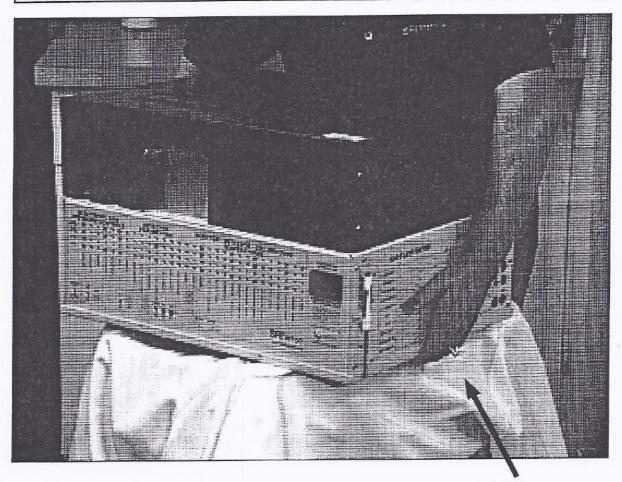
Chapter: Installation:

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.

Topic: Initial System Inspection And Setup

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.

Topic: Initial System Inspection And Setup (continued)

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 preferrable 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).

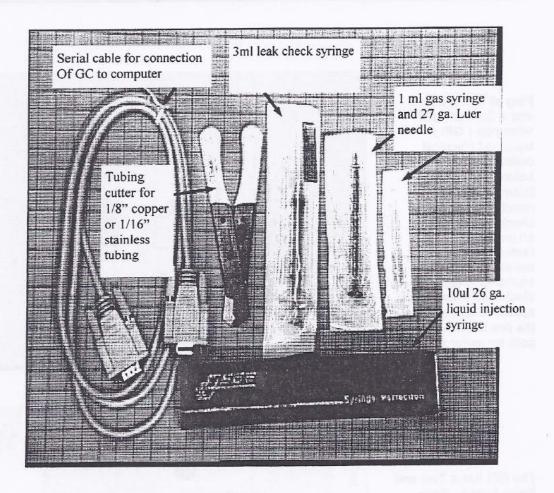
When routing the gas lines, care should be taken to avoid creating spots where moisture can gravitate and accumulate. Also, gas lines should not be routed near electrical outlets due to the potential for short circuiting created if the bare tubing were to come into contact with exposed electrical contacts, 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.

Topic: Contents of Accessories Kit included with GC



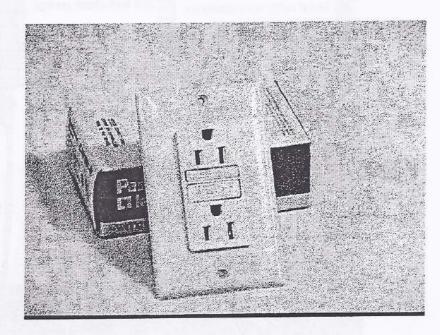
Contents of accessories kit shipped with new SRI GCs.

- 6' DB-9 serial cable for connection of GC to computer (Student model without data system will not have this item).
- Tubing cutter for easy installation of 1/8" copper or 1/16" stainless steel tubing
- 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

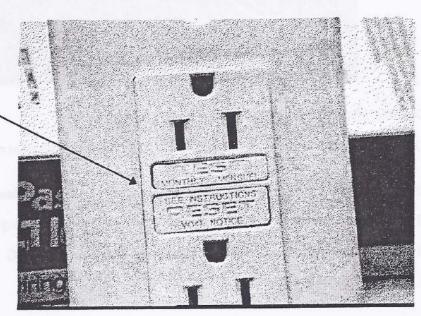
INSTALLATION:

ELECTRICAL POWER REQUIREMENTS

Plug all SRI products into a Ground Fault Interupter (GFI) equipped electrical outlet. The GFI will trip before a electrical failure in the GC can result in a dangerous shock to the operator, an important safety feature. If your outlet is not already GFI equipped, have your electrician install an approved GFI such as the one shown which sells for under \$10.



The GFI has a Test and Reset button. If the GFI trips, you must reactivate the GFI by pressing the Reset.



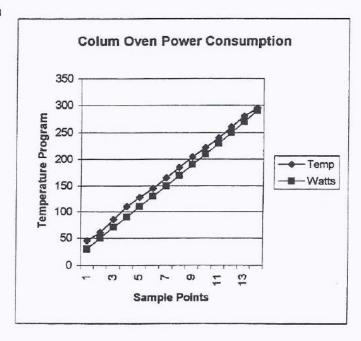
Basal Power

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
280	270	270-290



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

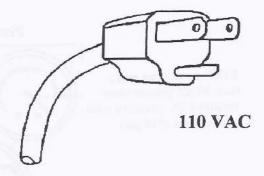
Total Power = (Basal + Detectors + Column Oven) = 400W

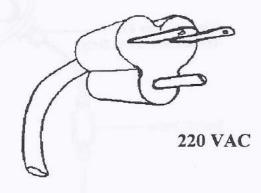
Topic: Power Supplies and Space Requirements

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).

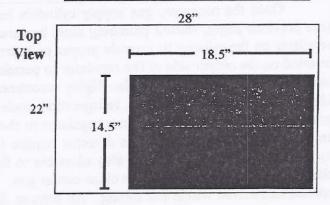
The SRI model 8610C chromatograph requires AC power at either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, depending on the AC power ordered. Both AC power supplies are equipped with a three prong grounded outlet (see diagrams to the right). Proper grounding is required for safe operation. Do not disable 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.

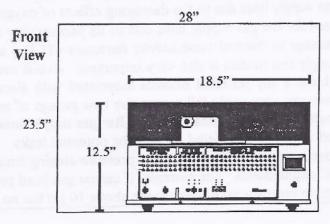
A standard SRI 8610C GC measures 18.5" X 14.5" X 12.5" and requires a minimum counter space of 28" X 22" X 23.5" for proper operation (see diagram to the right). Roughly 8" of clearance beside the left side control panel is needed for data cable, gas line and power switch access. 6" of clearance to the 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 ventallation. 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.



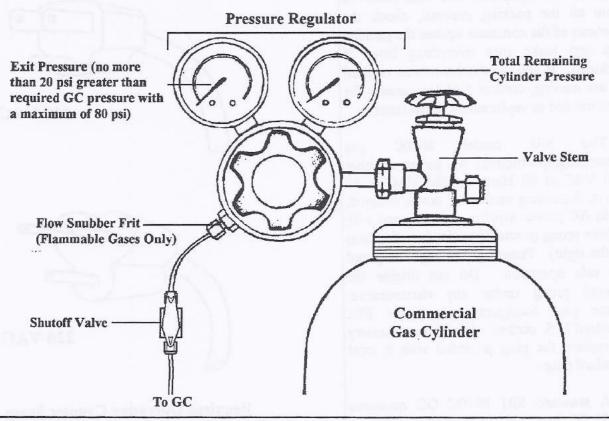


Required Operating Counter Space





Topic: Installing Gas Supply



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.

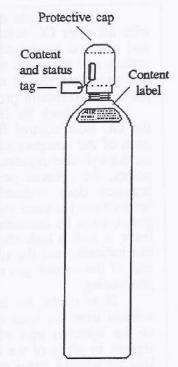
Topic: Gas Supply Selection

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. 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 CH

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.

