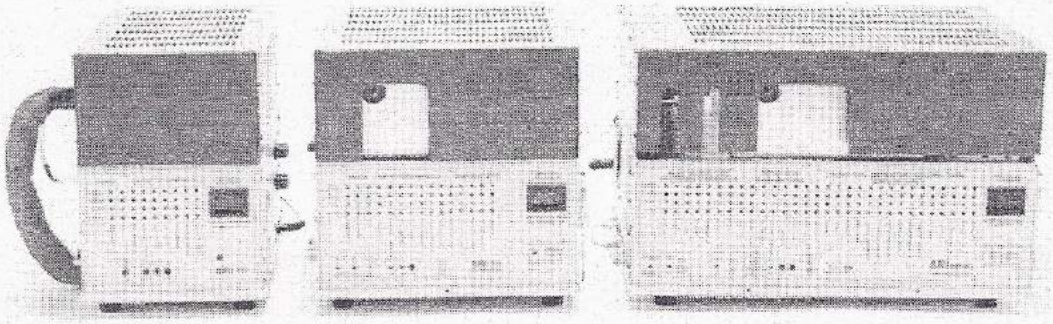
SRI Instruments
Operation Manual and Reference Guide



Model 110 Standalone Detector Chassis

Model 310 Ultra-compact GC

Model 8610C Compact GC

SRI Instruments
20720 Earl St.
Torrance CA 90503

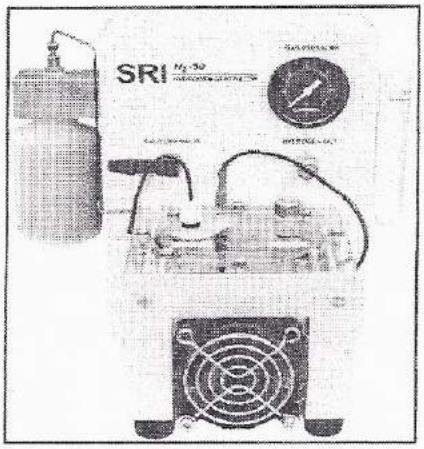
Phone: 310-214-5092
Fax: 5097

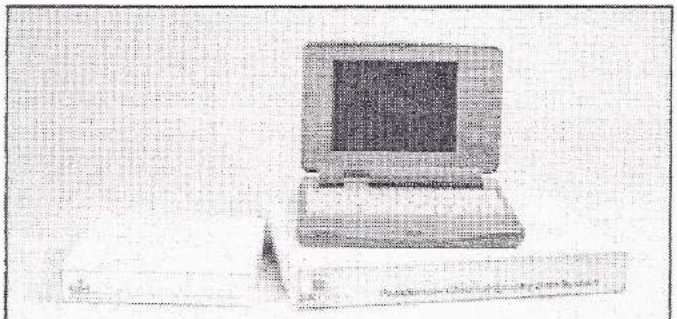
Web: http://www.srigc.com

E-mail: sales@srigc.com

January 2005

Model H2-50 Stand-alone Hydrogen Generator





Model 203 Single Channel Data System

Model 202 Four Channel Chromatography Data System

UNITS OF PRESSURE EQUIVALENCE (EQUAL TO 1 PSI)

1 psi = 2.036 in. Hg (inches of mercury)

= 27.68 in. w.c. (inches of water column)

= 51.715 mmHg or torr

= 0.068947 bar

= 0.06804 atm (atmospheres)

= 6.8947 kilopascals or KPa

= 0.0703 kg/cm^2

= 2.307 ft. H_20

WARRANTY AND WARNINGS

WARRANTY:

SRI will repair or replace any defective parts within two years from the date of shipment. Consummable items such as lamps, heaters, septa, NPD beads, ECD detector cell, DELCD heaters, FPD photomultiplier tubes, traps, filters, TCD filaments, columns, syringes, etc. are excluded. Replacement or repair shall be the purchaser's only remedy, and in no case shall SRI's liability exceed the original purchase price. The equipment is purchased without any other warranty expressed or implied, including, without limitation, any warranty of merchantability, any warranty arising from a course of dealing, performance of usage of trade and/or any warranty that the equipment is fit for any particular purpose or trade. The purchaser agrees to assume all risks of defects relating to the design, construction, purchase, operation, condition, maintenance, possession and use of the equipment, and to release SRI, to the maximum extent allowed by law, from any and all liabilities, claims or demands of any nature, including without limitation any claims based on incidental or consequential damages (forseeable or not), lost earnings, negligence (active or passive), strict liabitlity, breach of agreement or misconduct. The purchaser is aware of and waives the provisions of California Civil Code Section 1542, ("A general release does not extend to claims which the creditor does not know or suspect to exist in his favor at the time of executing the release, which if known by him must have materially affected his settlement with the debtor"), and/or all other laws, local, state, federal, or international, of similar intent, scope or purpose, relating to the release of unknown or unexpected claims. It is expressly agreed that the possibility of such unknown or undiscovered claims exist and has been explicitly taken into account in determining the equipment's purchase price and that consideration has been adjusted, having been bargained for in full knowledge of the possibility of such unknown claims. In the event the equipment is sold, loaned, or otherwise transferred, purchaser agrees to bind the third party to the terms of this agreement as a condition of transfer. Purchaser is aware of the dangers, and hazards inherent in operating chromatographs and data systems including but not limited to the warnings listed below. No agent, representative, distributor or employee of SRI has authority to amend this warranty in any way. In the event that any term or provision of this warranty is subject to valid claim of unenforceability, such term or provision shall be narrowly construed, the remaining provisions shall nevertheless survive, granting SRI the greatest possible protection then available under law.

WARNINGS AND HAZARDS:

Purchaser is aware of and accepts complete responsibility for operation of the equipment knowing that:

1) Flammable gases such as hydrogen and argon/methane are required for operation of some detectors, and adaquate precautions must be taken by the user to install safe and leak-free gas line tubing with flow snubbers, quick shutoff valves, etc. in accordance with all local fire department regulations. Flammable gases should not be used as carrier gas.

2) High temperatures may burn the operator. Safety gloves should be worn, and all surfaces touched only after making sure they are not hot.

3) High voltages on the PID lamp or FPD Photo-multiplier tube may shock the operator. Be sure the power is off before touching these parts.

4) Radioactive material is present inside the ECD detecter. It is the user's responsibility to comply with all regulations and safety precautions, and to dispose of the detecter in the manner prescribed by regulatory agencies. ECD detectors are transferred directly from Valco Inc.,Houston Texas, to the purchaser, and all licenses, details of operation, warranty, disposal, etc. are solely the responsibility of Valco and the purchaser.

5) Toxic, hazardous, or poisonous solvents such as N-Propanol are required for operation of the ELCD detecter. Other detectors may release or form toxic compounds, requiring operation under a fume hood or use of a respirator. Standards or samples required to calibrate the GC may be toxic, hazardous, or flammable.

6) Eye damaging ultra-violet light is emited by the PID lamp. Eye protection should be worn at all times when operating the GC.

7) Both qualitive and quantitive results from GC/Data System are subject to many sources of error. The magnitude of the error is variable, and must be statistically evaluated and controlled by the operator. Responsibility for the accuracy of the results obtained is solely the operator's. SRI makes no claims regarding the accuracy, bias, or precision of the results.

8) All SRI equipment is intended for operation by trained laboratory personnel only. It is the purchaser's responsibility to limit access so that only qualified laboratory technicians may operate the equipment and to ensure that they are provided with all neccesary safety apparatus, training, and procedures to minimize injury and/or damage in the event of an accident or malfunction (forseeable or not)

9) Errors and/or "bugs" may exist in integration software.

OPERATING THIS EQUIPMENT SHALL CONSTITUTE ACCEPTANCE OF ALL TERMS AND PROVISIONS ABOVE.

PLEASE READ!!!

0001.EPD

ATTENTION !
AVOID THESE COMMON ERRORS

1. In general, SRI Instruments does not recommend using nitrogen as a carrier gas. If nitrogen carrier must be used with a TCD, the current must be set to LOW. High current TCD operation with nitrogen carrier will destroy the filaments. Using nitrogen as carrier gas in capillary columns will drastically reduce the separating ability of the column. Use of helium carrier is suggested wherever possible. NEVER TURN ON THE TCD FILAMENT CURRENT BEFORE CARRIER AND REFERENCE GAS FLOWS EXIST THROUGH THE DETECTOR AND HAVE BEEN VERIFIED. THE TCD FILAMENTS WILL BE DESTROYED IF ENERGIZED WITHOUT THESE FLOWS! The 8610C gas chromatograph, in programmable and educational models, is equipped with a filament protection cutout circuit. This circuit will de-energize the filaments if the column carrier gas head pressure falls below a preset value (factory set to 3psi). This will prevent the filaments from incandescing if the carrier gas is interrupted to the detector by a removed septum nut, disconnected column, or empty carrier gas cylinder. It will not prevent filaments from damage if nitrogen carrier gas is used in conjunction with high filament current, which is prohibited and will void the warranty of the TCD detector. NEVER OPERATE THE TCD ON HIGH CURRENT WHEN USING NITROGEN CARRIER.

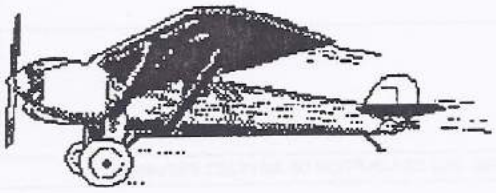
2. The SRI educational TCD-equipped gas chromatograph is equipped with a manual pressure regulator in lieu of a flow controller for carrier gas flow. The full-featured 8610C GC is equipped with programmable electronic pressure control (EPC) of carrier gas flow. Therefore, hydrogen carrier gas should not be used with either GC configuration. If a leak, column breakage, or other failure occurs, hydrogen gas could be released in dangerous concentrations, creating the potential for a fire or explosion to occur. Helium carrier offers an almost identical Van Deemter curve and performance to hydrogen carrier gas, and is the recommended substitute carrier gas.

3. When selecting a port address for the serial data acquisition interface built into your 8610C gas chromatograph, you must verify that you are not using a COM port on the data system host PC that is being shared with a mouse. Some PCs offer a DB-9 serial port labeled COM1, and have a DIN-plug type mouse with a separate, small, round plug and port. This may also be on the COM1 address. Consult your PC manual for information. If you are connecting to COM 2, and your PC is equipped with an internal modem, change the modem's COM port address from COM2 to COM3 or COM4 to avoid conflicts. Failure to do so will prevent the PC-based data system from communicating with the GC via the serial port. If you have any doubts regarding the configuration of your PC's COM ports, use the MS-DOS MSD.EXE utility to inspect your hardware settings.

4. If you have an NPD detector, do not use hydrogen as a carrier. The detector bead will overheat if the hydrogen flow is above 5 ml/min. Also, do not forget to install the NPD restrictor and resistor supplied, if your NPD also performs as a convertible FID detector. Do not turn the bead voltage above 4 volts or the detector bead will burn up. The voltage can be monitored with the digital display on the front of the unit. The display reads out in 1/100ths of a volt - 4 volts will be displayed as 400 units on the digital display.

Chapter: PREFACE

Topic: RETURNS OF EQUIPMENT FROM OUTSIDE U.S.A



In the event an item of SRI equipment needs to be returned to the factory from outside the U.S. A., please make a copy of the U. S. Customs form 3311 provided on the facing or reverse page, and include the filled out form with the shipping documents. This form will allow the equipment back into the U. S. without any customs duties, and will speed up customs clearance delay.

Before returning any goods, please obtain a RMA number (return material authorization) from SRI. At the time the RMA is issued, you will be advised on preferred methods of shipment and shipping companies. SRI will normally request pre-paid FEDX delivery.

To obtain an RMA contact:

SRI Instruments Technical Support
20720 Earl St.
Torrance CA 90503 U.S.A.
310-214-5092
fax 5097

DEPARTMENT OF THE TREASURY
UNITED STATES CUSTOMS SERVICE

Form Approved
OMB No. 1515-0043

DECLARATION FOR FREE ENTRY OF RETURNED AMERICAN PRODUCTS

19 CFR 10.1, 10.5, 10.6, 10.66, 10.67, 12.41, 123.4, 143.23, 145.35

1. PORT & DISTRICT	2. DATE	3. ENTRY NO. & DATE
4. NAME OF MANUFACTURER		5. CITY AND STATE OF MANUFACTURE
6. REASON FOR RETURN		7. U.S. DRAWBACK PREVIOUSLY ☐ CLAIMED ☐ UNCLAIMED 8. PREVIOUSLY IMPORTED UNDER TSUSA 864.05? ☐ YES ☐ NO

9. MARKS, NUMBERS, AND DESCRIPTION OF ARTICLES RETURNED	10. VALUE *

* If the value of the article is $10,000 or more and the articles are not clearly marked with the name and address of U.S. manufacturer, please attach copies of any documentation or other evidence that you have that will support or substantiate your claim for duty free status as American Goods Returned.

11. I declare that the information given above is true and correct to the best of my knowledge and belief; that the articles described above are the growth, production, and manufacture of the United States and are returned without having been advanced in value or improved in condition by any process of manufacture or other means; that no drawback bounty, or allowance has been paid or admitted thereon, or on any part thereof; and that if any notice(s) of exportation of articles with benefit of drawback ☐ was ☐ were filed upon exportation of the merchandise from the United States, such notice(s) ☐ has ☐ have been abandoned.

12. NAME OF DECLARANT	13. TITLE OF DECLARANT
14. NAME OF CORPORATION OR PARTNERSHIP (If any)	15. SIGNATURE (See note)

16. SIGNATURE OF AUTHORIZING CUSTOMS OFFICER

NOTE: If the owner or ultimate consignee is a corporation, this form must be signed by the president, vice president, secretary, or treasurer of the corporation, or by any employee or agent of the corporation who holds a power of attorney and a certificate by the corporation that such employee or agent has or will have knowledge of the pertinent facts.

Notice required by Paperwork Reduction Act of 1980: This information is needed to ensure that importers/exporters are complying with U.S. customs laws, to allow us to compute and collect the right amount of money, to enforce other agency requirements, and to collect accurate statistical information on imports. Your response is mandatory.

Statement required by 5 CFR 1320.21: The estimated average burden associated with this collection of information is 6 minutes per respondent or recordkeeper depending on individual circumstances. Comments concerning the accuracy of this burden estimate and suggestions for reducing this burden should be directed to U.S. Customs Service, Paperwork Management Branch, Washington, DC 20229, and the Office of Management and Budget, Paperwork Reduction Project (1515-0043), Washington, DC 20503.

ChromLab
A Chromatography Technology and Development Company

12 Vernon Lane
Malvern, PA 19355-2933
(610) 644-2260

GC & GC Data Handling Application Support and Training Services

Application Support and Training Services

ChromLab provides a full range of support, training and method development services for chromatography and data handling instrumentation, including:

- Complete installation and setup services for GC instrumentation and sample introduction systems including Headspace and Thermal Desorption.
- On-site training in GC and GC Data Handling for laboratory personnel.
- Development of effective operator manuals and in-house training programs.
- Method development for GC, HS/GC, capillary GC and fast GC analysis.

Benefits Provided to GC Customers

ChromLab gives your company the benefits of on-site chromatography expertise. We provide application support and training services for your GC instrumentation tailored to meet the needs of your laboratory when you need them and on a cost-effective basis.

- Reduced setup and training costs with on-site training for your personnel.
- On-site GC and GC data handling method setup and calibration for quick implementation of cost saving QC/QA chromatography methods.
- Customized Headspace GC, Thermal Desorption methods to solve difficult sample analysis challenges.
- Setup and training in regulatory compliance methods - EPA, OSHA, ASTM

ChromLab provides its support services to the following industries:

- Petroleum, Chemical, Energy, Environmental
- Flexible Packaging, Converting, Paper and Flooring Products, Inks and Coatings
- Pharmaceutical, Food and Beverage

Customized On-site GC Support and Training Services

ChromLab will train of your personnel in the efficient setup, operation and routine calibration of your GC equipment. We will assist in the transfer of new GC methods to your laboratory site. We will also optimize your GC data handling methods and develop standard operating procedures for your personnel to use after the training is complete. Training is available in the following areas:

- **Basic GC**
- **Capillary Column GC**
- **Headspace GC**
- **Thermal Desorption GC**
- **GC Data Handling.**

These support and training services will greatly reduce your GC downtime and overall operational costs to give your organization real long term saving. Your personnel will gain a solid understanding of their GC system and the confidence to perform routine calibration, maintenance and troubleshooting.

Providing Expert Technical Leadership

Burton S. Todd, is the Technical Director of ***ChromLab*** with over 30 years of hands-on expertise in providing application support and training in GC, HS/GC, GC/MS and GC data handling. He has worked with hundreds of companies, large and small assisting in the specification and setup of GC and data handling instrumentation. He has provided successful solutions to solve difficult analytical challenges and helped to build the confidence and skills of laboratory personnel through customized training tailored to meet their needs.

For More Information

For more information on these and other GC support and training services please contact ***ChromLab*** at 610-644-2260 or Fax your inquires and questions to Burton Todd. We will be glad to assist you in developing a support program to help meet your laboratory's goals.

Phone: (610) 644-2260 **FAX: (610) 644-2478**

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV

EPA
U.S. Environmental Protection Agency

ESTCP
Environmental Security Technology Certification Program

ornl
Oak Ridge National Laboratory

Joint Verification Statement

TECHNOLOGY TYPE:	**GAS CHROMATOGRAPHY**		
APPLICATION:	**MEASUREMENT OF EXPLOSIVES IN CONTAMINATED SOIL**		
TECHNOLOGY NAME:	**Model 8610C Gas Chromatograph/Thermionic Ionization Detection**		
COMPANY:	**SRI Instruments**		
ADDRESS:	**20720 Earl Street Torrance, CA 90503**	**PHONE: FAX:**	**(310) 214-5092 (310) 214-5097**
WEB SITE: EMAIL:	**www.srigc.com hagoldsmith@earthlink.net**		

The U.S. Environmental Protection Agency (EPA) has created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations, stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Department of Defense (DoD) has a similar verification program known as the Environmental Security Technology Certification Program (ESTCP). The purpose of ESTCP is to demonstrate and validate the most promising innovative technologies that target DoD's most urgent environmental needs and are projected to pay back the investment within 5 years through cost savings and improved efficiencies. ESTCP demonstrations are typically conducted under operational field conditions at DoD facilities. The demonstrations are intended to generate supporting cost and performance data for acceptance or validation of the technology. The goal is to transition mature environmental science and technology projects through the demonstration/ validation phase, enabling promising technologies to receive regulatory and end user acceptance in order to be field tested and commercialized more rapidly.

The Oak Ridge National Laboratory (ORNL) is one of the verification organizations operating under the Site Characterization and Monitoring Technologies (SCMT) program. SCMT, which is administered by EPA's National Exposure Research Laboratory, is one of 12 technology areas under ETV. In this verification test, ORNL evaluated the performance of explosives detection technologies. This verification statement provides a summary of the test results for SRI Instruments' Model 8610C gas chromatograph with thermionic ionization detection (GC/TID). This verification was conducted jointly with DoD's ESTCP.

VERIFICATION TEST DESCRIPTION

This verification test was designed to evaluate technologies that detect and measure explosives in soil. The test was conducted at ORNL in Oak Ridge, Tennessee, from August 21 through 30, 2000. Spiked samples of known concentration were used to assess the accuracy of the technology. Environmentally contaminated soil samples, collected from DoD sites in California, Louisiana, Iowa, and Tennessee and ranging in concentration from 0 to approximately 90,000 mg/kg, were used to assess several performance characteristics. The primary constituents in the samples were 2,4,6-trinitrotoluene (TNT); isomeric dinitrotoluene (DNT), including both 2,4-dinitrotoluene and 2,6-dinitrotoluene; hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). The results of the soil analyses conducted under field conditions by the GC/TID were compared with results from reference laboratory analyses of homogenous replicate samples analyzed using EPA SW-846 Method 8330. Details of the verification, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Explosives Detection Technology—SRI Instruments, GC/TID*, EPA/600/R-01/065.

TECHNOLOGY DESCRIPTION

The SRI Model 8610C gas chromatograph (GC) is a transportable instrument that can provide on-site analysis of soils for explosives. Coupling this transportable gas chromatograph with a thermionic ionization detector (TID) allows for the determination of explosives in soil matrices following simple sample preparation procedures. Samples are extracted in acetone, diluted, and injected directly onto the GC column within a heated injection port. The high temperature of the injection port instantaneously vaporizes the solvent extract and explosives, allowing them to travel as a vapor through the GC column in the presence of the nitrogen carrier gas. The stationary phase of the GC column and the programmable oven temperature separate the components present in sample extracts based on their relative affinities and vapor pressures. Upon elution from the column's end, compounds containing nitro groups are ionized on the surface of the thermionic bead, and the increased conductivity of atmosphere within the heated detector is measured with a collector electrode. In this verification test, the instrument was verified for its ability to detect and quantify 2,4-dinitrotoluene (2,4-DNT), RDX, and TNT. Analytical run times were typically less than 7 min and reporting limits were typically 0.5 mg/kg.

VERIFICATION OF PERFORMANCE

The following performance characteristics of SRI's GC/TID were observed.

Precision: The mean relative standard deviations (RSDs) for 2,4-DNT, RDX, and TNT were 15%, 14% and 23%, respectively, indicating that the determinations of all analytes were precise.

Accuracy: Accuracy was assessed using the performance evaluation (PE) soil samples, which were spiked to nominal TNT and RDX concentrations of 0, 10, 50, 100, 250, and 500 mg/kg each by an independent laboratory. The mean percent recoveries for RDX and TNT were 91% and 97%, respectively, indicating that the analyses were unbiased.

False positive/false negative results: Of the 20 blank soils, SRI reported TNT in five samples (25% false positives). No false positives were reported for 2,4-DNT and RDX. False positive and false negative results were also estimated by comparing the GC/TID result to the reference laboratory result for the environmental and spiked samples (e.g., whether SRI reported a result as a nondetect that the reference laboratory reported as a detection, and vice versa). For these soils, 3% of the 2,4-DNT results and 7% of the TNT results were reported as false positives relative to the reference laboratory results, but none of the RDX results were reported as false positives. Similarly, a small percentage of the results were reported as nondetects by SRI (i.e., false negatives) when the laboratory reported a detection (2% for RDX , 4% for TNT, none for 2,4-DNT).

Completeness: The GC/TID generated results for all 108 soil samples for a completeness of 100%.

Comparability: A one-to-one sample comparison of the GC/TID results and the reference laboratory results was performed for all samples (spiked and environmental) that were reported as detects. The correlation coefficient (r) for the comparison of the entire soil data set for TNT (excluding one suspect measurement for the reference laboratory) was 0.95 (slope (m) = 1.32). When comparability was assessed for specific concentration ranges, the r value did not change dramatically for TNT, ranging from 0.89 to 0.93 depending on the concentrations selected. RDX correlation coefficient with the reference laboratory for all soil results was slightly lower than TNT ($r = 0.85$, $m = 0.91$). The GC/TID's results for RDX correlated better with the reference laboratory for concentrations <500 mg/kg ($r = 0.96$, $m = 0.83$) than for samples where concentrations were >500 mg/kg ($r = 0.49$, $m = 0.56$). For the limited number of data points where both the reference laboratory and SRI reported results for 2,4-DNT ($n = 14$), the correlation was 0.44 ($m = 0.33$).

Sample Throughput: Throughput was approximately three samples per hour. This rate was accomplished by two operators and included sample preparation and analysis.

