Scroll down until you find the [Lawson] section. Place the cursor at the last line of the [Lawson] section and type "**LoopbackFactor=X**", and then press **ENTER**. **X** is the value you calculated earlier. For example "**LoopbackFactor=0.1003**".



```
[Statistics]
R2IncludeZero=0          // 0=exclude 1=include changes r-sq statistic

[Lawson]
Debug=0                  // debug = 1 for ON-writes lawsonXX.err on failure
DebugRead=0              // 1=write serial data stream to disk-large files
SingleChannel=0          // 1 = single-channel high speed data mode enabled
SampleRate=0             // Hz, 0=calculated internally for best resolution
Average=1                // 0= no averaging
Filter=2                 // 2 sets internal A/D filter cutoff= 400 hz
Interval=0               // secs, 0=calculated internally
Calibrate=1              // 0=no 1=yes calibrate during sign-on sequence
RelaySense=00            // 00-FF in hexadecimal sets TTL logic state
BaudRate=1               // 0=9600 1=4800 2=2400 3=1200 4=600 5=300
Timeout=5000             // millisecs before automatic restart, 0 for never
MaxCount=1               // Any Value sets temp prog to .01 degree resolution
EdgeTrigger=2            // 0= no edge trigger 1= positive edge 2= neg. edge
Gain=1                   // Sets A/D board gain
LineFrequency=60         // Sets line freq for Model 203 board only
LocalAverage=4           // n=(1-20) Sets running average for 203 board
Model203=0               // n=0,1 Set to 1 to assume Model 203 connected
LoopbackFactor=0.098

[PRINT]
TopMargin=000           // #of dots to leave as page margin
```

Press **ALT-F** then **S** to save the file. Press **ALT-F** then **X** to exit. Restart **PeakSimple** and load the loopback.con control file you saved earlier. Run the loopback test again. The accuracy of the D/A converter should be improved. (Channel 1 toluene area counts should closely match the channel 4 toluene area counts).

End of Test:

Turn off G.C. power and re-connect the original A/D board wiring. The loopback test is completed.

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

In the LOAD position:

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

In the INJECT position:

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

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

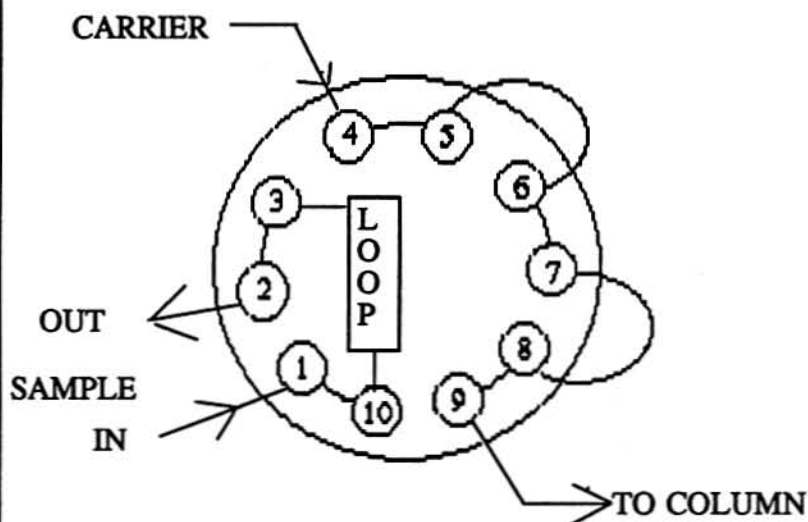
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

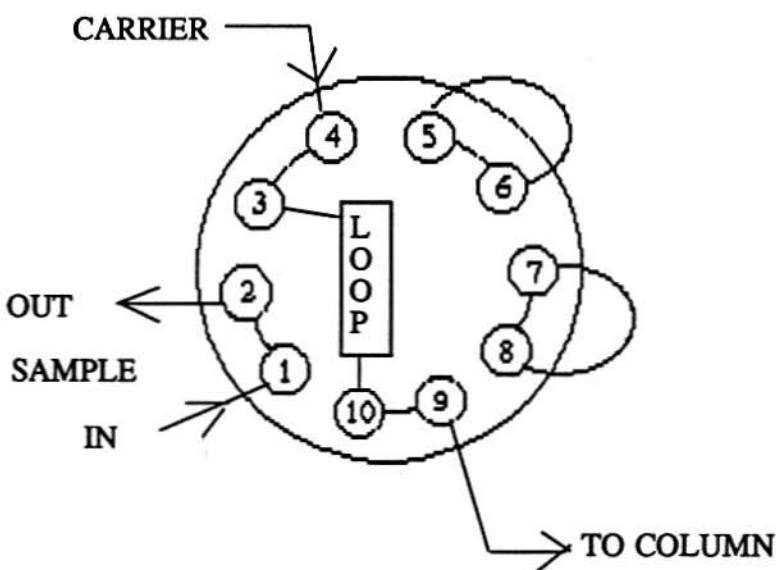
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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