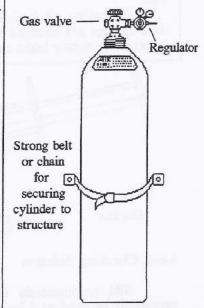
IMPORTANT SAFETY NOTE

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

Topic: Checking for Gas Leaks

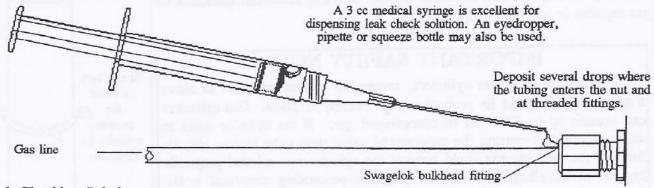
Once all of the appropriate gas supply sources and lines have been properly installed, along with all other GC columns and connections, the entire system should be systematically pressurized and checked for possible leaks. Begin by opening all of the compressed gas cylinder valves and setting exit pressures 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 pessure indicator should not noticeably decrease for at least five minutes. If pressure does noticably 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 interal 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

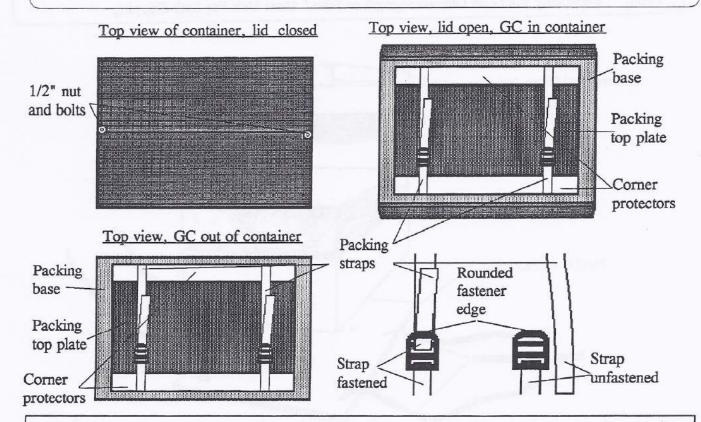
Following all the instructions above will assure the operator that the system is leak free. Any time fittings are changed or the GC is relocated, the system should be rechecked for leaks. Failure to properly repair leaks can cause safety risks as well as operational malfunctions.



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.

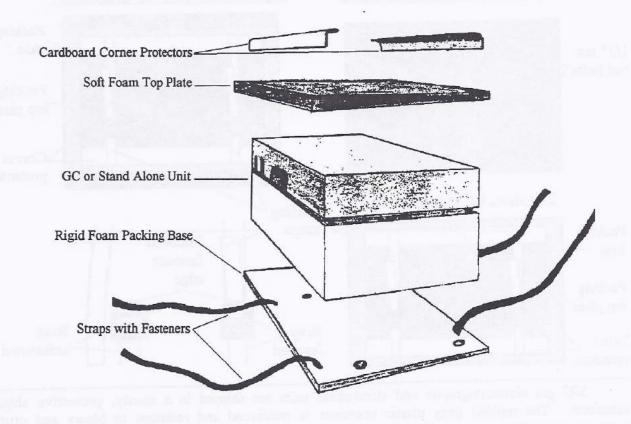
Topic: Removing The Chromatograph From The Shipping Container



SRI gas chromatographs and stand-alone units are shipped in a sturdy, protective shipping 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: Repacking Your Gas Ghromatograph 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.

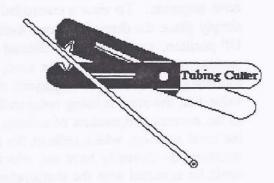
Topic: Tubing Cutter - For Facilitating Gas Connections

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.

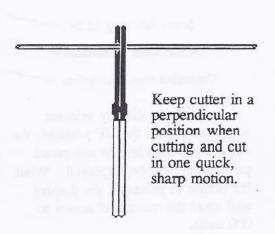
Users can make up to ten connections in the time that it took to cut, file, ream and connect one single tubing connection. Since the tubing is sheared and not twisted or stressed, the inside passage is not deformed or restricted, enabling the user to cut very small internal diameter stainless steel tubing (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 acheived without having to dissassemble 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.



Chapter:

INSTALLATION

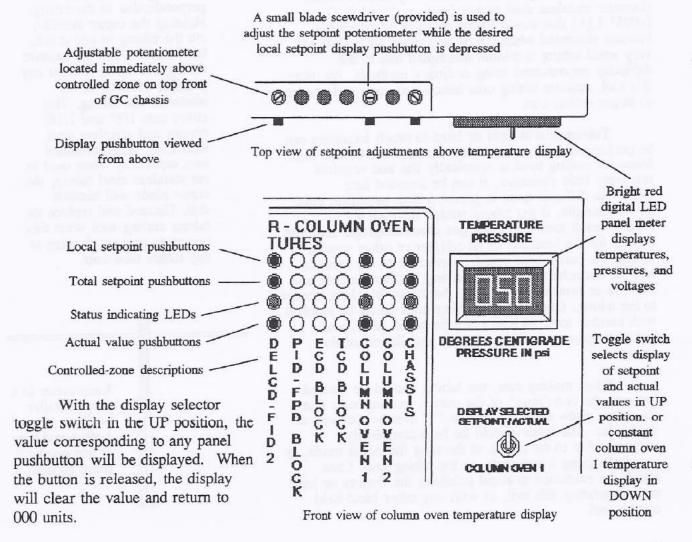
Topic:

Setting and Adjustment of Controlled Heated / Cooling Zones on 8610C GC

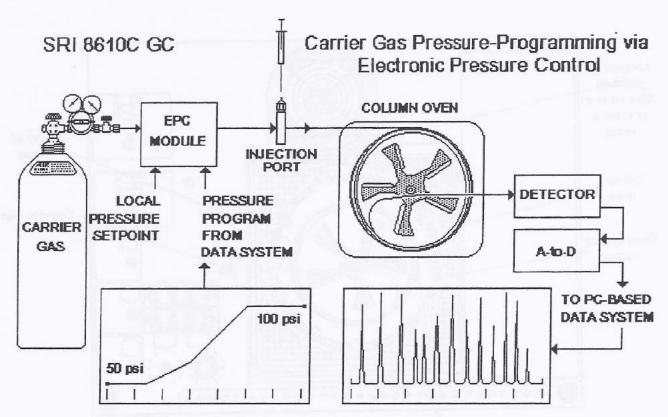
The 8610C gas chromatograph permits easy display and adjustment of all controlled zone setpoints. To view a controlled zone, simply place the display selector switch in the UP position, and depress the desired feature pushbutton. Depending on the zone, the following values may be displayed: the actual value that the zone is being measured at, such as the current temperature of column oven 1; the local setpoint, which reflects the adjustable

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

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.



Topic: Pressure-Programmed Carrier Gas Operation Using EPC System

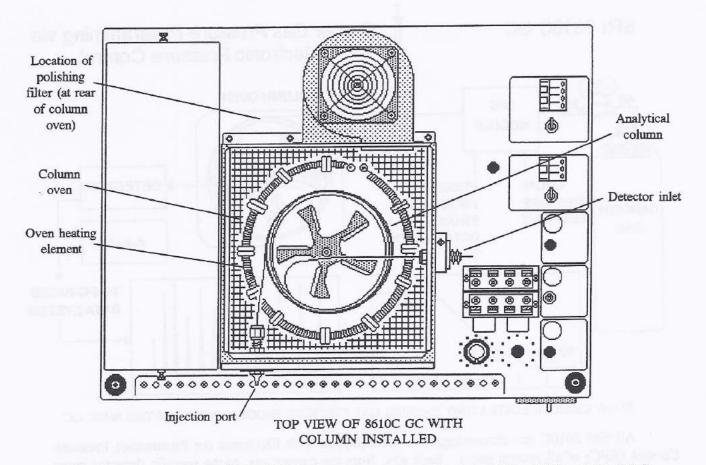


FLOW CHART ILLUSTRATING CARRIER GAS PRESSURE PROGRAMMING ON THE 8610C GC

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.

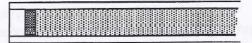
The PeakSimple serial data system interface offers two rampable voltage outputs - one to program the column oven, and the other to program carrier gas pressure. Outputting a 0 to 5VDC variable signal, the EPC module will permit an output pressure of from 0 to 100psi (the carrier pressure shown is actually 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.

Topic: Analytical Column Installation



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.