Ease of Use: No particular level of educational training is required for the operator, but knowledge of chromatographic techniques and experience in field instrument deployments would be advantageous.

Overall Evaluation: The overall performance of the GC/TID for the analysis of 2,4-DNT, RDX, and TNT was characterized as precise and unbiased. As with any technology selection, the user must determine if this technology is appropriate for the application and the project's data quality objectives. For more information on this and other verified technologies, visit the ETV web site at http://www.epa.gov/etv.

Gary J. Foley, Ph.D.
Director
National Exposure Research Laboratory
Office of Research and Development

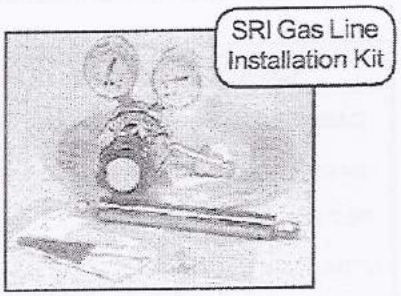
W. Frank Harris, Ph.D.
Associate Laboratory Director
Biological and Environmental Sciences
Oak Ridge National Laboratory

Jeffrey Marqusee, Ph.D.
Director
Environmental Security Technology Certification Program
Department of Defense

NOTICE: EPA and ESTCP verifications are based on evaluations of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. EPA, ESTCP, and ORNL make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements. Mention of commercial product names does not imply endorsement or recommendation.

Quick Start SRI GC Installation Guide

I. Gas Installation & Connection



1. To connect your GC to a gas supply, we recommend the following:
   - A 50 foot length of copper tubing
   - A stainless steel gas line filter
   - At least 2 sets of stainless steel Swagelok nuts and brass ferrules (it is a good idea to keep a few extras on hand)
   - A cylinder pressure regulator with 0-100psi output

NOTE: each type of cylinder has a different CGA connection to the regulator (CGA = Compressed Gas Association). Air is typically CGA 590 or 346. Helium and nitrogen are CGA 580. Hydrogen and argon-methane are CGA 350.

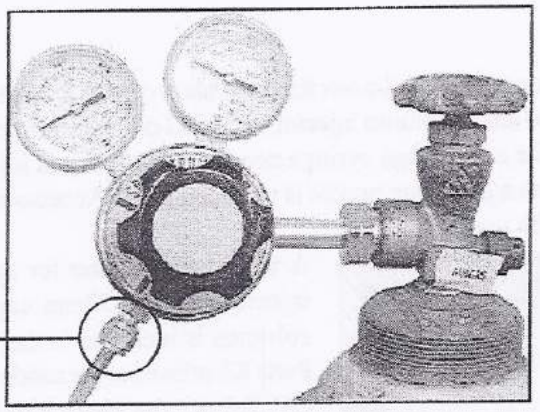
Gas line installation kits that include everything you need are available from SRI:

8600-C590	Air gas line kit (with both CGA 590 and 346 inlet fittings)
8600-C580	Helium/nitrogen gas line kit
8600-C350	Hydrogen/argon-methane gas line kit (the hydrogen CGA is equipped with a flow restrictor to limit the escape of gas in the event of a leak)

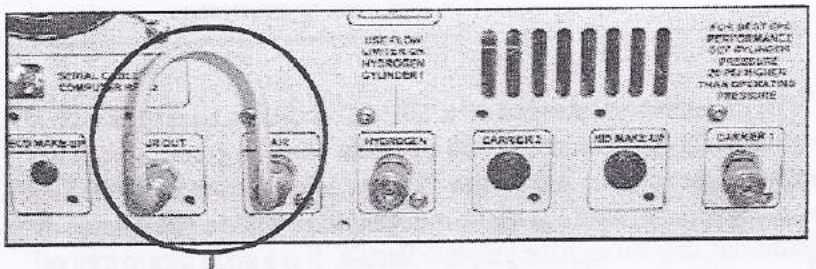
These kits include everything in the list of recommended supplies above, plus a tubing cutter. Each regulator is supplied with a 1/8" Swagelok fitting for easy connection to the copper tubing.

2. Using the appropriate CGA connection as described above, attach the regulator securely to the gas cylinder.

3. Secure one end of the 1/8" copper tubing to the regulator with a Swagelok nut and ferrule. Cut the tubing to the desired length before connecting it to the GC. Make sure to leave it long enough to allow you to move your GC around your work area.



4. If you don't already filter your gas, install gas line filter(s) in the gas line(s) where it is convenient to replace when needed.



5. Connect the gas or gases to the inlets on the left-hand side of the GC as labeled.

NOTE: the GC shown here is equipped with a built-in air compressor. When using the internal air compressor instead of cylinder air, a jumper tube is secured to the air inlet and outlet. If you ordered your GC with an air compressor, it is shipped with the jumper tube in place as shown.

Quick Start GC Installation Guide

("Gas Installation & Connection" *continued*)

GAS FLOW RATES

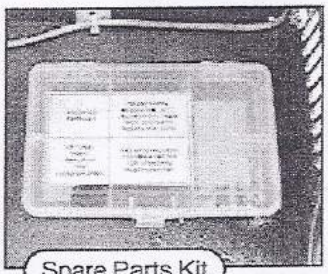
CARRIER 1:		7	PSI =	10	ml/min
CARRIER 2:			PSI =		ml/min
P&T PURGE:			PSI =		ml/min
HYDROGEN 1:	FID	21	PSI =	25	ml/min
HYDROGEN 2:			PSI =		ml/min
AIR 1:	FID	9	PSI =	250	ml/min

6. The pressure that correlates with the flow rate for the column, make-up gases, and detector supplies is labeled on the right-hand side of the GC. For best EPC performance, set the incoming gas pressure(s) 15-20psi higher than the operating pressure listed on the right-hand side of the GC.

II. Column Installation

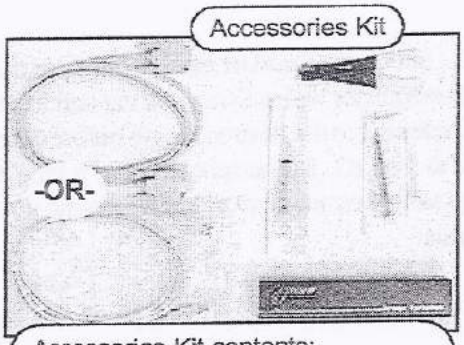
1. If you ordered a column with your GC, it is shipped installed in the column oven and you can skip this section. Otherwise, open the GC lid and the column oven lid.

2. These instructions will cover the installation of a 0.53mm capillary column into an on-column injector. The SRI on-column injector is designed for a 26 gauge syringe needle; a 10µL liquid injection syringe with a 26 gauge needle is included in the Accessories Kit shipped with your GC.



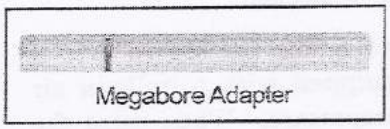
Spare Parts Kit

A megabore adapter for syringe injection onto 0.53mm capillary columns is included in the Spare Parts Kit affixed to the inside of the GC lid on the right-hand rear corner.



Accessories Kit

Accessories Kit contents:
- 6' Serial *or* USB cable
- Tubing cutter
- 10µL liquid injection syringe
- 1mL gas injection syringe & needle
- 3mL leak check syringe



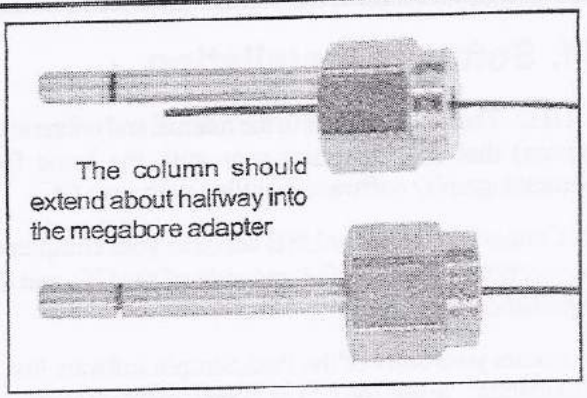
Megabore Adapter

3. The megabore adapter is a 1" x 1/8"OD stainless steel tube with a perpendicular gash cut into it, and a conical entry to guide the syringe needle into the column. A 0.53mm capillary column connects to the SRI on-column injector with a graphite reducing ferrule and a 1/8" Swagelok nut. Insert one end of the column through the nut, then through the graphite ferrule. It is a good idea to trim off about one inch of the column to avoid possible peak tailing from any graphite shavings left behind after inserting the column through the ferrule; make sure the cut is clean, with no jagged edges.

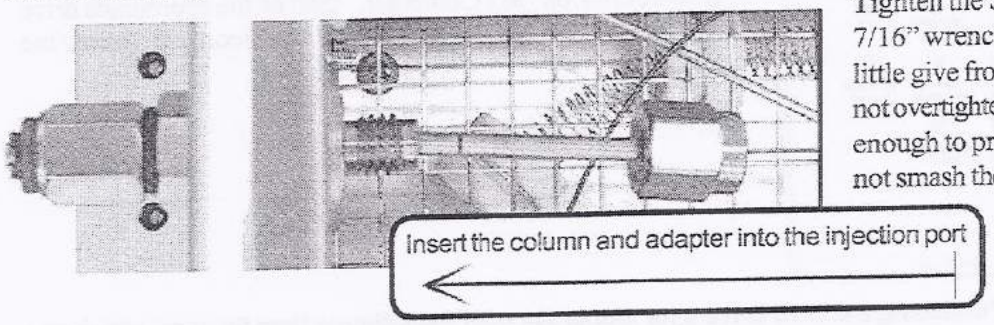
Quick Start GC Installation Guide

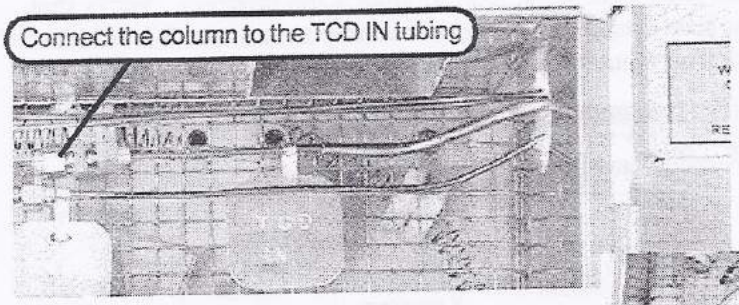
("Column Installation" *continued*)

4. Insert the column end with the graphite ferrule and Swagelok nut about halfway into the megabore adapter and tighten it with the nut and ferrule.



5. After inserting the column into the adapter, insert the column and adapter together into the injection port. Tighten the Swagelok nut with a 7/16" wrench. You should feel a little give from the ferrule, but do not overtighten it. You want it tight enough to prvent leakage, but do not smash the ferrule.

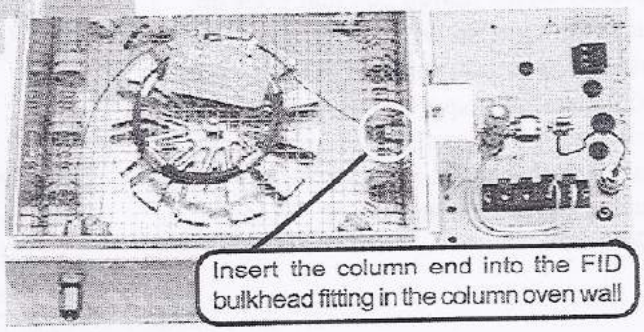




6. Slide another 1/8" Swagelok nut and graphite ferrule over the other end of the column. For a TCD detector, connect the nut to the fitting labeled "TCD IN" in the column oven.

For an FID detector, leave about 1" of the column protruding through the nut and ferrule. Insert the column into the FID bulkhead fitting in the column oven wall and tighten the Swagelok nut.

Please see "Analytical Column Installation" in the INSTALLATION section of your manual for more detailed information on column installation.



Quick Start GC Installation Guide

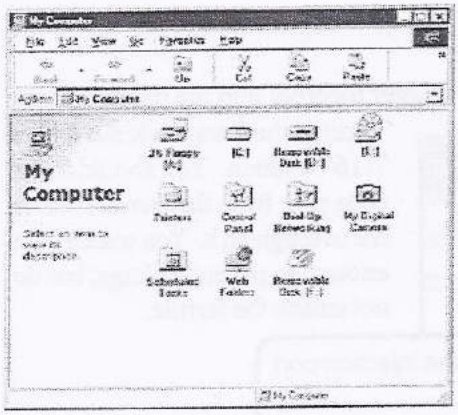
III. Software Installation

NOTE: There are tutorials in the manual and online at www.srigc.com (click on the "Download Our Documents" button) that will acquaint you with the basic functions of the PeakSimple chromatography software included with your GC.

1. Connect the serial or USB cable to your computer and the GC. The serial port connection is on the left-hand side of the GC, and the USB connection is on the right-hand side.

2. Locate your copy of the PeakSimple software just inside the front cover of your SRI manual. Insert the CD or floppy disks into your computer's appropriate drive.

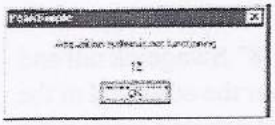
SRI Manual



3. Double click on "My Computer," then on the appropriate drive to open it. Double click on the "setup.exe" icon, and follow the instructions.

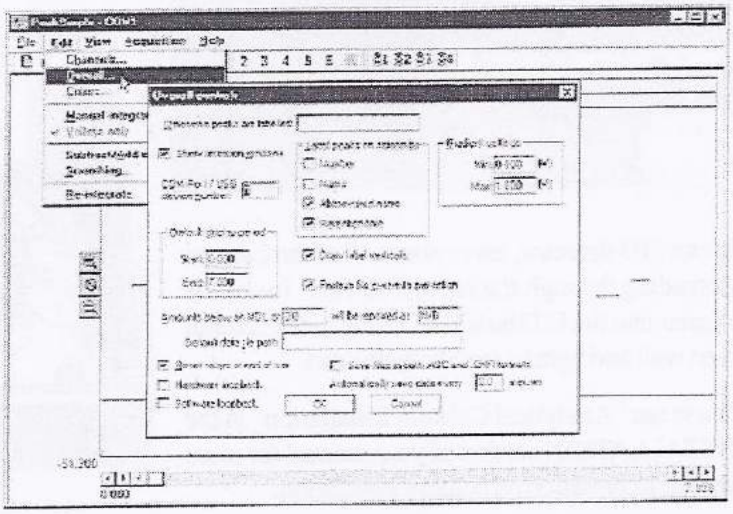
Setup

4. For USB, refer to "**Installing the USB Drivers for Model 302 USB PeakSimple Data System**" which you will find immediately behind these instructions in your manual, or online at www.srigc.com. Return to step #5 below when you are finished installing the USB drivers. For serial port, proceed to the next step.



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

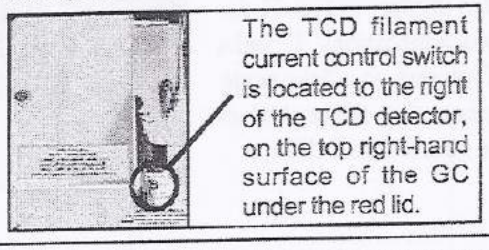
6. Open the Edit menu and choose Overall. In the dialog box that pops up, enter the number of the COM port to which you have connected the GC. For USB, enter the unique USB device number that is printed on your PeakSimple disk(s), and on the back of the GC. It is a 4-digit number that always begins with "5" (5093, 5276, etc.).



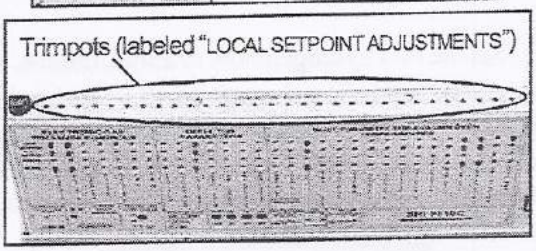
IV. Detector Activation

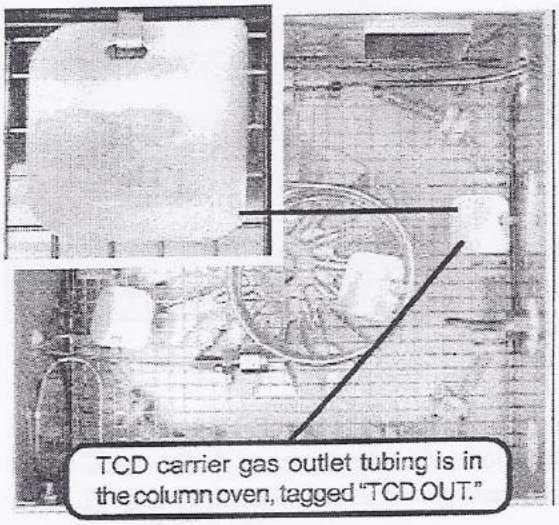
IMPORTANT: If you have a pre-configured GC system, please see the manual section for instructions on operating procedures. The manual is organized into sections with labeled tabs. In addition to preconfigured GCs, there are sections on detectors, injectors, autosamplers, valves, and more.

A. TCD Detector



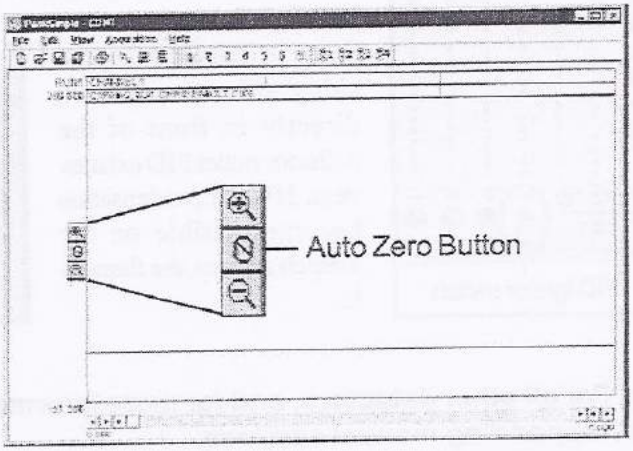
1. Your GC power should still be ON, and the filaments should still be OFF. The TCD oven is set to 150°C at the factory. It is adjustable by turning the trimpot while observing the TCD CELL LOCAL SETPOINT temperature on the LED display. The trimpots are located on the top edge of the GC front control panel. Allow the TCD to reach desired operating temperature and stabilize.





2. TCD filaments will be damaged or destroyed if current is applied in the absence of flowing carrier gas. Therefore, always verify that carrier gas is exiting the TCD carrier gas outlet before energizing the TCD filaments. The TCD carrier gas outlet tubing is in the column oven, labeled "TCD OUT." Place the end of the tubing in some liquid; if no bubbles are exiting the tube, there is a flow problem. *DO NOT* turn the TCD current ON if you cannot detect carrier gas flow. A filament protection circuit prevents filament damage if carrier gas pressure is not detected at the GC, but it cannot prevent filament damage under all circumstances. Correct any lack of carrier gas flow before proceeding.

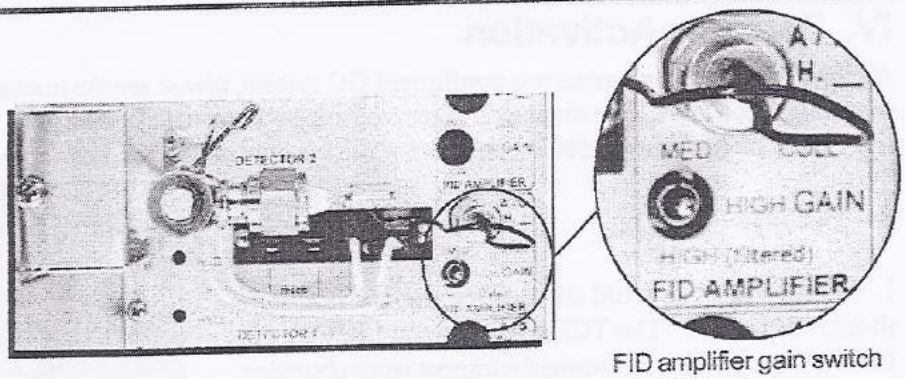
3. With the TCD filaments still OFF, zero the data system signal by clicking on the Auto Zero icon on the left side of the chromatogram. Switch the TCD current to LOW. The data system signal's deflection should not be more than 5-20mV for a brand-new TCD detector. There is also a HIGH current TCD filament setting, but to avoid filament damage, we recommend you use only the LOW setting until you are familiar with your GC and TCD detector.



Quick Start GC Installation Guide

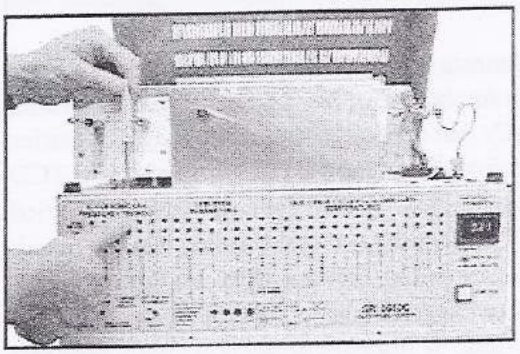
B. FID Detector

1. Set the FID amplifier gain switch to HIGH for most applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM.

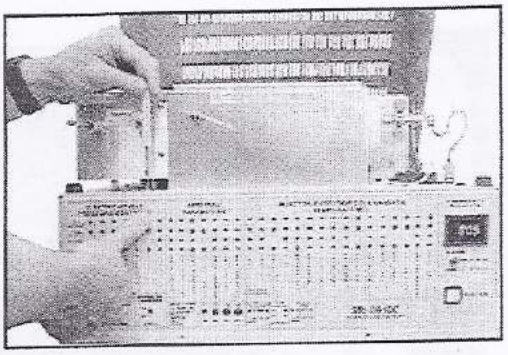


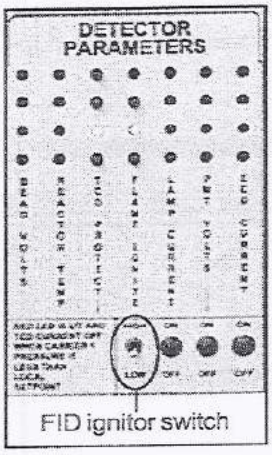
FID amplifier gain switch

2. Set the FID hydrogen flow to 25mL/minute, and the FID air to 250mL/minute. The approximate pressures required for this flow through your GC are labeled on the right-hand side of the GC chassis. In most cases, the pressure will have been set correctly at the factory. Check the hydrogen and air flow settings by pressing the LOCAL SETPOINT button while observing the LED display. The gas flow settings are adjusted using the trimpots on the top edge of the GC front control panel.



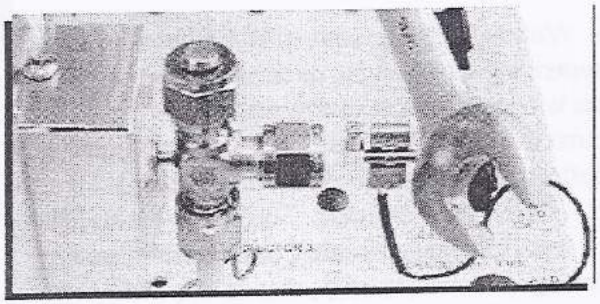
Turn the trimpot while holding down the "LOCAL SETPOINT" button until you read your desired setting in the LED display.