REV. 9-7-91

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

In the LOAD position:

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

In the INJECT position:

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

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

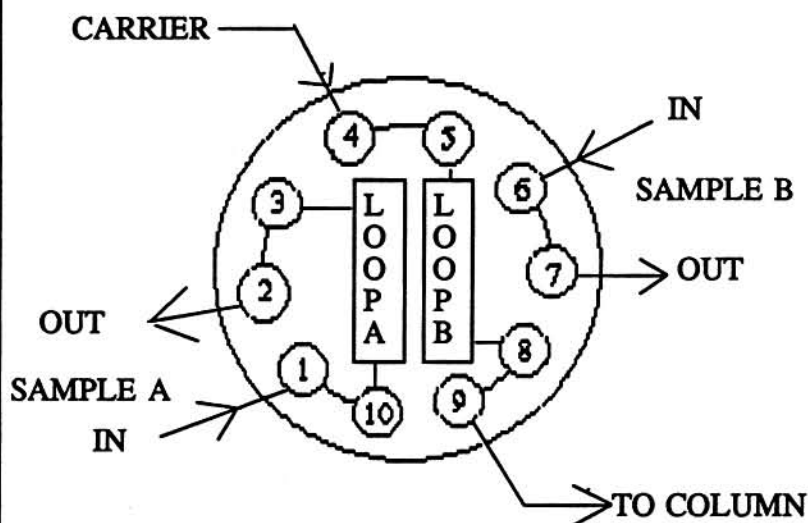
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

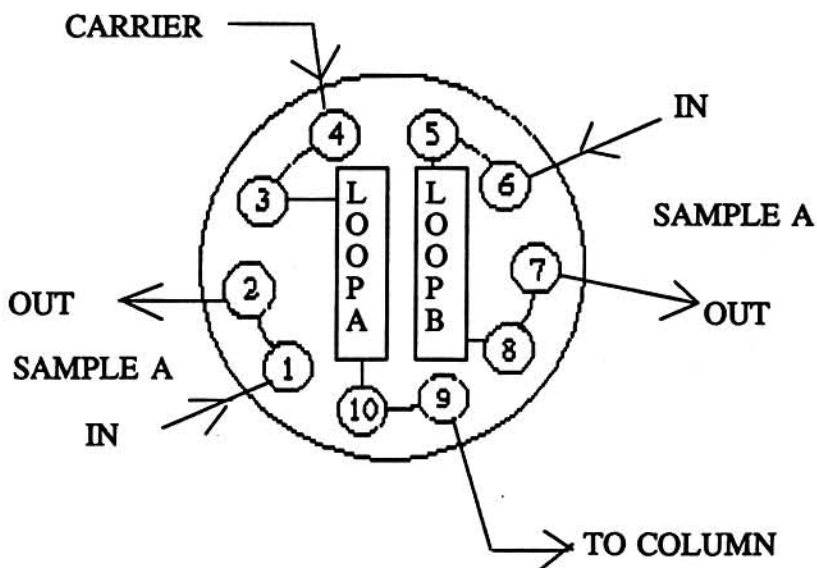
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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

In the LOAD position:

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

In the INJECT position:

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

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

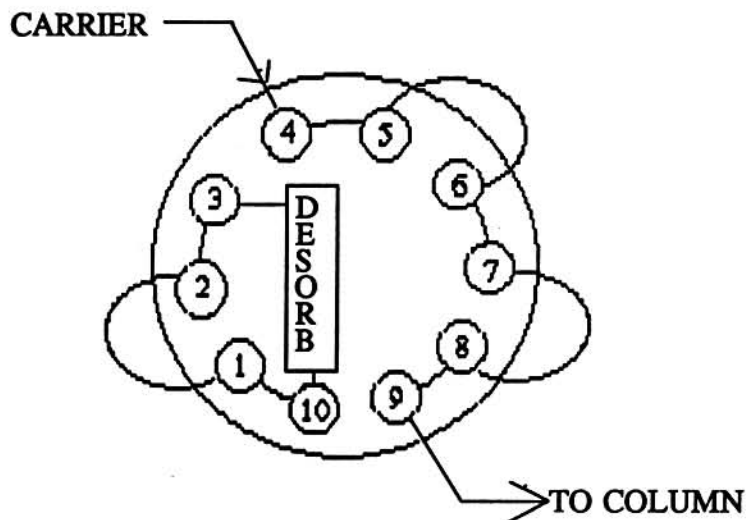
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