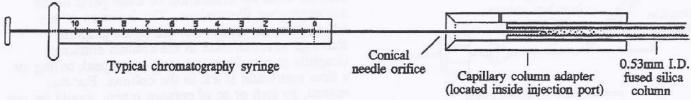
0.25 to 0.32mm I.d fused silica tubing coated on inside surface with stationary phase film 0.1 to 1.0 microns thick 0.53mm I.D. fused silica or fused silica-lined stainless steel tubing coated on the inside surface with a stationary phase film 0.1 to 5.0 micron thick



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 woll plug retains the packing inside the tubing

Topic: Analytical Column Installation (continued)

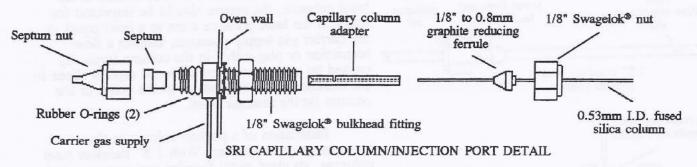
The injection port of the SRI chromatograph is designed specifically for direct injection onto a 0.53mm I.D. wide-bore capillary column. A sample, injected using a chromatography syringe equipped with a 26 gauge needle, is deposited directly into the column. The injector is supplied with a 1/8" O.D. 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.

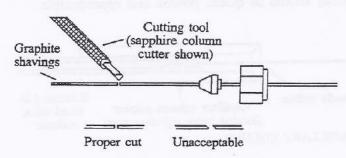
The capillary column adapter is located within the assembly that forms the injection port when a 0.53mm I.D. column is in use. The injection port is constructed from a 1/8" stainless steel Swagelok® bulkhead fitting that has been modified to permit the connection of a gas source directly into the fitting 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.

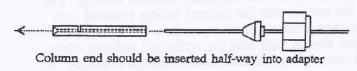


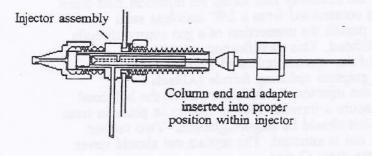
The injection port is compact and has a low thermal mass. Since most of the injector body is located within the column oven, the injector and oven temperatures are always equal (the standard injector is not supplied with any provision for independent heating. Heated on-column injection is available as 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.

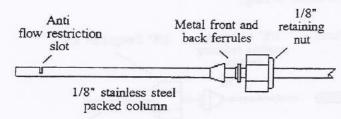
Topic: Analytical Column Installation (continued)

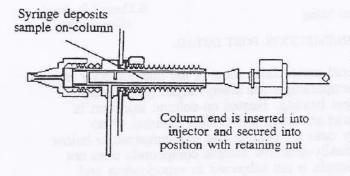
Graphite reducing ferrule 1/8" Swagelok retaining nut









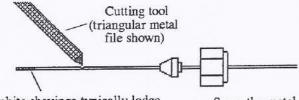


When installing a capillary column in the chromatograph, a graphite reducing ferrule must be used to secure the capillary tubing in the 1/8" retaining nut (Alltech RF200/0.8-G for 0.53mm) tubing, RF200/0.5-G for 0.32mm tubing and RF200/0.4-G for 0.25mm tubing). The column is inserted first through the 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).

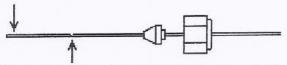
Installation of a packed column in the chromatograph is simpler. With 1/8" stainless steel columns, standard metal ferrules are used to secure the column at the retaining nut. The ferrules are placed onto the column end as shown, and then the column end is inserted into the injector. The capillary column 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.

Topic: Analytical Column Installation (continued)

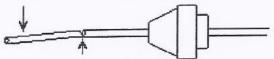
Graphite reducing ferrule 1/8" Swagelok retaining nut



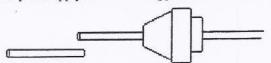
Graphite shavings typically lodge in tip of column when passing through graphite reducing ferrule Score the metal tubing with the file



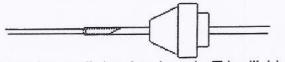
After scoring the metal column, wipe away any shavings and apply force to the tubing at the score



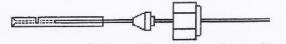
Bend the metal tubing downward until the score opens into a break in the metal Stop and apply force in the opposite direction



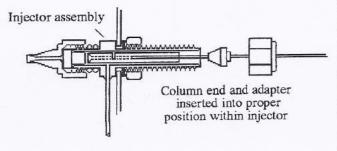
The front piece of tubing should break away cleanly



Insert a syringe needle into the column tip. This will debur the tip and permit the needle to slide in during injection



Column end properly located in capillary column adapter



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 corse, 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.

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

> Combo Detector

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 MINISTER DE L'ANDRES DE L'ANDR chromatogram. Benzene and carbon Method 5030 CARROLL MARCOLL MARCOL tetrachloride are common target analytes Purge & Trap which co-elute. The FID responds to both. The PID responds only to benzene, while tetrachloride. PID Detector FID/DELCD

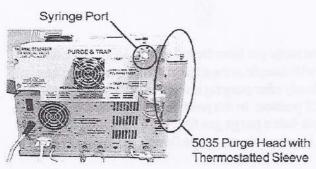
60m Capillary Column

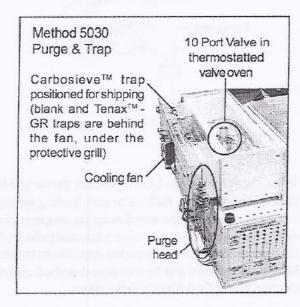
inside Column Oven

the DELCD responds only to carbon 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

Method 5030/5035 Purge & Trap on an Environmental GC

On-Column Injector

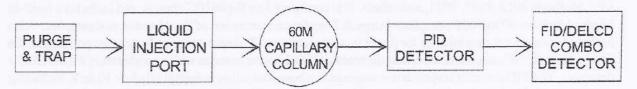




tankless field operation.

POPULAR CONFIGURATION GCs BTEX & Environmental

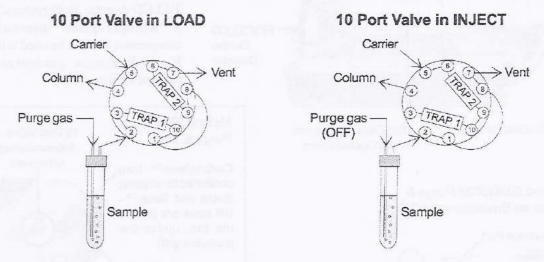
Theory of Operation



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 5μ 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.



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 1 mL or less for gas, and $5 \mu \text{L}$ 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 27 gauge needle for manual headspace injections

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 "OF F"			
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 "OF F"			
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.

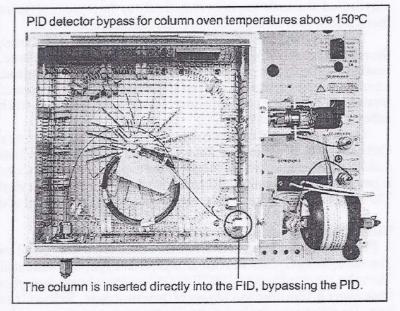
Dual T	rap Event	Table (Epap&t2c.evt)				
EVENT TIME	EVENT	EVENT FUNCTION				
0.000	ZERO	Zero signal				
0.100	E "ON"	Purge "ON" Purge "OFF" Trap 2 (Carbosieve) heat "ON" Valve in "INJECT"				
5.100	E "OFF"					
6.000	C "ON"					
8,000	G "ON"					
8.100	F "ON"	Trap 1 (Tena×GR) heat "ON				
8.500	G"OFF"	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"CN" (+50"C)				
14.900	F "OFF"	Trap 1 "OF F"				
15.000	E "OFF"	Purge "OFF"				
15.100	C "OFF"	Trap 2 "OF F"				
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 $10\mu L$ GC syringe with a 26 gauge needle. Fill the syringe with sample, and work out any air bubbles. Depress the plunger until $1\mu L$ 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.
- Begin the analysis by pressing the RUN button on the GC or the computer keyboard spacebar.
- N Auto Zero button

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

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.

