

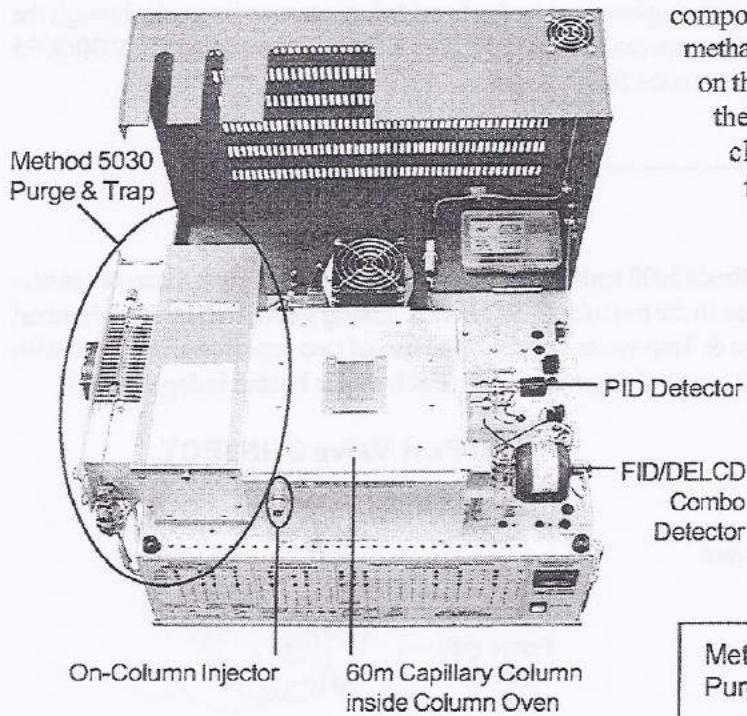
## POPULAR CONFIGURATION GCs BTEX & Environmental

### System Overview

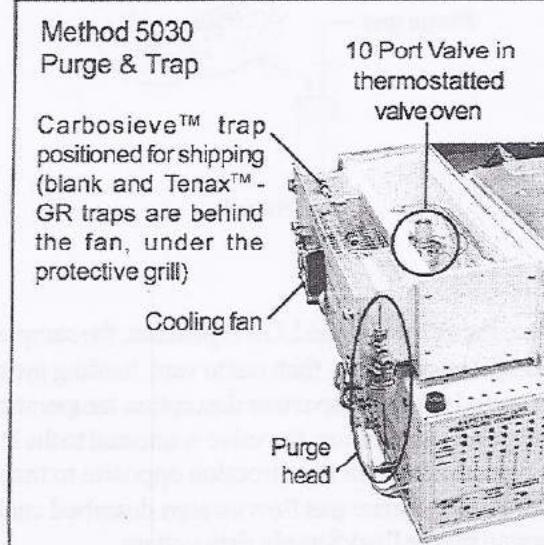
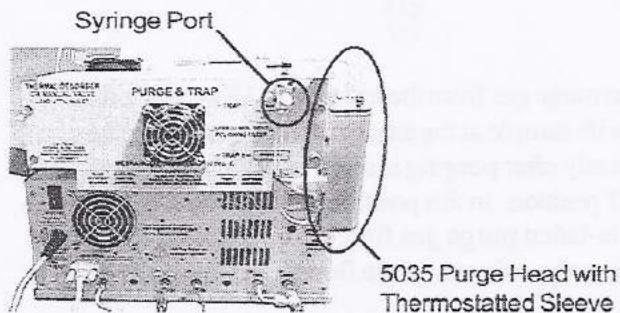
Your SRI Environmental GC is equipped with everything you need to generate certification quality data for EPA Methods 8010, 8015, 8021, and others. It is configured on the 8610C chassis, and includes a built-in Method 5030 or 5030/5035 compliant Purge & Trap for concentration of liquid and/or soil samples. 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 H<sub>2</sub>-50 hydrogen generator for tankless field operation.



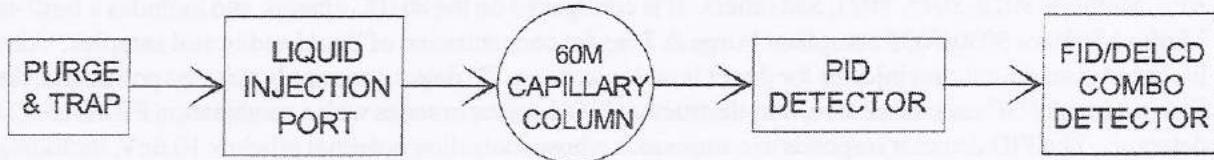
Method 5030/5035 Purge & Trap on an Environmental GC



## POPULAR CONFIGURATION GCs

### BTEX & Environmental

#### Theory of Operation

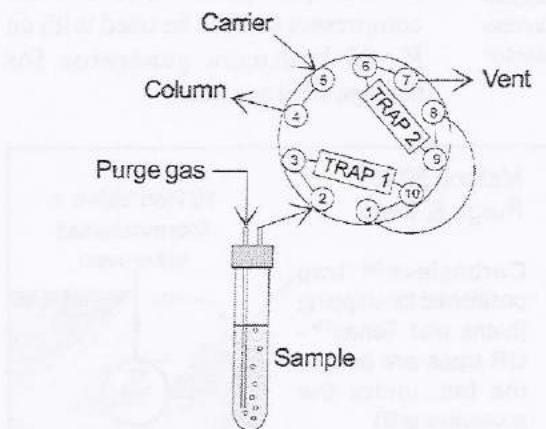


The versatile BTEX/Environmental GC systems can analyze gas, water, and soil samples. Four types of injection techniques can be used: purge and trap, direct liquid injection, TO-14 type gas sample concentration, and manual headspace injection. The Purge & Trap concentrator may be used for gas, liquid, and solid samples. For liquid samples up to 5 $\mu$ L 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.

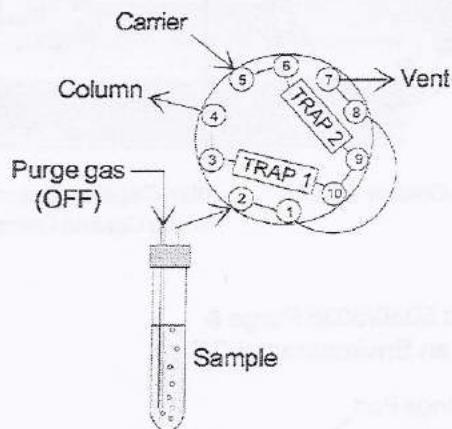
#### Purge & Trap Injection

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.

**10 Port Valve in LOAD**



**10 Port Valve in INJECT**



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**

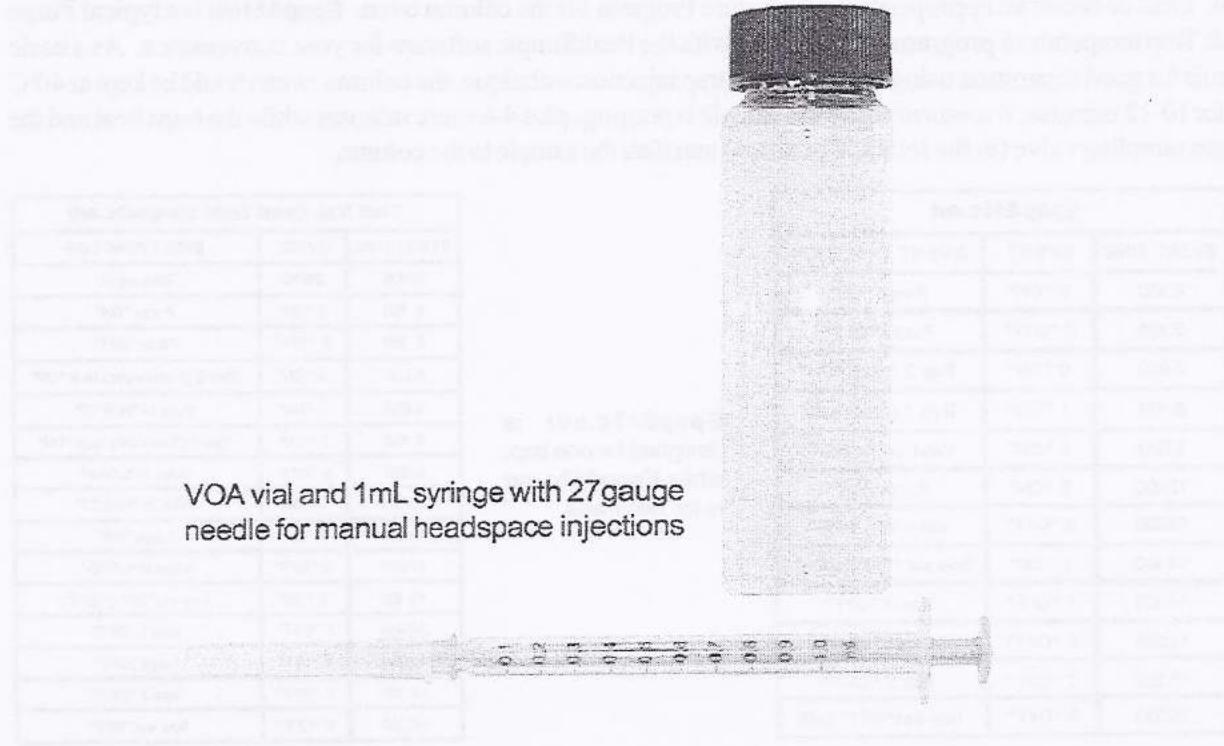
Direct injection with the BTEX or Environmental GC systems is simple and straightforward. This method uses the on-column injector to inject the sample directly into the column, bypassing the entire purge and trap injection system. Sample size for this technique is 1mL or less for gas, and 5 $\mu$ L or 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.srgc.com](http://www.srgc.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 (Carbo sieve) heat "ON"
6.000	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"

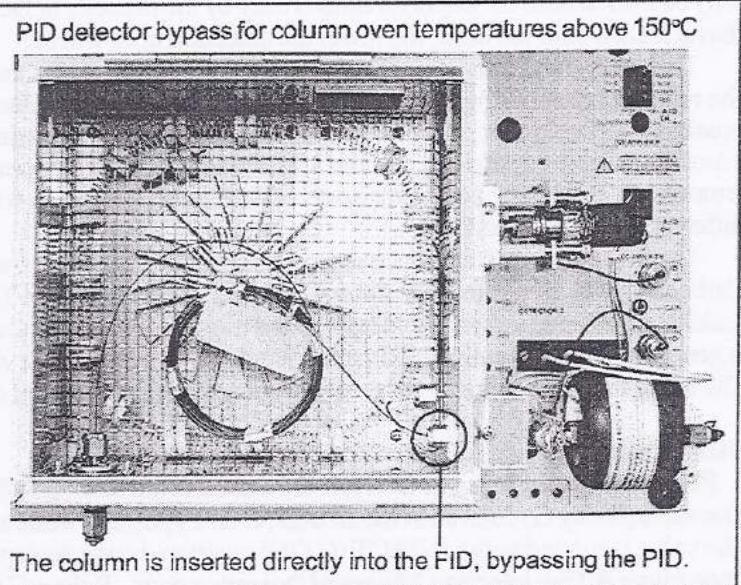
*General Operating Procedures continued*

**Direct Injection**

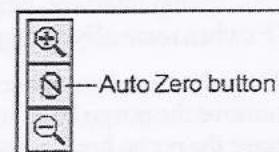
This technique is useful for volatile and semi-volatile compounds, but is typically used for diesel and other compounds that don't purge well from aqueous or soil samples.

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**

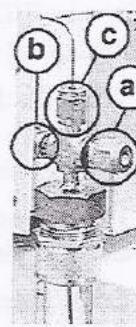
This TO-14 type technique injects a gas or air sample using either a large syringe (60mL) or a Tedlar bag (1L). A vacuum pump may be used to pull the sample through the sorbent traps. The amount of sample that may be loaded onto the trap(s) is limited only by the capacity of the trap's adsorbent packings.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

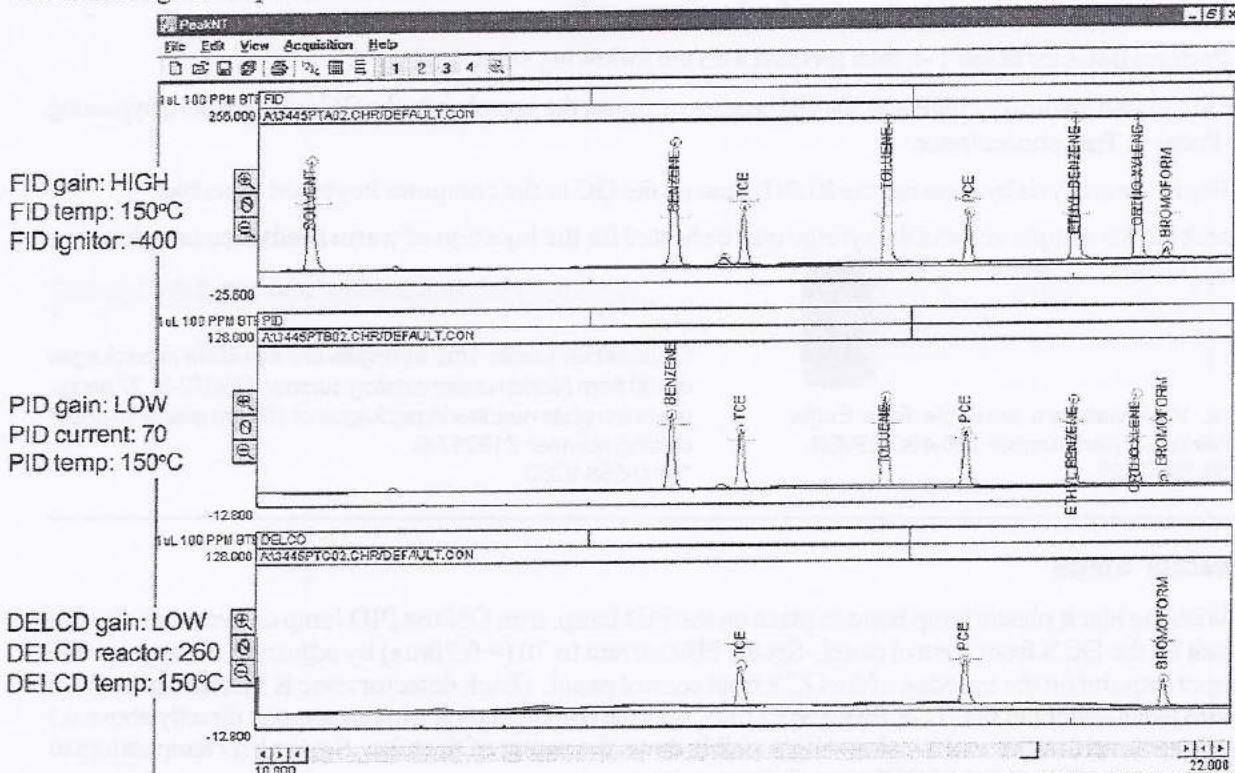
1. With the black plastic lamp hood in place on the PID lamp, turn ON the PID lamp current with the flip switch on the GC's front control panel. Set the PID current to 70 (=0.70ma) by adjusting the appropriate trimpot setpoint on the top edge of the GC's front control panel. (Each detector zone is labeled 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