FID ignitor switch

3. Ignite the FID by holding the ignitor switch up for a couple of seconds, until you hear a small POP. The ignitor switch is located on the front panel of your GC under the "DETECTOR PARAMETERS" heading, with a vertical label reading "FLAME IGNITE." Verify that the flame is lit by holding the shiny side of a wrench directly in front of the collector outlet/FID exhaust vent. If water condensation becomes visible on the wrench surface, the flame is lit.



C. For all other detectors, and for more information on the TCD and FID, please see the corresponding manual sections.

V. Inject Your Sample

NOTE: If you are injecting with a Purge & Trap, TO-14, or Headspace concentrator, a thermal desorber, an autosampler, or any of the heated on-column injectors (PTV, Split/Splitless, etc.), please see the corresponding manual section for operating procedures.

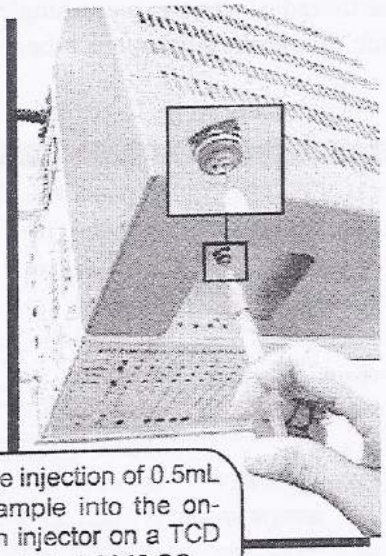
A. Syringe Injection

1. Enter a temperature program for the column oven. The temperature program is determined by the sample and the goals of the analysis.

2. For gas samples, fill the 1mL gas syringe with 0.5-1mL. For liquid samples, fill the 10µL liquid syringe with 1µL, removing the bubbles before injecting.

The Auto Zero button is on the left side of the chromatogram window.

3. Click on the Auto Zero button to zero the data system signal. Hit the computer keyboard spacebar.



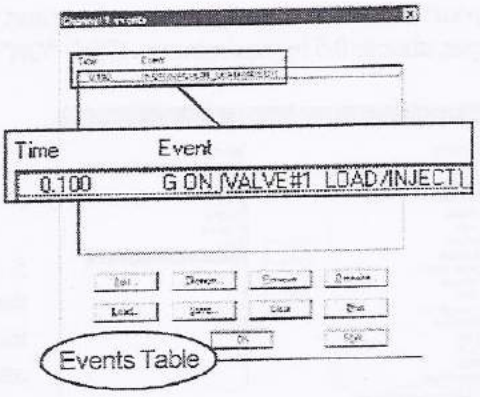
Syringe injection of 0.5mL gas sample into the on-column injector on a TCD equipped Model 310 GC

4. Pierce the septum in the on-column injector with the syringe needle. Insert the needle straight into the on-column injector port; avoid bending the needle. Depress the syringe plunger to inject the sample, then withdraw the syringe. For the best and most consistent results, use an easily reproducible injection technique with quick, smooth movements.

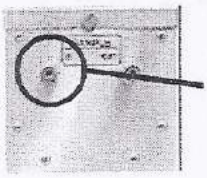
B. Valve Injection

1. Set the valve oven temperature between ambient and 175°C using the trimpot on the top edge of the front control panel. Enter a temperature program for the column oven.

2. Enter an event program to automatically inject the contents of the valve sample loop. The valve is usually in the LOAD position (default), during which Relay G is OFF. When relay G is activated, the valve is rotated to the INJECT position, in which the carrier gas stream sweeps the contents of the sample loop onto the column(s). Set the valve to INJECT (Relay G ON) 0.1 minutes into the run unless you have specific run parameters that require different timing.



Events Table

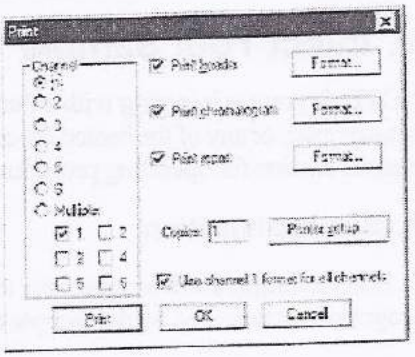


3. Sample is injected into the bulkhead fitting labeled "SAMPLE IN" on the front of the valve oven. The fitting is equipped with a 1/8" Swagelok nut for easy connection of sample streams.

4. Press the computer keyboard spacebar to initiate the run. The valve will automatically rotate to the INJECT position at 0.1 minutes (or whatever time you entered in the Events Table).

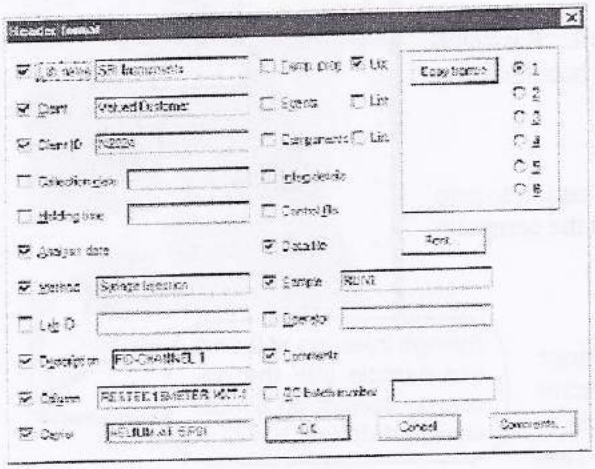
Quick Start GC Installation Guide

VI. Print Your Chromatogram

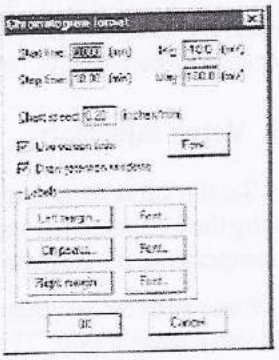


1. Choose File / Print from the main menu bar.

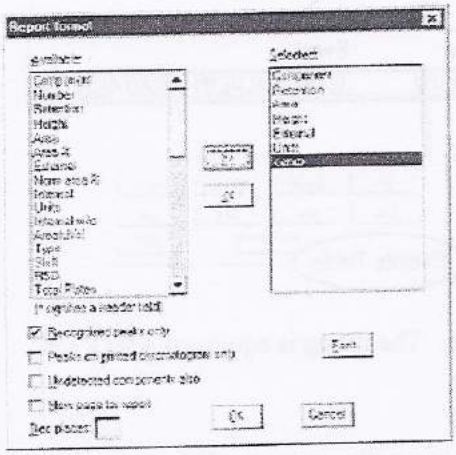
2. In the Print screen, designate which channel(s) you want printed. Use the radio buttons to pick a single channel, or select "Multiple:" and click the checkboxes to select the channels you want to print.



3. Click the checkbox to select "Print header," then click on the "Format..." button to set up the Header. The Header is printed above the chromatogram on the page, and can contain such information as the analysis date, the sample and injection type, column and carrier gas used, client and lab names, and any special comments about the analysis that you want printed with the chromatogram. Click "OK" when finished formatting your header. The Print screen is still open.



4. In the Print screen, click the checkbox to select "Print chromatogram," then click on the "Format..." button. Choose "Use screen limits" to print the chromatogram as you see it onscreen. You can also choose the chart speed, which determines the number of inches per minute displayed in the chromatogram timeline. For example, if your chromatogram is 10 minutes long and you want it to occupy 5 inches on the paper, choose 0.5 inches/minute. Click "OK" when finished.

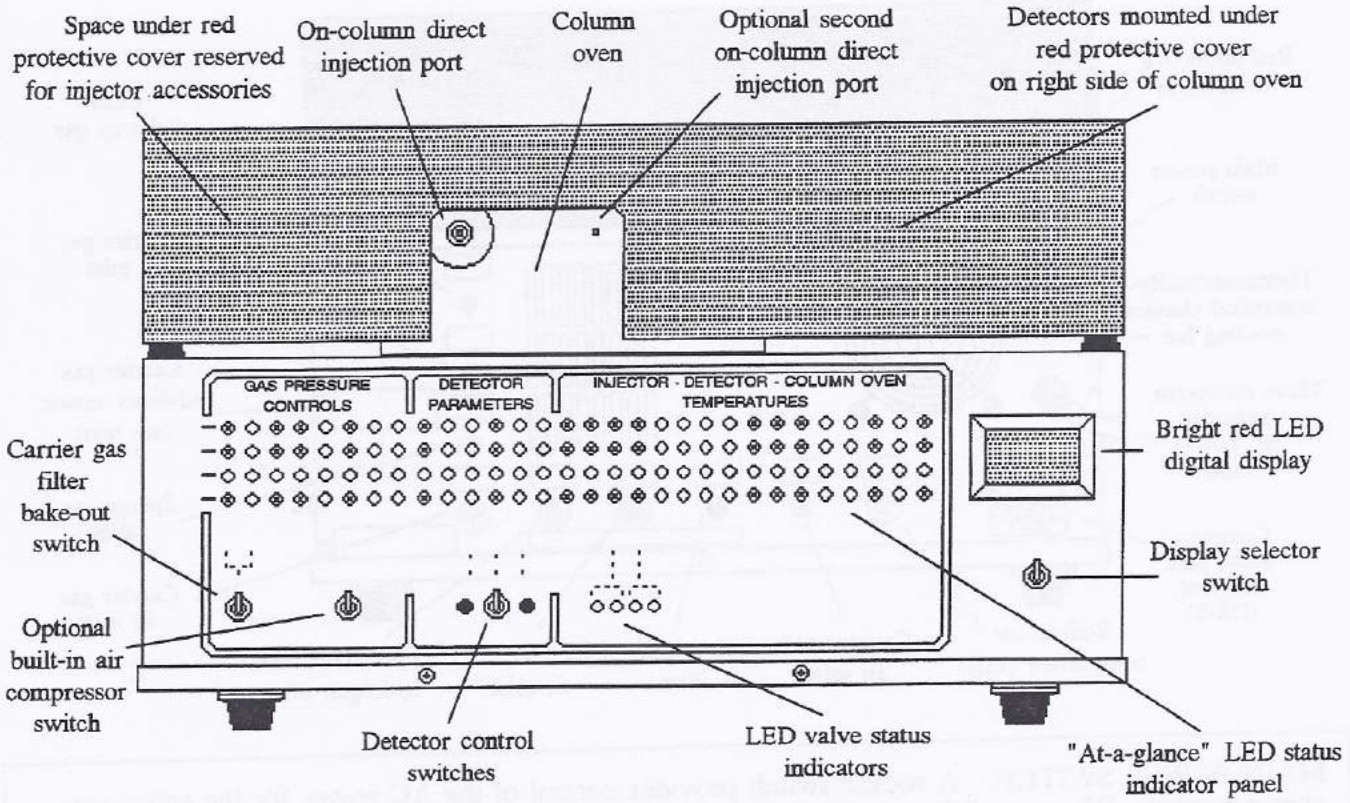


5. In the Print screen, click the checkbox to select "Print report," then click on the "Format..." button to choose the data that will be included in the report at the bottom, such as the component name, retention time, peak area and height, etc. Click "OK" when finished.

6. Now that your chromatogram is ready to print, click on the Print button in the Print screen.

Chapter: INSTALLATION

Topic: FRONT PANEL ORIENTATION - 8610C GC CHASSIS



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

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

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

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

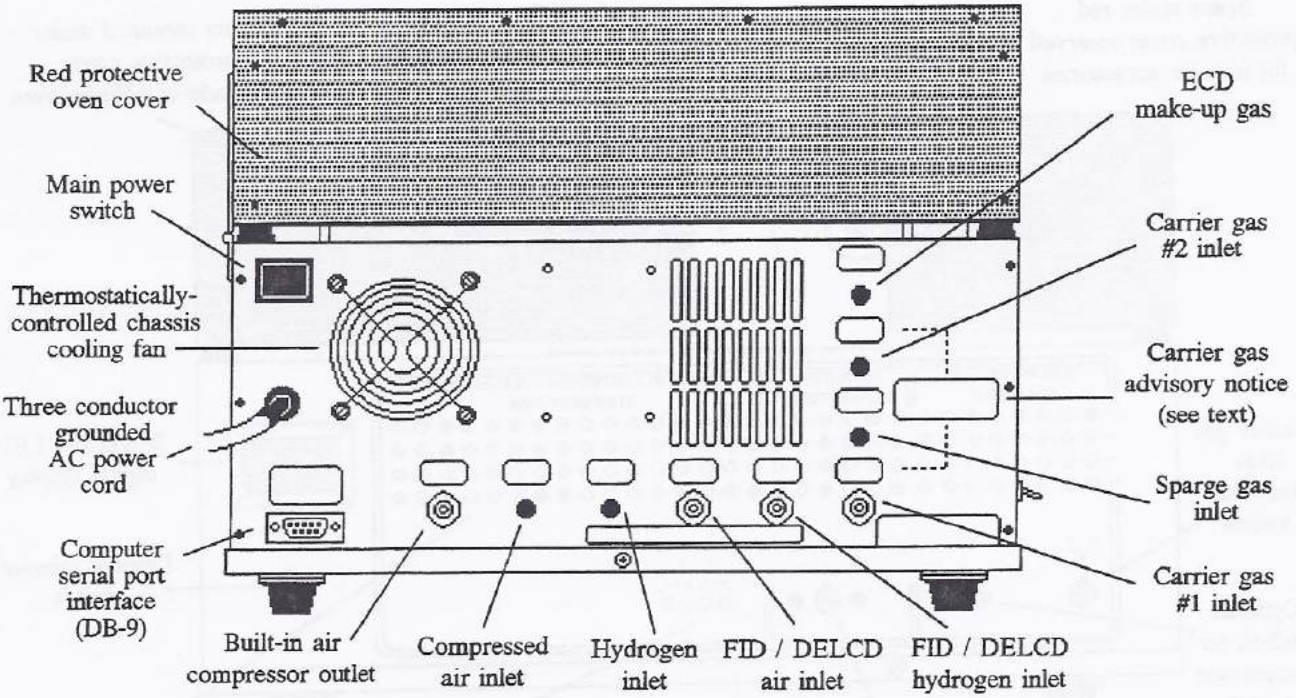
INJECTOR PORT - A direct on-column inject port is provided, and supports the use of both packed and capillary analytical columns. A capillary column adapter is provided for installation of wide-bore capillary columns. Optional heated injection ports and heated split-splitless injection ports are available.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

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

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

Chapter: INSTALLATION

Topic: LEFT SIDE PANEL ORIENTATION - 8610C GC CHASSIS



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

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

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

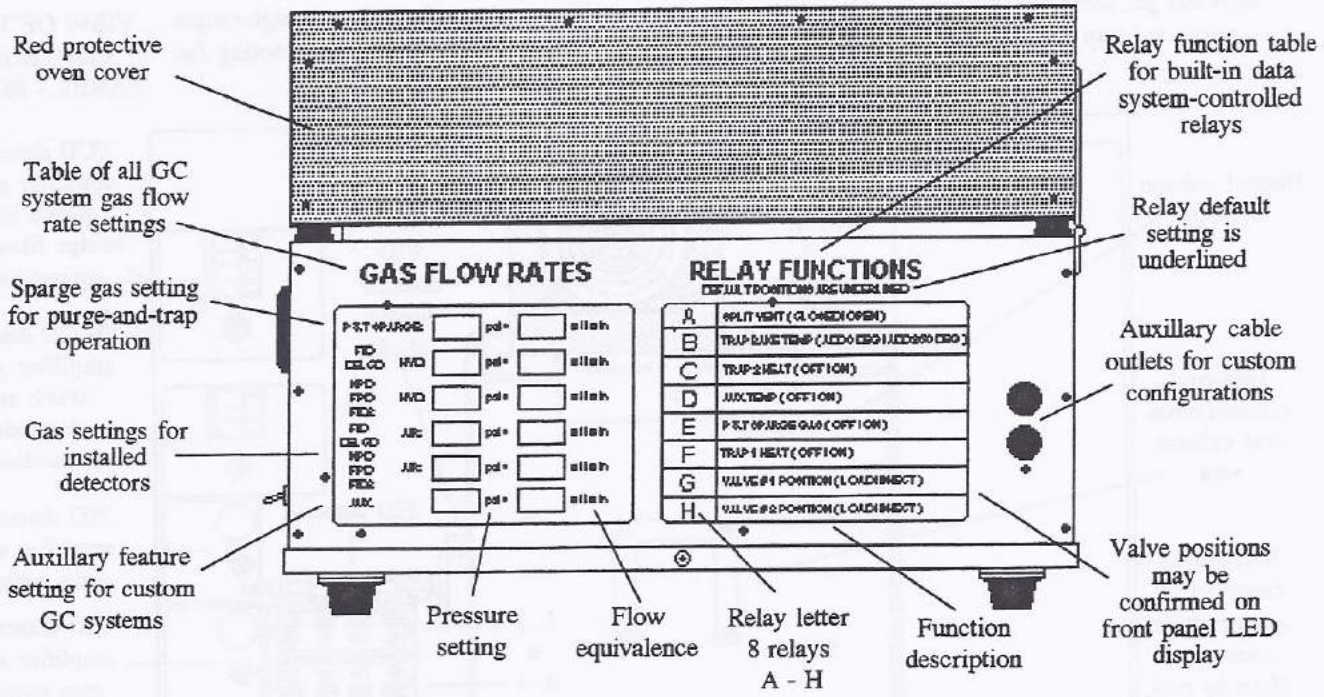
GAS INLETS: Stainless steel gas bulkhead fittings are provided for connection of all system gases. Separate inlets are provided for sparge, FID, DELCD, and ECD gases. If the GC is equipped with a built-in air compressor, a compressed air outlet is also provided.

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

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

Chapter: INSTALLATION

Topic: RIGHT SIDE PANEL ORIENTATION - 8610C GC CHASSIS



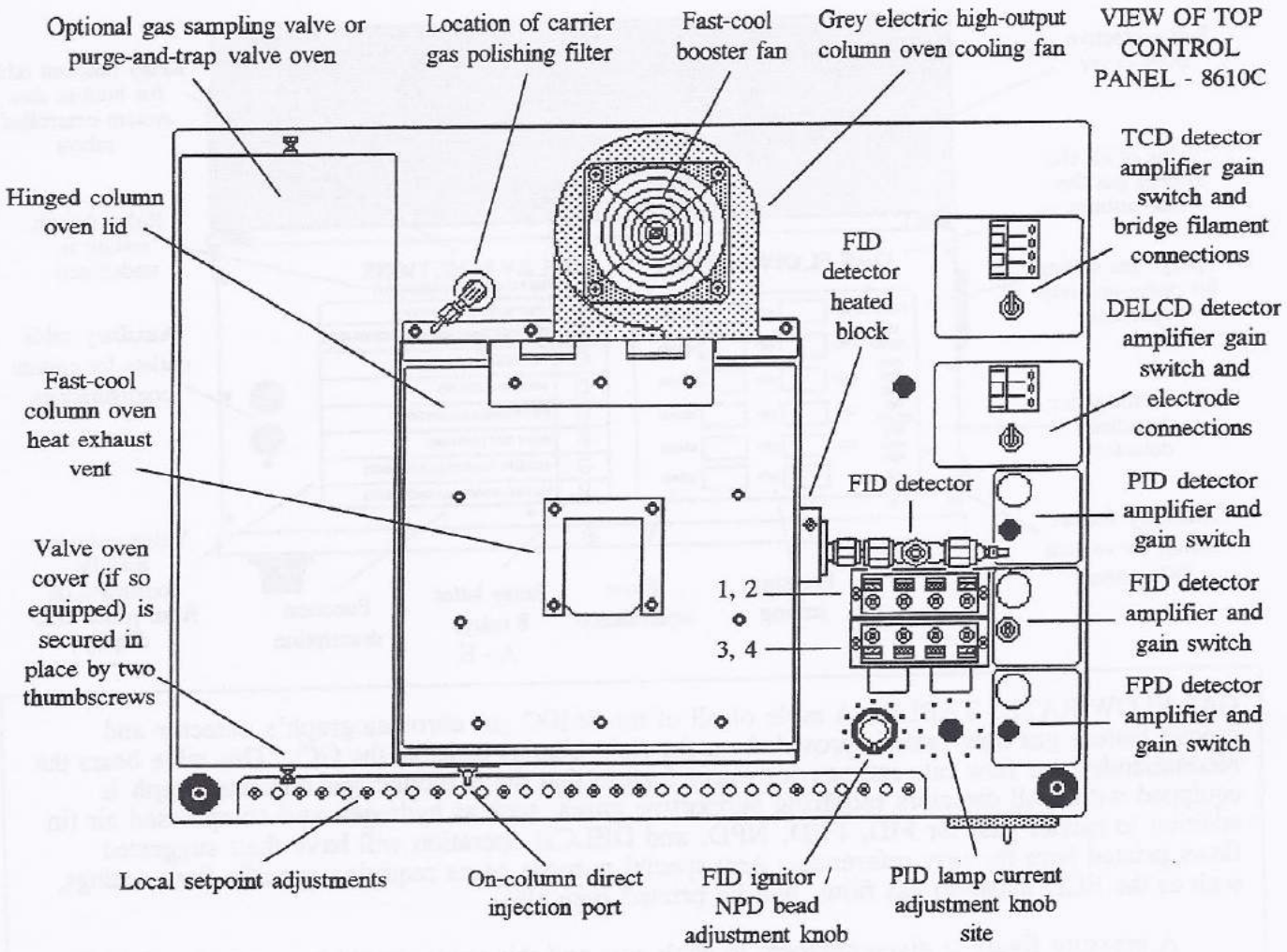
GAS FLOW RATES TABLE: A table of all of the 8610C gas chromatograph's detector and special feature gas flow rates is provided on the right chassis panel of the GC. This table bears the recommended gas flow rate settings for every feature that the particular gas chromatograph is equipped with. All detectors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