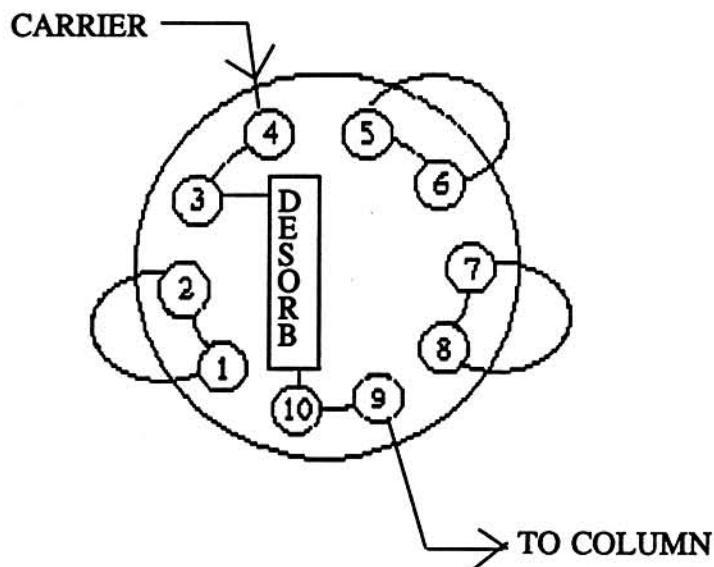
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



Chapter: INJECTORS & GAS VALVES

Topic: SIMULTANEOUS INJECTION OF THE SAME
SAMPLE INTO TWO SEPARATE COLUMNS

REV. 9-7-91

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

In the LOAD position:

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

In the INJECT position:

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

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

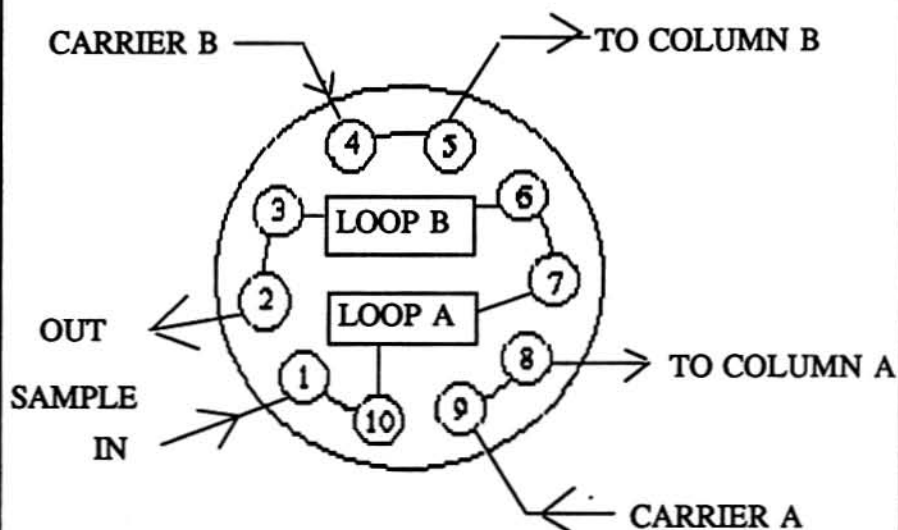
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

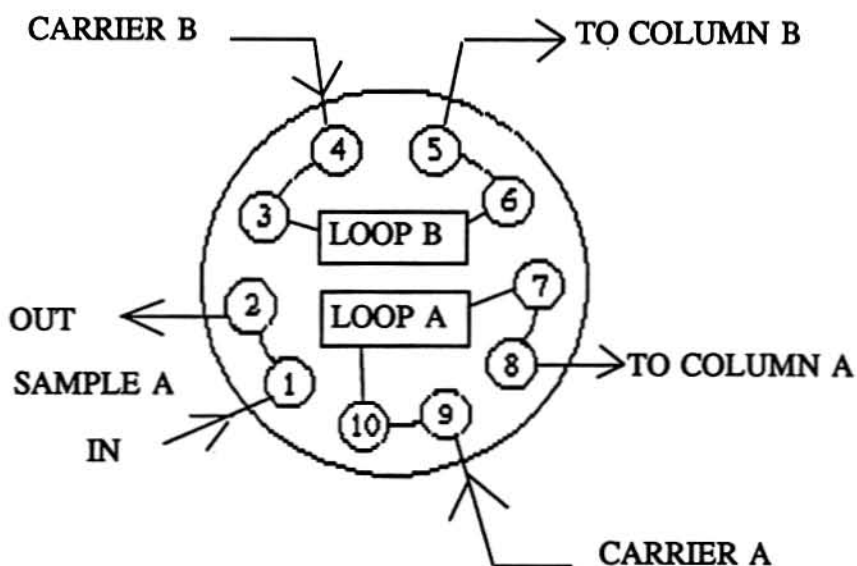
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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