Events (5030.evt):	
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:  
(Epap&t.tem)

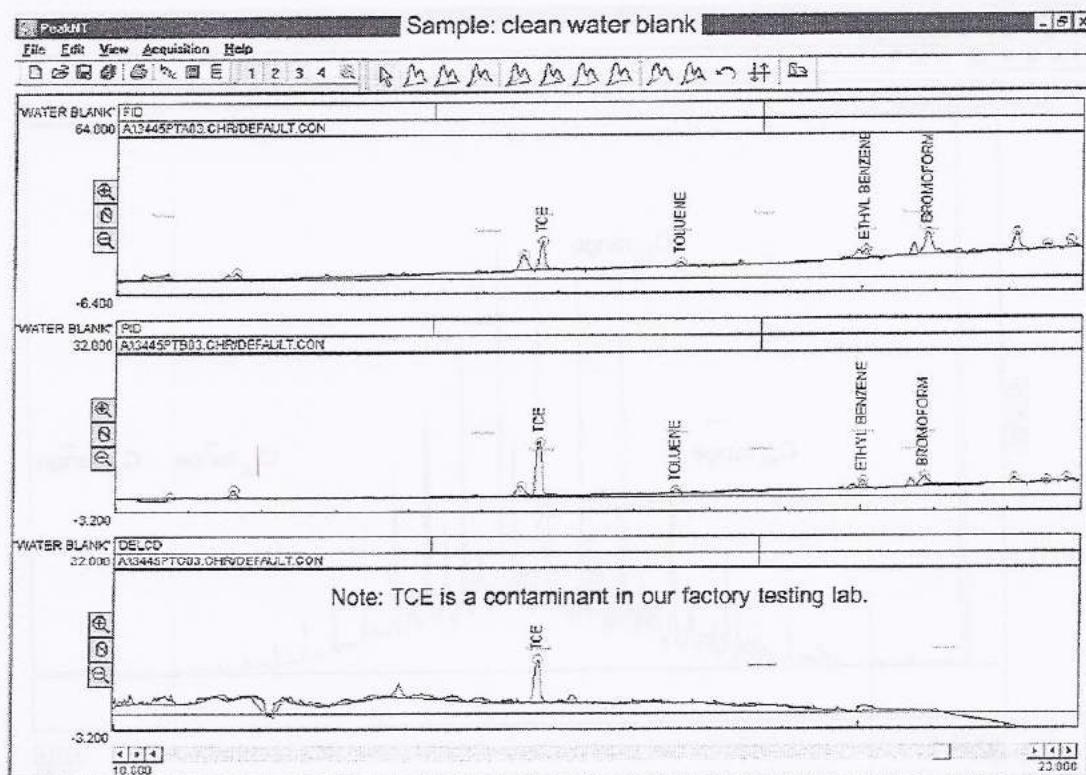
Initial Hold Ramp Final  
40°C 10.00 10.00 180°C

## 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:

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

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

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

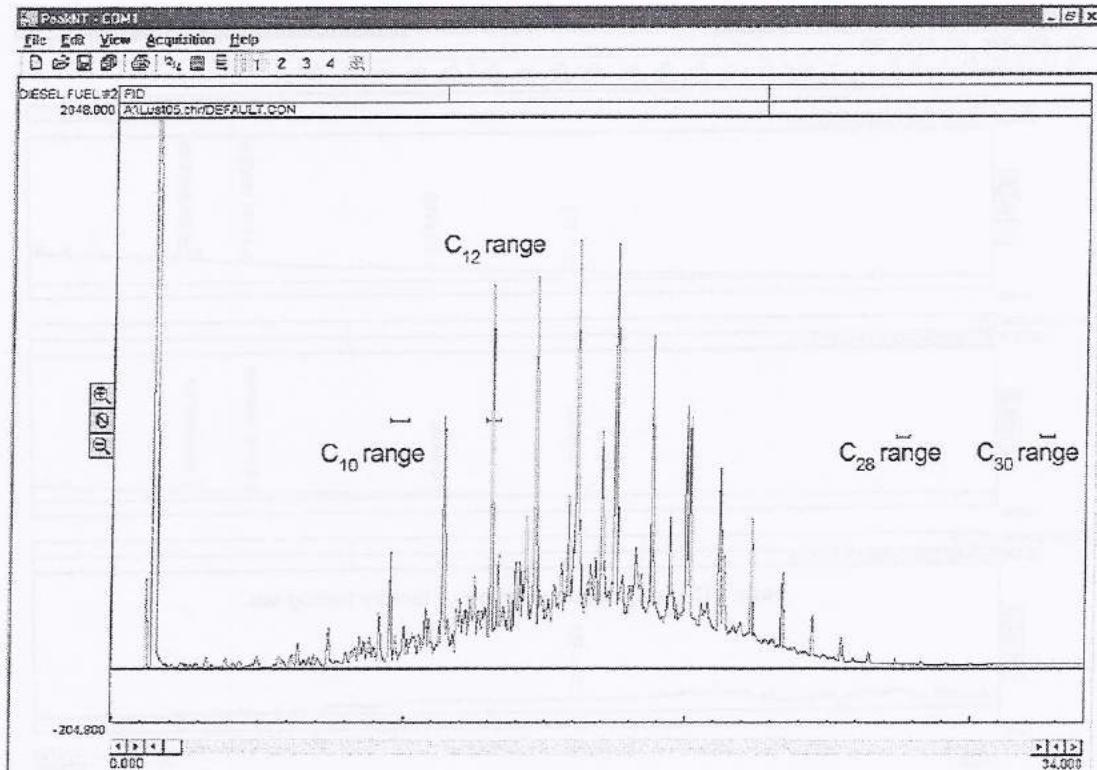
(x represents the ppb concentration of the carryover)

## 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<sub>10</sub>, C<sub>12</sub>, C<sub>28</sub>, and C<sub>30</sub>.



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

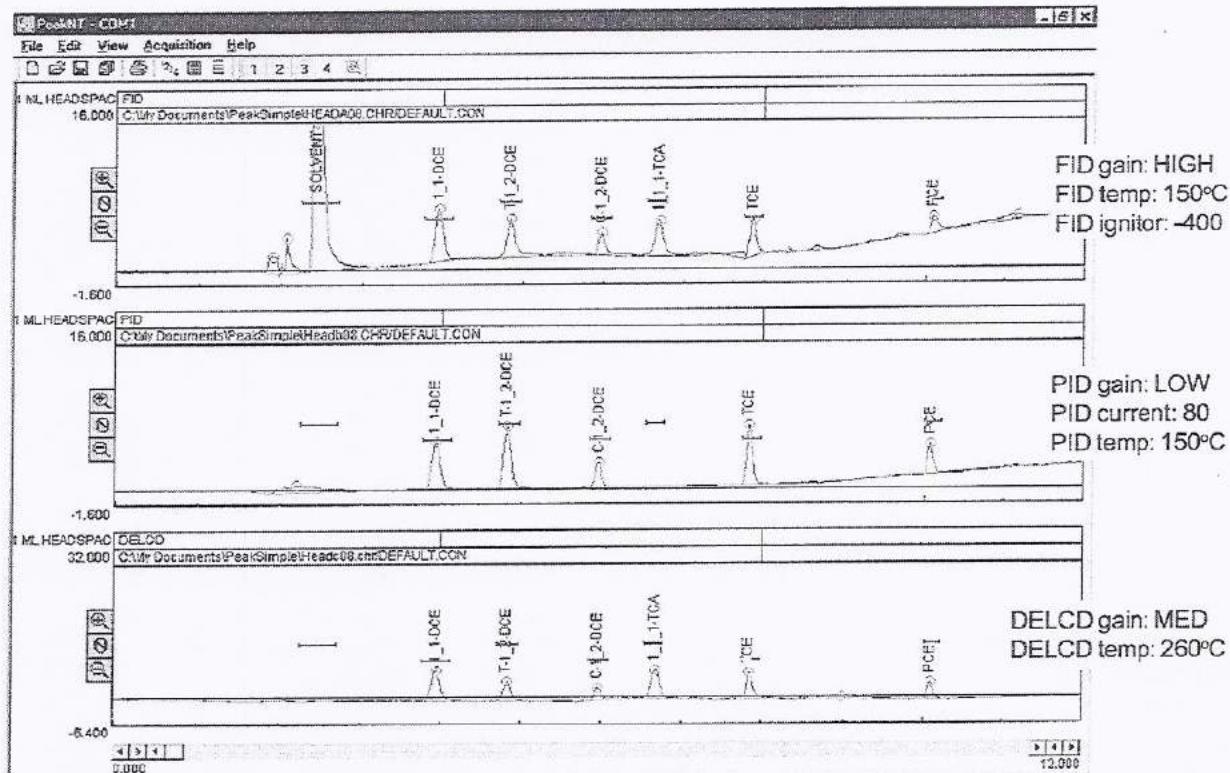
FID gain: HIGH  
FID temp: 325°C  
FID ignitor: -400

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	0.603	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:**

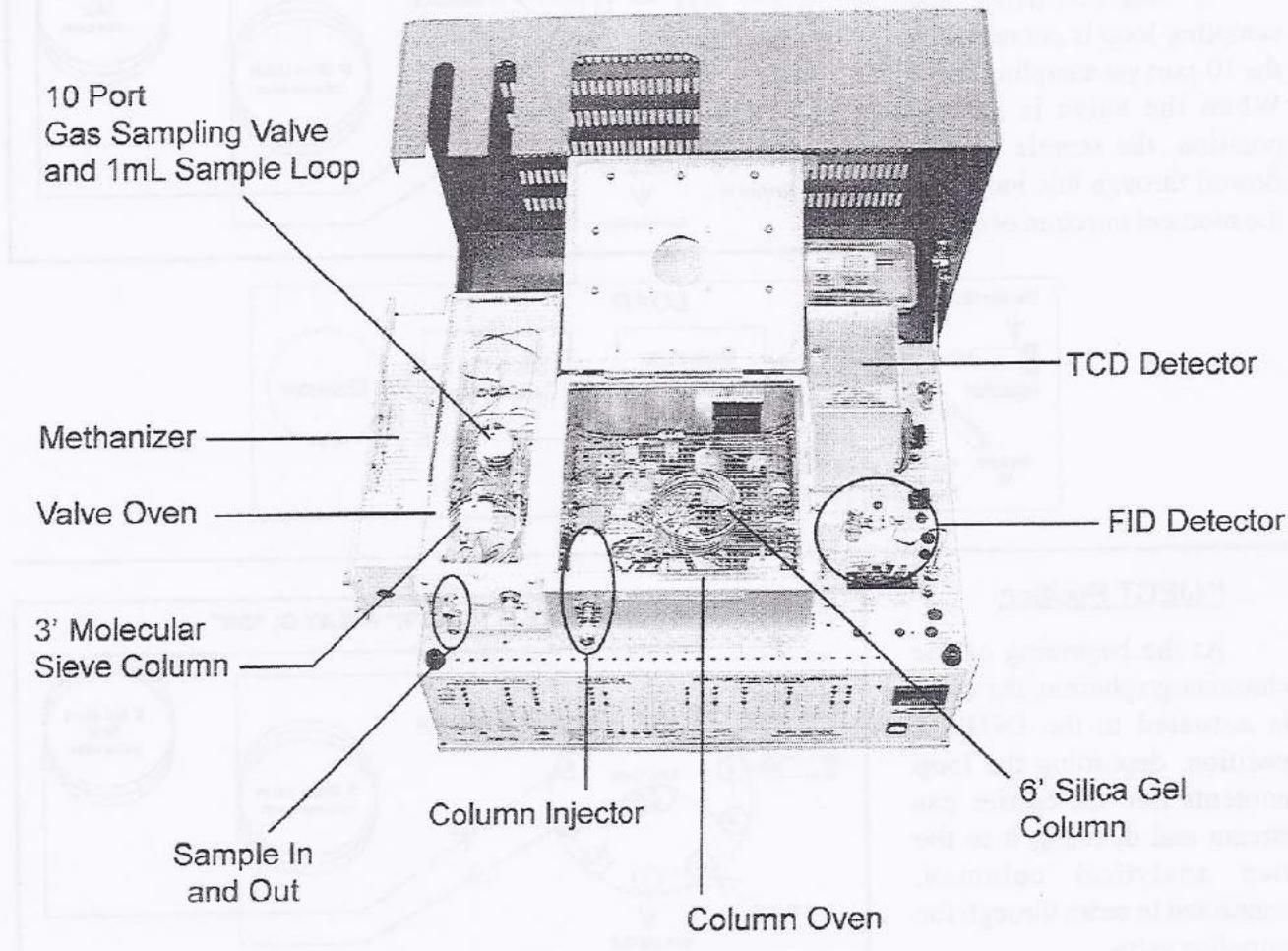
Component	Retention	Area
1_1-DCE	3.933	63.1790
T-1_2-DCE	4.816	38.0780
C-1_2-DCE	5.950	18.0560
1_1_1-TCA	6.666	53.2210
TCE	7.833	39.6900
PCE	10.083	20.8340
Total		233.0580

## POPULAR CONFIGURATION GCs

### Multiple Gas Analyzer #1

#### System Overview

Your SRI Multiple Gas Analyzer GC is pre-plumbed and ready to resolve H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, Methane, CO, Ethane, CO<sub>2</sub>, Ethylene, NOx, Acetylene, Propane, Butanes, Pentanes, and C<sub>6</sub> through C<sub>8</sub>. The basic version of the Multiple Gas Analyzer GC has a TCD detector only. A TCD-HID detector combination is also available. A third version, shown below, has a TCD, Methanizer, and FID.



The Multiple Gas Analyzer #1 configuration allows you to obtain complete analyses of the fixed and natural gases listed above with a single sample injection. This is achieved using a 10 port gas sampling valve, a 1mL sample loop, and two independent analytical columns--a Silica Gel packed column and a Molecular Sieve packed column. The Silica Gel column is located in the Column Oven, while the Molecular Sieve column, 1mL sample loop, and the gas sampling valve are located in the Valve Oven.

# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #1

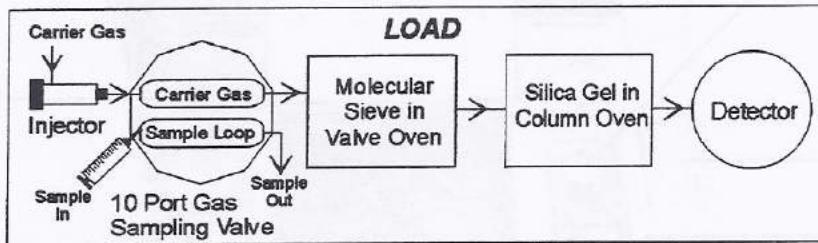
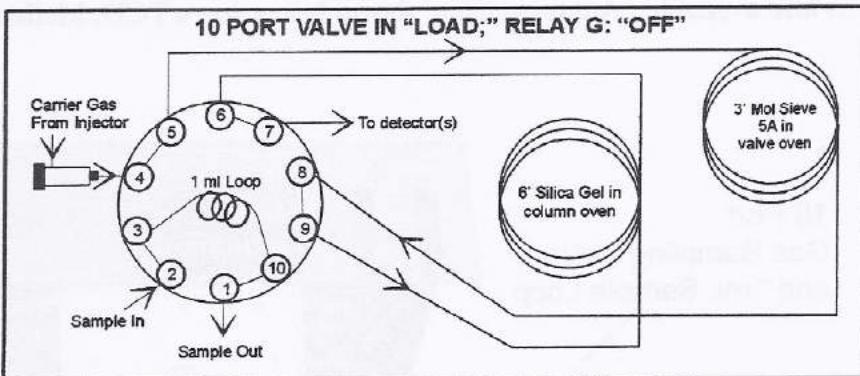
### Theory of Operation

#### 10 Port Gas Sampling Valve Plumbing Connections

The Multiple Gas Analyzer #1 configuration uses two analytical columns and one 10-port gas sampling valve to analyze hydrogen, oxygen, nitrogen, methane, ethane, propane, butanes, pentanes, carbon monoxide and carbon dioxide.

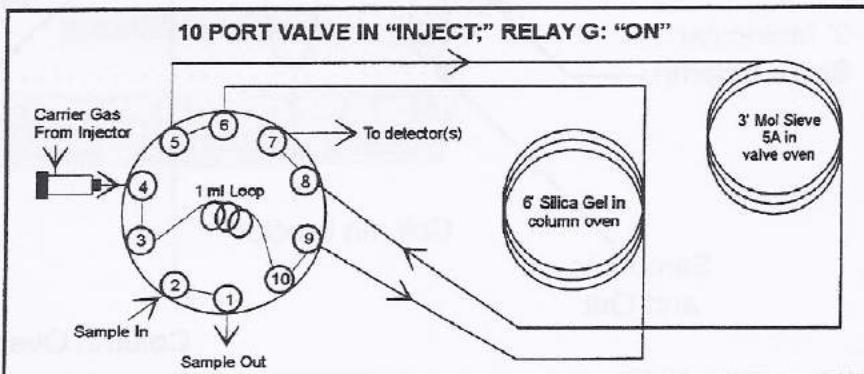
#### LOAD Position

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

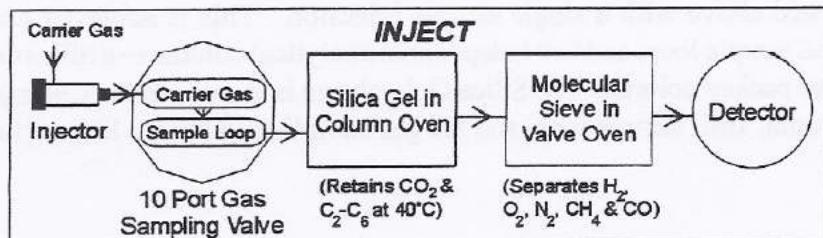


#### INJECT Position

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



*\*\*Column sequence is reversed while the flow direction remains the same\*\**



# POPULAR CONFIGURATION GCs

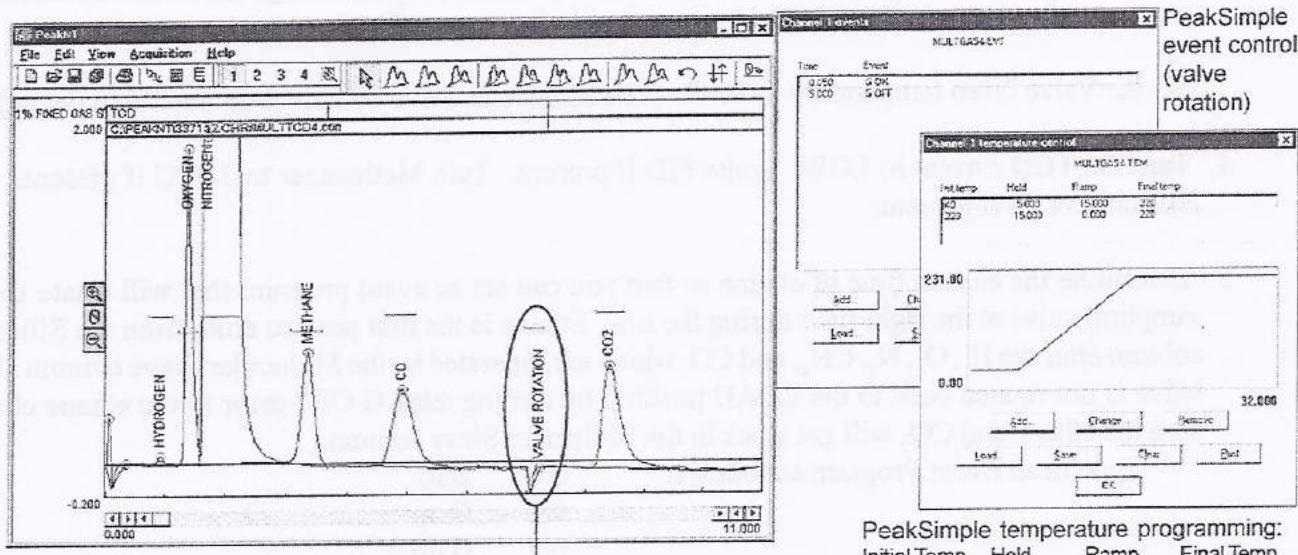
## Multiple Gas Analyzer #1

### Theory of Operation

#### 10 Port Gas Sampling Valve Plumbing Connections Continued

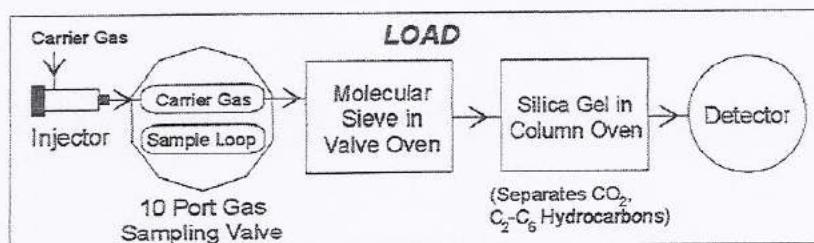
The sample is deposited first into the Silica Gel packed column, at 40°C in the column oven, where the ethane, propane, butanes, pentanes and carbon dioxide are retained. The remainder of the sample containing hydrogen or helium (whichever is not being used as a carrier), as well as oxygen, nitrogen, methane and any carbon monoxide, continues on to the Molecular Sieve column. During a chromatographic run with the sampling valve in the INJECT position, the hydrogen or helium, oxygen, nitrogen and methane components are the first to elute through the columns and into the detector. This is due to the Silica Gel column's long retention time at 40°C of C<sub>2</sub>, CO<sub>2</sub> and higher hydrocarbons. Under programmed temperature and event control using the data system, the sampling valve is actuated back into the LOAD position immediately following the elution of the carbon monoxide peak.

#### Multiple Gas Analyzer TCD chromatogram with temperature programming and sample valve rotation



PeakSimple temperature programming:  
Initial Temp Hold Ramp Final Temp  
40°C 5.00 15.00 220°C  
220°C 15.00 0.00 220°C

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.



## **POPULAR CONFIGURATION GCs**

### **Multiple Gas Analyzer #1**

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#### **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 side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.
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 energizing the TCD. Labelled for identification, the carrier gas outlet is located inside the Column Oven. Place the end of the tube in liquid and observe (a bit of 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.
3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)
4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.
5. Determine the elution time of ethane so that you can set an event program that will rotate the gas sampling valve at the right time during the run. Ethane is the first peak to elute from the Silica Gel column after the H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, and CO, which are separated by the Molecular Sieve column. If the valve is not rotated back to the LOAD position by turning relay G OFF prior to the ethane elution, then the ethane and CO<sub>2</sub> will get stuck in the Molecular Sieve column.

Type in an Event Program as follows:

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 and then immediately reverse the columns so the sample will not enter the Molecular Sieve column. In this mode of operation, the elution time of ethane can be easily determined.

6. Set the Column Oven temperature program as follows:  
40°C hold 6 minutes then ramp at 10°/min to 200°C

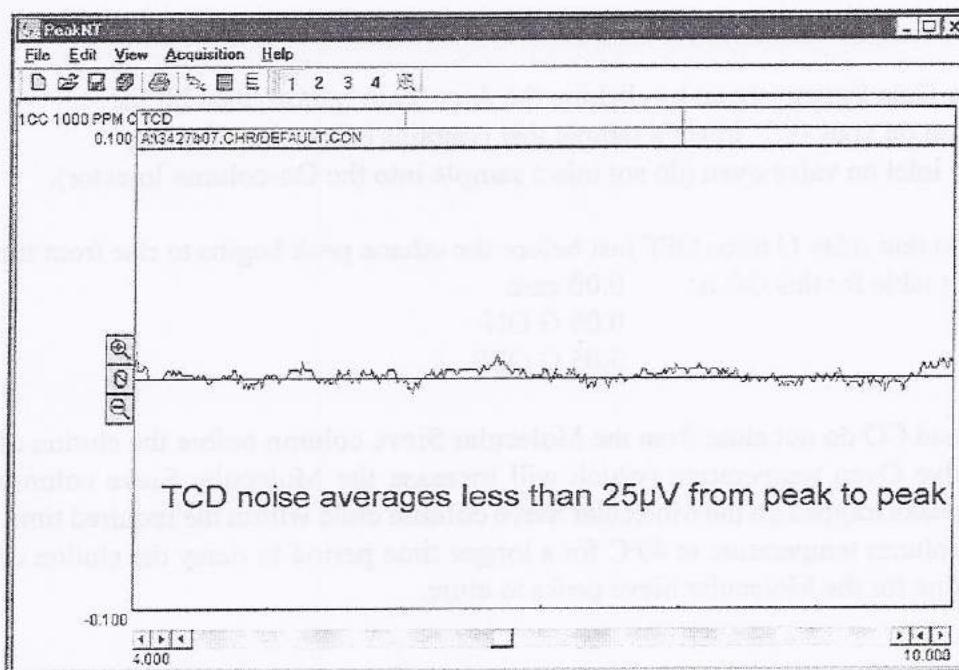
***General Operating Procedure Continued***

7. In PeakSimple, zero the Data System signal by clicking the Auto Zero button, then hit the spacebar or the run button on your GC. Inject a sample that contains ethane into the Gas Sampling Valve through inlet on valve oven (do not inject sample into the On-column Injector).
8. Revise the event table so that relay G turns OFF just before the ethane peak begins to rise from the baseline. A typical event table for this GC is:  
0.00 zero  
0.05 G ON  
5.05 G OFF
9. If the H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, and CO do not elute from the Molecular Sieve column before the elution of ethane, increase the Valve Oven temperature (which will increase the Molecular Sieve column temperature) so that all peaks trapped on the Molecular Sieve column elute within the required time. Or, hold the Silica Gel column temperature at 40°C for a longer time period to delay the elution of ethane, allowing more time for the Molecular Sieve peaks to elute.

# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #1

### Expected Performance



#### TCD noise run

Columns: 1m Mol. Sieve,  
2m Hayesep-D,  
30m MXT-1  
Carrier: Helium @ 10mL/min  
TCD gain = LOW  
TCD temp = 100°C  
Valve temp = 110°C

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

### Factory Test Analysis of Natural Gas Standard

#### Temperature program:

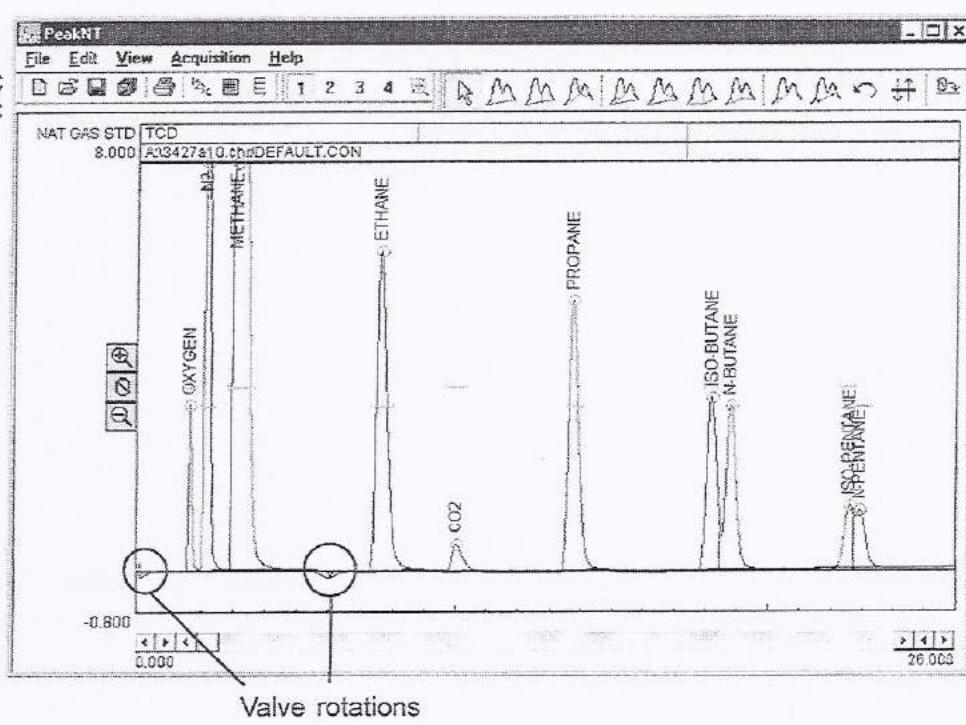
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