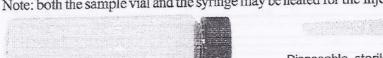
Using a vacuum pump:

- 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.

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.
- 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.
- 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

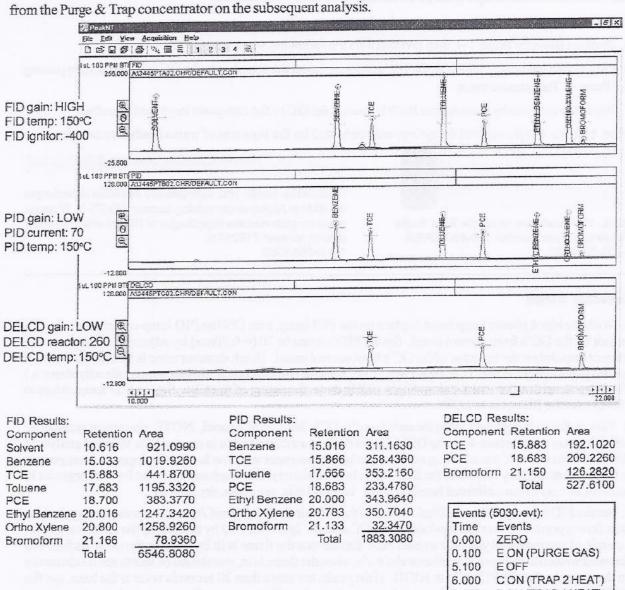
Detector Steps

- 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.
- 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.



Sample: 1µL 100ppm BTEX Plus standard dissolved in 10mL of water to yield 10ppb

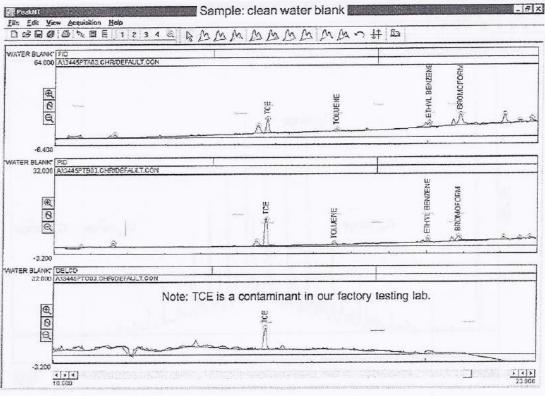
of each analyte

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

Events	(5030.evt):
Time	Events
0.000	ZERO
0.100	E ON (PURGE GAS)
5.100	EOFF
6.000	C ON (TRAP 2 HEAT)
6.050	FON (TRAP 1 HEAT)
8.000	G ON (VALVE INJECT)
12.000	EON
12.900	B ON (BAKE)
13.000	G OFF (VALVE LOAD)
14.900	FOFF
15.100	COFF
15.300	EOFF
15.500	BOFF

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:			PID Results:			DELCD Results:		
Component	Retention	Area	Component	Retention	Area	Component	Retention	Area
TCE	15.766	58.9100	TCE	15.750	58.1920	TCE	15.750	46.0340
Toluene	17.566	17.4000	Toluene	17.533	4.3400			
Ethyl Benzene	20.033	51.9080	Ortho Xylene	20.850	20.8720			
Ortho Xylene	20.833	91.5290		Total	609.1300			
To	tal	219.7470						

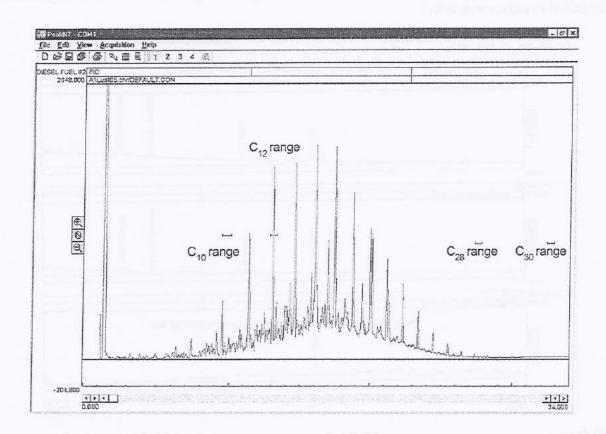
Determine the carryover level by comparing the areas of the two PID Toluene peaks resulting from the sample and blank runs:

 $\frac{4}{353} = \frac{x}{10ppb}$ 353x = 40ppb x = 0.1133ppb

(x represents the ppb concentration of the carryover)

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} .

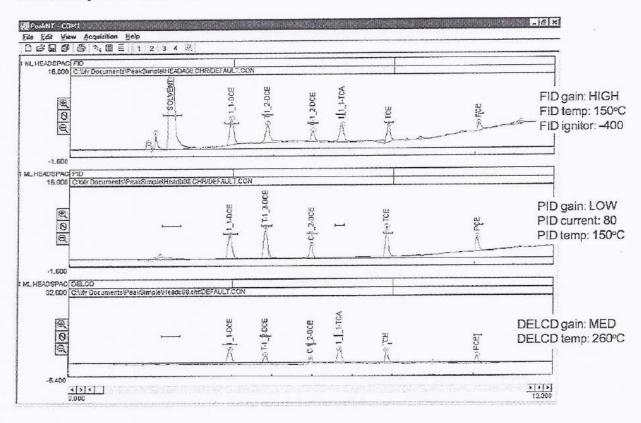


Sample: diesel fuel #2 Method: direct injection Column: 60m MXT-VOL Carrier: helium @ 10mL/min

FID gain: HIGH FID temp: 325℃ 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

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

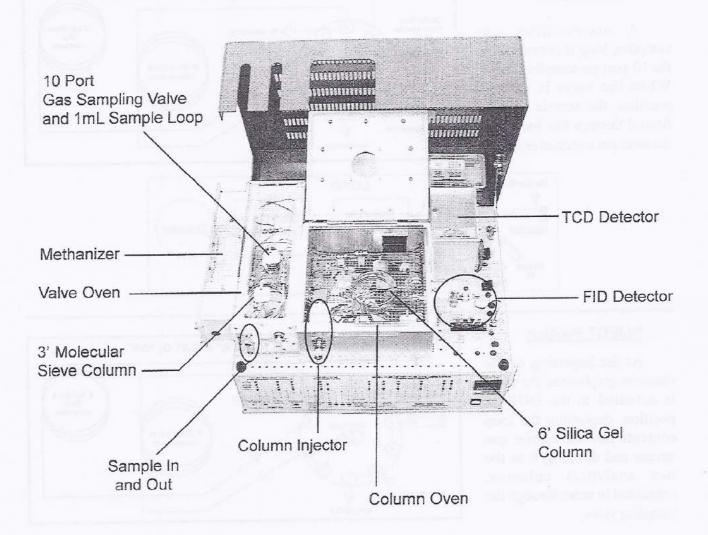
Column: 60m MXT-VOL Carrier: helium @ 10mL/min Temperature program: Initial Hold Ramp Final 40°C 2.000 15.000 220°C

220°C 10.00 0.000 220°C

FID Results:			PID Results:			DELCD Resu	ults:	
Component	Retention	Area	Component	Retention	Area	Component	Retention	Area
Solvent	2.416	290.1100	Solvent	2.183	22.7450	1_1-DCE	3.933	63.1790
1 1-DCE	3.933	39,6100	1 1-DCE	3.916	39.4070	T-1 2-DCE	4.816	38.0780
T-1 2-DCE	4.833	34.3780	T-1 2-DCE	4.800	45.0050	C-1 2-DCE	5.950	18.0560
C-1 2-DCE	5.966	18.6020	C-1 2-DCE	5.950	15.7380	1 1 1-TCA	6.666	53.2210
1 1 1-TCA	6.683	29.6320	TCE	7.816	33.7270	TCE	7.833	39.6900
TCE	7.850	23,4490	PCE	10.066	16.2780	PCE	10.083	20.8340
PCE	10.083	10.7560		Total	172.9000		Total	233.0580
	Total	446.5370						

System Overview

Your SRI Multiple Gas Analyzer GC is pre-plumbed and ready to resolve H_2 , O_2 , N_2 , Methane, CO, Ethane, CO_2 , Ethylene, NOx, Acetylene, Propane, Butanes, Pentanes, and C_6 through C_8 . The basic version of the 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.