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

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

Chapter: INSTALLATION

Topic: Top Control Panel Orientation - 8610C GC Chassis



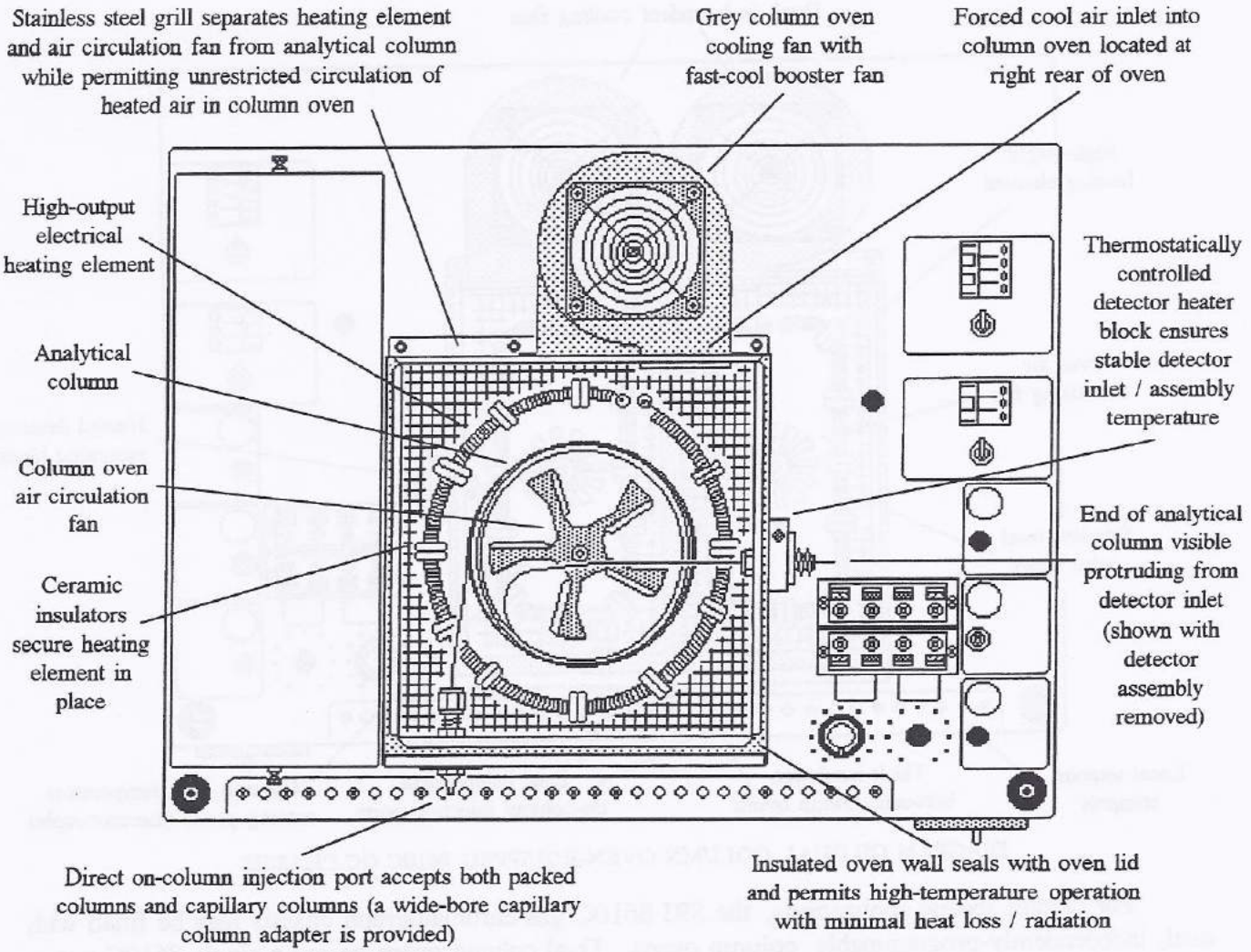
The 8610C top control panel is divided into four main areas:

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

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

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

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



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

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

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

Chapter: INSTALLATION

Topic: Dual Oven 8610C Gas Chromatograph Chassis

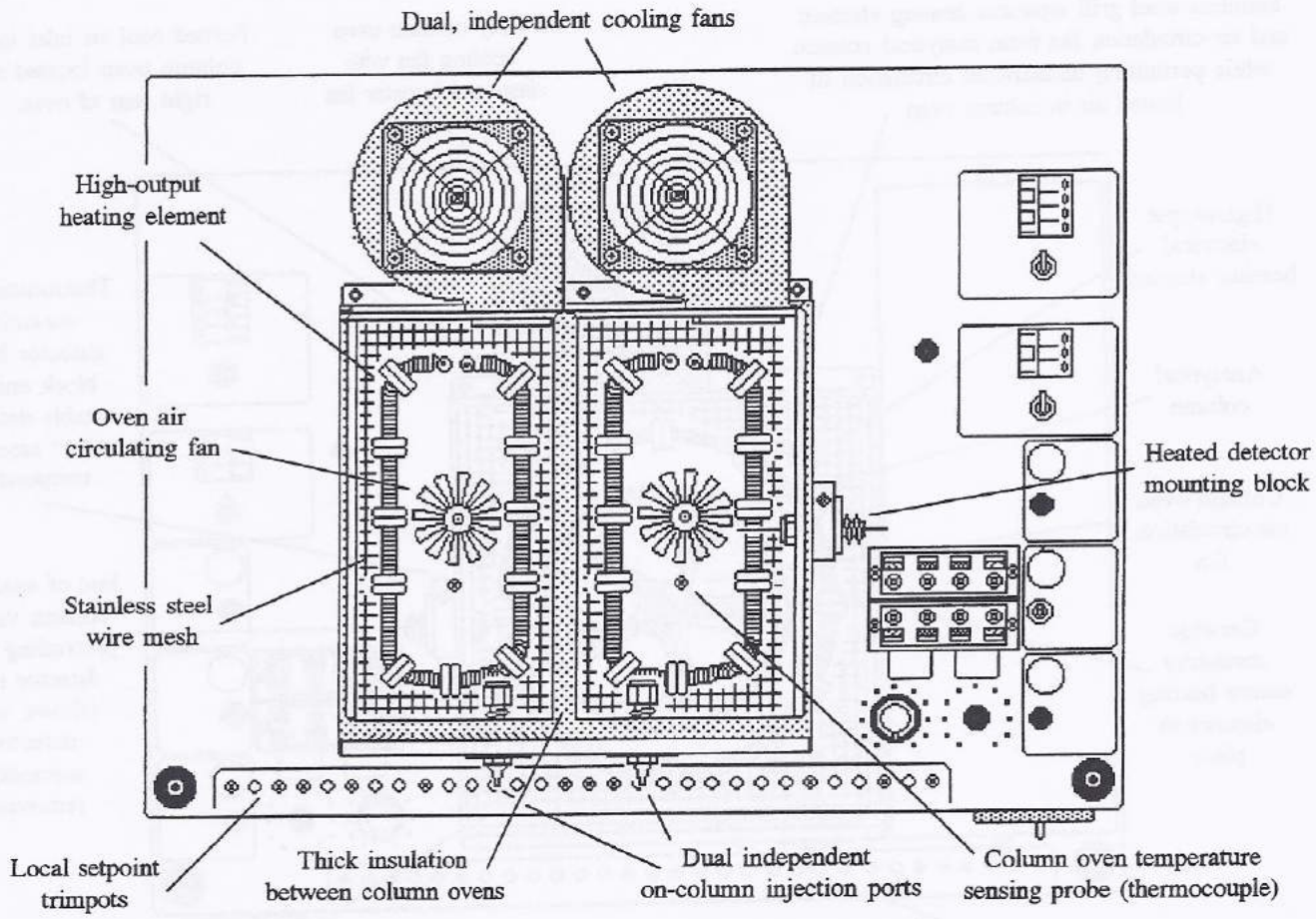


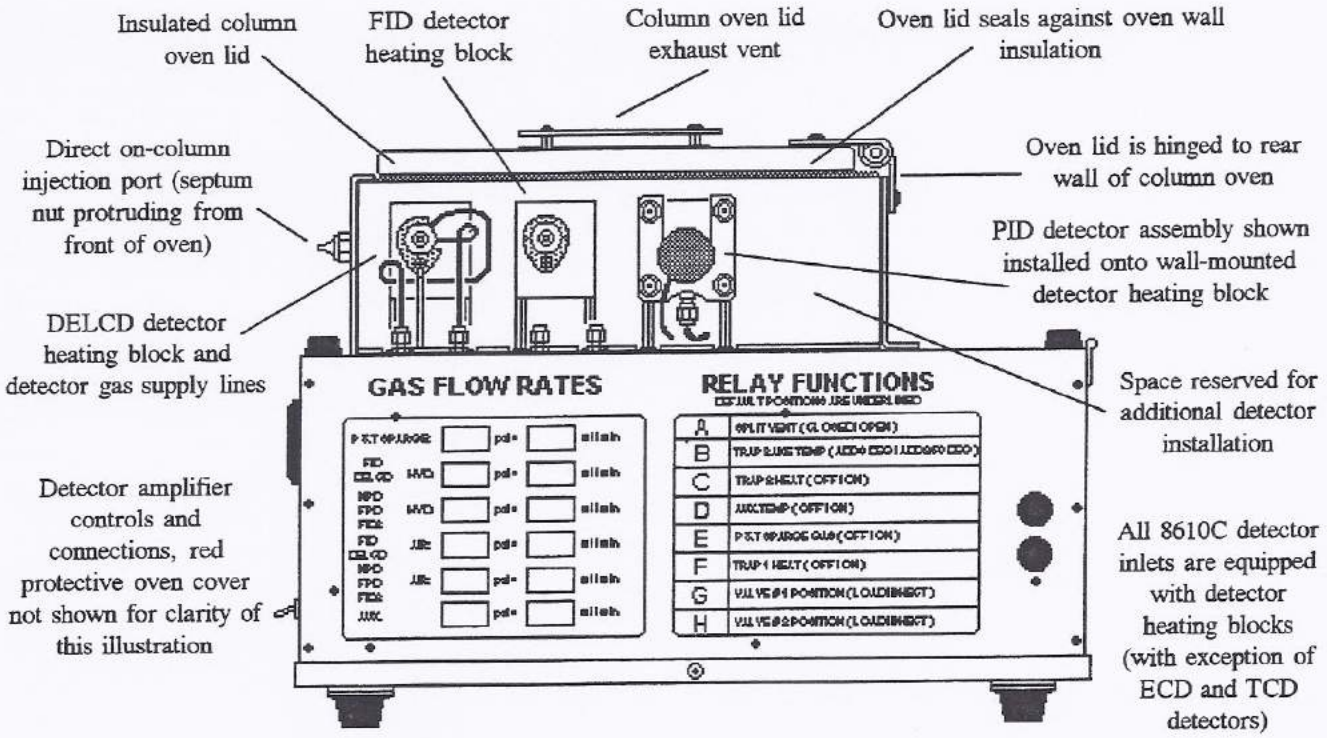
DIAGRAM OF DUAL COLUMN OVEN-EQUIPPED 8610C GC CHASSIS

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

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

Chapter: INSTALLATION

Topic: Detector Mounting Platforms on Column Oven Wall



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

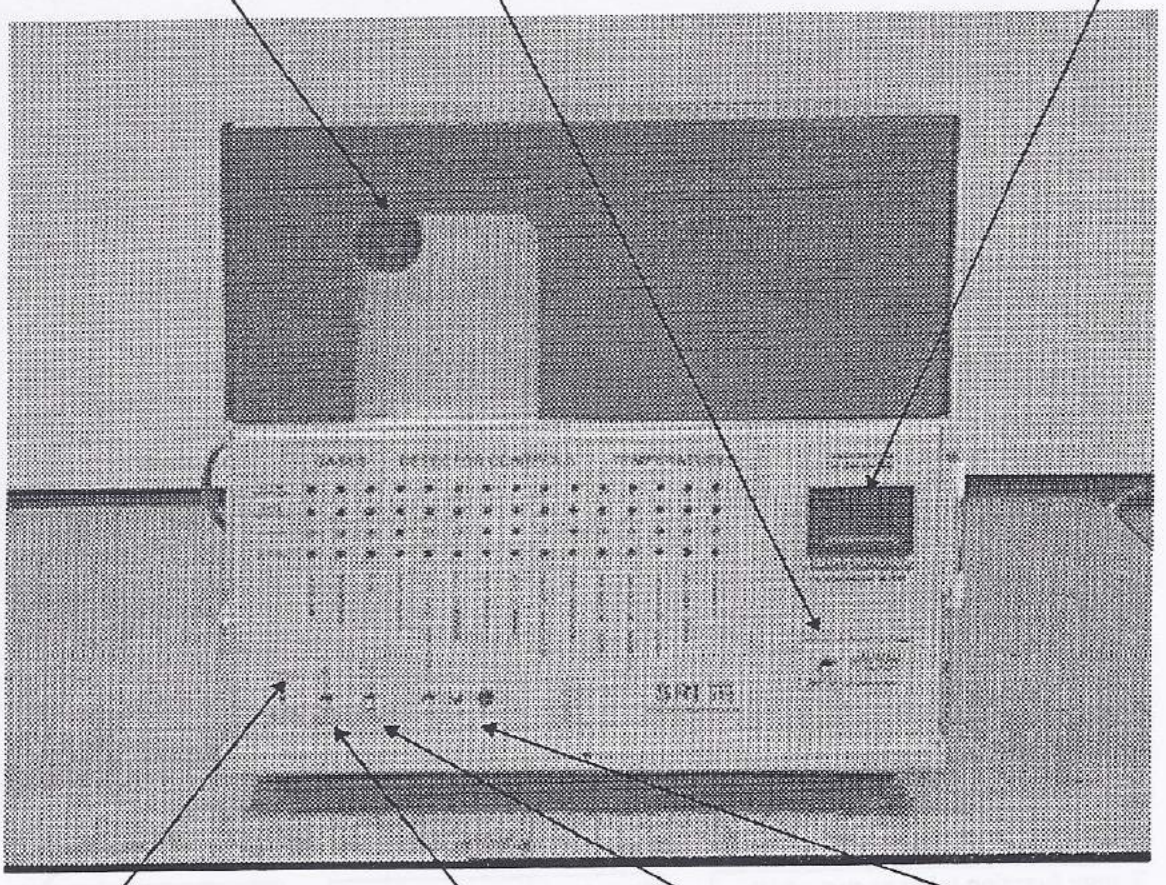
All 8610C gas chromatographs are equipped with a thermostatically-controlled heating block mounted at the base (or inlet) of each detector. This new feature permits the user to preset the temperature of the detector inlet. This is convenient for methods prescribing a specific detector operating temperature,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

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

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

Chapter: MODEL 310 GC CHASSIS

Topic: FRONT PANEL ORIENTATION



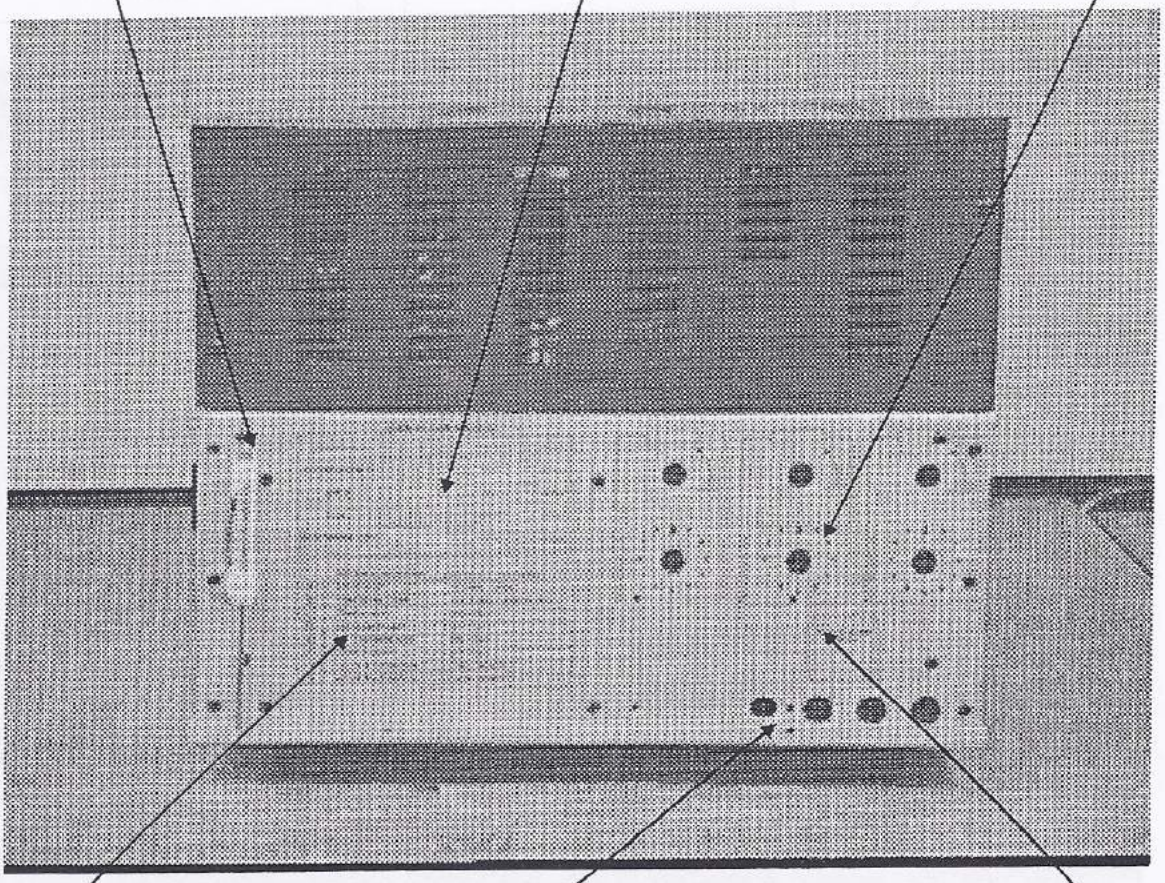
Chapter: MODEL 310 GC CHASSIS

Topic: RIGHT SIDE PANEL ORIENTATION

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

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

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



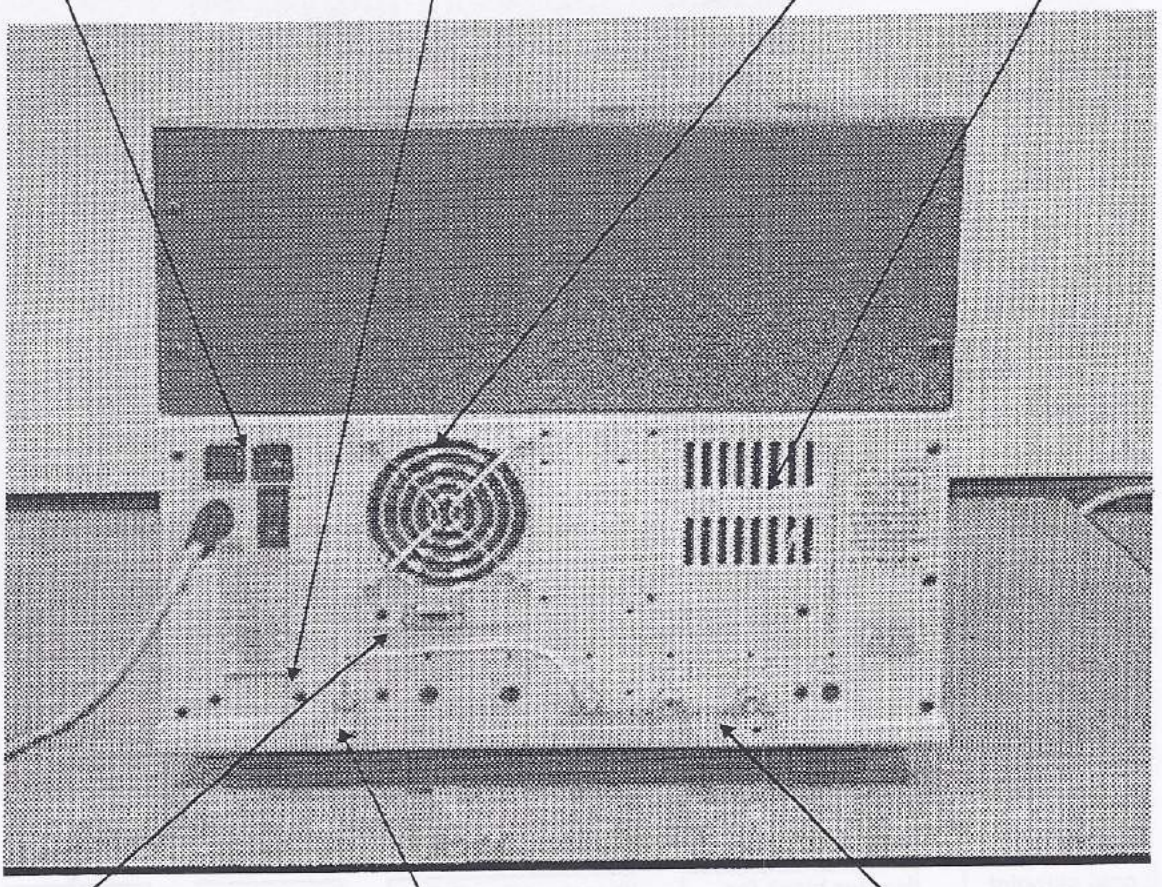
Relay function table shows which function each of the 8 (A-H) data system relays is assigned. Depending on GC configuration, some relays may have no function.