#### Events:

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

#### 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



# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #1

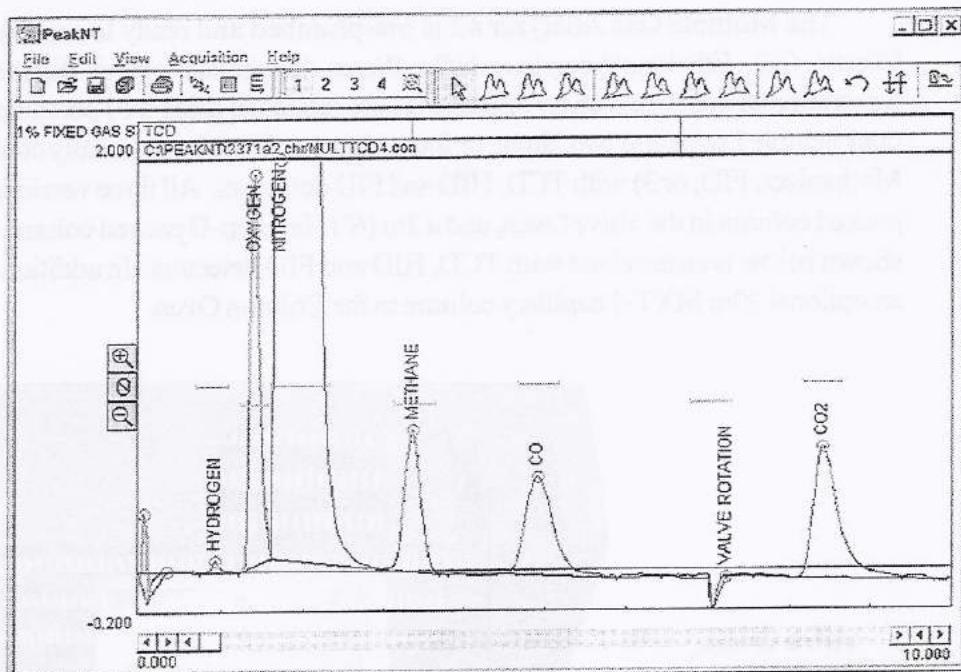
### Expected Performance

#### Factory Test Analysis of 1% Fixed Gas Standard on a TCD Multiple Gas Analyzer #1

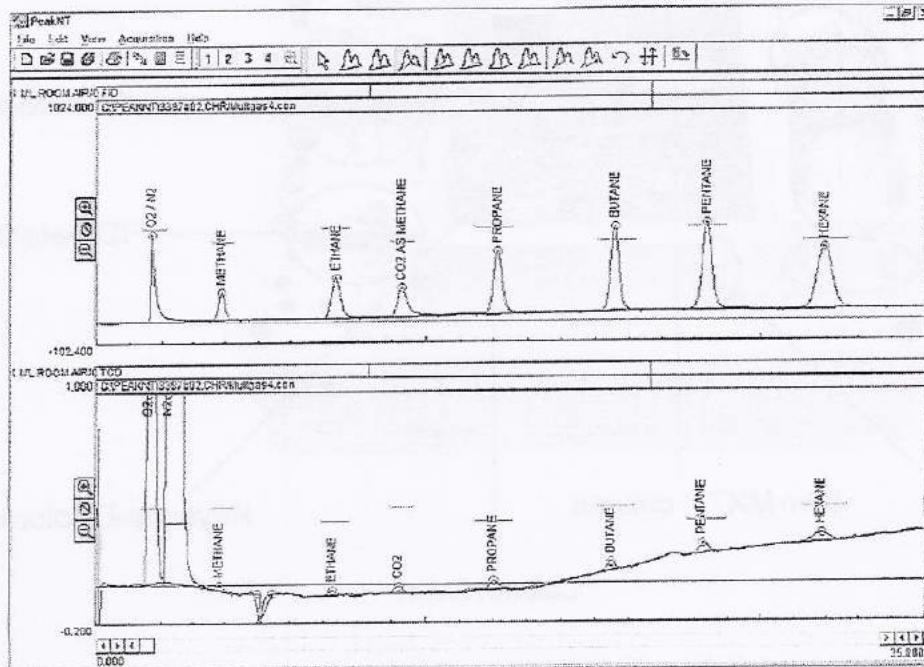
TCD current = LOW  
 TCD temp = 100°C  
 Valve Oven temp = 90°C

##### RESULTS:

Component	Retention	Area
Hydrogen	0.916	0.4710
Oxygen	1.383	14.9530
Nitrogen	1.700	1049.4940
Methane	3.333	9.4875
CO	4.900	11.4460
Valve Rotate	7.200	0.6460
CO <sub>2</sub>	8.383	13.8000
total		1100.2775



#### Factory Test Analysis of Room Air & C<sub>1</sub>-C<sub>6</sub> Hydrocarbons on a dual-channel TCD-Methanizer-FID Multiple Gas Analyzer #1



##### FID Results:

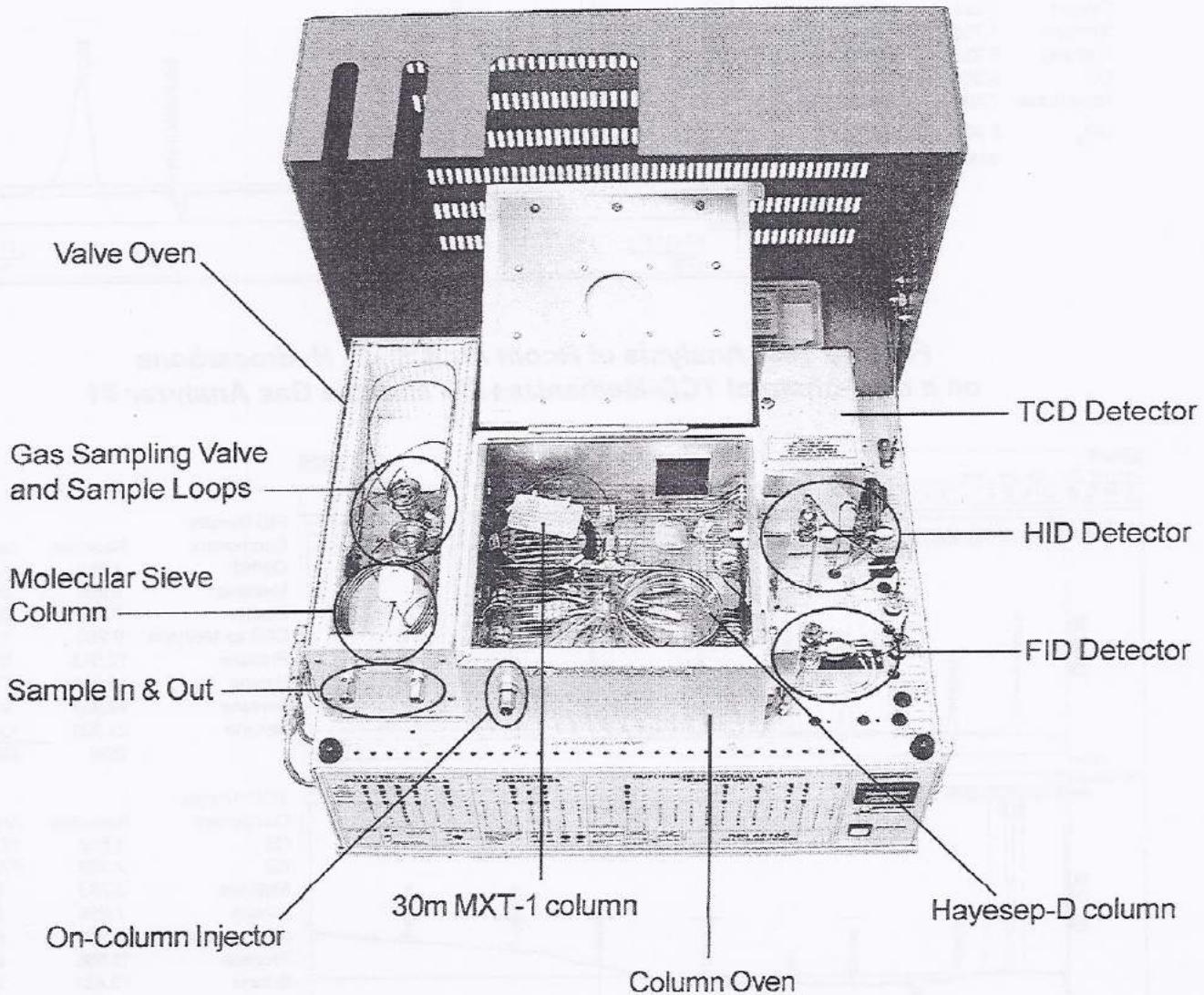
Component	Retention	Area
O2/N2	1.650	4731.2140
Methane	3.866	2008.6000
Ethane	7.316	3854.7300
CO2 as Methane	9.250	3142.1040
Propane	12.083	5379.8755
Butane	15.533	7326.4440
Pentane	18.333	9136.3340
Hexane	21.900	10408.3160
Total		45987.6175

##### TCD Results:

Component	Retention	Area
O2	1.566	181.0650
N2	2.166	675.1440
Methane	3.783	0.3880
Ethane	7.216	0.4670
CO2 as Methane	9.150	0.6780
Propane	11.966	0.8210
Butane	15.433	1.1210
Pentane	18.200	1.2710
Hexane	21.816	1.6480
Total		862.8130

## System Overview

The Multiple Gas Analyzer #2 is pre-plumbed and ready to resolve H<sub>2</sub>, He, O<sub>2</sub>, N<sub>2</sub>, Methane, CO, Ethane, CO<sub>2</sub>, Ethylene/Acetylene, NOx, Water, Alcohols, Propane, Butanes, Pentanes, and C<sub>6</sub> through C<sub>20</sub>. Separation of this wide variety of peaks is accomplished using a 10 port automated Gas Sampling Valve with dual Sample Loops and two, three, or four columns. It can be optionally configured with 1) a TCD, 2) a TCD, Methanizer, FID, or 3) with TCD, HID and FID detectors. All three versions have a 1m (3') Molecular Sieve packed column in the Valve Oven, and a 2m (6') Hayesep-D packed column in the Column Oven. The model shown below is customized with TCD, HID and FID detectors. In addition to the Hayesep-D column, it has an optional 30m MXT-1 capillary column in the Column Oven.



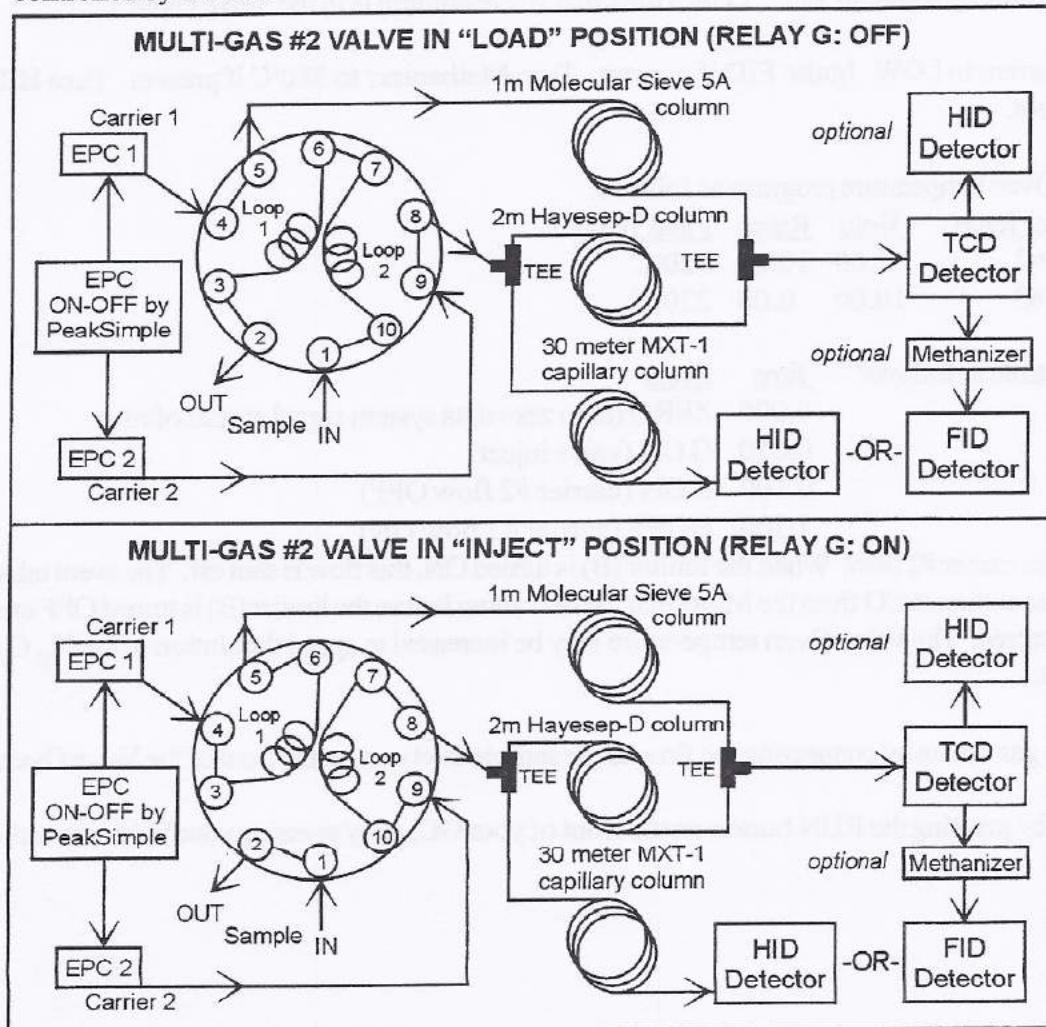
# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #2

### Theory of Operation

The Multiple Gas Analyzer #2 GC uses a single automated 10 port Gas Sampling Valve and multiple columns to separate a wide variety of peaks. The system achieves this by turning the carrier gas flow to each column on at different times during the run. This procedure allows the Molecular Sieve column in the Valve Oven to completely separate H<sub>2</sub>, He, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> and CO before the carrier flow to the Hayesep-D column in the Column Oven is turned on. The Hayesep-D column then separates all compounds in the C<sub>1</sub>-C<sub>6</sub> range. An optional 30m MXT-1 capillary column in the Column Oven separates the remaining hydrocarbons out through C<sub>20</sub>, using the same carrier gas flow as the Hayesep-D column and an FID or HID detector.

This configuration uses two carrier gas flows, each regulated by Electronic Pressure Control (EPC) using the PeakSimple data system. Carrier 1 flows to the Molecular Sieve column, then on through the "Tee" to the TCD detector, and it is always on; if not, the lack of carrier gas flow triggers the TCD filament protection circuit. Carrier 2 flows to another "Tee" where it splits to enter the Hayesep-D column and also the MXT-1 column. The flow from the Hayesep-D column continues to the TCD detector, and the flow from the MXT-1 goes to the FID or HID detector. The carrier #2 flow (EPC 2) is turned on and off by PeakSimple, controlled by the user.