In the LOAD position:

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

In the INJECT position:

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

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

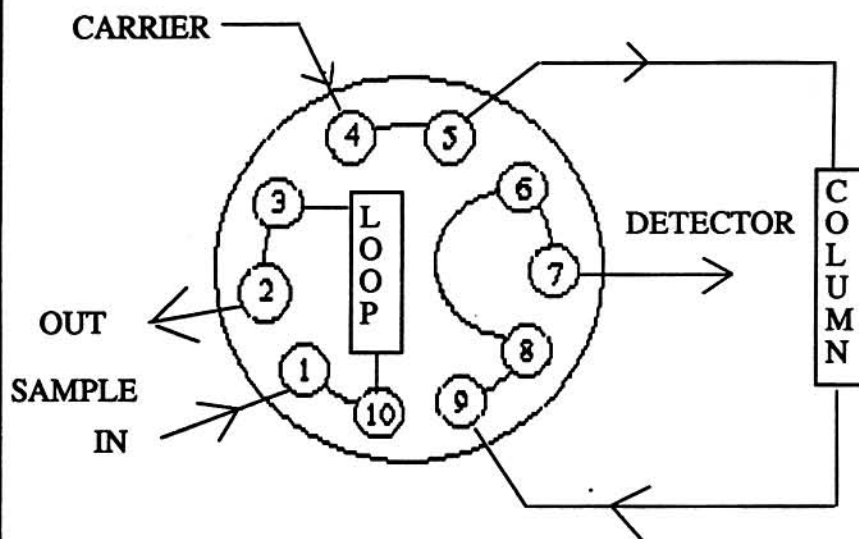
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

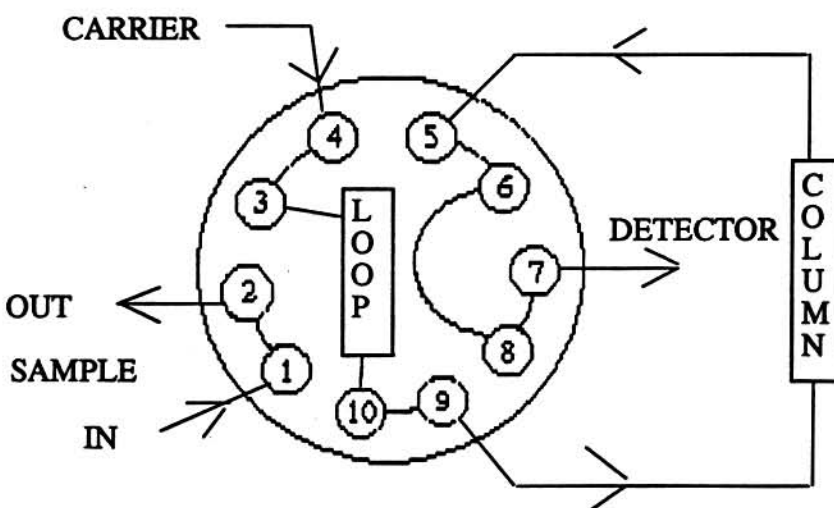
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



Chapter: INJECTORS & GAS VALVES

Topic: LOOP SAMPLING WITH BACKFLUSH
OF PRE-COLUMN TO VENT

REV. 9-7-91

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

In the LOAD position:

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

In the INJECT position:

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

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

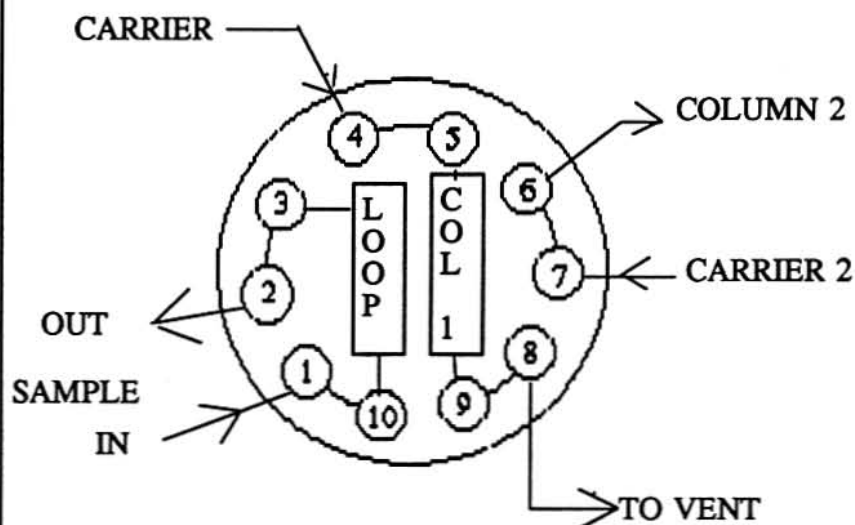
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed by:

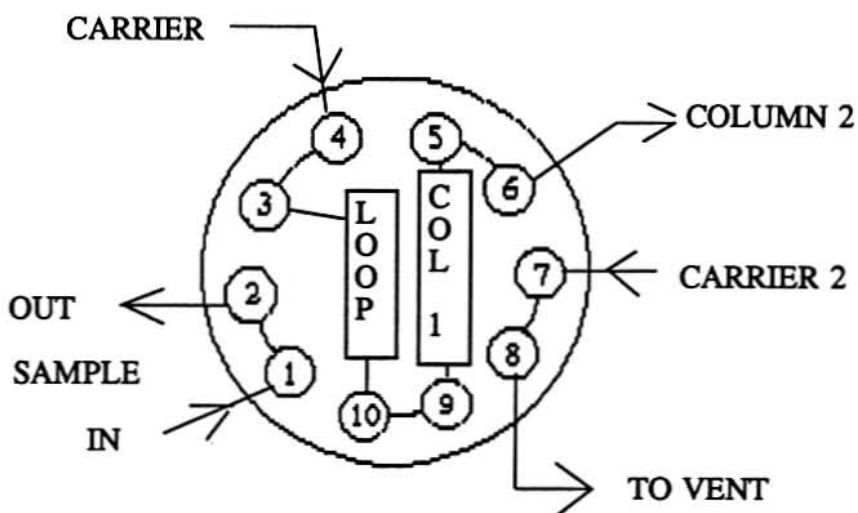
Date:

GC serial number:

LOAD POSITION - TURN SHAFT CCW



INJECT POSITION - TURN SHAFT CW



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

In the LOAD position:

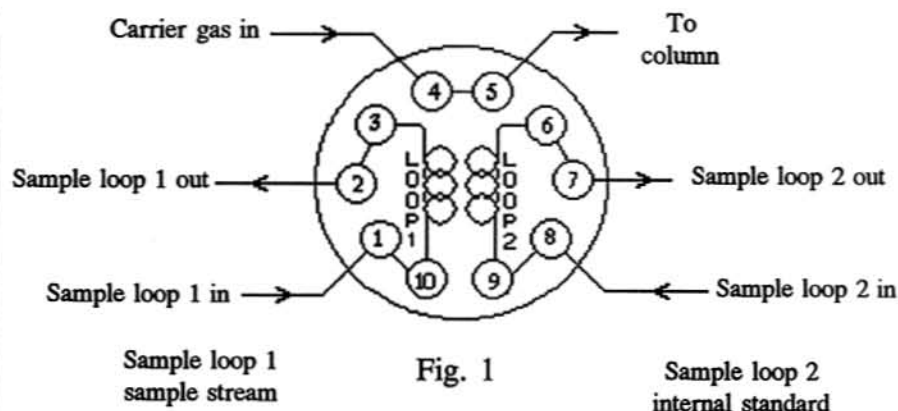
Two separate loop circuits exist in this configuration. Sample loop #1 receives sample through port #1, and vents through port #2. Sample loop #2 receives sample through port #8 and vents through port #7. Meanwhile the carrier gas is routed into port #4 from the injector, through the valve, and out through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

In the INJECT position:

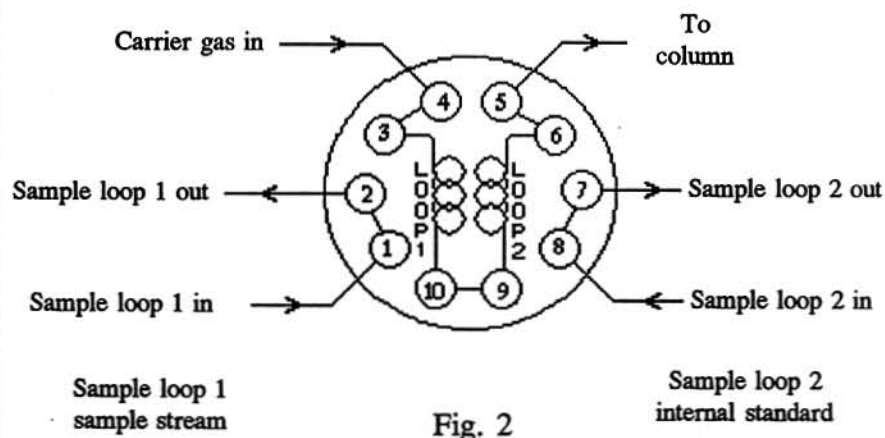
When the valve is rotated to the INJECT position, both sample loop inlets and outlets are isolated (ports #1 and #2, and #7 and #8). The carrier gas entering the valve at port #4 is diverted into loop #1 through port #3. Loop #1 is now in series with loop #2 and the contents of both loops is swept out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

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

10 Port Gas Sampling Valve in LOAD Position



10 Port Gas Sampling Valve in INJECT Position



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

Chapter: INJECTORS & VALVES

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

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

In the LOAD position:

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

In the INJECT position:

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

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

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

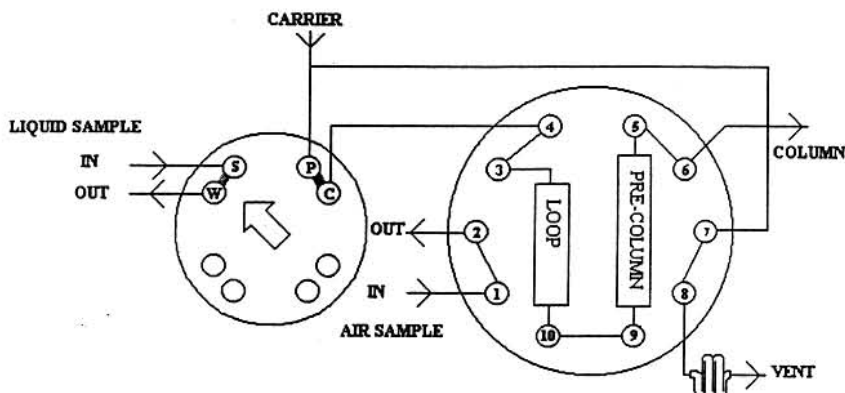
VALCO
BOX 55603
HOUSTON, TX 77055
(800) 367-8424
(713) 688-9345

Your Valve was plumbed
by:

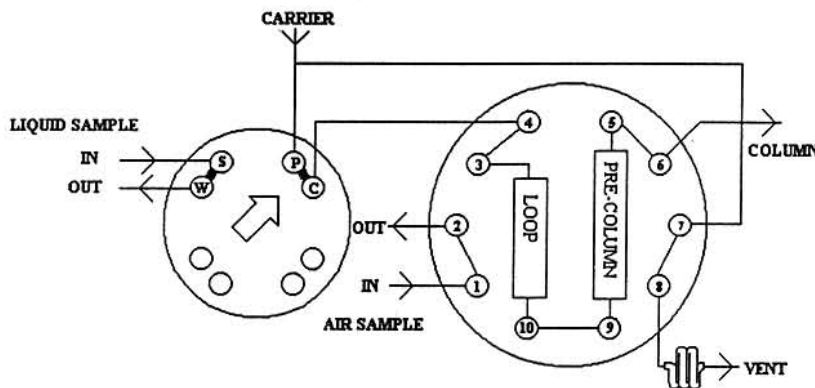
Date:

GC serial number:

LOAD POSITION



INJECT POSITION



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

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

A critical requirement in the cleanup of contaminated soil sites is an accurate determination of the nature and extent of soil contamination. The primary U.S. EPA-recommended method (EPA/SW-846-5030A and 8260A) for measurement of volatile organic compounds (VOCs) in soils is purge-and-trap,^{1,24,25} followed by gas chromatography/mass spectroscopy. Under this protocol, organic-free water containing internal standards and surrogates is mixed with a soil sample and heated to 40 °C. An inert gas is bubbled through the solution at ambient temperature, and the vapor is passed through a sorbent column, where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb

the components onto a gas chromatographic column.¹ Use of purge-and-trap to measure VOCs in soil is based on the assumption that soil VOCs rapidly equilibrate with soil water. Recent studies,^{2–5} however, strongly question this assumption and indicate that soil desorption is a biphasic process with an initial rapid surface desorption followed by a much slower, diffusion-limited, desorptive phase from the interior of the soil matrix.^{2–4,6–11}