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

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

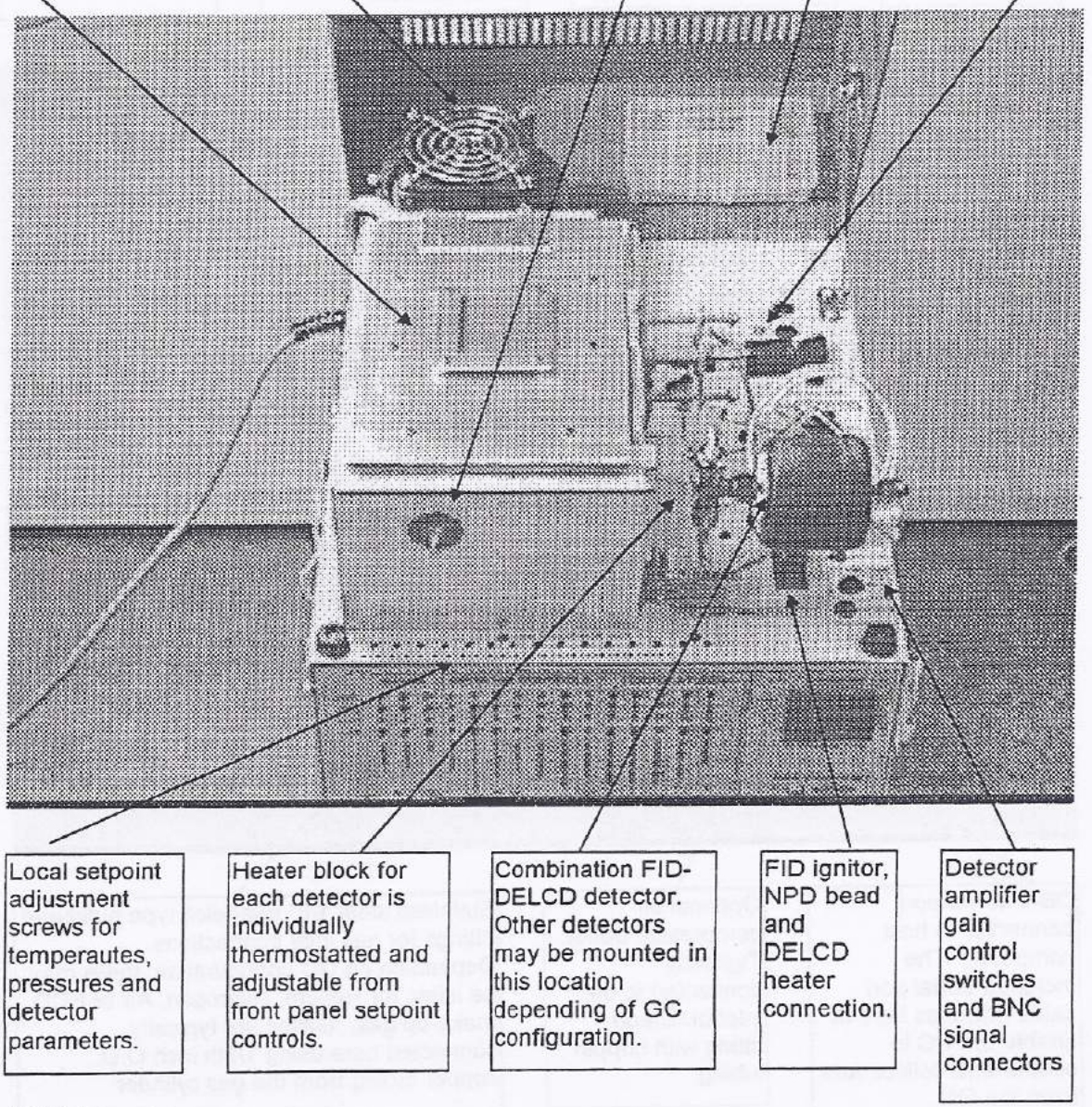
Chapter: MODEL 310 GC CHASSIS

Topic: LEFT SIDE PANEL ORIENTATION



Chapter: MODEL 310 GC CHASSIS

Topic: TOP PANEL ORIENTATION



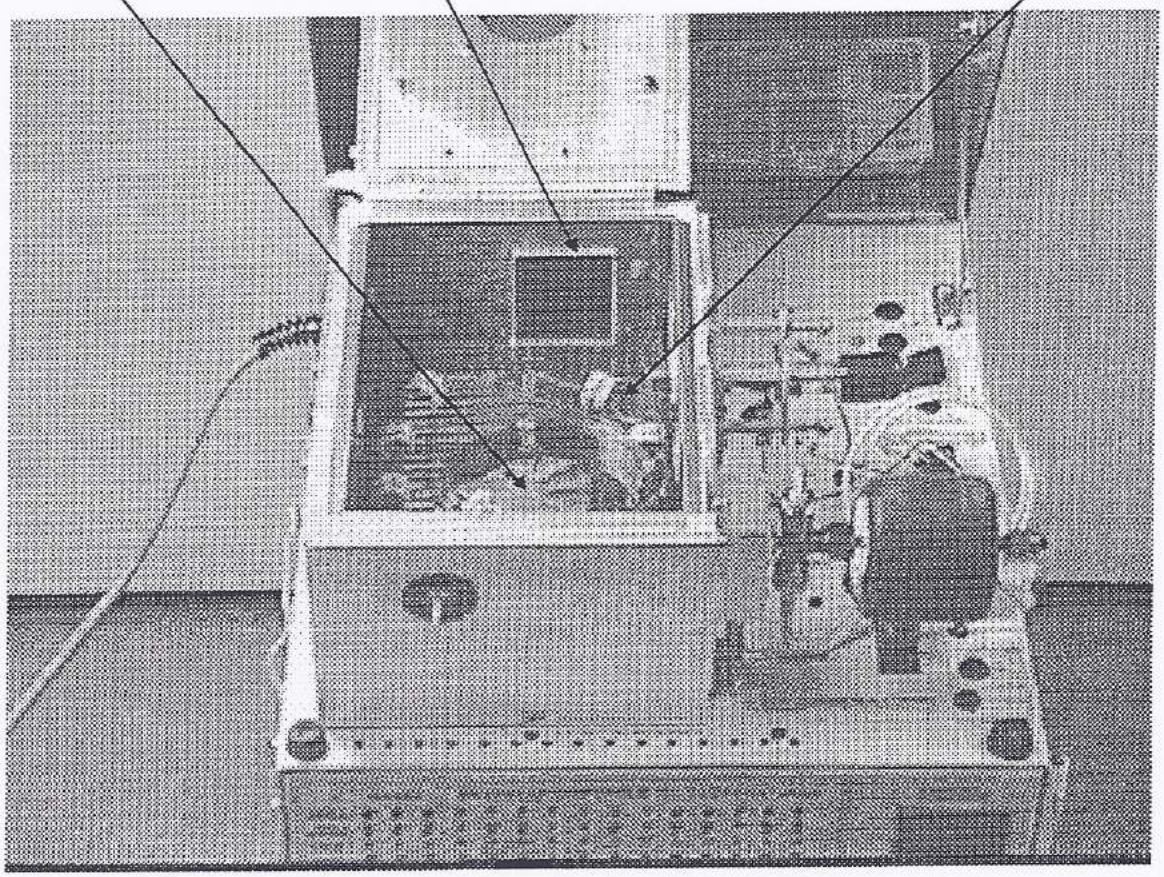
Chapter: MODEL 310 GC CHASSIS

Topic: COLUMN OVEN INTERIOR

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

Duct for cooling air from oven cooling fans.

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



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

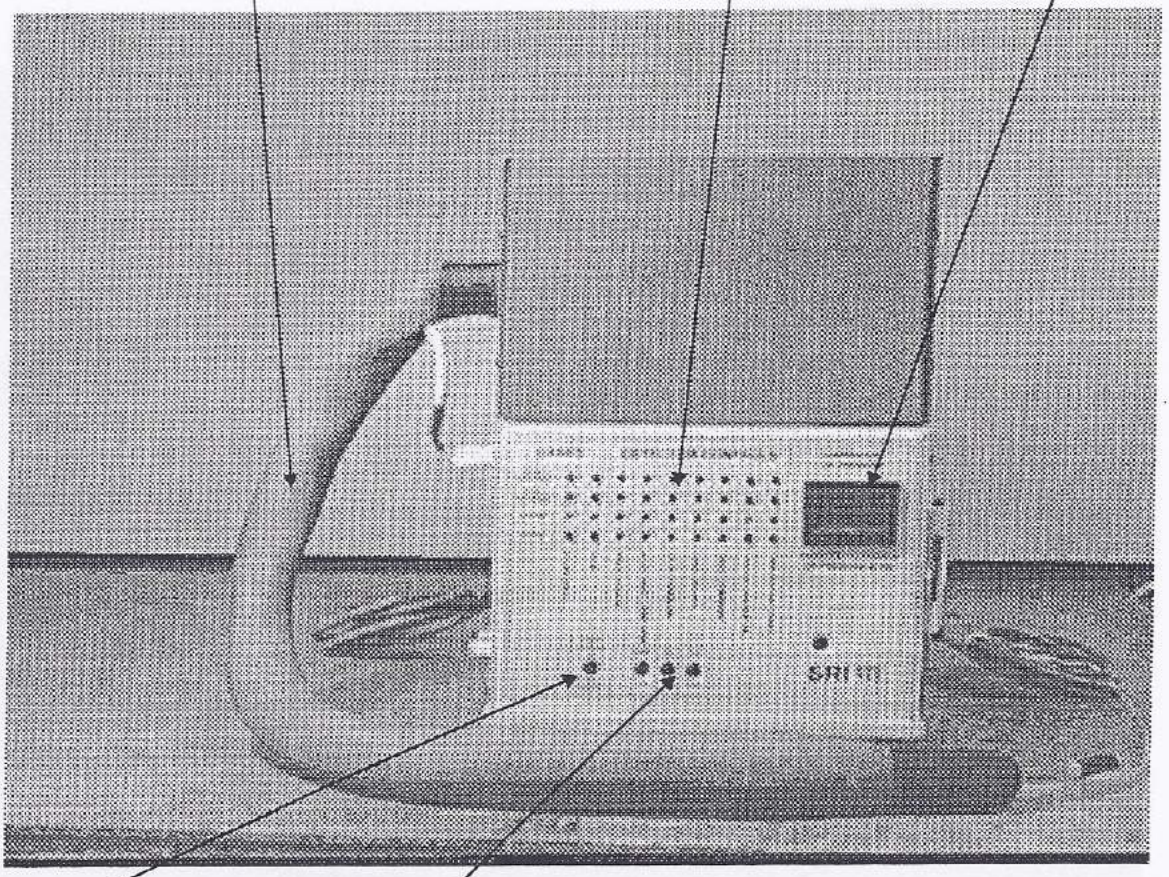
Chapter: MODEL 110 GC CHASSIS

Topic: FRONT PANEL ORIENTATION

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

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

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



On/off switch for optional built-in air compressor

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

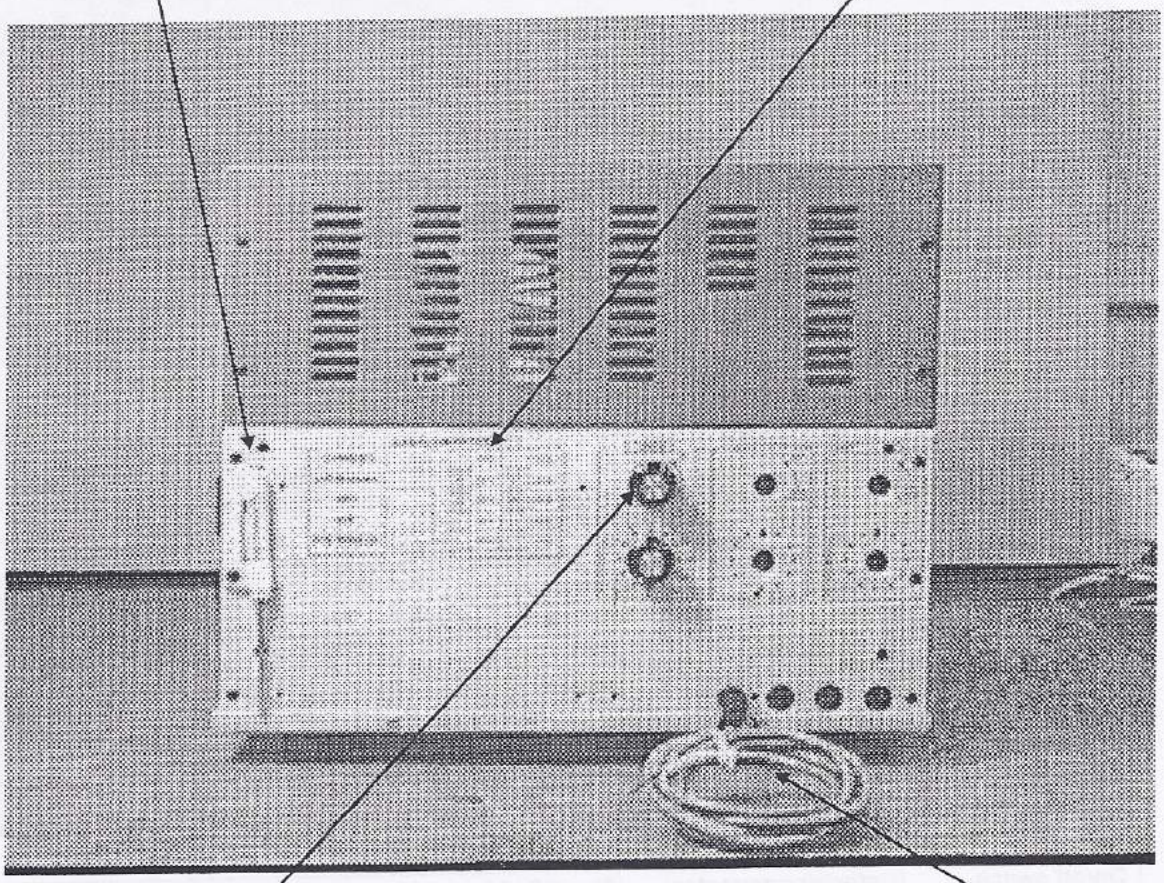
The SRI Model 110 chassis is used primarily as a mounting platform for stand-alone GC detectors. The heated transfer line makes it easy to connect the detector to the host GC since only a small opening into the host GC's column oven is required. User's should note that because the heated transfer line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

Chapter: MODEL 110 GC CHASSIS

Topic: RIGHT SIDE PANEL ORIENTATION

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

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



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

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

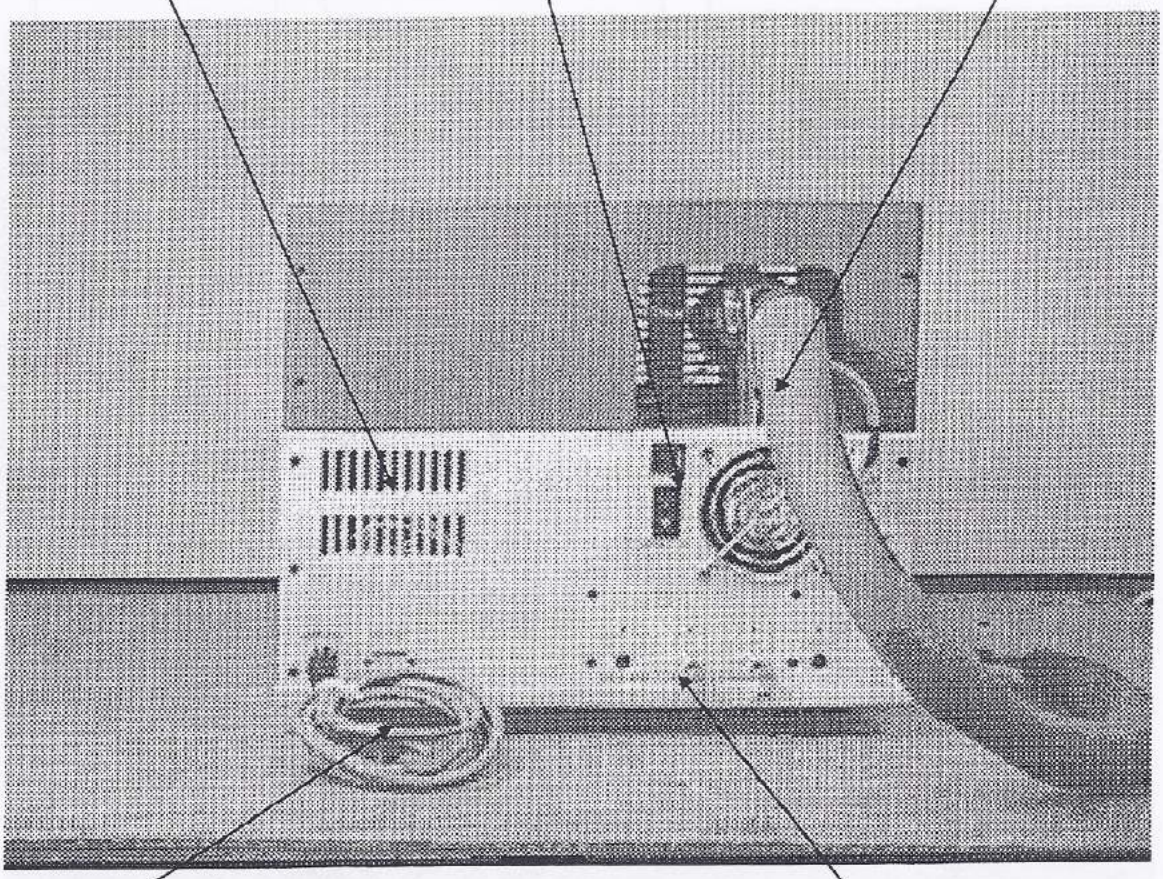
Chapter: MODEL 110 GC CHASSIS

Topic: LEFT SIDE PANEL ORIENTATION

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

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

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



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

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

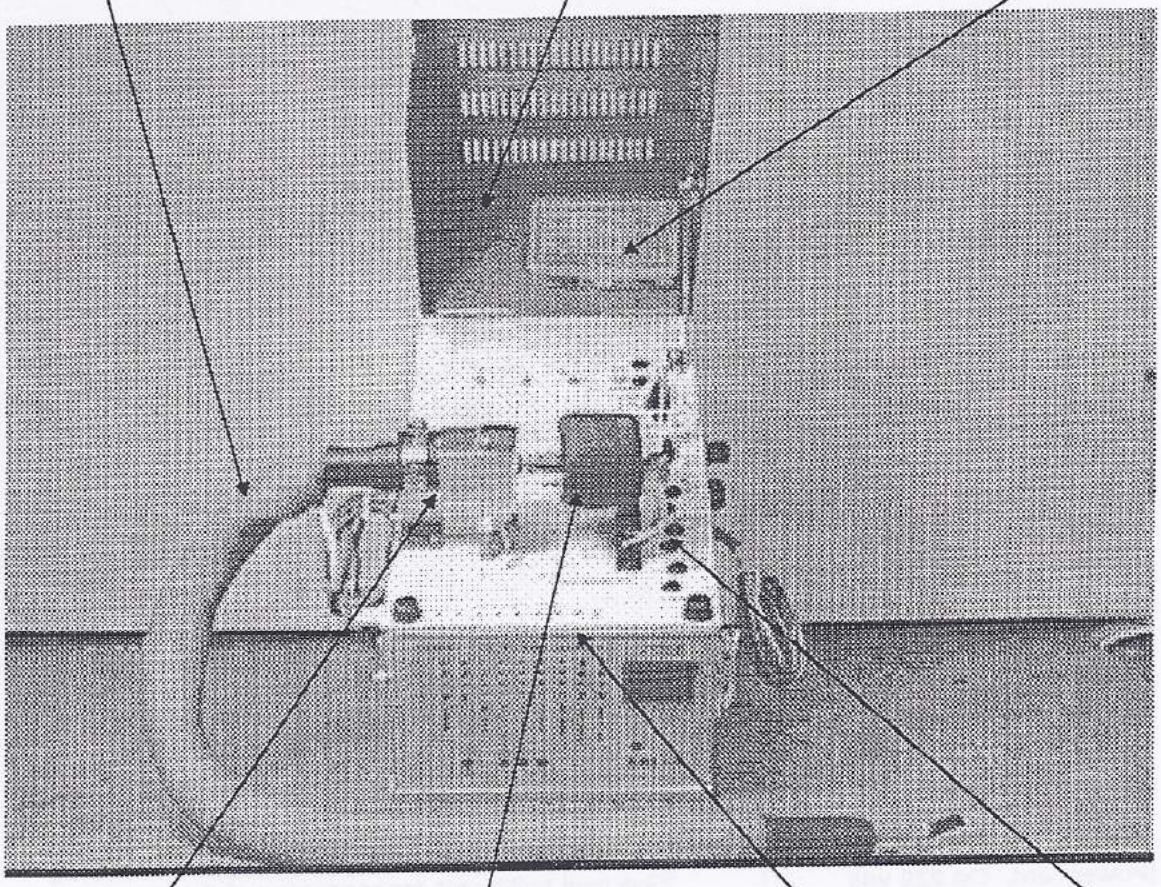
Chapter: MODEL 110 GC CHASSIS

Topic: TOP PANEL ORIENTATION

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

Red lid hinges up to allow access to detectors

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



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

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

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

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

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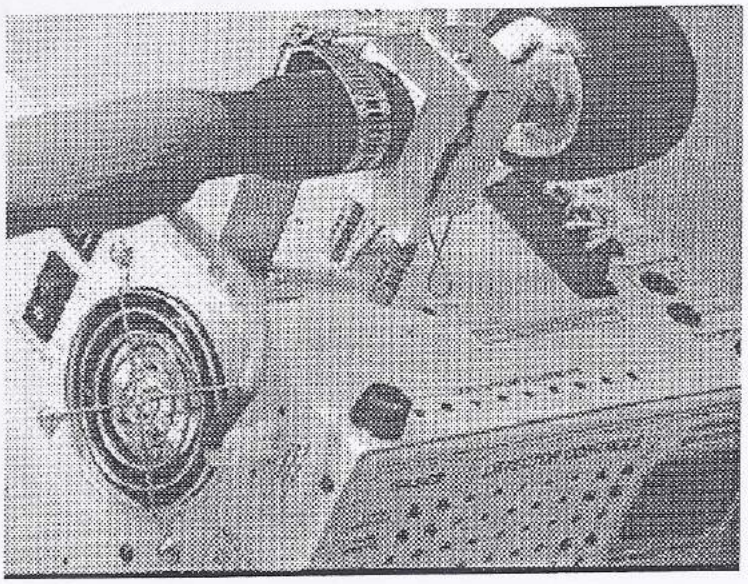
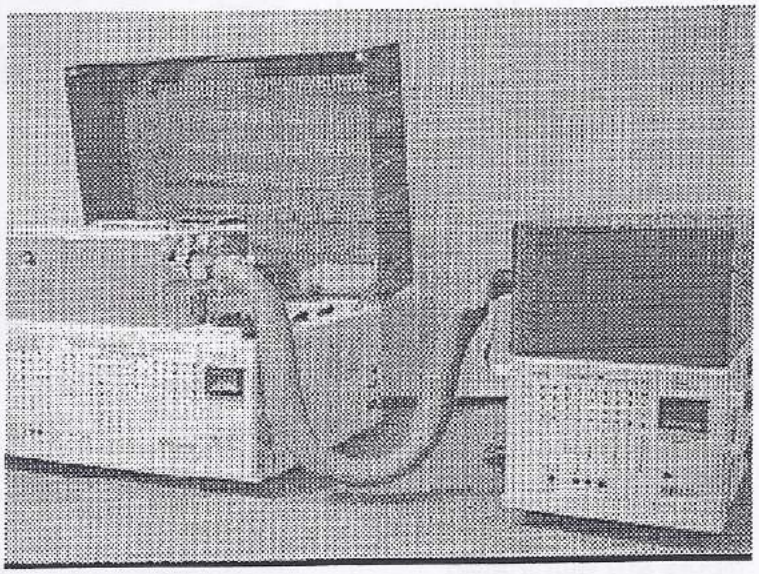
Chapter: MODEL 110 GC CHASSIS

Topic: HEATED TRANSFER LINE

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



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

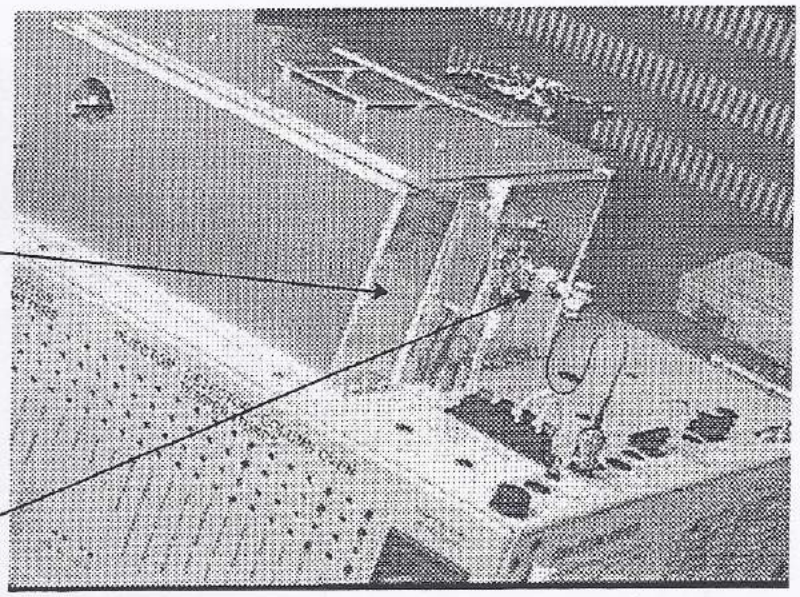


Chapter: MODEL 110 GC CHASSIS

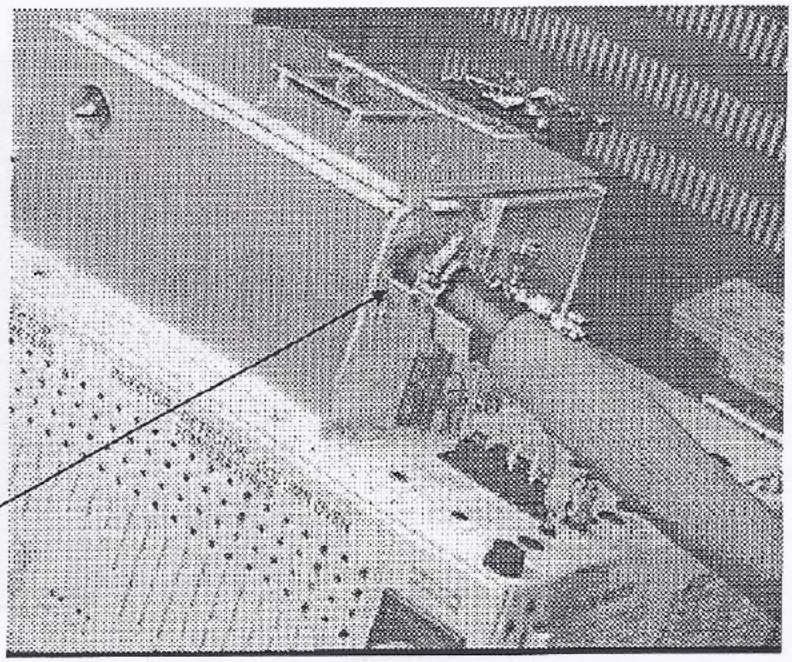
Topic: CONNECTING TRANSFER LINE TO GC

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

FID detector installed



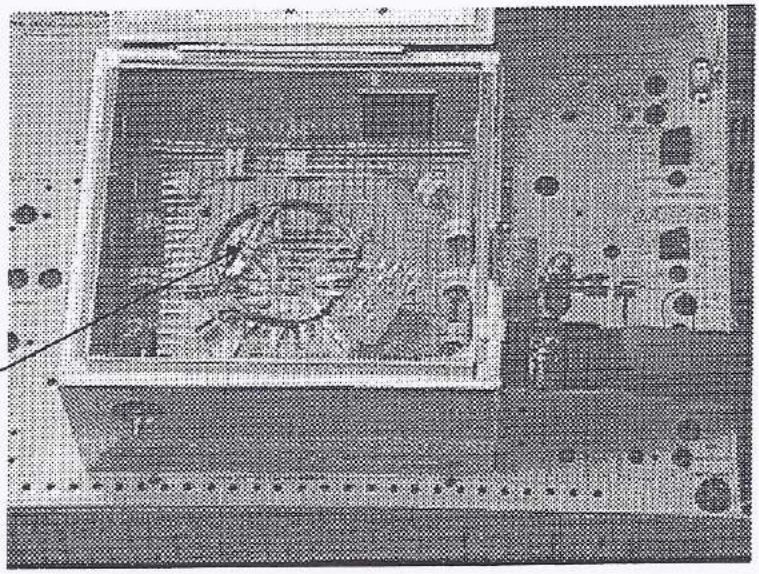
Replace one of the blank cover plates with the Transfer Line Mounting Plate (SRI part# 8670-9836) by removing the two screws at the base of the plate. The nuts on the underside of the chassis must be accessed by removing the bottom plate of the GC. The heated transfer line is then lightly secured to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