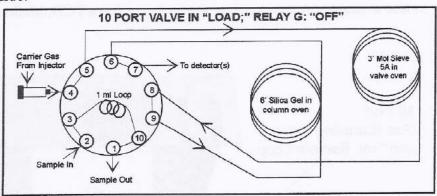
The Multiple Gas Analyzer #1 configuration allows you to obtain complete analyses of the fixed and natural gases listed above with a single sample injection. This is achieved using a 10 port gas sampling valve, a 1mL sample loop, and two independent analytical columns—a Silica Gel packed column and a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

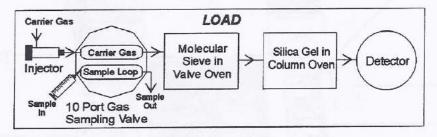
Theory of Operation 10 Port Gas Sampling Valve Plumbing Connections

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

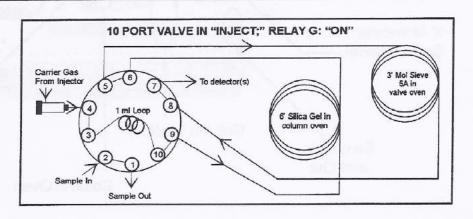
A one-milliliter gas sampling loop is connected to the 10-port gas sampling valve. When the valve is in load position, the sample may be flowed through this loop until the moment injection occurs.



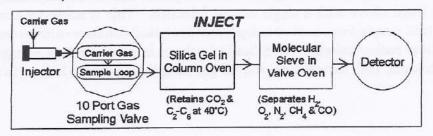


INJECT Position

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.



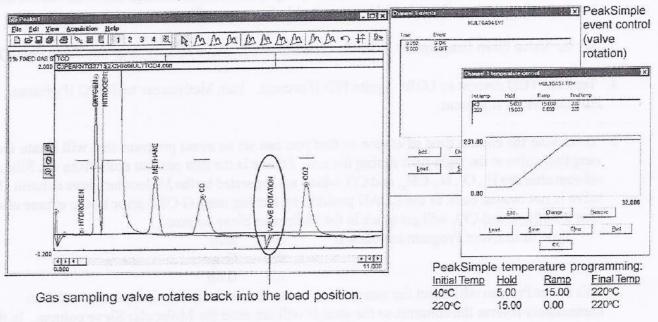
**Column sequence is reversed while the flow direction remains the same **



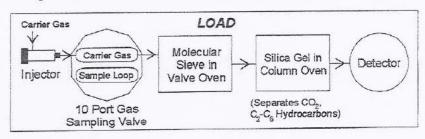
Theory of Operation 10 Port Gas Sampling Valve Plumbing Connections Continued

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 C₂, CO₂ 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



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.



General Operating Procedure

- 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.)
- 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 H₂, O₂, N₂, CH₄, 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, 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 Progam 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

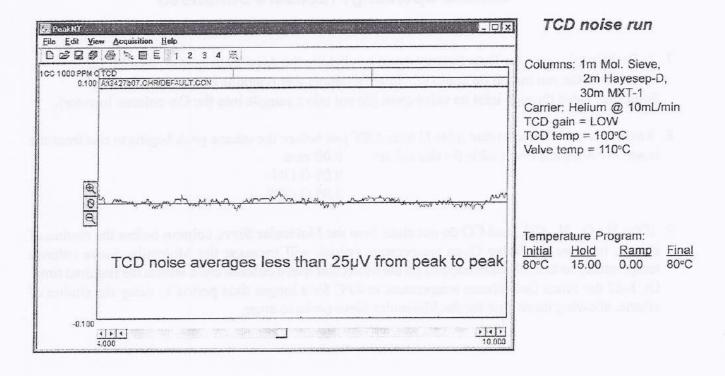
- 7. In PeakSimple, zero the Data System signal by clicking the Auto Zero button, then hit the spacebar or the run button on your GC. Inject a sample that contains ethane into the Gas Sampling Valve through inlet on valve oven (do not inject sample into the On-column Injector).
- 8. Revise the event 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

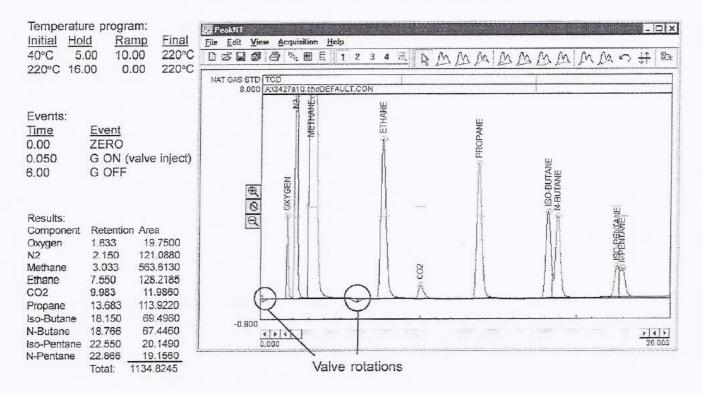
0.05 G ON 5.05 G OFF

9. If the H₂, O₂, N₂, CH₄, 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.

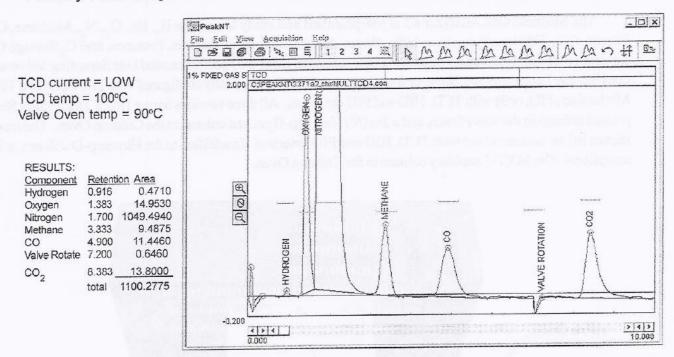
Expected Performance



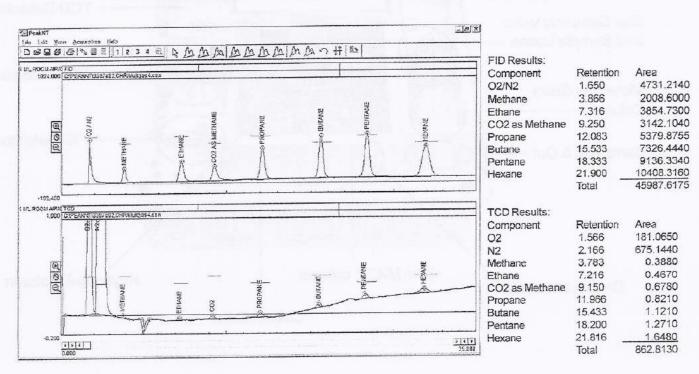
Factory Test Analysis of Natural Gas Standard



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

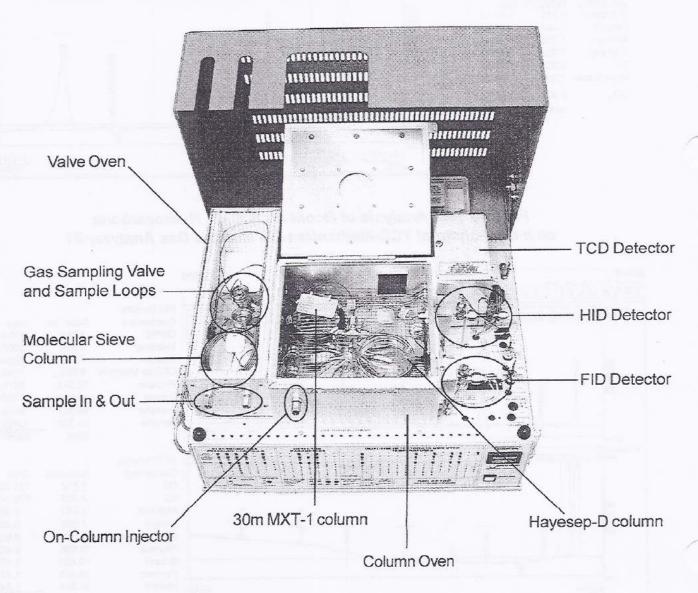


Factory Test Analysis of Room Air & C,-C, Hydrocarbons on a dual-channel TCD-Methanizer-FID Multiple Gas Analyzer #1