The biphasic nature of desorption casts doubt on the widely used, EPA-recommended purge-and-trap method. When soil has been in contact with VOCs for a long time period (aged soils), VOCs diffuse into soil micropores, where they are unavailable for purge-and-trap measurement. Except for a single study⁶ involving 1,2-dibromoethane (EDB), the impact of soil aging on the effectiveness of VOCs measurement techniques is largely unexplored. The present study focuses on the effectiveness of three commonly used techniques for measuring VOCs concentrations in aged soils: purge-and-trap, methanol immersion, and hot methanol extraction. Since vapor extraction is a currently popular technique for removal of VOCs from soils, we subjected one soil sample to air stripping to evaluate the effect of vapor extraction on the extraction efficiency of purge-and-trap measurements.

EXPERIMENTAL PROCEDURE

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

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

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[†] University of Tennessee.

[‡] Chemical and Analytical Sciences Division, ORNL.

[§] U.S. Department of Energy.

^{*} Health Sciences Research Division, ORNL.

(1) Environmental Protection Agency. *Code of Federal Regulations*, Part 261; Title 40: U.S. Government Printing Office: Washington, DC, 1994; Chapter 1, App. III, p 67 (July 1, 1994).

Table 1. Comparison of Three Methods for measuring VOCs in Soils

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

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

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

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

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

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

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

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

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

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

Extraction Following Air Stripping. The Louisiana sample was mixed in a 7:3 ratio with calcium oxide to prevent solidification during air stripping. The sample was then placed in a Buchner

funnel with a vacuum running from the bottom of the funnel through a flask. Ambient air was passed through the sample using a vacuum of 450–675 mmHg for 1 week. Occasional mixing of the soil was performed throughout this time. The soil sample was divided into three subsamples, and each subsample was then subjected to one of the three methods as previously described.

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

RESULTS

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

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

DISCUSSION

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

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

The soil desorption process is known to involve two distinct phases: a rapid desorption from the soil surface occurring within 24 h and a much slower diffusion-limited desorption from the interior of the soil matrix occurring over a period of days to years.^{5,19} This biphasic desorption pattern is most pronounced in aged soils, where a significant fraction of contamination is located in the interior of the soil matrix. For example, despite its high volatility and degradability, 1,2-dibromoethane (EDB), a soil fumigant, was found⁸ in agricultural topsoil 19 years after its last known application. The persistence of EDB was attributed to desorption half-times of 2–3 decades at 25 °C.² For trichloroethylene (TCE), a continuous desorption study¹⁰ of long-contaminated soils revealed persistence of 18% of the initial TCE concentration after desorption with 24 000 pore volumes of water. In a subsequent study³ on simultaneous desorption of TCE, tetrachloroethylene, toluene, and xylene, a substantial portion (48–94%) of the sorbed contaminant mass resisted desorption after 7 days of contact time. TCE soil concentrations at the Picatinny

Arsenal were found to be 1–3 orders of magnitude greater than predicted using soil–gas concentrations and equilibrium conditions.¹⁸ The present study found that the purge-and-trap method, as compared to hot solvent extraction, recovered only 42 and 4.8%, respectively, of TCE in long-contaminated clays and silty loam soils.