Chapter: MODEL 110 GC CHASSIS

Topic: COLUMN/TRANSFER LINE CONNECTION

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

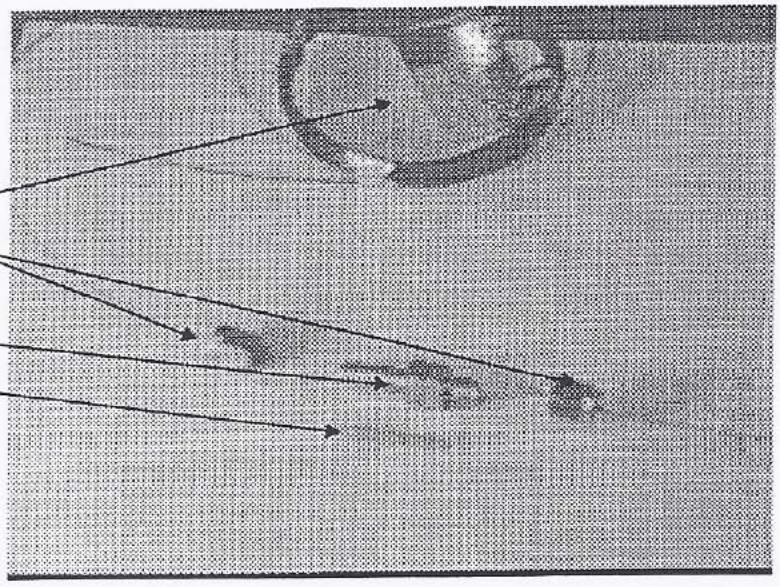


The separate parts of the union and column to transfer line connection hardware consist of:
GC column

Nut with graphite ferrule (2)

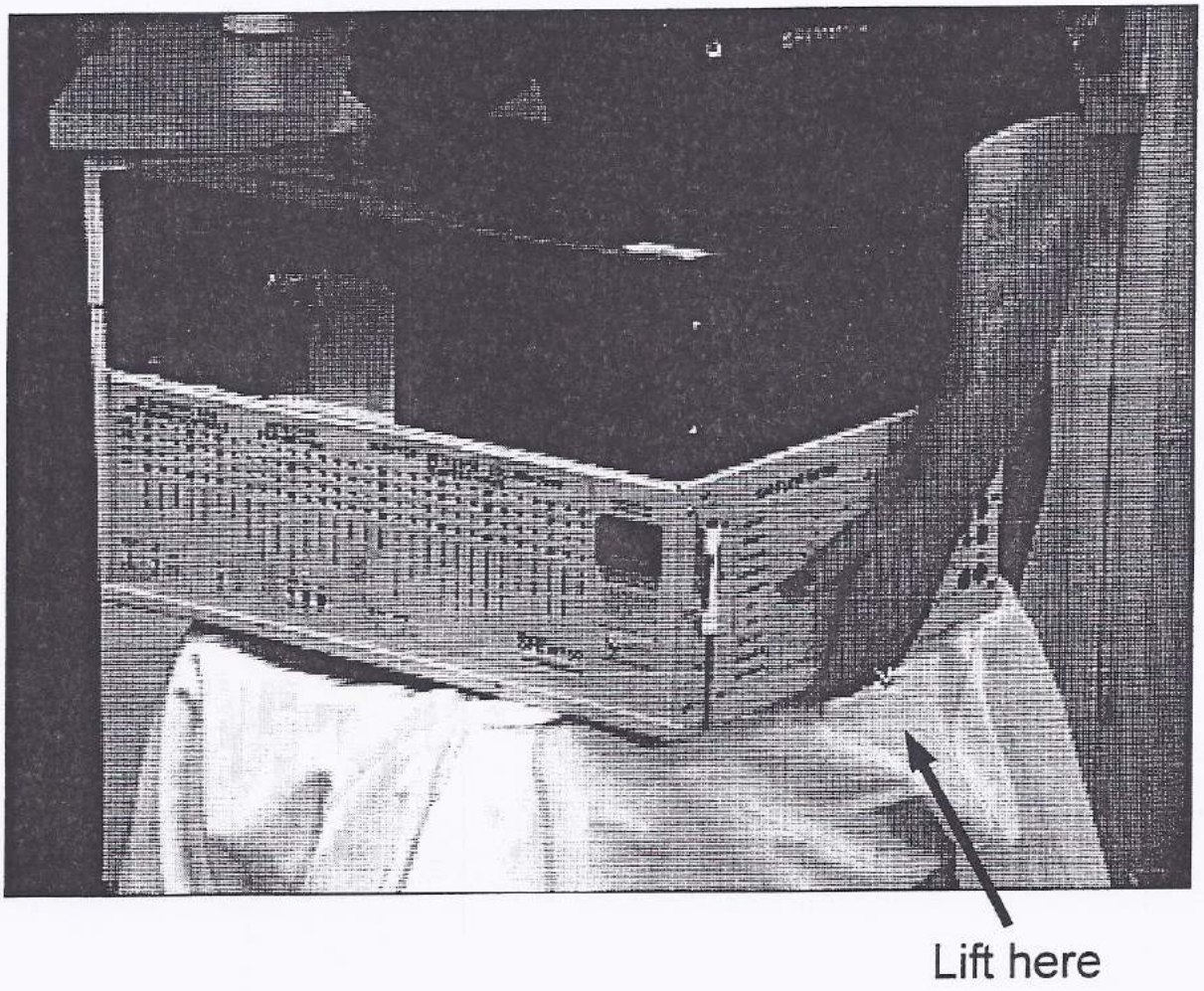
Stainless Steel bulkhead

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



Chapter: Installation:

Topic: Lifting the 8610C and 310 GCs



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.

Upon receiving the chromatograph and data system from the freight carrier, immediately inspect the containers for visible signs of damage. If any external damage is observed, notify the delivery person immediately. If no external signs of damage are present, proceed to inspect the contents of the containers. If the materials appear to have been damaged in shipping, immediately contact the carrier and submit a written report describing the extent of the damage. All packing materials and containers should be retained if damage is discovered until the carrier has been able to inspect the damaged goods. If no damage is discovered, packing should be retained until proper unit operation has been established. The chromatograph, serial data interface cable and manual are shipped in one container, along with the GC accessory kit. This container is a reusable plastic shipping container. These containers are rugged and shipped easily via freight carriers. Most importantly, the plastic container protects larger, more complex and delicate instruments from costly damage to external accessories. Save the packing materials after removing the chromatograph, for future transportation.

The contents of the containers should be checked against the packing slip accompanying the shipment. Verify that all specified accessory items ordered such as columns, syringes and the like have actually been shipped. If any items have been omitted or are missing from the shipment, contact SRI Instruments for location and/or replacement of the item.

The SRI model 8610C gas chromatograph requires either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, dependent on which AC power supply was specified when ordered. Both AC power supplies support the 3-prong grounded outlet. Proper grounding is required to minimize AC line interference and eliminate ground loops. The 220 VAC plug is keyed so that it cannot be inserted into a 110 VAC receptacle. A generator or high-current inverter may be used for operation from a vehicular power source. If an AC power generator is used, as is done in the field, line voltage and/or current may fluctuate. Appropriate steps should be taken to minimize any inconveniences caused by line noise or an irregular AC waveform.

A standard model 8610C gas chromatograph measures approximately 18.5" x 14.5" x 12.5" and requires a counter surface space of about 32" x 22". Eight inches of clearance are needed in front of the left side control panel for the fan, gas line access and the AC power switch. Another six inches of clearance are suggested in front of the right control panel and to the rear of the unit for safe operation and ease of access during routine service. The red oven cover requires a clearance of at least 24" (measured from the counter top) in order to provide adequate access to the column oven for service. If the chromatograph is equipped with optional accessories such as the 10 station purge and trap autosampler for the optional built-in EPA Style purge and trap, the access to the left side of the chromatograph must be increased by a minimum of an additional 12". The compact footprint of the system is economical on lab counter space and is ideal for mobile environmental installations.

Prior to placing the chromatograph into service, the gas supply and related plumbing should be installed and routed. The gas cylinders should be located outside the lab where possible, with only the lines plumbed inside to the chromatograph. Gas cylinders should be secured in place with chain or nylon strap to prevent a cylinder from falling and snapping off the valve. **A gas cylinder contains up to 2700psi and can become a deadly projectile if the valve stem were snapped off.** A regulator should be used to set the supply a gas pressure reduced to a value appropriate for introduction into the GC. Gas pressures at each cylinder pressure regulator should be maintained reasonably above the carrier gas regulator setting in order to provide a range of control (a supply pressure set to no more than 20psi greater than the EPC setpoint is recommended). A block valve should be inserted on the output side of the regulator to permit line service when needed, and to permit immediate shut-off in case of emergency.

Refrigeration-grade copper tubing may be used for all of the gas supply lines to the chromatograph. Plastic tubing should never be used as it permits contaminants, includung 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.

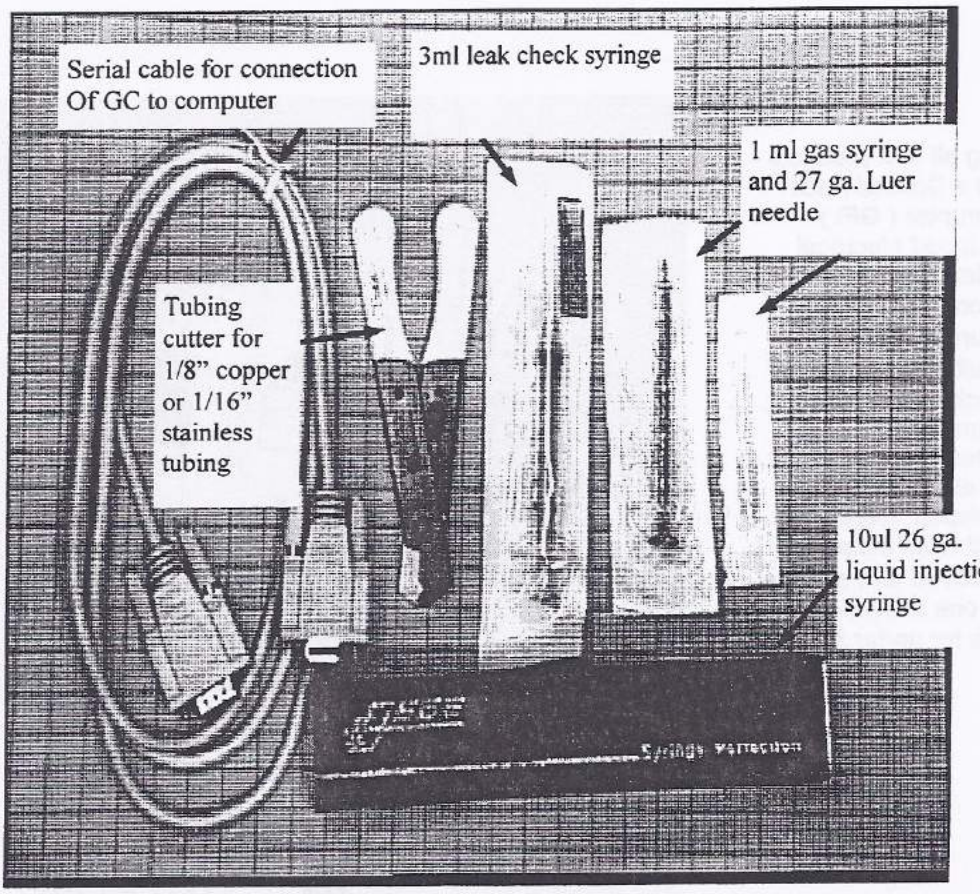
Once the gas line connections have been made and leak-tested, and the gas chromatograph has been located in the counter-top position where it will be used, plug the GC into a properly grounded AC outlet, and energize the unit. Gas pressures may then be adjusted to proper operating conditions by means 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.

Chapter: INSTALLATION

Topic: Contents of Accessories Kit included with GC



Contents of accessories kit shipped with new SRI GCs.

1) 6' DB-9 serial cable for connection of GC to computer (Student model without data system will not have this item).

2) Tubing cutter for easy installation of 1/8" copper or 1/16" stainless steel tubing

3) 3ml leak check syringe (fill with alcohol/water mix to check fitting for leaks)

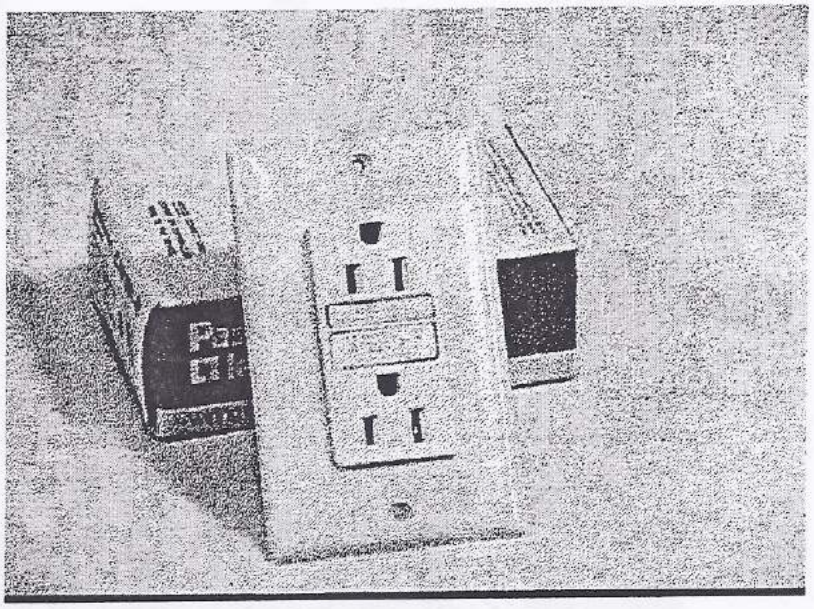
4) 1ml gas syringe and needle for injection of gas samples into GC

5) 10ul liquid injection syringe

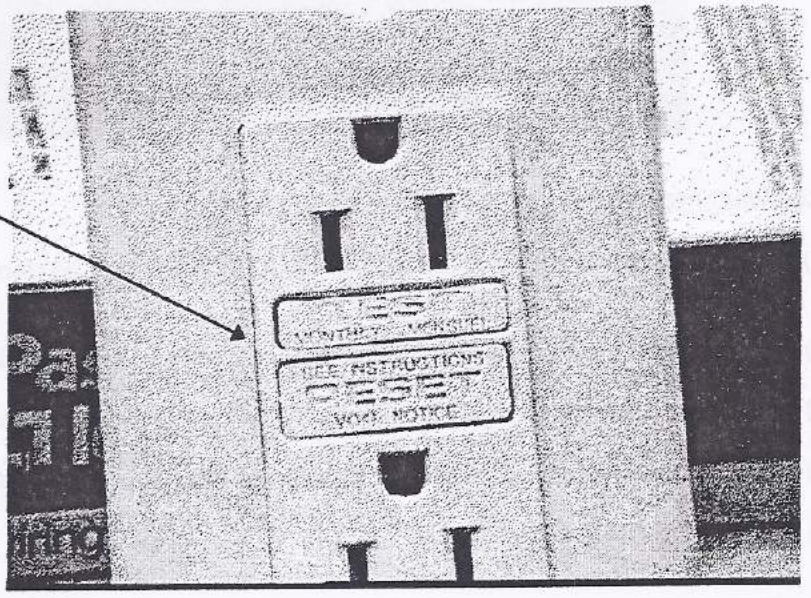
INSTALLATION:

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 re-activate the GFI by pressing the Reset.



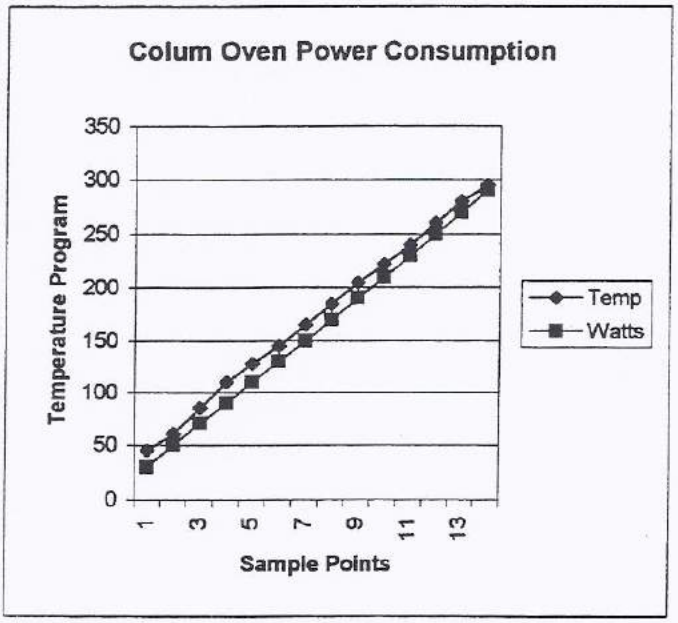
8610C Power Consumption

7/16/2002

Basal Power
With no zones heating, Power Usage = 50W
With 2 Detector zones heating = 150W
With Detector Zones Stabilized, Total Basal Power = 100W

Column Oven
Temperature Program 40C to 300C @ 5C/min

Average Temp	Watts	Temp Range
45	30	40-50
60	50	50-70
85	70	70-100
110	90	100-120
127.5	110	120-135
145	130	135-155
165	150	155-175
185	170	175-195
205	190	195-215
222.5	210	215-230
240	230	230-250
260	250	250-270
280	270	270-290
295	290	290-300



Maximum Power Usage
Ballistic Heating to 300C = 675W
Total Power = (Basal + Detector + Column Oven) = 825W

Isothermal Power Usage
Column Oven Stabilized @ 300C
2 detectors @ 150C
Total Power = (Basal + Detectors + Column Oven) = 400W

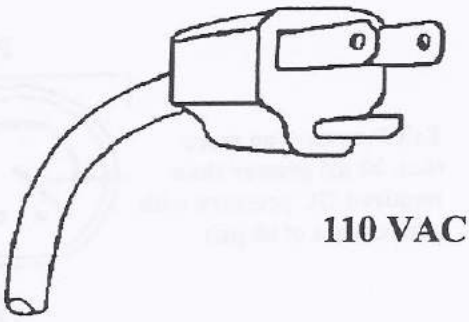
Chapter: INSTALLATION

Topic: Power Supplies and Space Requirements

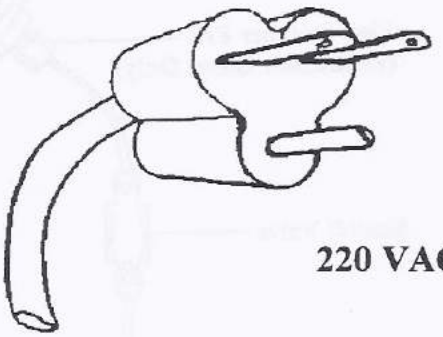
Once the equipment has been removed from all the packing material, check the contents of the container against the packing slip and make sure everything listed is included. If any item(s) have been omitted or are missing, contact SRI Instruments for location and or replacement of the item(s).

The SRI model 8610C gas chromatograph requires AC power at either 110 VAC at 60 Hertz or 220 VAC at 50 Hertz, depending on the AC power ordered. Both AC power supplies are equipped with a three prong grounded outlet (see diagrams to the right). Proper grounding is required for safe operation. Do not disable the ground prong under any circumstance. These plug configurations are for EIA standard U.S. outlets. It may be necessary to replace the plug provided with a local standard plug.

A standard SRI 8610C GC measures 18.5" X 14.5" X 12.5" and requires a minimum counter space of 28" X 22" X 23.5" for proper operation (see diagram to the right). Roughly 8" of clearance beside the left side control panel is needed for data cable, gas line and power switch access. 6" of clearance to the rear of the GC and 11" of clearance above the GC is required. This will provide adequate access to the column oven for maintenance and provide space for proper GC 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.