System Overview

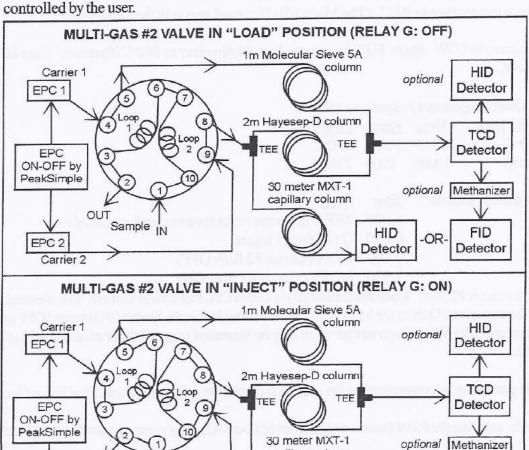
The Multiple Gas Analyzer #2 is pre-plumbed and ready to resolve H₂, He, O₂, N₂, Methane, CO, Ethane, CO₂, Ethylene/Acetylene, NOx, Water, Alcohols, Propane, Butanes, Pentanes, and C₆ through C₂₀. Separation of this wide variety of peaks is accomplished using a 10 port automated Gas Sampling Valve with 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.



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_2 , H_2 , H_3 , H_4 , $H_$

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.



capillary column

HID

Detector

FID

Detector

-OR-

OUT

EPC 2

Carrier 2

Sample

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.

General Operating Procedure

- 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.)
- 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:

Initial Temp	Hold	Ramp	Final Temp
50°C	The second second		220°C
220°C	10.00	0.00	220°C

6. Type in an event table as follows:

Time Event

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)

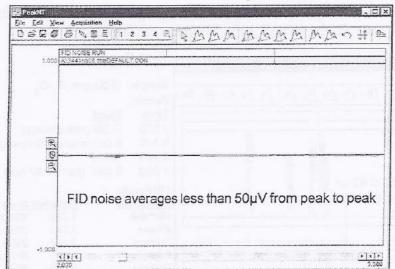
7.500 B OFF (carrier #2 flow ON)

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_2 , O_2 , N_2 , CH_4 , 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

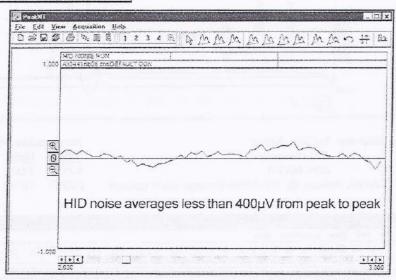
Columns: 1m Mol. Sieve, 2m Hayesep-D, 30m MXT-1

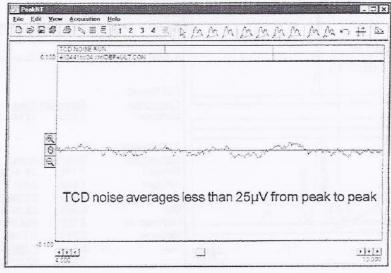
Carrier: Helium @ 10mL/min

HID gain = HIGH HID current = 70 HID temp = 200°C Valve temp = 110°C

Temperature Program:

Initial <u>Hold</u> Ramp Final 15.00 0.00 80°C 80°C





TCD noise run

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

Carrier: Helium @ 10mL/min

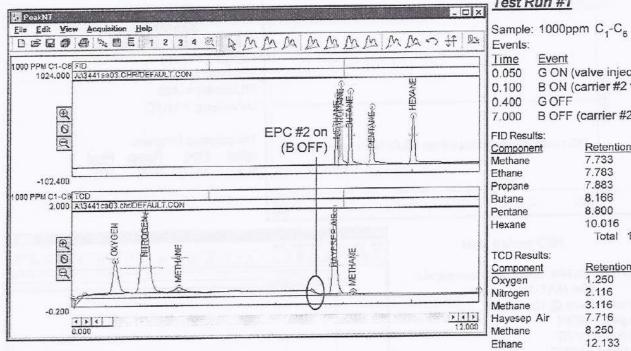
TCD gain = LOW TCD temp = 100°C Valve temp = 110°C

Temperature Program:

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

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_1 - C_6 sample; the second, a 1% fixed gas standard sample.



Test Run #1

Events:	
Time	Event
0.050	G ON (valve inject)
0.100	B ON (carrier #2 flow off)
0 100	0.000

7.000 B OFF (carrier #2 flow on)

FID Results:		
Component	Retention	on 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

Component	Retentio	n Area
Oxygen	1.250	7.9800
Vitrogen	2.116	27.9765
Methane	3.116	2.0210
layesep Air	7.716	23.5150
Methane	8.250	0.4950
thane	12.133	1.0240
	Total	63.0115

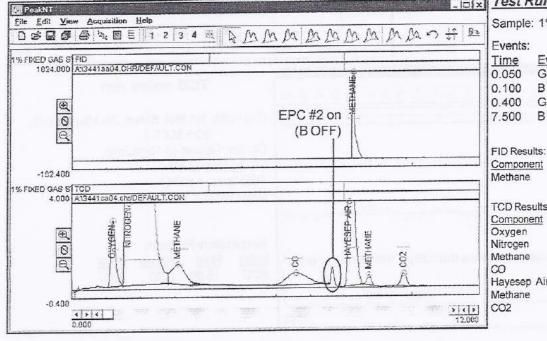
Columns: 1m Mol. Sieve,

2m Havesep-D. 30m MXT-1

Carrier: Helium @ 10mL/min through each column

Temperature Program:

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



-IDIXI Test Run #2

Sample: 1% fixed gas standard

Events:

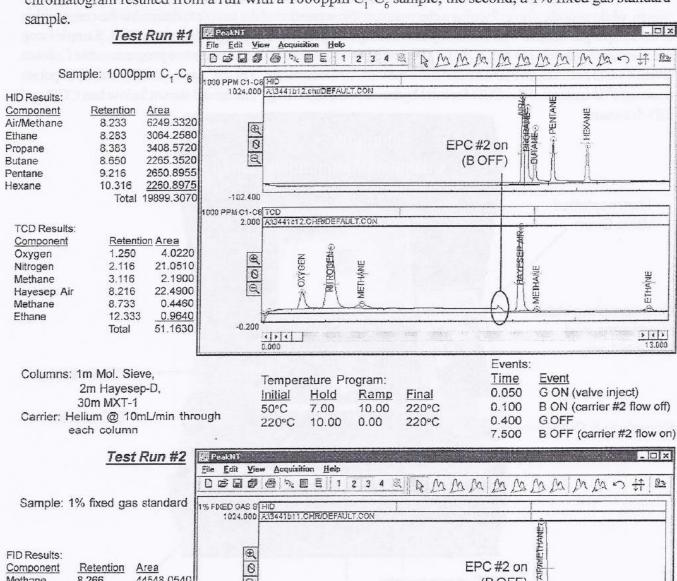
lime	Event
0.050	G ON (valve inject)
0.050 0.100	B ON (carrier #2 flow off)
0.400	GOFF
7.500	B OFF (carrier #2 flow on)