When the 10 port Gas Sampling Valve is in LOAD position, the two carrier gas flows bypass the Sample Loops through the Valve and travel on to the columns.

When the 10 port Gas Sampling Valve is in INJECT position, the two carrier gas flows sweep through the Sample Loops, sending their contents to the columns and detectors.

## 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 side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.
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 energizing the TCD. Labelled for identification, the carrier gas outlet is located inside the Column Oven. Place the end of the tube in liquid and observe (a bit of 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.
3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)
4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.
5. Set the Column Oven temperature program as follows:

<u>Initial Temp</u>	<u>Hold</u>	<u>Ramp</u>	<u>Final Temp</u>
50°C	7.00	10.00	220°C
220°C	10.00	0.00	220°C
6. Type in an event table as follows:

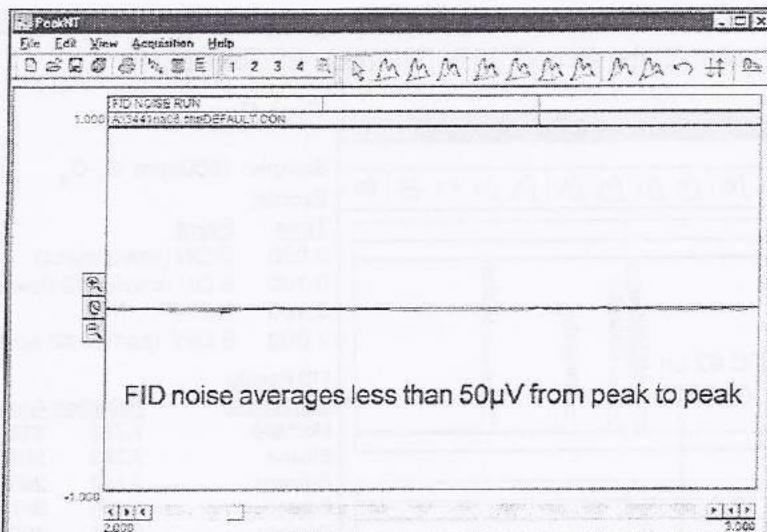
<u>Time</u>	<u>Event</u>
0.000	ZERO (auto zero data system signal at start of run)
0.050	G ON (valve inject)
0.100	B ON (carrier #2 flow OFF)
7.500	B OFF (carrier #2 flow ON)

EPC #2 controls the carrier #2 flow. When the limiter (B) is turned ON, this flow is shut off. The event table should allow for the elution of CO from the Molecular Sieve column before the limiter (B) is turned OFF and carrier #2 flow restored. The Valve Oven temperature may be increased to speed the elution of the H<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>, and CO.
7. Load your sample gas stream by connecting the flow to the sample inlet port on the front of the Valve Oven.
8. Start the analysis by pressing the RUN button on the front of your GC, or by pressing your PC keyboard's spacebar.

## POPULAR CONFIGURATION GCs

### Multiple Gas Analyzer #2

#### Expected Performance



#### FID noise run

Columns: 1m Mol. Sieve, 2m Hayesep-D,  
30m MXT-1

Carrier: Helium @ 10mL/min

FID gain = HIGH

FID temp = 150°C

FID ignitor = -400

Valve temp = 110°C

Temperature Program:

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

#### HID noise run

Columns: 1m Mol. Sieve, 2m Hayesep-D,  
30m MXT-1

Carrier: Helium @ 10mL/min

HID gain = HIGH

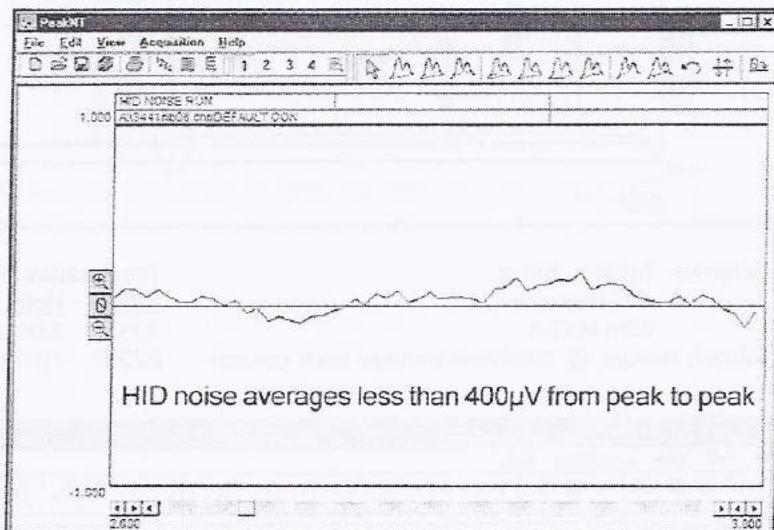
HID current = 70

HID temp = 200°C

Valve temp = 110°C

Temperature Program:

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



#### TCD noise run

Columns: 1m Mol. Sieve, 2m Hayesep-D,  
30m MXT-1

Carrier: Helium @ 10mL/min

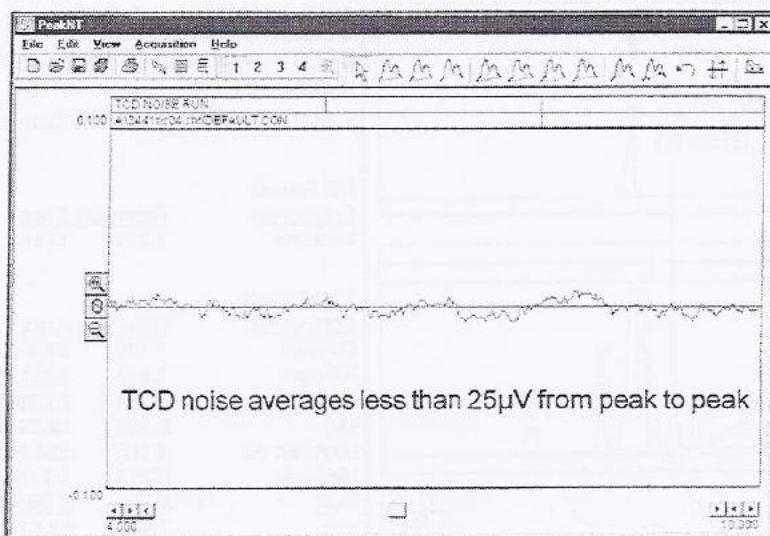
TCD gain = LOW

TCD temp = 100°C

Valve temp = 110°C

Temperature Program:

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

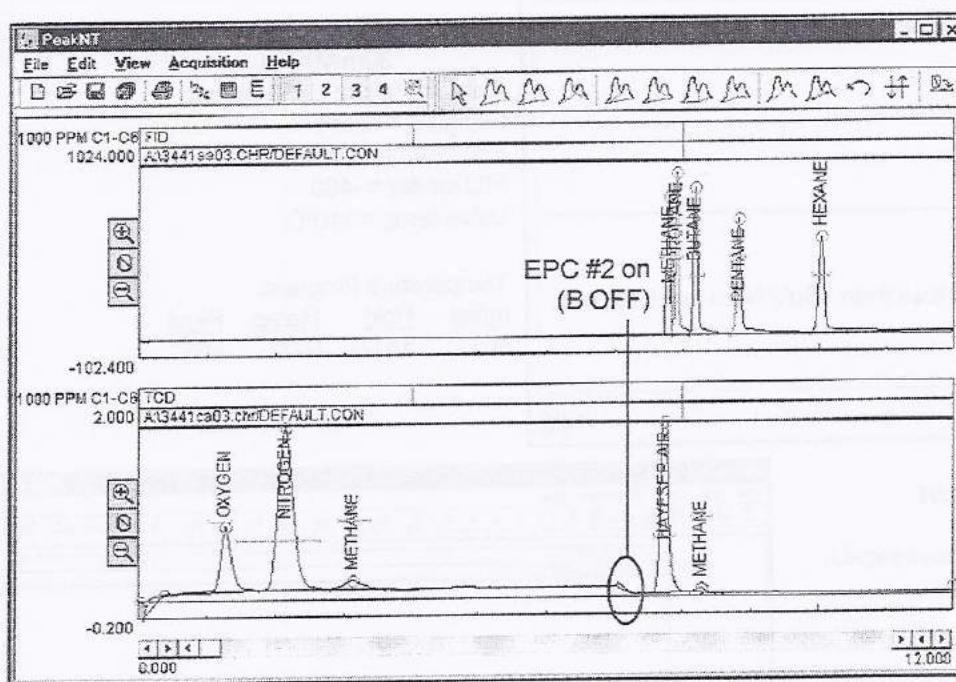


# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #2

### Expected Performance: FID & TCD Detectors

These two factory test runs utilized the same carrier flow and temperature program. The first chromatogram resulted from a run with a 1000ppm C<sub>1</sub>-C<sub>6</sub> sample; the second, a 1% fixed gas standard sample.



Columns: 1m Mol. Sieve,  
2m Hayesep-D,  
30m MXT-1  
Carrier: Helium @ 10mL/min through each column

Temperature Program:  

Initial	Hold	Ramp	Final
50°C	7.00	10.00	220°C
220°C	10.00	0.00	220°C

#### Test Run #1

Sample: 1000ppm C<sub>1</sub>-C<sub>6</sub>

Events:

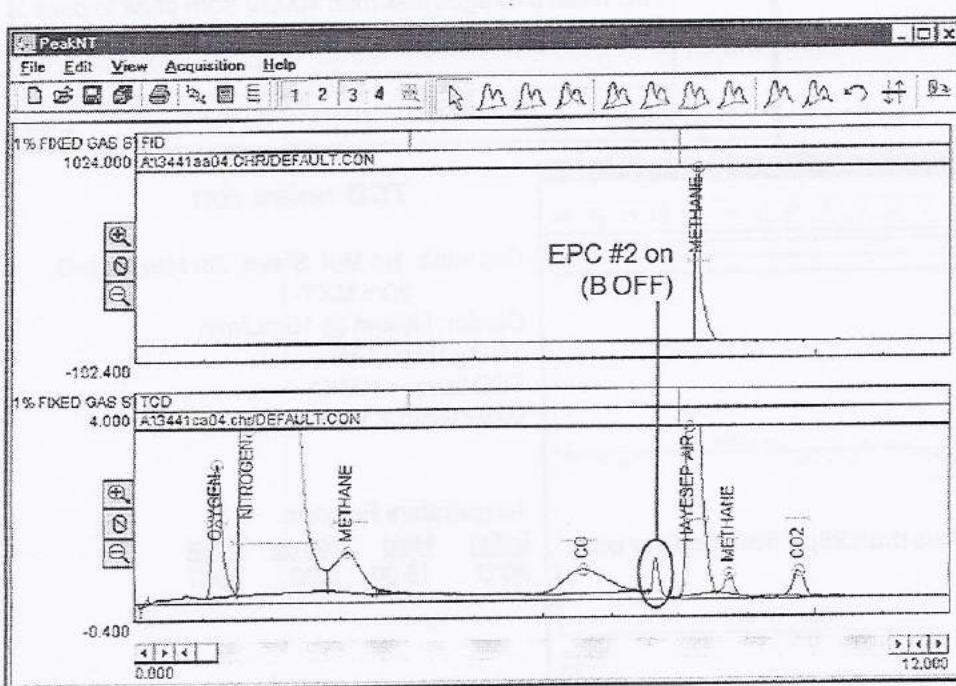
Time	Event
0.050	G ON (valve inject)
0.100	B ON (carrier #2 flow off)
0.400	G OFF
7.000	B OFF (carrier #2 flow on)

FID Results:

Component	Retention Area
Methane	7.733 838.3160
Ethane	7.783 2066.2065
Propane	7.883 2953.3865
Butane	8.166 3479.4540
Pentane	8.800 4021.5110
Hexane	10.016 3512.6800
Total	16871.5540

TCD Results:

Component	Retention Area
Oxygen	1.250 7.9800
Nitrogen	2.116 27.9765
Methane	3.116 2.0210
Hayesep Air	7.716 23.5150
Methane	8.250 0.4950
Ethane	12.133 1.0240
Total	63.0115



#### Test Run #2

Sample: 1% fixed gas standard

Events:

Time	Event
0.050	G ON (valve inject)
0.100	B ON (carrier #2 flow off)
0.400	G OFF
7.500	B OFF (carrier #2 flow on)

FID Results:

Component	Retention Area
Methane	8.233 12144.3770

TCD Results:

Component	Retention Area
Oxygen	1.166 26.4920
Nitrogen	1.633 2251.7140
Methane	3.083 23.0975
CO	6.566 22.2440
Hayesep Air	8.116 524.2010
Methane	8.716 3.7730
CO <sub>2</sub>	9.750 6.3940
Total	63.0115

# POPULAR CONFIGURATION GCs

## Multiple Gas Analyzer #2

### Expected Performance: HID & TCD Detectors

These two factory test runs utilized the same carrier flow, temperature program, and event table. The first chromatogram resulted from a run with a 1000ppm C<sub>1</sub>-C<sub>6</sub> sample; the second, a 1% fixed gas standard sample.