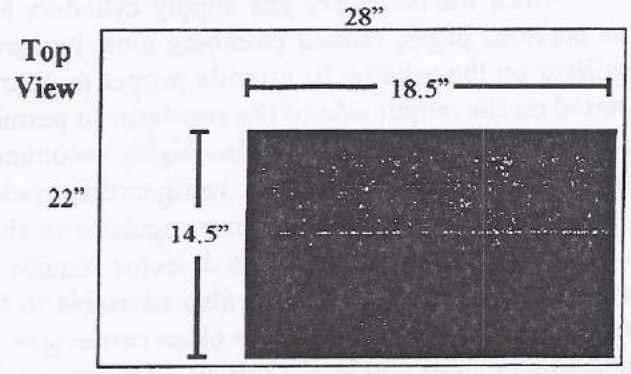


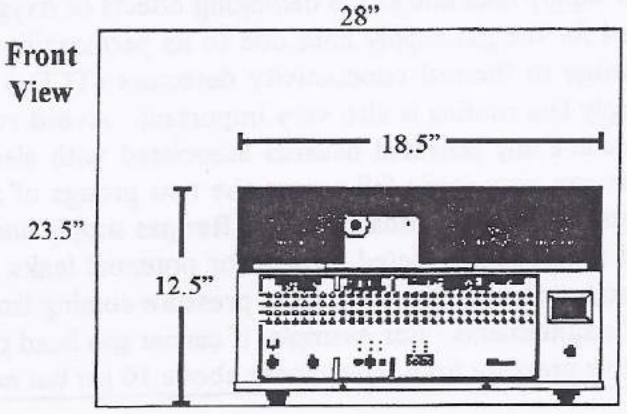
110 VAC



220 VAC

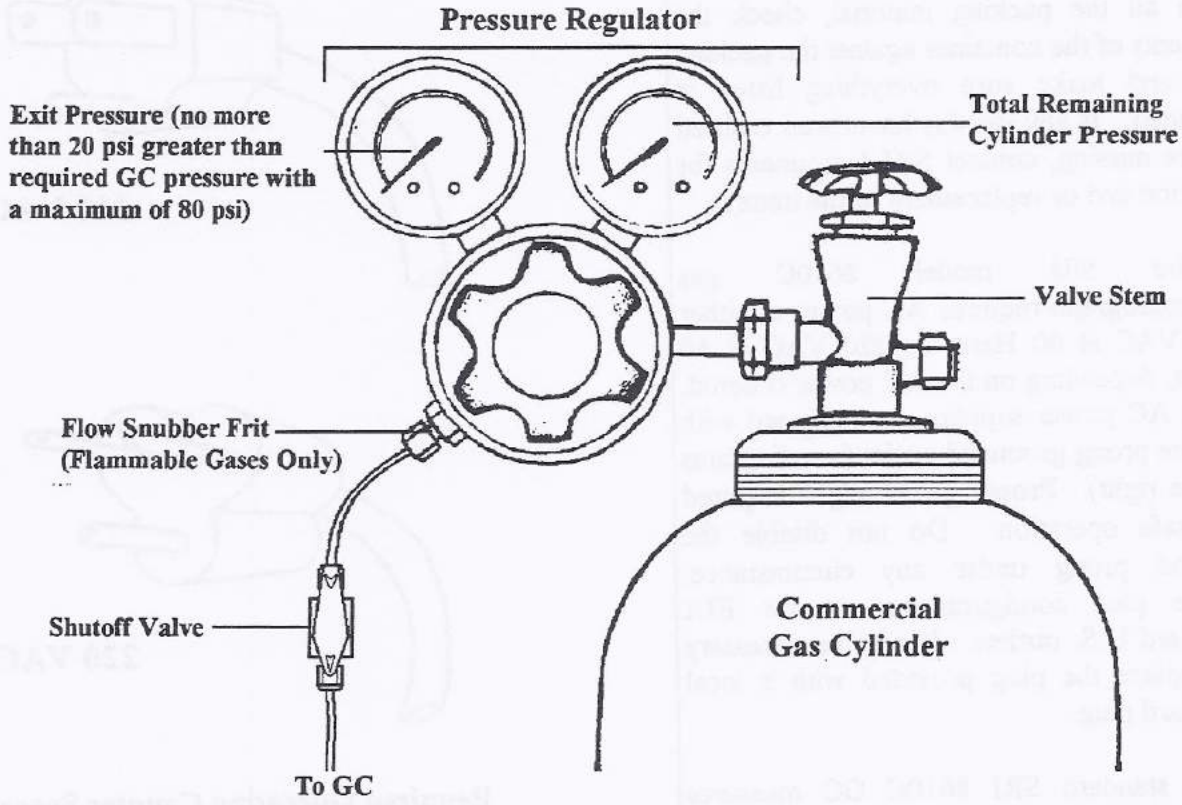
Required Operating Counter Space





Chapter: INSTALLATION

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.

Chapter: INSTALLATION

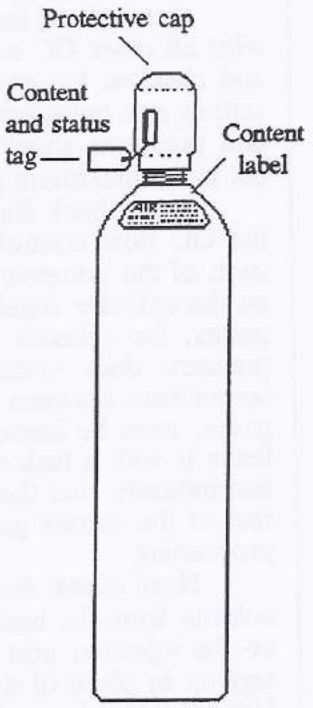
Topic: Gas Supply Selection

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. The recommended make-up gas is argon/methane which provides the best sensitivity and largest dynamic range for the ECD, but nitrogen is a readily available, cost effective alternative (see the manual section on the ECD for more details). FIDs, FPDs, and NPDs all require hydrogen and air in order to create the combustible fuel mixture for the detector flame. Hydrogen is an extremely flammable gas and must be handled appropriately. Always consult local safety regulatory agencies for proper procedures for handling compressed and/or flammable gases. An internal air compressor is an available SRI GC option as a source of air. GCs with a purge and trap accessory also require some type of sparge gas. Generally helium can be used as both a carrier and a sparge gas supply. Methanizer accessories require hydrogen gas as a reactant in the catalytic reduction of CO and CO_2 to CH_4.

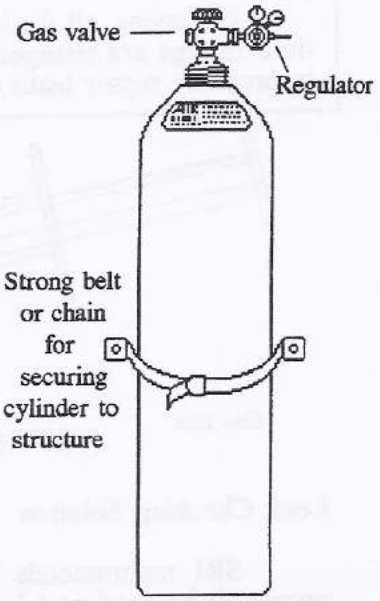
We recommend the use of medium to high quality gas sources for all required gases in order to prevent any operational problems associated with low quality gas. ECDs require an extremely pure carrier gas source of 99.9995% or higher. SRI GCs are equipped with small internal molecular sieve polishing filters 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

Chapter: INSTALLATION

Topic: Checking for Gas Leaks

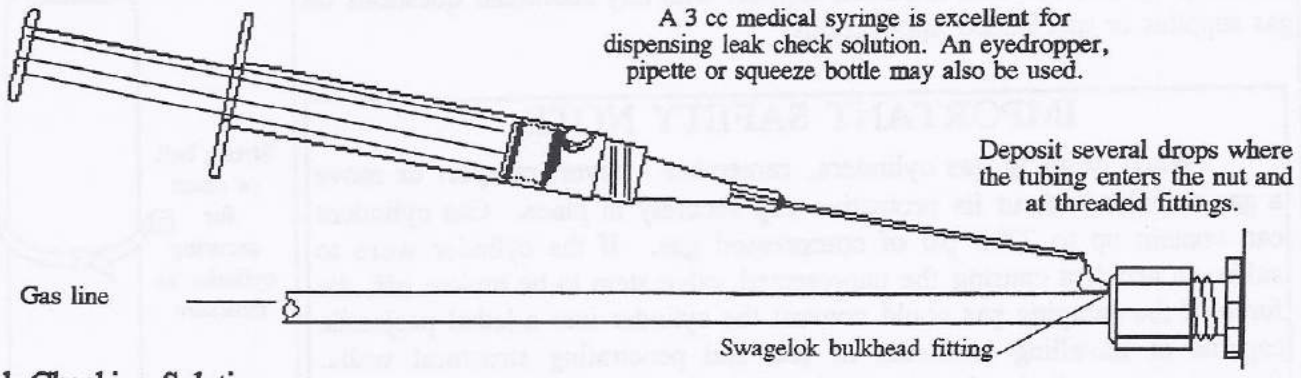
Once all of the appropriate gas supply sources and lines have been properly installed, along with all other GC columns and connections, the entire system should be systematically pressurized and checked for possible leaks. Begin by opening all of the compressed gas cylinder valves and setting exit pressures 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.



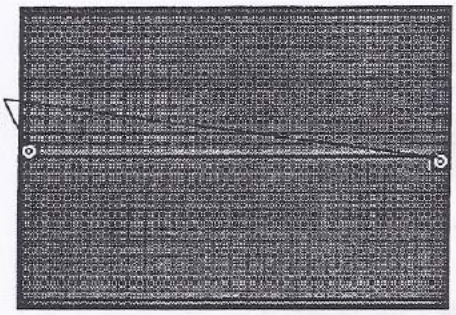
Leak Checking Solution

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.

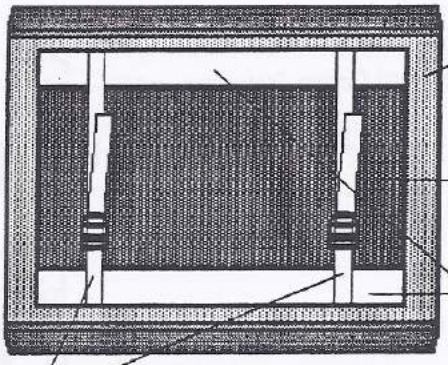
Chapter: INSTALLATION

Topic: Removing The Chromatograph From The Shipping Container

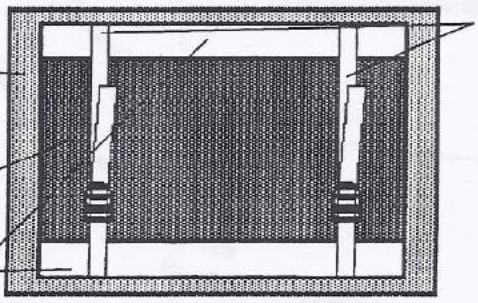
Top view of container, lid closed

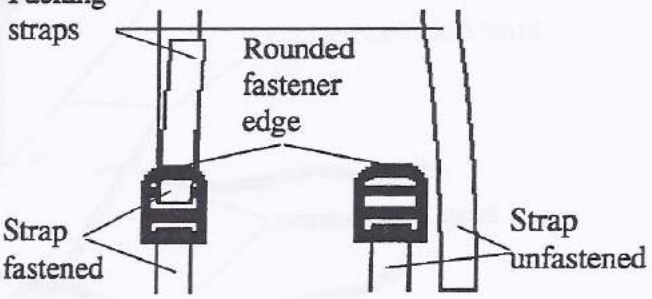


Top view, lid open, GC in container



Top view, GC out of container

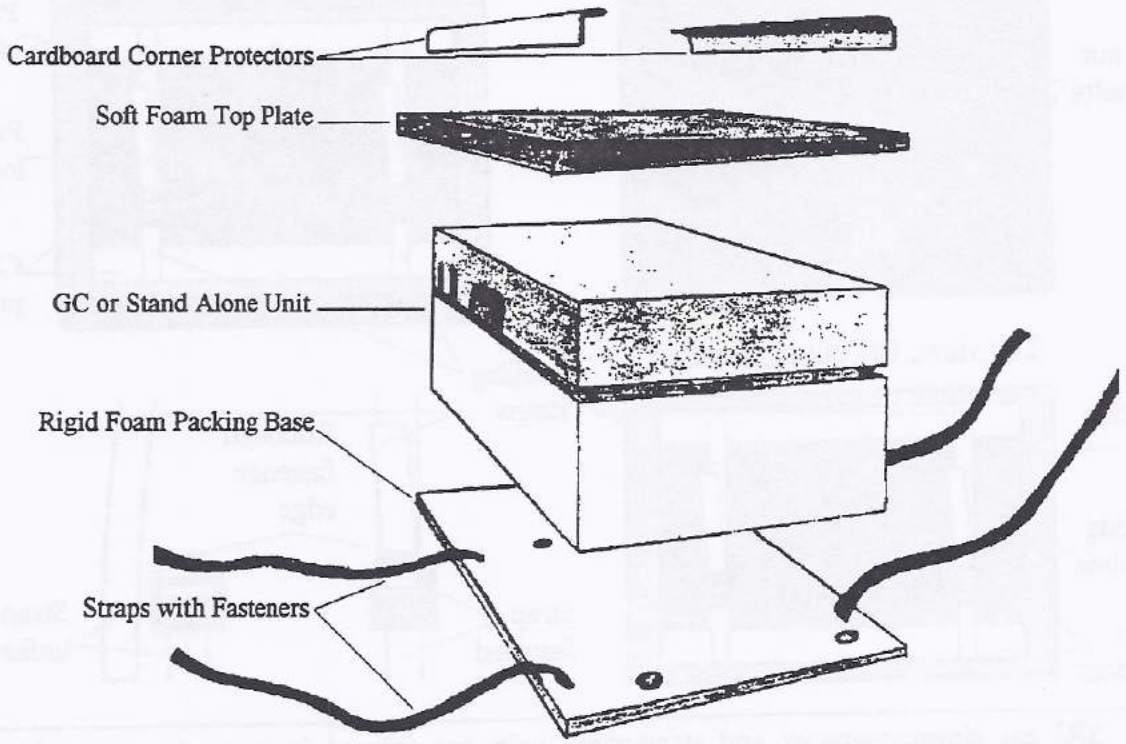




SRI gas chromatographs and stand-alone units are shipped in a sturdy, protective shipping container. The molded gray plastic container is reinforced and resistant to blows and crushing pressures typically encountered while en route to the customer or job site. Upon receipt, check to see that there is no obvious damage to the exterior of the shipping container. Notify the delivery person immediately of any such damage. The lid of the shipping container is secured closed by a 1/2" nut and bolt set each located on either side of the container. To open the lid of the shipping container, completely remove the two nut and bolt set and simply open lid. Screw the nuts back onto the bolts and place in shipping container for future use. The GC is held in place within the shipping container in custom packing material consisting of (1) rigid foam bottom packing base, (1) soft foam top plate, (2) cardboard corner protectors, and (2) straps with fasteners to bind GC within packing material. Some SRI GCs can weigh more than 70 pounds, and care must be taken to prevent injury when removing from shipping container. To properly remove the GC from within the shipping container, firmly grasp the two visible straps running across the soft foam top plate between the two cardboard corner protectors. Being careful to properly bend your knees, lift the entire GC, still contained within the packing material, straight up and out of the shipping container. To remove the packing material from around the GC, begin by removing the two straps holding it all in place. Place your fingers beneath the rounded strap fastener edge and pull up and back. When the strap loosens up, pull the free end of the strap completely through the fastener. Once both straps have been unfastened, remove the two cardboard corner protectors along with the soft foam top plate and place back in the empty shipping container for safe keeping. Next, slide your fingers between the metal GC base plate and the rigid foam bottom packing base, and firmly grasp the bottom of the GC with both hands. Once again being careful to properly bend your knees, lift the GC up and out of the packing base. Place the packing base, with straps still attached, in the shipping container with the other packing materials. Be sure to save all packing materials along with the shipping container for all future shipping needs.

Chapter: *Installation*

Topic: 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.

Chapter: INSTALLATION

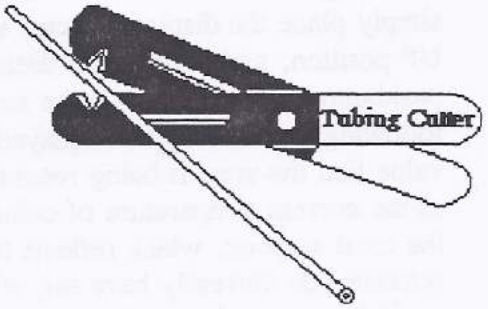
Topic: Tubing Cutter - For Facilitating Gas Connections

Included in the optional gas line installation kits that may be purchased with each SRI Instruments gas chromatograph is a disposable tubing cutter. This tool is capable of producing clean, fast cuts in chromatography tubing that rival more time-consuming tubing cutting methods. The hardened, beveled cutting surface of the tool enables the user to effect a through-and-through cut upon the tubing in one motion, cutting copper and stainless steel tubing with ease. The cut obtained allows both metal and graphite ferrules to slide onto the tubing without the normal filing or reaming necessary after cutting tubing using other methods. No smearing or burring is produced if this tool is used as directed.

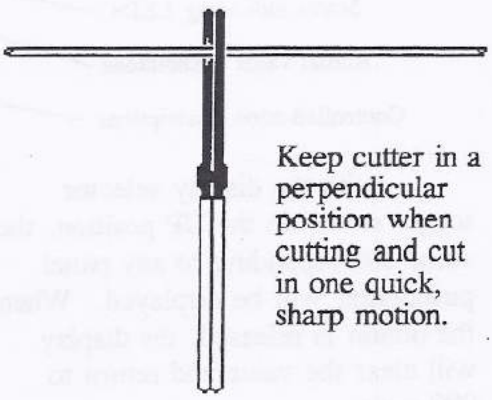
Users can make up to ten connections in the time that it took to cut, file, ream and connect one single tubing connection. Since the tubing is sheared and not twisted or stressed, the inside passage is not deformed or restricted, enabling the user to cut very small internal diameter stainless steel tubing (such as 1/16" O.D. x 0.005" I.D.) that would likely collapse or otherwise become restricted when cut by any other tool. Cuts on very small tubing is seldom attempted due to the difficulty encountered using ordinary methods. By using this tool, delicate tubing cuts become as easy and routine as larger tubing cuts.

Tubing cuts in tight or hard-to-reach locations can be performed without difficulty with the use of this tool. Since the cutting head is practically flat and requires relatively little clearance, it can be inserted into otherwise difficult spots to perform high precision cuts. As an example, if gas tubing routed through a hard-to-reach area inside the gas chromatograph required cutting for the insertion of an adapter or other special fitting, the cutter head could be inserted to the location and the cut 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.



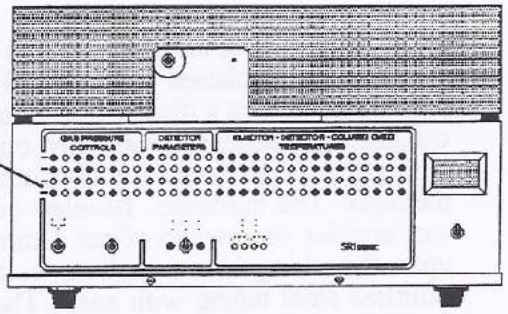
Keep cutter in a perpendicular position when cutting and cut in one quick, sharp motion.

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 setpoint you currently have set, which, in the case of column oven 1, would be an offset value that could be summed with the temperature signal being sent from the data system; and the total setpoint, which is the sum of any signal being sent from the data system to the controlled zone, in addition to any local setpoint value you have set (for example, if column oven 1 has a local setpoint of 50 degrees, and the data system is instructing the GC to heat the column oven to 100 degrees, the total setpoint should display 150 degrees). Most zones will only display the local setpoint and actual value. Each zone also displays its status via a light-emitting diode (LED) that glows when the zone is active.

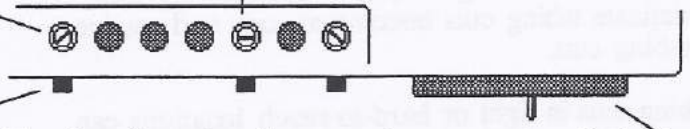
"At-a-glance" display panel also permits viewing of actual and setpoint values



A small blade scewdriver (provided) is used to adjust the setpoint potentiometer while the desired local setpoint display pushbutton is depressed

Adjustable potentiometer located immediately above controlled zone on top front of GC chassis

Display pushbutton viewed from above



Top view of setpoint adjustments above temperature display

Bright red digital LED panel meter displays temperatures, pressures, and voltages

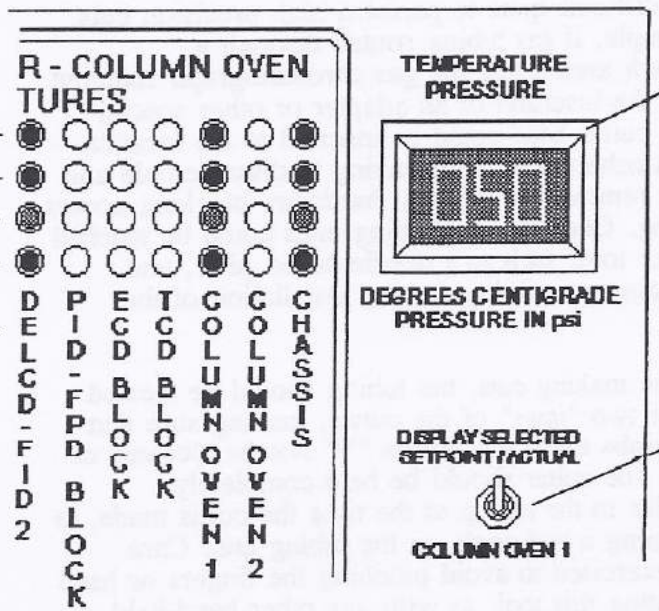
Local setpoint pushbuttons

Total setpoint pushbuttons

Status indicating LEDs

Actual value pushbuttons

Controlled-zone descriptions



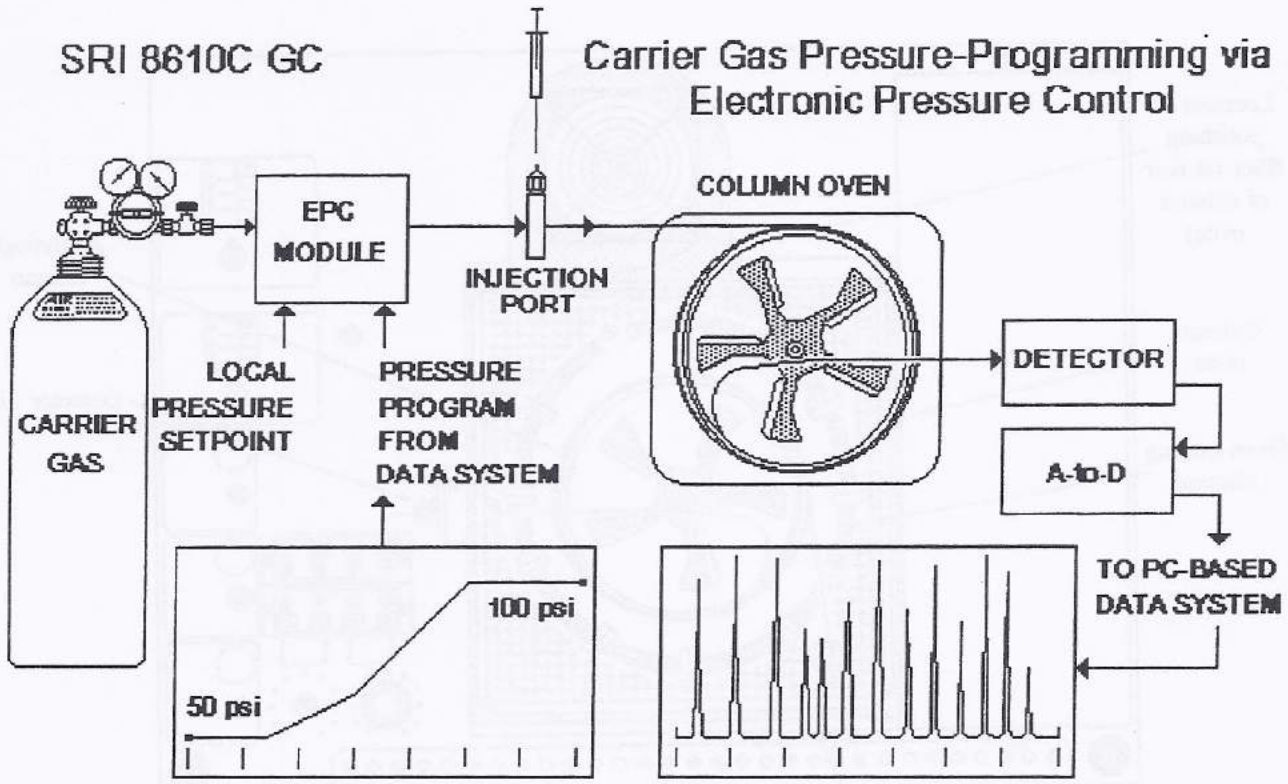
Front view of column oven temperature display