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

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

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

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

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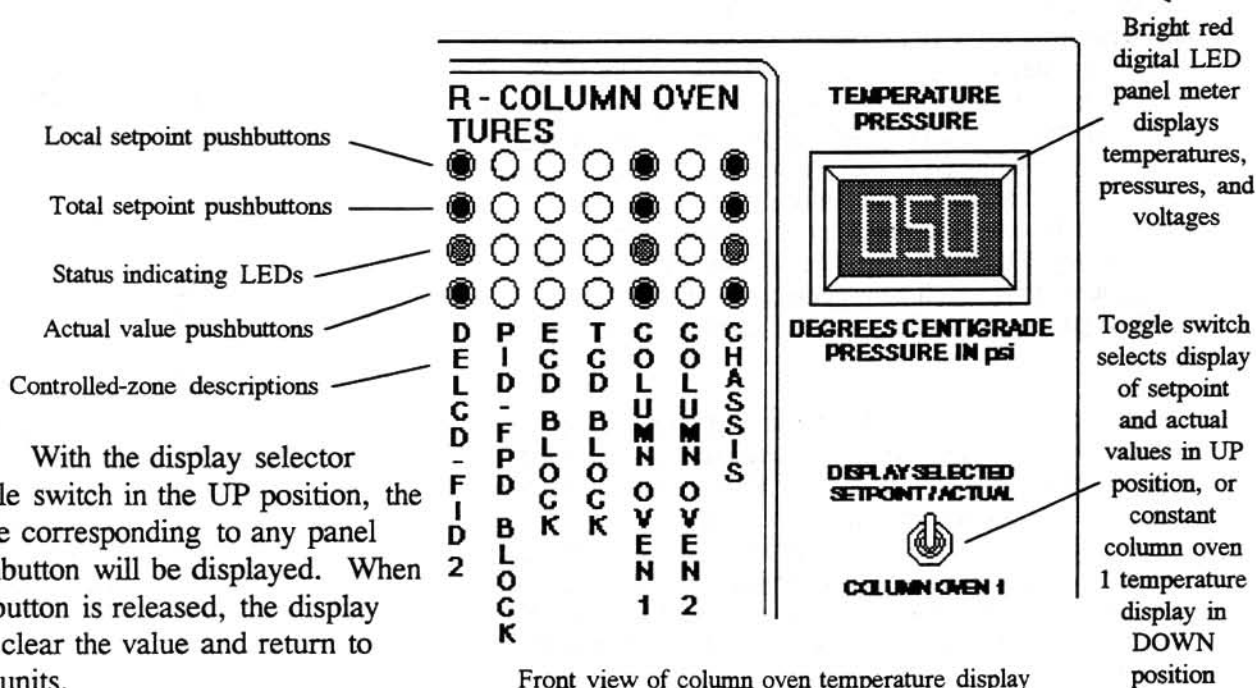
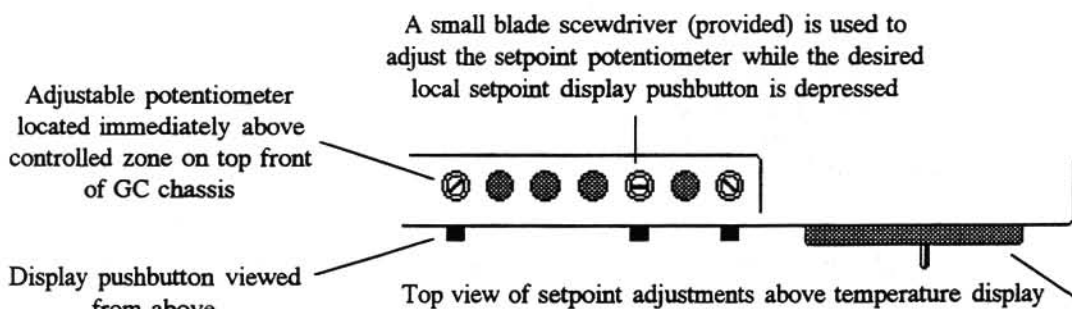
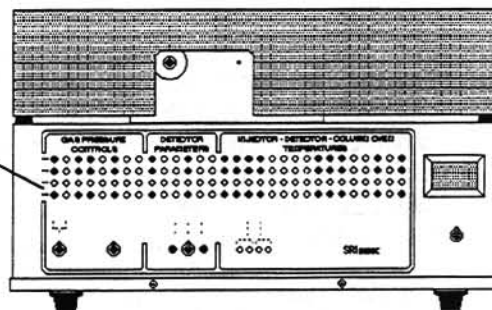
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The 8610C gas chromatograph permits easy display and adjustment of all controlled zone setpoints. To view a controlled zone, simply place the display selector switch in the UP position, and depress the desired feature pushbutton. Depending on the zone, the following values may be displayed: the actual value that the zone is being measured at, such as the current temperature of column oven 1; the local setpoint, which reflects the adjustable setpoint you currently have set, which, in the case of column oven 1, would be an offset value that could be summed with the temperature signal being sent from the data system; and the total setpoint, which is the sum of any signal being sent from the data system to the controlled zone, in addition to any local setpoint value you have set (for example, if column oven 1 has a local setpoint of 50 degrees, and the data system is instructing the GC to heat the column oven to 100 degrees, the total setpoint should display 150 degrees). Most zones will only display the local setpoint and actual value. Each zone also displays its status via a light-emitting diode (LED) that glows when the zone is active.

"At-a-glance" display panel also permits viewing of actual and setpoint values



With the display selector toggle switch in the UP position, the value corresponding to any panel pushbutton will be displayed. When the button is released, the display will clear the value and return to 000 units.

Front view of column oven temperature display