#### Test Run #1

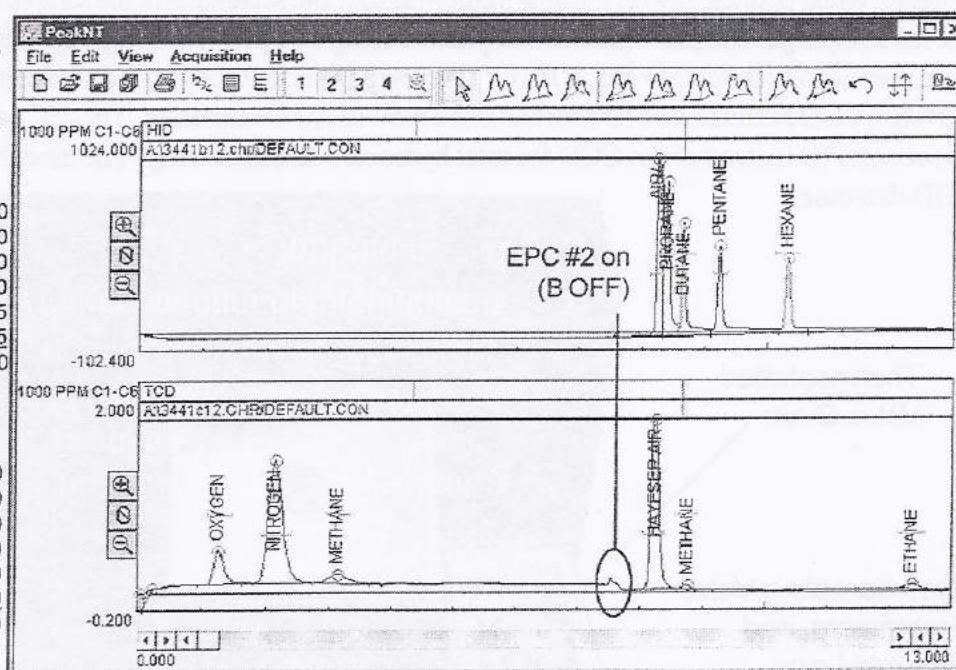
Sample: 1000ppm C<sub>1</sub>-C<sub>6</sub>

##### HID Results:

Component	Retention	Area
Air/Methane	8.233	6249.3320
Ethane	8.283	3064.2580
Propane	8.383	3408.5720
Butane	8.650	2265.3520
Pentane	9.216	2650.8955
Hexane	10.316	2260.8975
Total		19899.3070

##### TCD Results:

Component	Retention	Area
Oxygen	1.250	4.0220
Nitrogen	2.116	21.0510
Methane	3.116	2.1900
Hayesep Air	8.216	22.4900
Methane	8.733	0.4460
Ethane	12.333	0.9640
Total		51.1630



Columns: 1m Mol. Sieve,  
2m Hayesep-D,

30m MXT-1

Carrier: Helium @ 10mL/min through  
each column

##### Temperature Program:

Initial	Hold	Ramp	Final
50°C	7.00	10.00	220°C
220°C	10.00	0.00	220°C

##### Events:

Time	Event
0.050	G ON (valve inject)
0.100	B ON (carrier #2 flow off)
0.400	G OFF
7.500	B OFF (carrier #2 flow on)

#### Test Run #2

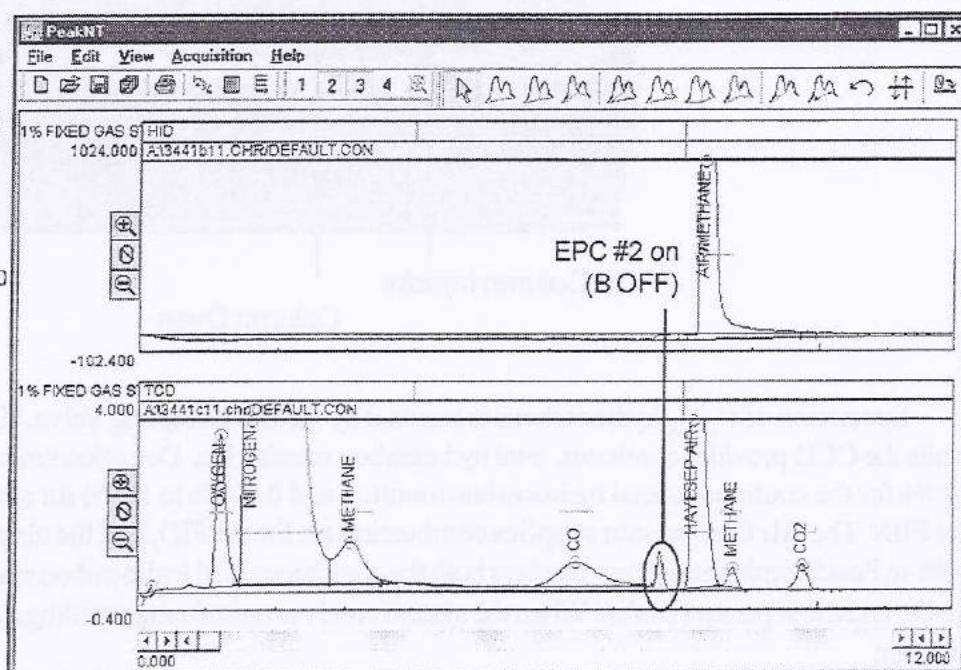
Sample: 1% fixed gas standard

##### FID Results:

Component	Retention	Area
Methane	8.266	44548.0540

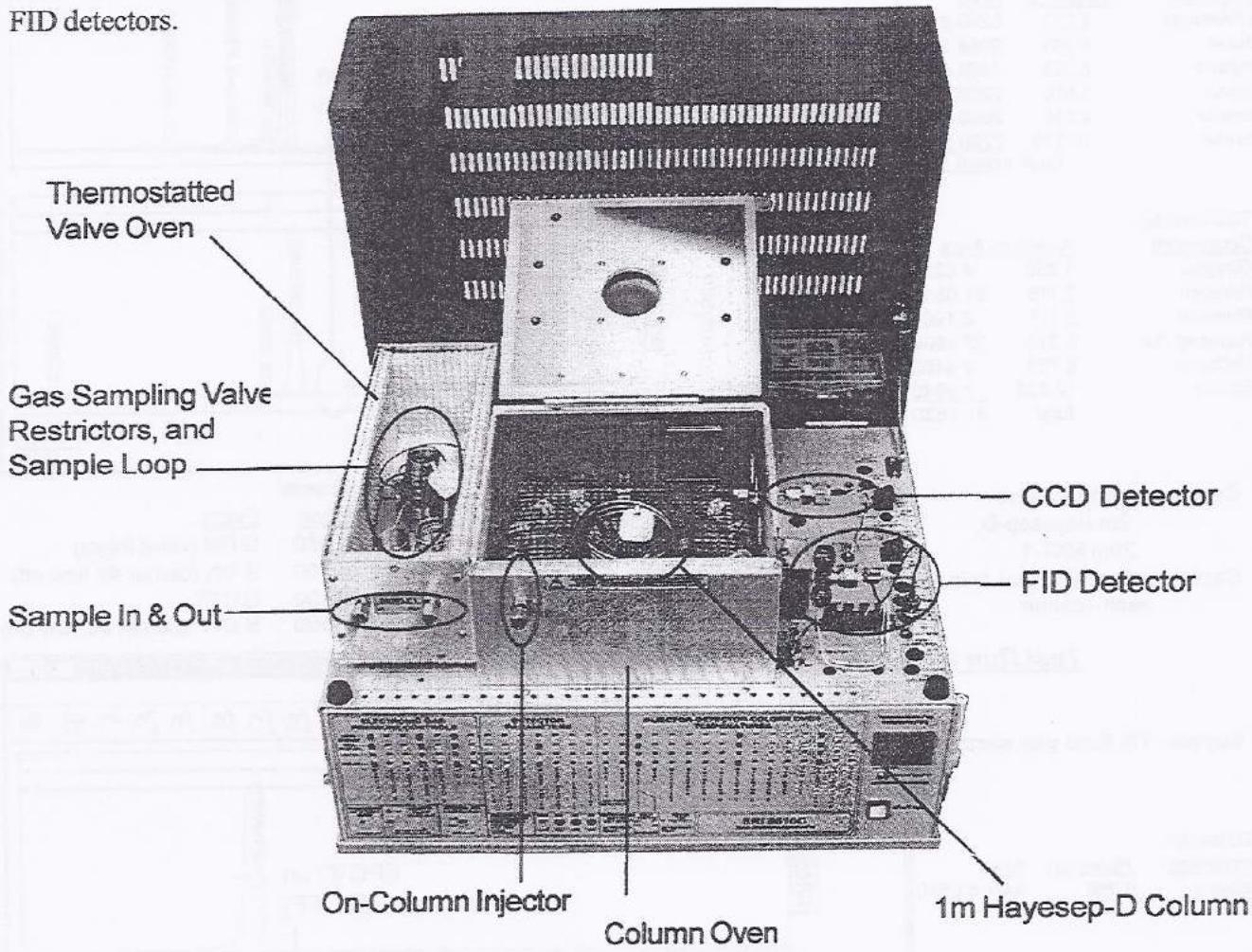
##### TCD Results:

Component	Retention	Area
Oxygen	1.166	31.0260
Nitrogen	1.616	2261.6430
Methane	3.050	12.6240
CO	6.400	22.4410
Hayesep Air	8.116	542.6790
Methane	8.716	3.5950
CO <sub>2</sub>	9.750	6.6920
Total		2880.7000



## 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 $\mu$ 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.



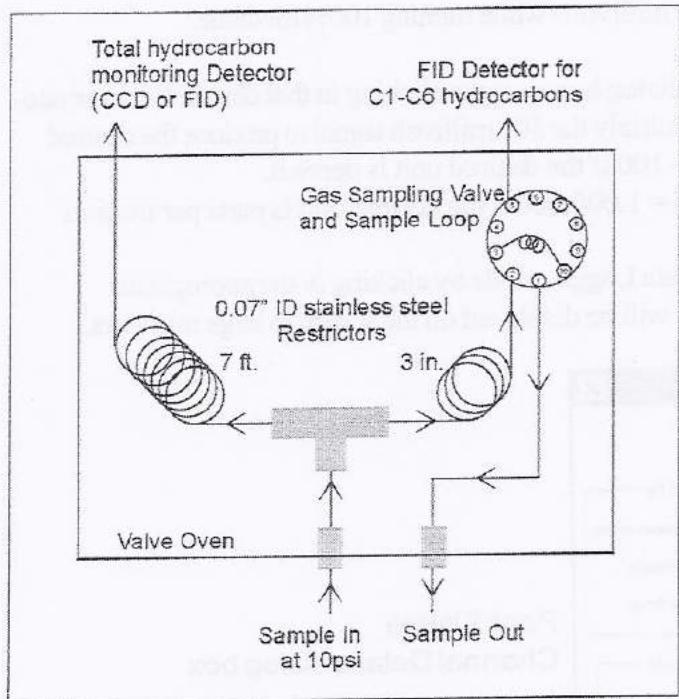
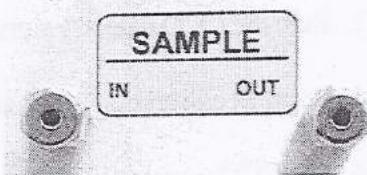
Speciation of C<sub>1</sub>-C<sub>6</sub> hydrocarbons is handled by the Gas Sampling Valve, Hayesep-D column, and FID while the CCD provides continuous, total hydrocarbon monitoring. Detection limits for this system are 0.1% to 100% for the continuous total hydrocarbon monitor, and 0.005% to 100% for 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

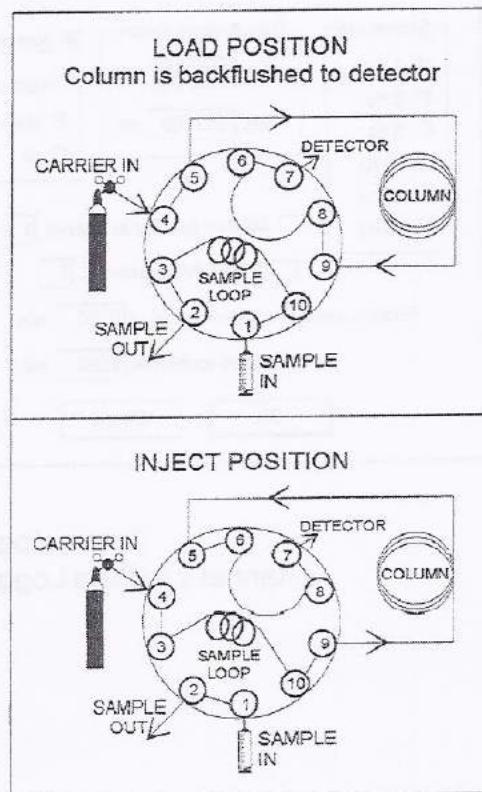
The sample gas stream is connected to a bulkhead fitting on the system's thermostatted Valve Oven where it flows through the sampling loop of the 10 port Gas Sampling Valve, and also to the CCD detector. The fitting labelled "Sample In" (pictured at right) on the front of the Valve Oven is the sample 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.



10 Port Gas Sampling Valve

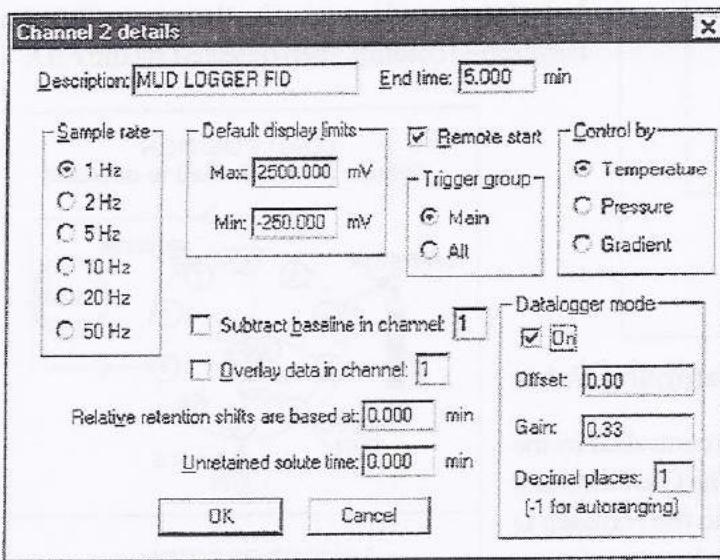
At an automatically repeating time interval controlled by the user with the built-in PeakSimple data system, the Gas Sampling Valve injects the contents of its sample loop into the Hayesep D packed column where it is separated into the constituent hydrocarbon ( $C_1$ - $C_6$ ) peaks and detected by the 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 $\mu$ 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.

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 to 5mL/min 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.



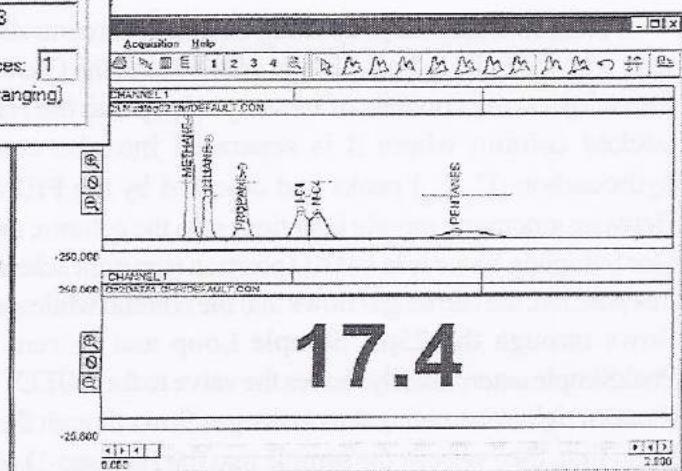
### General Operating Procedure Part 1: Total Hydrocarbons Using the CCD Detector

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.