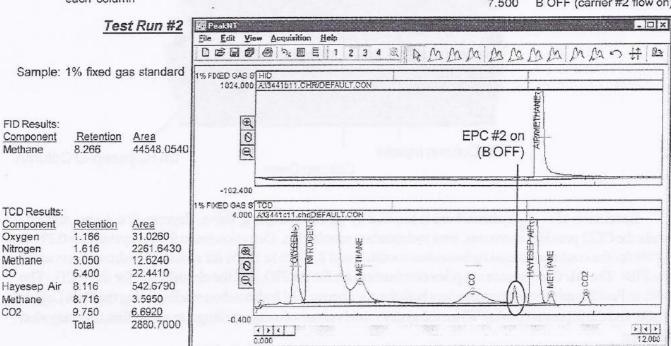
Component		Retention Area		
	Methane	8.233	12144.377	
	TCD Results:			
	Component	Retention	on Area	
	Oxygen	1.166	26.4920	
Contract of	Nitrogen	1.633	2251.7140	
	Methane	3.083	23.0975	
	co	6.566	22.2440	
1	Havesep Air	8.116	524.2010	
ALC: NO.	Methane	8.716	3.7730	
STATE OF	CO2	9.750	6.3940	
The Party of the P		Total	63.0115	

POPULAR CONFIGURATION GCs Multiple Gas Analyzer #2

Expected Performance: HID & TCD Detectors

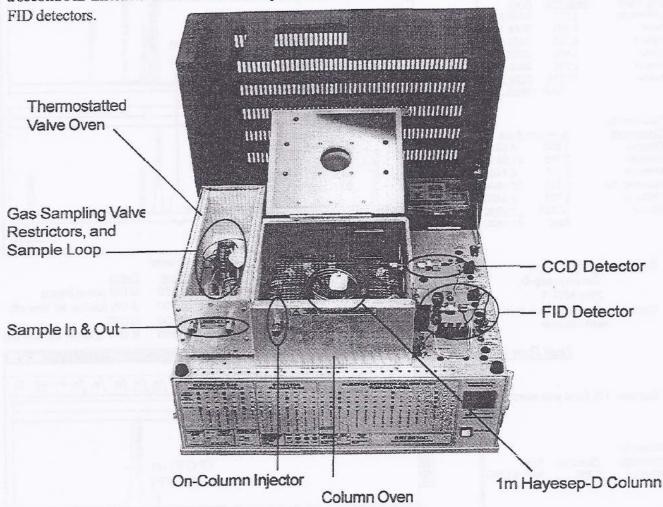
These two factory test runs utilized the same carrier flow, temperature program, and event table. The first chromatogram resulted from a run with a 1000ppm $\rm C_1$ - $\rm C_6$ sample; the second, a 1% fixed gas standard





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 25µL 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



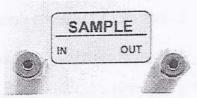
Speciation of C₁-C₆ hydrocarbons is handled by the Gas Sampling Valve, Hayesep-D column, and FID while the CCD provides continuous, total hydrocarbon monitoring. Detection limits for this system are 0.1% to 100% for the continuous total hydrocarbon monitor, and 0.005% to 100% for speciated hydrocarbons 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.

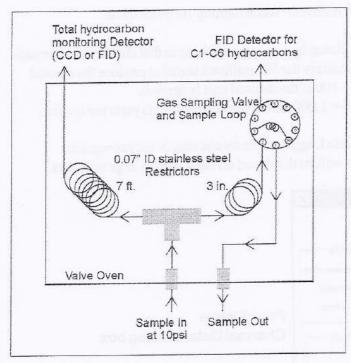
POPULAR CONFIGURATION GCs Mud-Logger

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.

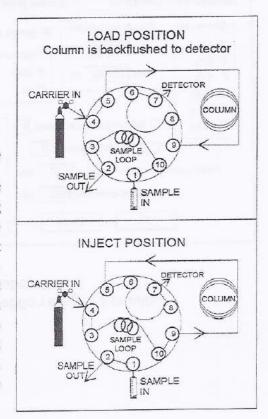




Once the sample enters the inlet, its path is T'd through two restrictors and on to the detectors. To avoid damaging the CCD, the maximum pure hydrocarbon flow to reach this detector is 5mL/min. The restrictors regulate the flow to the CCD to 5mL/min when the sample inlet pressure is 10psi. The remainder 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.

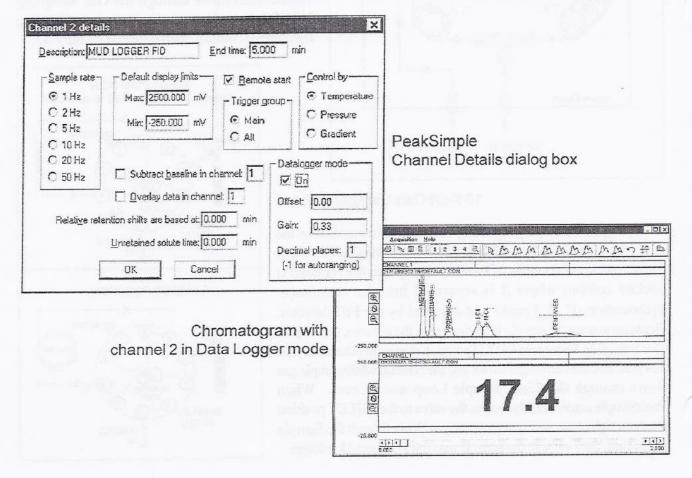
10 Port Gas Sampling Valve

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 though 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 x .33 = 100 if the desired unit is percent. 300 x 3333 = 1,000,000 if the desired unit is parts per million
- 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.



General Operating Procedure Part 2: Speciated Hydrocarbons Using the FID Detector

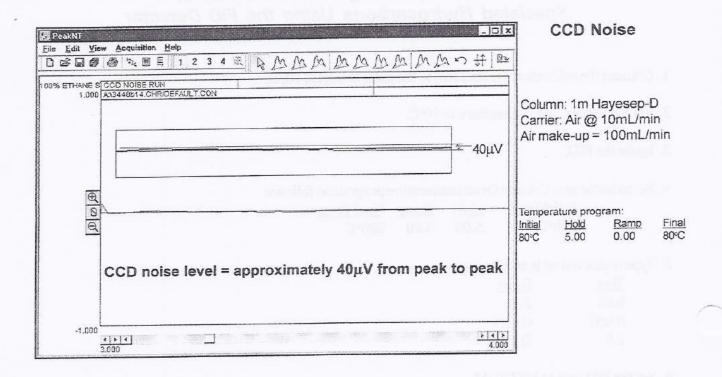
- Connect the calibration gas standard to the sample inlet at 10psi.
- 2. Set the Valve Oven temperature to 90°C.
- 3. Ignite the FID.
- 4. Set an isothermal Column Oven temperature program as follows:

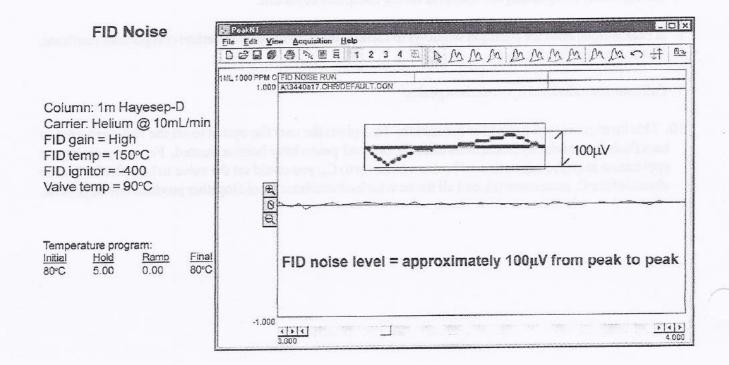
Initial Temp	Hold Ramp		Final Temp	
200°C	5.00	0.00	200°C	

5. Type in an even table as follows:

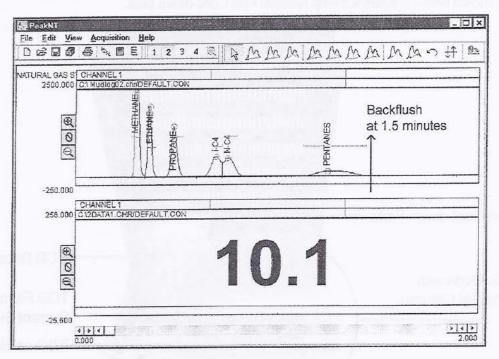
Time	Event	
0.00	Zero	
0.050	G ON	
1.5	G OFF	

- 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.