Chromatogram with  
channel 2 in Data Logger mode

PeakSimple  
Channel Details dialog box



## 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:

<u>Initial Temp</u>	<u>Hold</u>	<u>Ramp</u>	<u>Final Temp</u>
200°C	5.00	0.00	200°C

5. Type in an even table as follows:

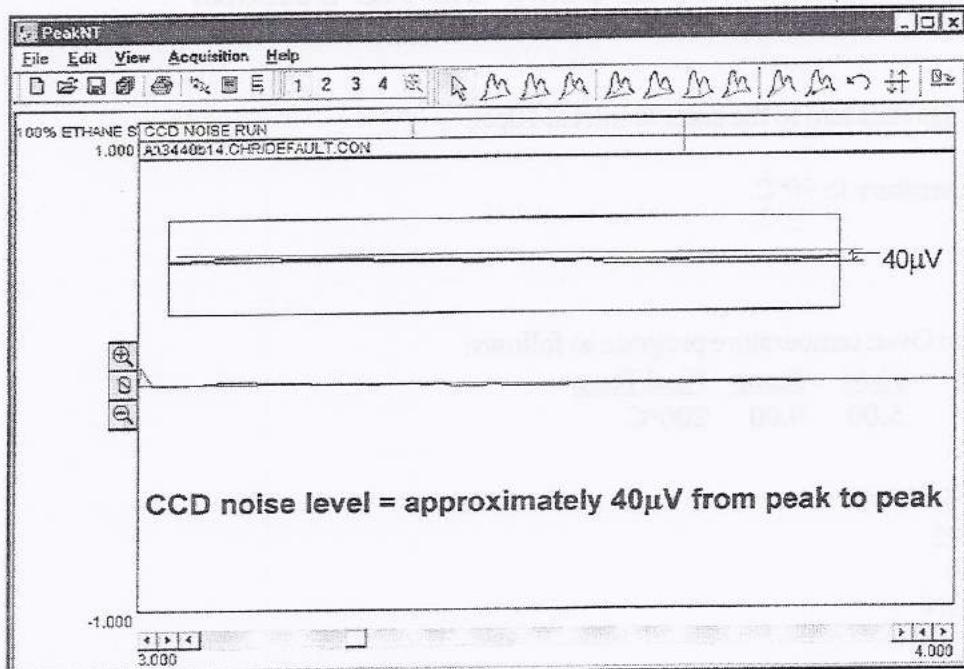
<u>Time</u>	<u>Event</u>
0.00	Zero
0.050	G ON
1.5	G OFF

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 hydrocarbons up to C<sub>5</sub>, you could set the valve to backflush after the elution of the C<sub>5</sub> component(s), and all the heavier hydrocarbons would together produce one large peak.

# POPULAR CONFIGURATION GCs

## Mud-Logger

### Expected Performance



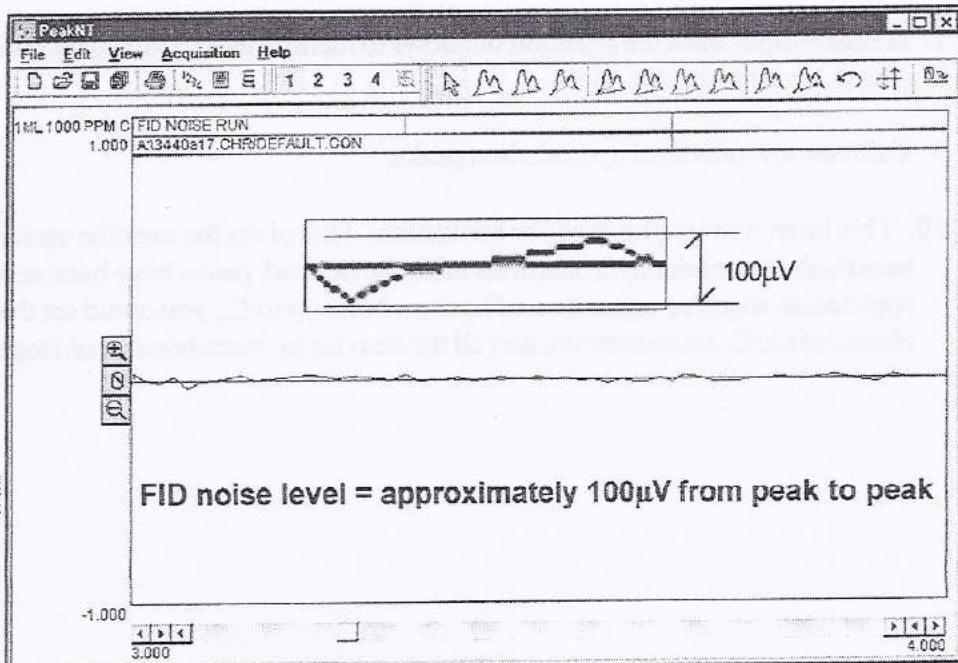
Column: 1m Hayesep-D  
Carrier: Air @ 10mL/min  
Air make-up = 100mL/min

Temperature program:  
Initial Hold Ramp Final  
80°C 5.00 0.00 80°C

### 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

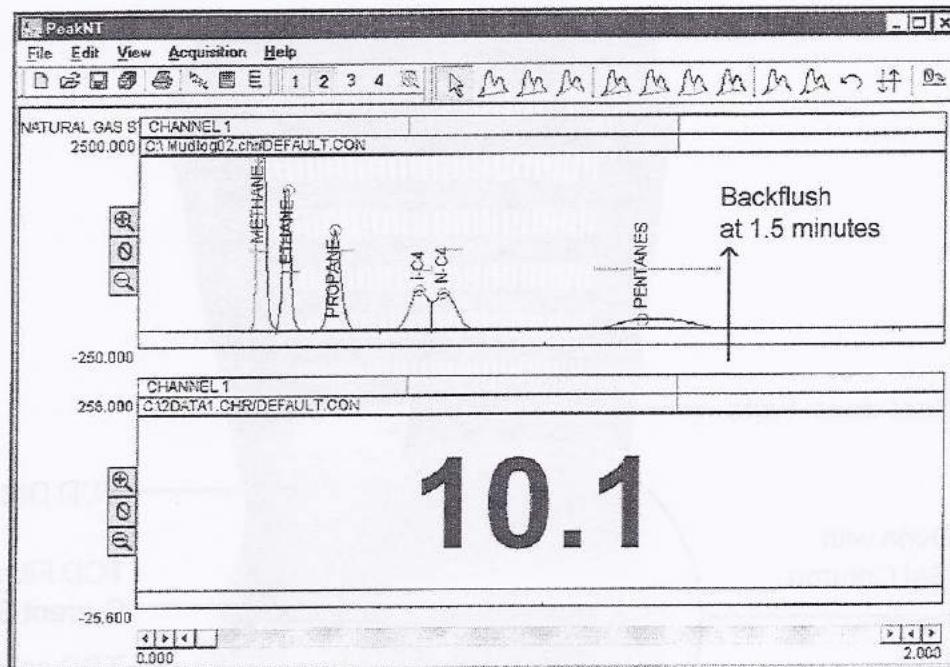


# POPULAR CONFIGURATION GCs

## 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

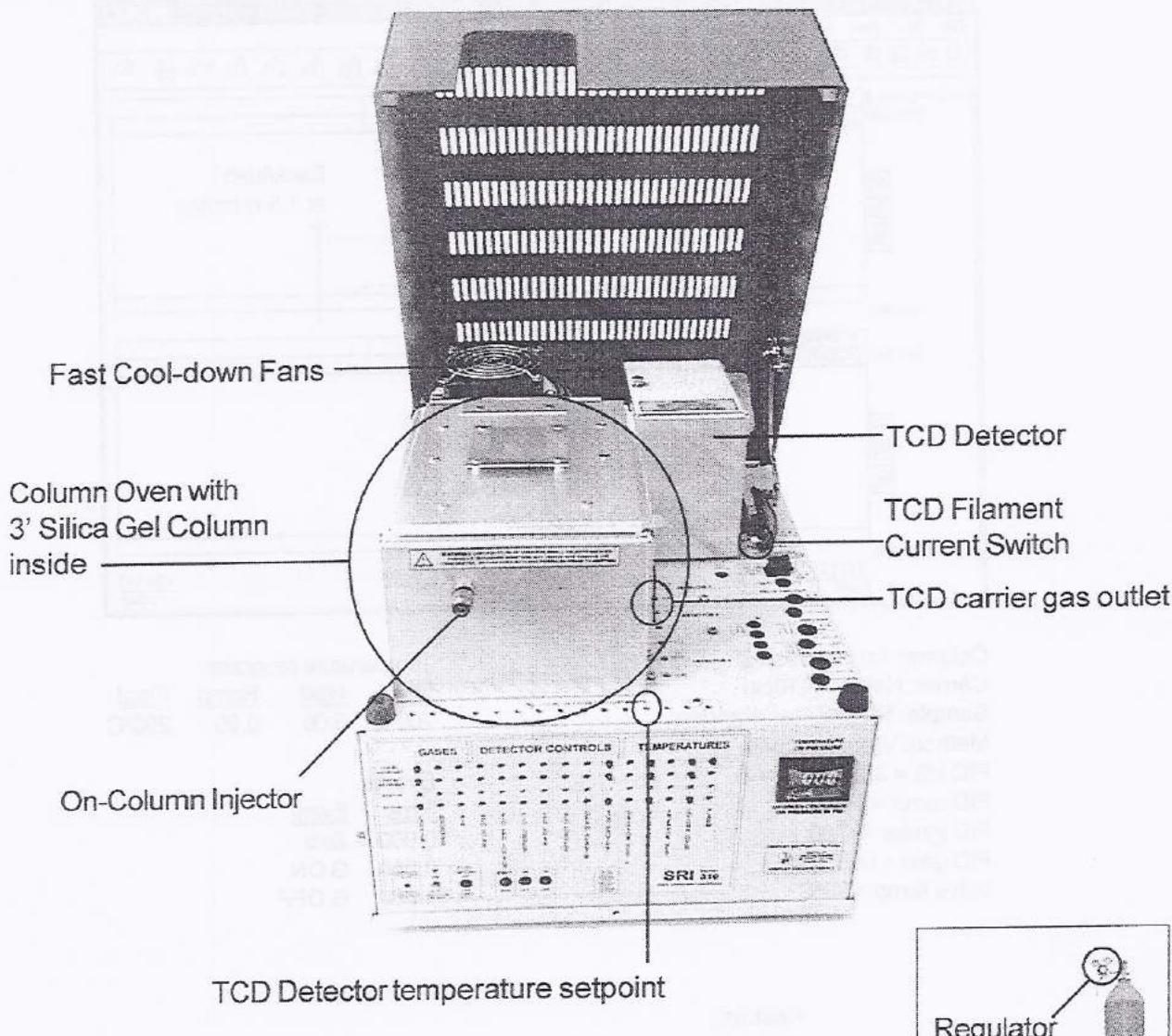
Temperature program:  
Initial Hold Ramp Final  
200°C 5.00 0.00 200°C

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

## 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.

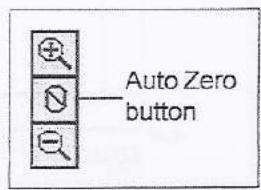
The TCD Detector requires helium to operate, which must be supplied by a gas cylinder and regulator. The helium cylinder pressure is normally set at 30psi, which is 10-20psi higher than the column head pressure.

## 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:

<u>Initial Temp.</u>	<u>Hold</u>	<u>Ramp</u>	<u>Final Temp.</u>
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.

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

# POPULAR CONFIGURATION GCs

## Educational TCD

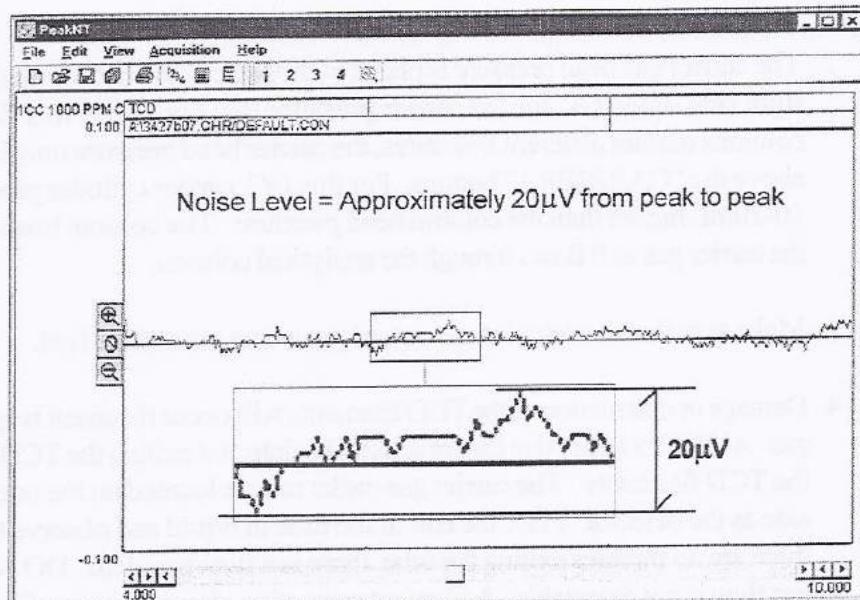
### Expected Performance

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 O<sub>2</sub>, N<sub>2</sub>, CO & CO<sub>2</sub>.