Factory Test Run of a Standard Mud-Logging System (FID and CCD)



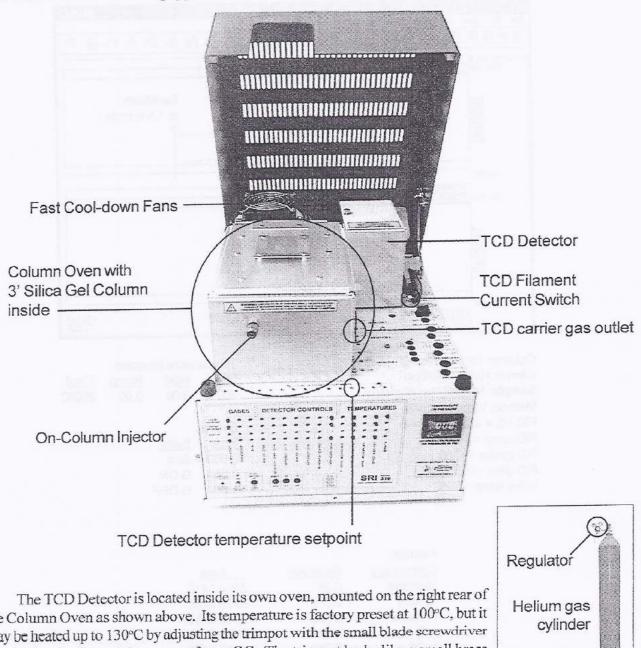
Column: 1m Hayesep-D
Carrier: Helium @10psi
Sample: Natural Gas standard
Method: Valve injection
FID H2 = 30, FID air = 6
FID temp = 150°C
FID ignitor = -750
FID gain = MEDIUM
Valve temp = 90°C

Temperature program: Initial Hold Ramp Final 200°C 5.00 0.00 200°C Events: Time Event Zero 0.000 0.050 GON 1.500 GOFF

Results:			
Component	Retention	on on	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 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.

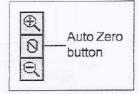
The TCD Detector requires helium to operate, which must be supplied by a gas cylinder and regulator. The helium cylinder pressure is normally set at 30psi, which is 10-20psi higher than the column head pressure.

General Operating Procedure

- 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.



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

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 $\rm O_2$, $\rm N_2$, $\rm CO \& CO_2$.

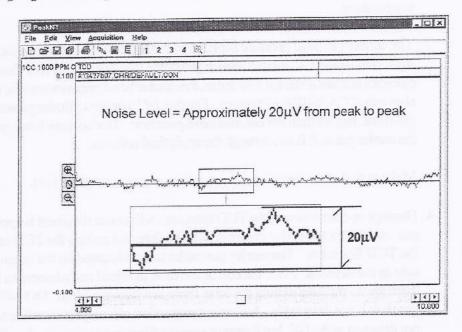
TCD Detector Noise

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

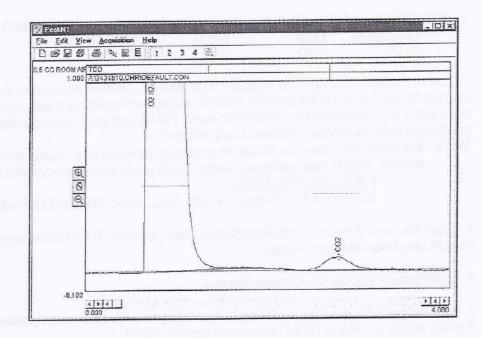
RESULTS:	Detention	Area
Component	Retention	
O2 N2	0.450	1252.9980
CO2	2.500	13.6460
Total		1266.6440

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

 O2 N2
 0.716
 1021.3830

 CO2
 2.766
 1.5060

 Total
 1022.8890

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

Suggested Class Experiment: "Waiting to Exhale"

 CO_2 is a natural by-product of human respiration. Our lungs get oxygen when we inhale and release CO_2 when we exhale. When we hold our breath, the concentration of 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 CO_2 concentration. Have a contest for the highest CO_2 concentration: the student with the most 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:

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:

<u>VWR (800-932-5000):</u> BD-309587 Syringe-Needle, 3mL Sub-Q 26g 5/8 Luer-lokTM

- 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 Peak Simple chromatogram with the student's name for the sample identification. Typical results are about 12-14 area counts per 1% of 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:

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

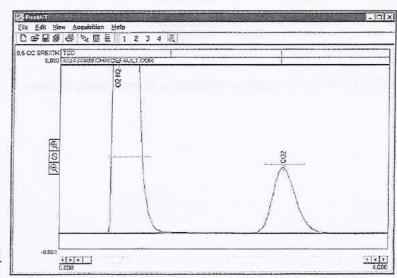
Results:

 Component
 Retention
 Area

 O₂ N₂
 0.700
 1379.4740

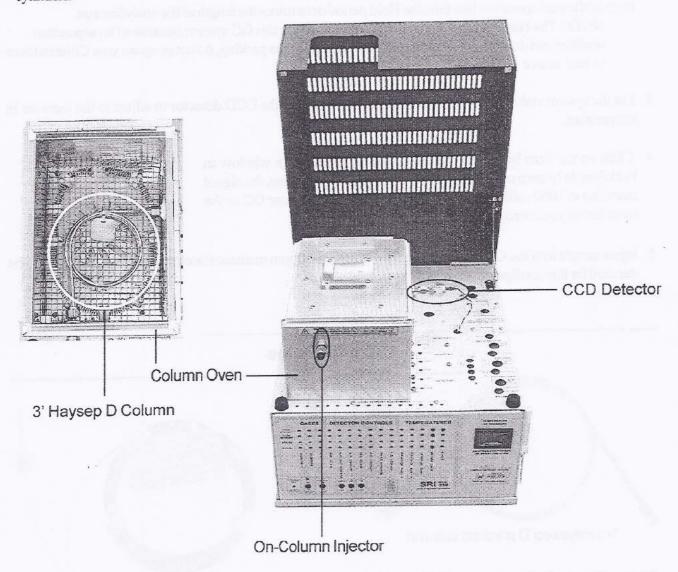
 CO₂
 2.700
 61.9540

 Total
 1441.4280



System Overview

Your SRI Gas-lessTM 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 cooldown 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

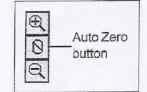
Final Temp 130.00

130.00

0.00 10.00

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.



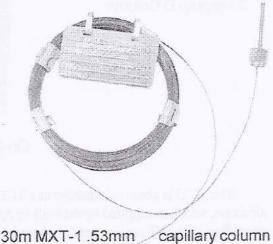
5. Inject sample into the On-Column Injector. A $1\mu L$ 1000ppm methanol/acetone sample is the factory test standard for this configuration.

Column Notes

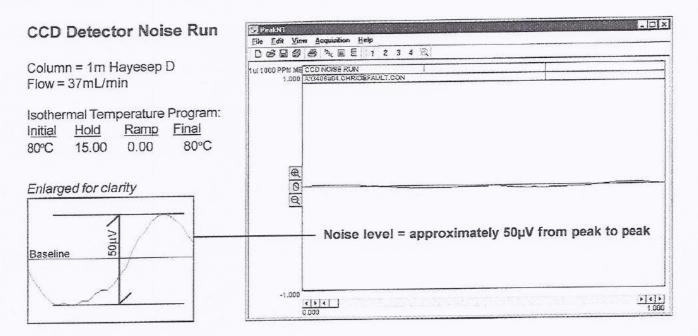


1m Havesep D packed column

Hayesep 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.



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.



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