#### TCD Detector Noise

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

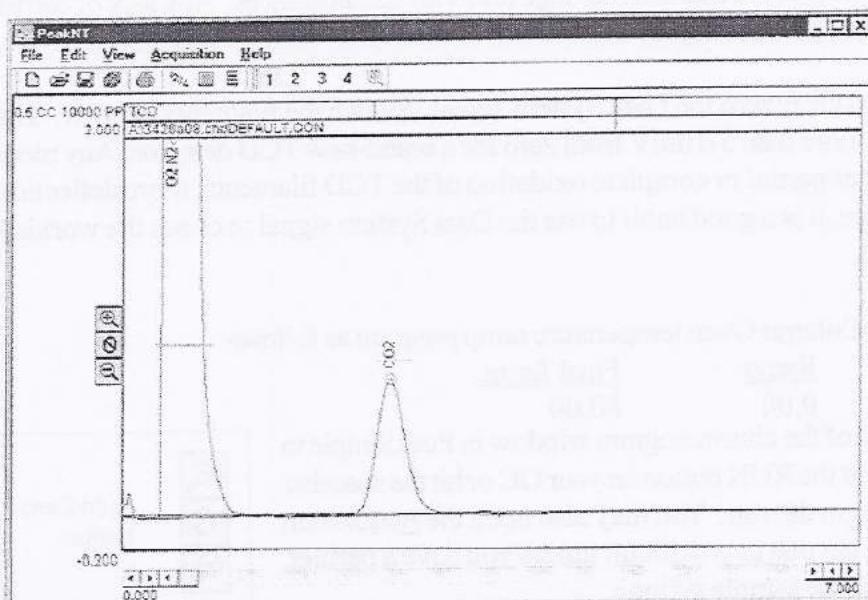
Temperature Program:  
Initial Hold Ramp Final  
80°C 10.00 0.00 80°C



#### Factory test run of an Educational TCD GC

Column = 1m Silica Gel  
Carrier = Helium at 10mL/min  
Sample = 0.5cc 10,000ppm CO<sub>2</sub>  
TCD current = LOW  
TCD Temp = 100°C

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



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

## POPULAR CONFIGURATION GCs

### Educational TCD

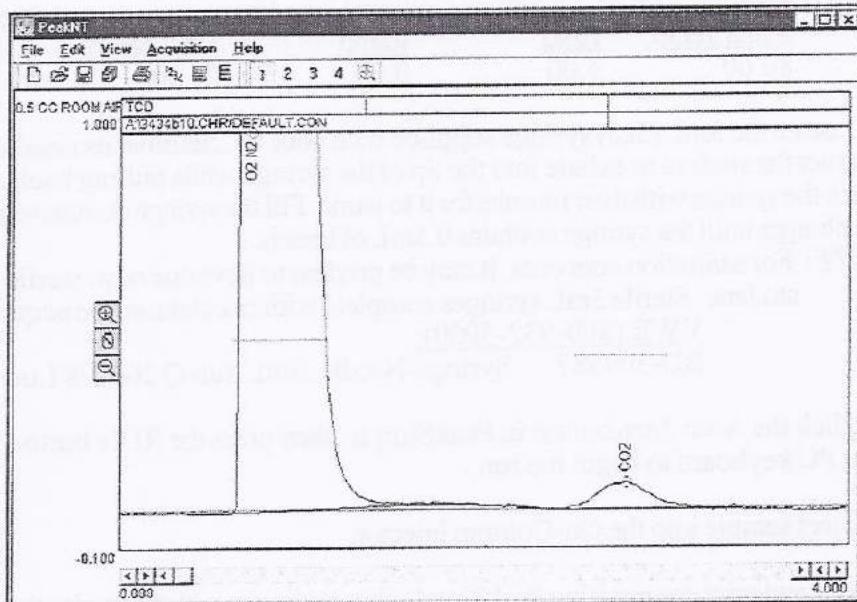
#### Expected Performance

#### 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 <sub>2</sub>	0.716	1021.3830	
N <sub>2</sub>			
CO <sub>2</sub>	2.766	1.5060	
Total		1022.8890	

The CO<sub>2</sub> content of the room air analyzed is approximately 350ppm.

## POPULAR CONFIGURATION GCs

### Educational TCD

#### **Suggested Class Experiment: "Waiting to Exhale"**

$\text{CO}_2$  is a natural by-product of human respiration. Our lungs get oxygen when we inhale and release  $\text{CO}_2$  when we exhale. When we hold our breath, the concentration of  $\text{CO}_2$  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  $\text{CO}_2$  concentration. Have a contest for the highest  $\text{CO}_2$  concentration: the student with the most  $\text{CO}_2$  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:

<u>Initial Temp.</u>	<u>Hold</u>	<u>Ramp</u>	<u>Final Temp.</u>
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  $\text{CO}_2$ .

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

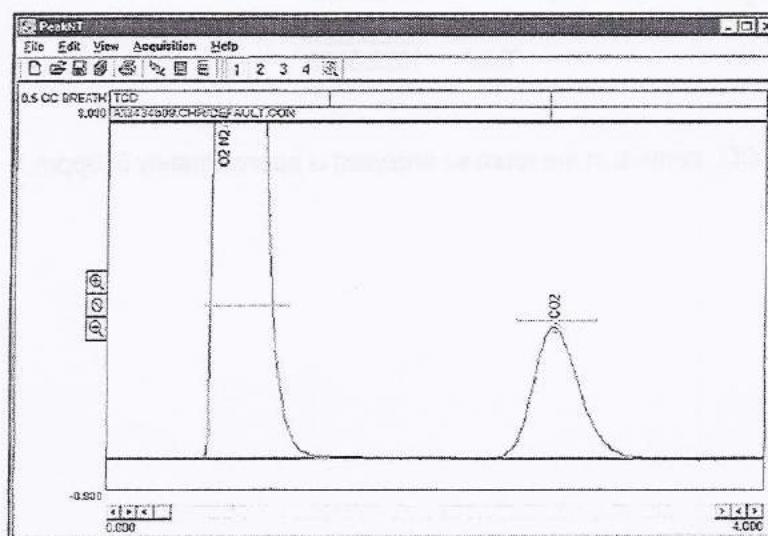
TCD temperature: 100°C

Temperature Program:

<u>Initial</u>	<u>Hold</u>	<u>Ramp</u>	<u>Final</u>
80°C	24.00	0.00	80°C

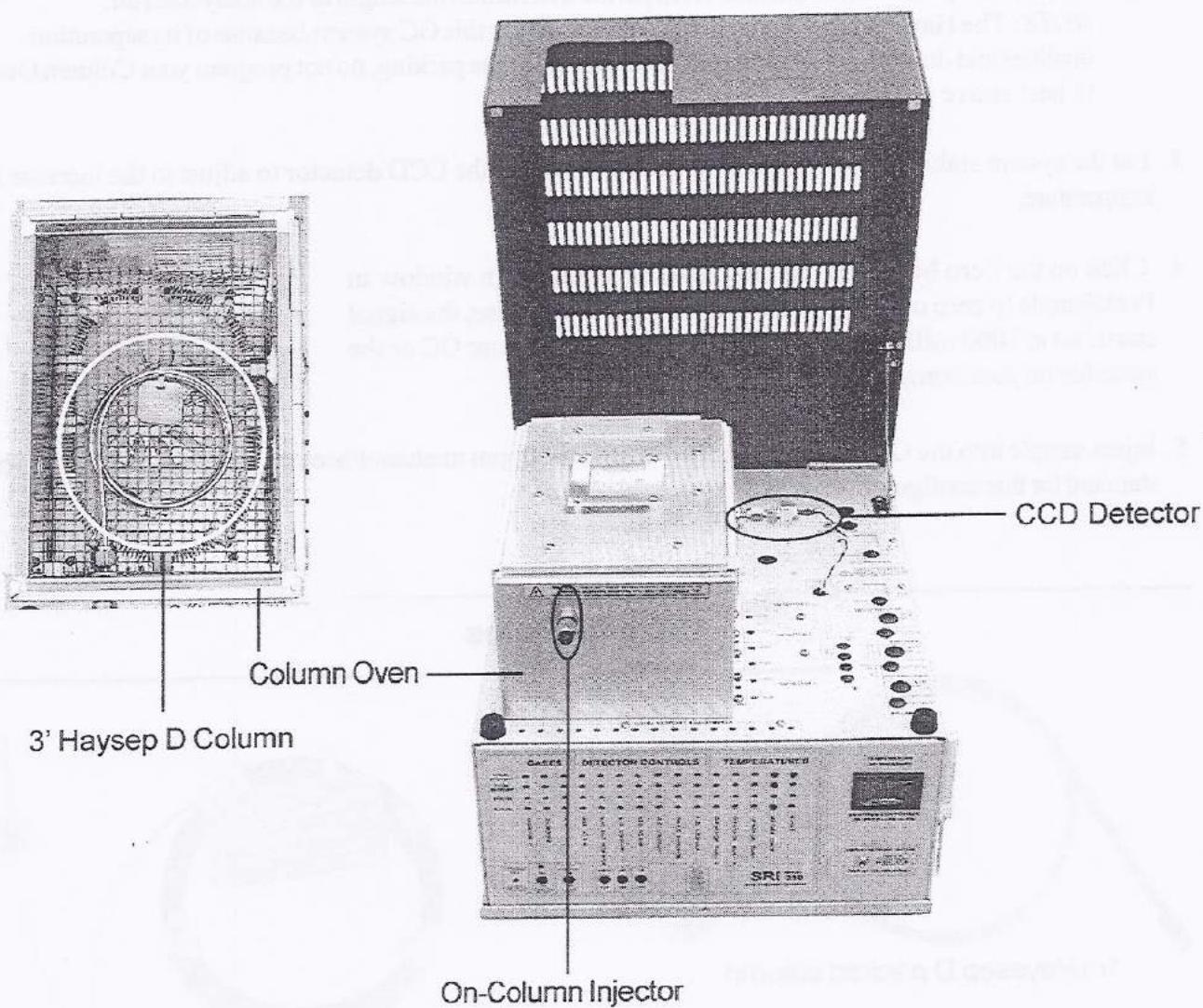
Results:

<u>Component</u>	<u>Retention</u>	<u>Area</u>
$\text{O}_2$	0.700	1379.4740
$\text{CO}_2$	2.700	61.9540
Total		1441.4280



## 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

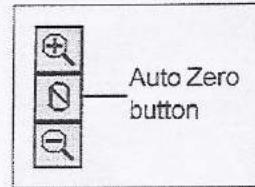
1. Connect your GC to your Windows PC with PeakSimple installed. Plug in your GC and turn its power on.
2. Set the Column Oven temperature to 130°C in PeakSimple as follows:

Initial Temp	Hold	Ramp	Final Temp
130.00	10.00	0.00	130.00

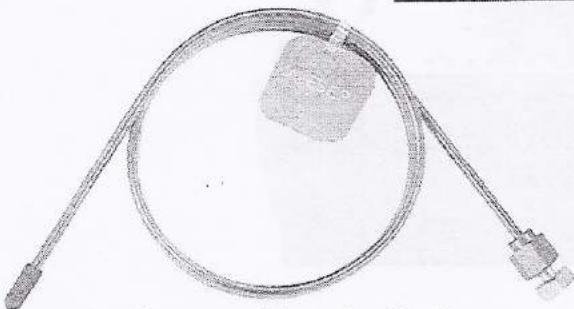
In an isothermal operation like this, the Hold period determines the length of the analytical run.

*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.

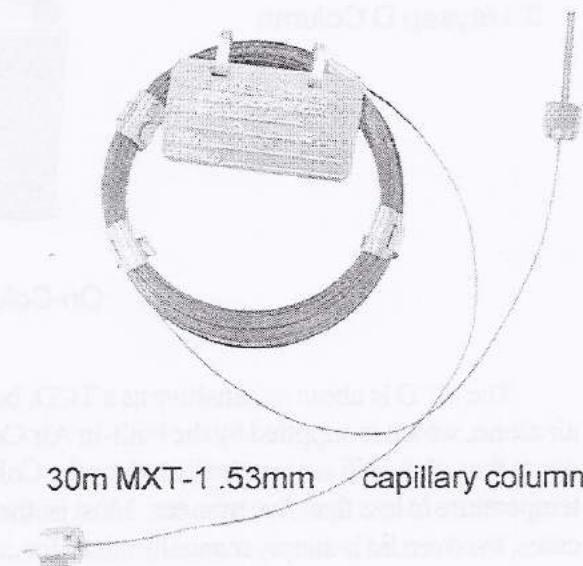


### Column Notes



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

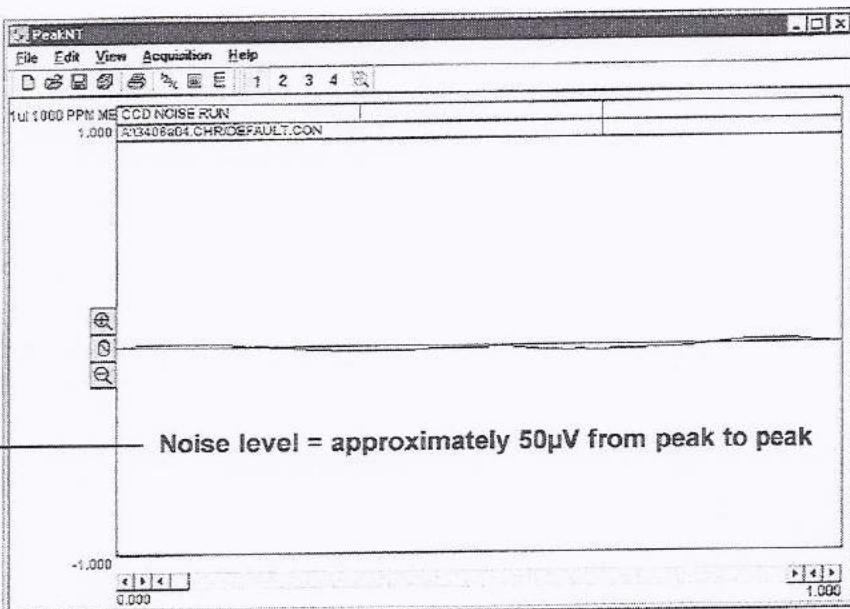
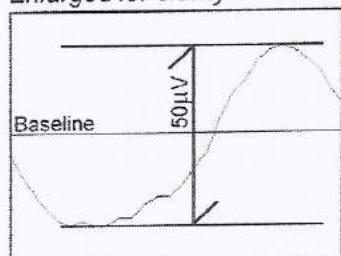
The CCD Detector in your Gas-less™ Educational GC is mounted on the wall of the Column Oven in a brass housing. It consists of a tiny coil of platinum wire embedded in a catalytic ceramic bead. This catalytic ceramic bead is housed in a plastic shell. A 150 milliamp current heats the bead to around 500°C. 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

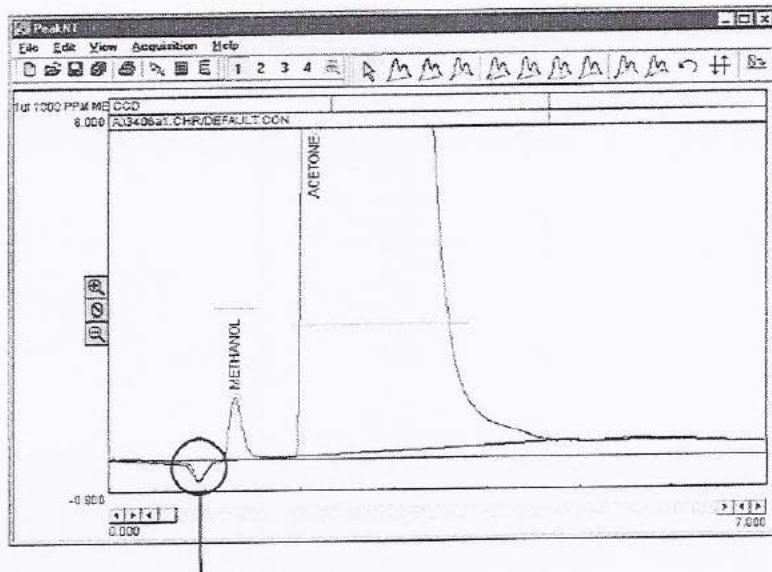
Column = 1m Hayesep D  
Flow = 37mL/min

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



Negative water peak

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

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

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