Toggle switch selects display of setpoint and actual values in UP position, or constant column oven 1 temperature display in DOWN position

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

Chapter: INSTALLATION

Topic: Pressure-Programmed Carrier Gas Operation Using EPC System



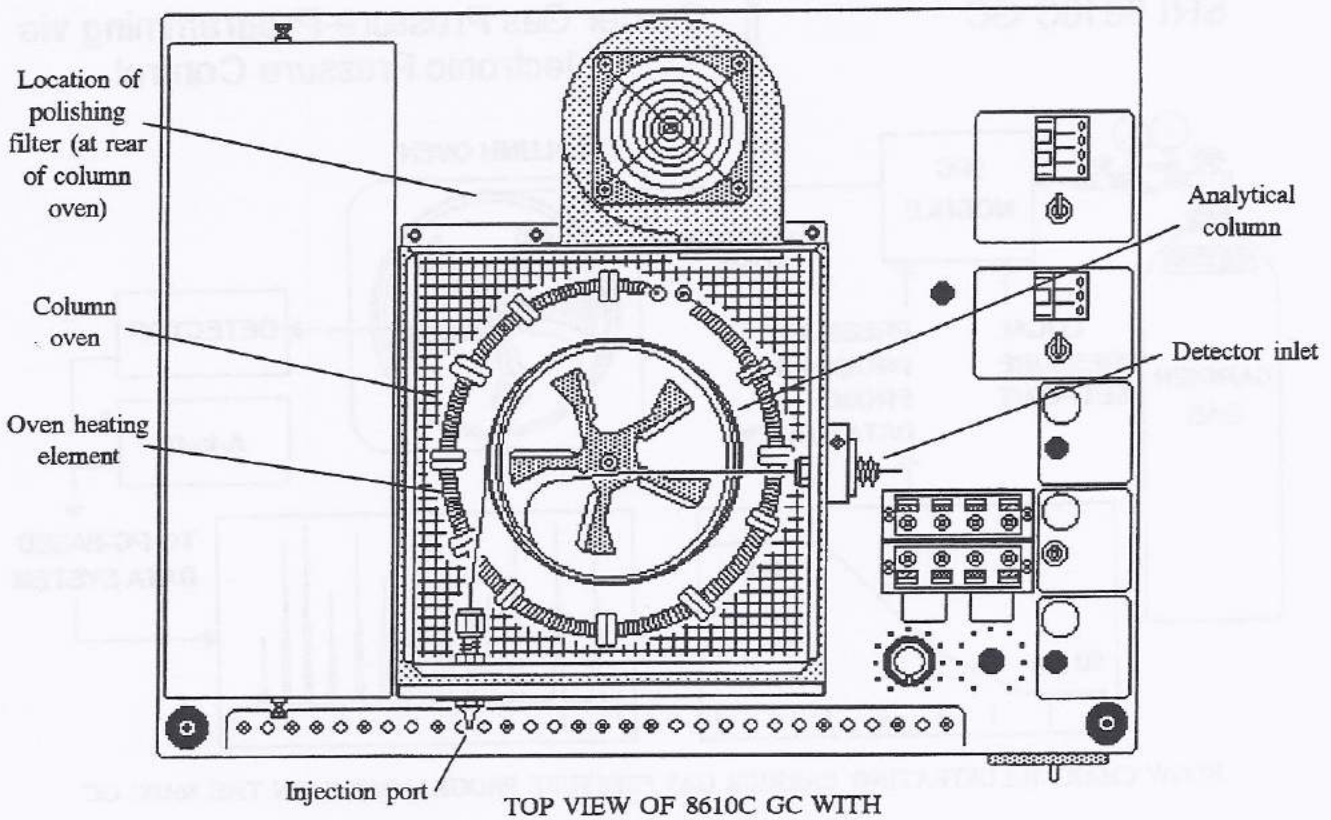
FLOW CHART ILLUSTRATING CARRIER GAS PRESSURE PROGRAMMING ON THE 8610C GC

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.

Chapter: INSTALLATION

Topic: Analytical Column Installation

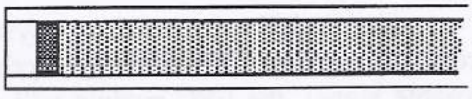


TOP VIEW OF 8610C GC WITH COLUMN INSTALLED

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.

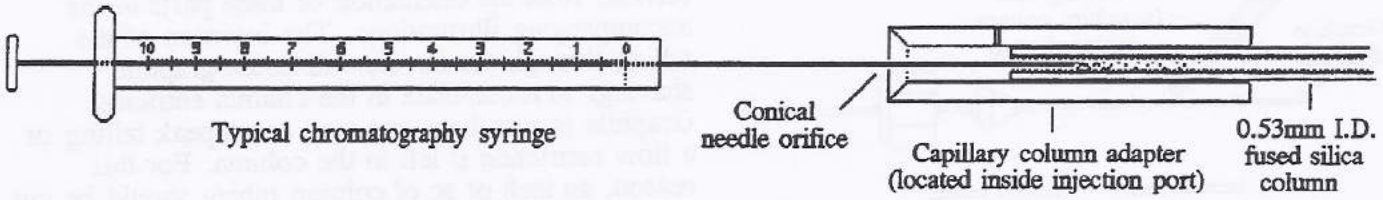
0.25 to 0.32mm I.d fused silica tubing coated on inside surface with stationary phase film 0.1 to 1.0 microns thick

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

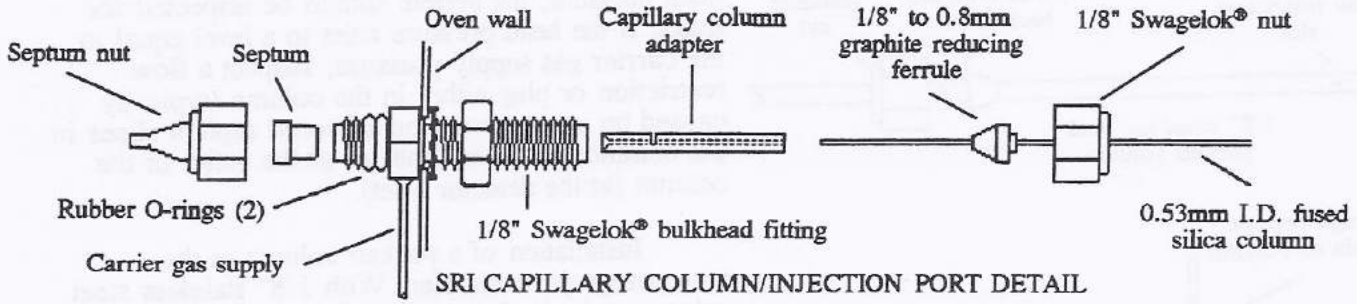
The injection port of the SRI chromatograph is designed specifically for direct injection onto a 0.53mm I.D. wide-bore capillary column. A sample, injected using a chromatography syringe equipped with a 26 gauge needle, is deposited directly into the column. The injector is supplied with a 1/8" O.D.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

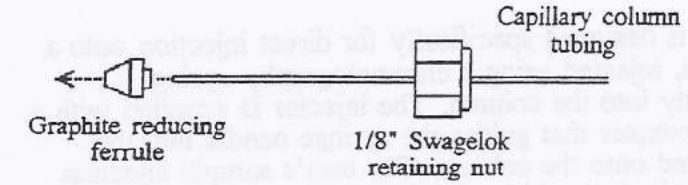
The wide-bore capillary column adapter is machined from 1/8" stainless steel and accepts the insertion of 0.8mm O.D. tubing (the outer dimension of 0.53mm I.D. capillary column tubing). The injection end of the adapter is conical and "funnels" the needle into the column tubing inserted into the adapter 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.

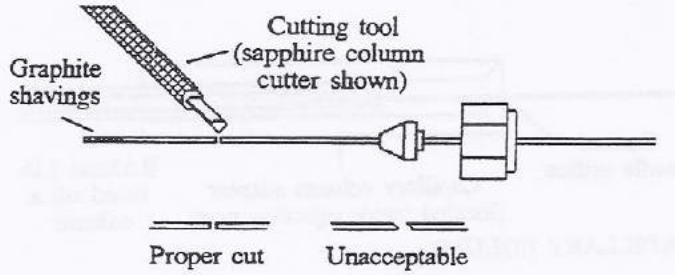
The capillary column adapter is located within the assembly that forms the injection port when a 0.53mm I.D. column is in use. The injection port is constructed from a 1/8" stainless steel Swagelok® bulkhead fitting that has been modified to permit the connection of a gas source directly into the fitting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

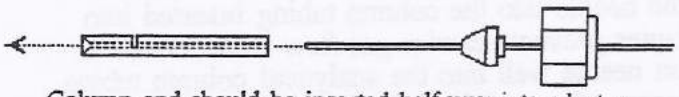


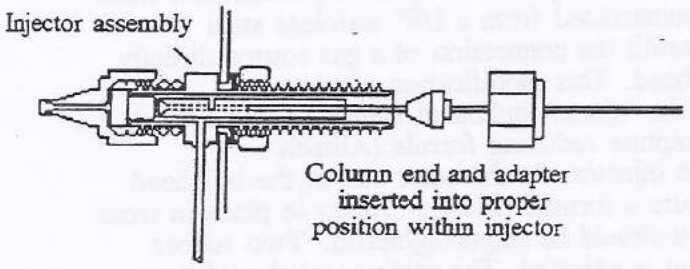
SRI CAPILLARY COLUMN/INJECTION PORT DETAIL

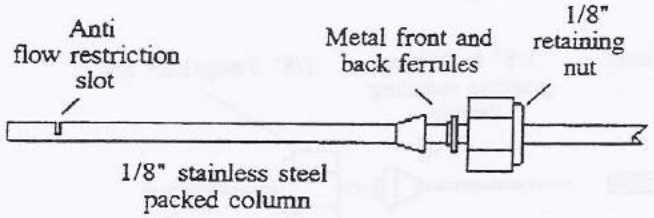
The injection port is compact and has a low thermal mass. Since most of the injector body is located within the column oven, the injector and oven temperatures are always equal (the standard injector is not supplied with any provision for independent heating. Heated on-column injection is available as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

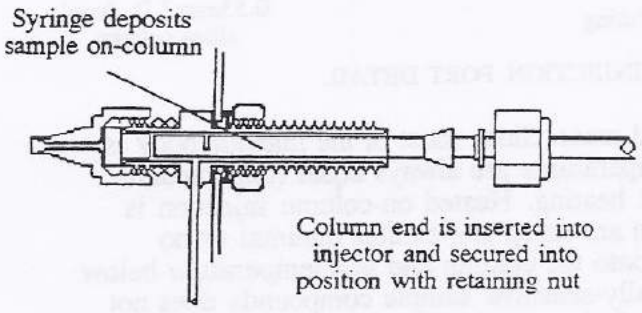






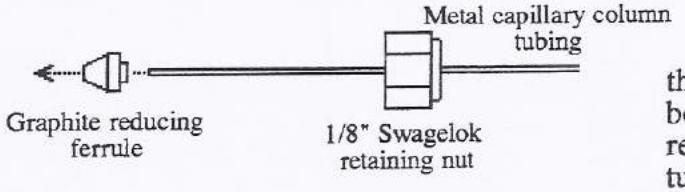


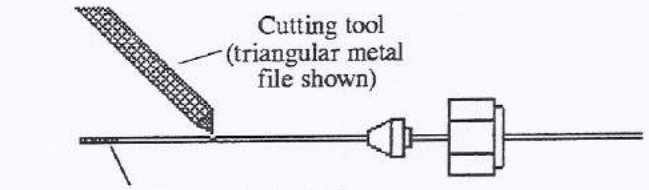


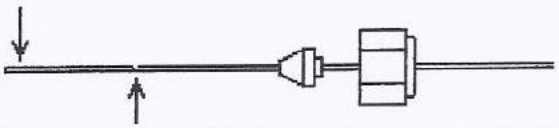


When installing a capillary column in the chromatograph, a graphite reducing ferrule must be used to secure the capillary tubing in the 1/8" retaining nut (Alltech RF200/0.8-G for 0.53mm tubing, RF200/0.5-G for 0.32mm tubing and RF200/0.4-G for 0.25mm tubing). The column is inserted first through the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

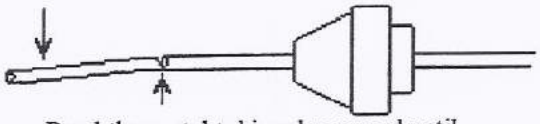
Installation of a packed column in the chromatograph is simpler. With 1/8" stainless steel columns, standard metal ferrules are used to secure the column at the retaining nut. The ferrules are placed onto the column end as shown, and then the column end is inserted into the injector. The capillary column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



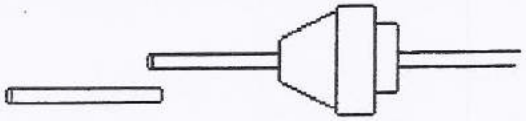




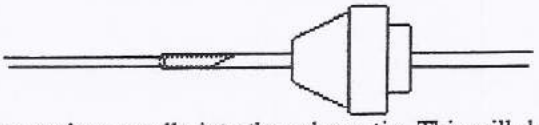
After scoring the metal column, wipe away any shavings and apply force to the tubing at the score



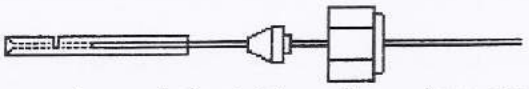
Bend the metal tubing downward until the score opens into a break in the metal
Stop and apply force in the opposite direction



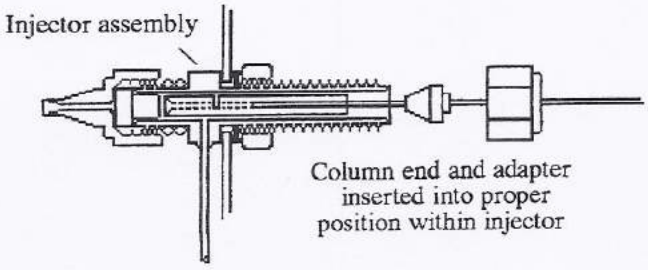
The front piece of tubing should break away cleanly



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

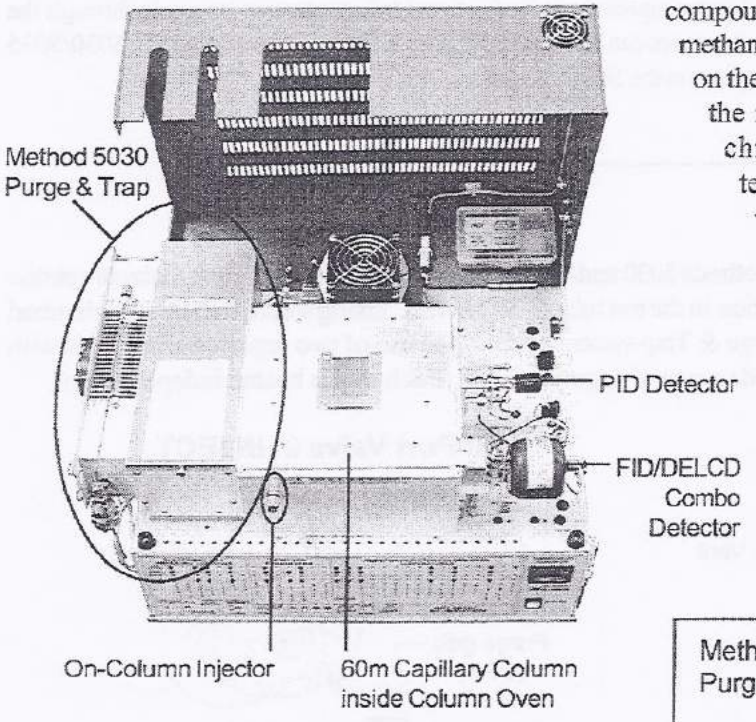


When installing a metal capillary column in the chromatograph, a graphite reducing ferrule must be used to secure the capillary tubing in the 1/8" retaining nut (Alltech RF200/0.8-G for 0.53mm I.D tubing, RF200/0.5-G for 0.32mm I.D. tubing and RF200/0.4-G for 0.25mm I.D. tubing). The column is inserted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

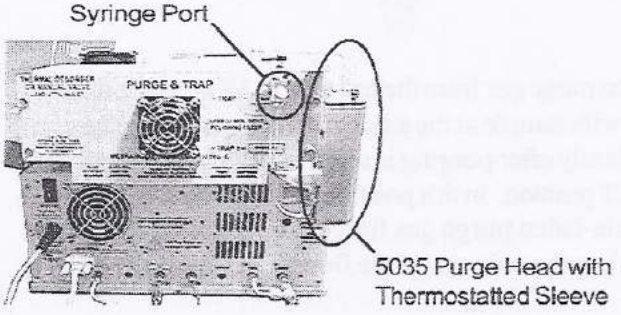
System Overview

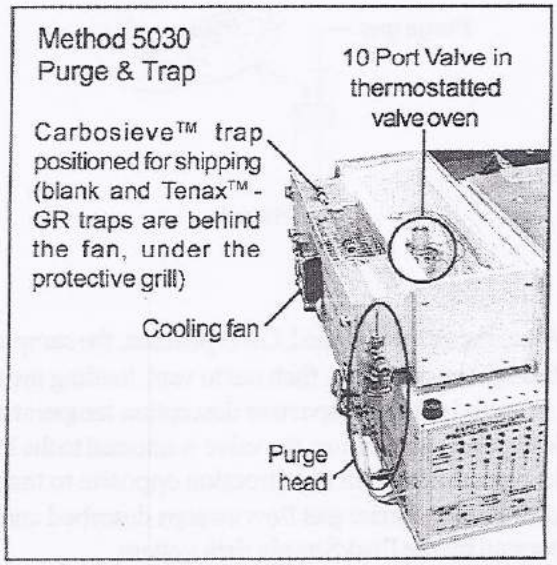
Your SRI Environmental GC is equipped with everything you need to generate certification quality data for EPA Methods 8010, 8015, 8021, and others. It is configured on the 8610C chassis, and includes a built-in Method 5030 or 5030/5035 compliant Purge & Trap for concentration of liquid and/or soil samples. 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 hydrocarbon overload. The PID is blind to certain compounds which can cause interference, such as methanol, and is recommended by the EPA. Peaks on the FID chromatogram that are obscured by the methanol peak are visible on the PID chromatogram. Benzene and carbon tetrachloride are common target analytes which co-elute. The FID responds to both. The PID responds only to benzene, while the DELCD responds only to carbon tetrachloride.

The BTEX GC is the same as the Environmental GC without the DELCD detector. Both systems have a "whisper quiet" internal air compressor and can be used with an H_2-50 hydrogen generator for tankless field operation.



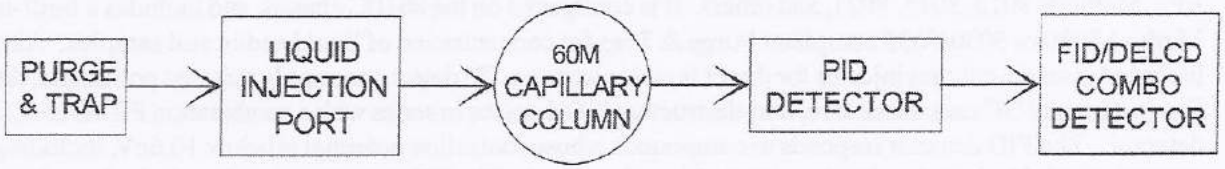
Method 5030/5035 Purge & Trap on an Environmental GC





POPULAR CONFIGURATION GCs
BTEX & Environmental

Theory of Operation

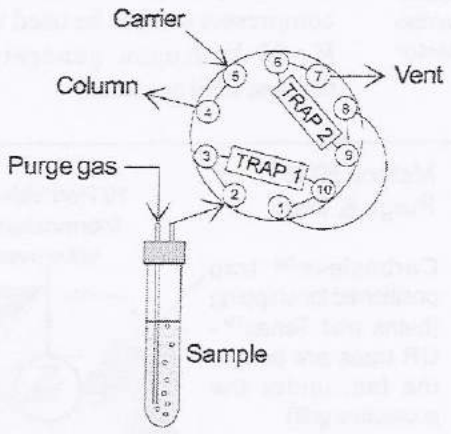


The versatile BTEX/Environmental GC systems can analyze gas, water, and soil samples. Four types of injection techniques can be used: purge and trap, direct liquid injection, TO-14 type gas sample concentration, and manual headspace injection. The Purge & Trap concentrator may be used for gas, liquid,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

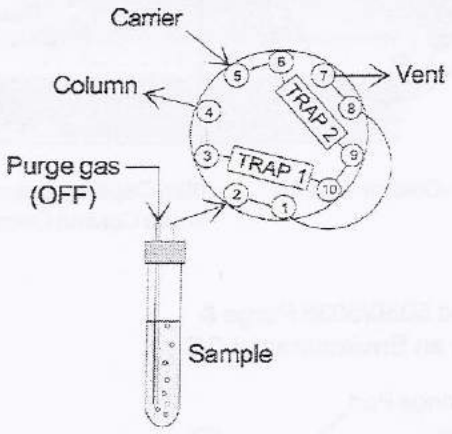
Purge & Trap Injection

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

10 Port Valve in LOAD



10 Port Valve in INJECT



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

Theory of Operation continued

Direct Injection

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

Gas Sample Concentration

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

Room Temperature Manual Headspace Injection

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

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

General Operating Procedures

EPA Style Purge & Trap Injection

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

1. The purge gas flow is controlled with an Electronic Pressure Controller (EPC). Set the purge flow (measurable at the trap vent at the rear of the purge and trap system); 40mL/min is a typical purge flow. The pressure required for 40mL/min through a single Tenax trap is printed on the right panel of the GC. ***NEVER use hydrogen as a purge gas***. SRI recommends helium purge gas.

2. TRAP 1 is in the lower position in the Purge & Trap, and TRAP 2 is in the upper position. The trap temperatures are factory set at 200°C for desorption. For adsorption temperatures, trap 1 is set at 30°C and trap 2 is set at 35°C. Trap heating will be controlled by the timed Event Table during the run. NOTE: the actual temperatures typically run 5°C over the setpoint. See the instructions in the Purge & Trap section of the manual for adjusting the trap adsorption temperature settings.

3. Load or create an Event Table that is appropriate to the sample to be analyzed, or that is designed for compliance with a particular EPA Method (such as **Epap&t1c.evt** for a single trap or **Epap&t2c.evt** for dual traps included in version 2.66 or higher of the PeakSimple software).

4. Load or create an appropriate Temperature Program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software for your convenience. As a basic rule for good separation using the purge and trap injection technique, the column oven should be kept at 40°C for 10-12 minutes: 6 minutes while the sample is purging, plus 4-6 more minutes while the traps heat and the gas sampling valve (in the INJECT position) transfers the sample to the column.

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

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

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

General Operating Procedures continued

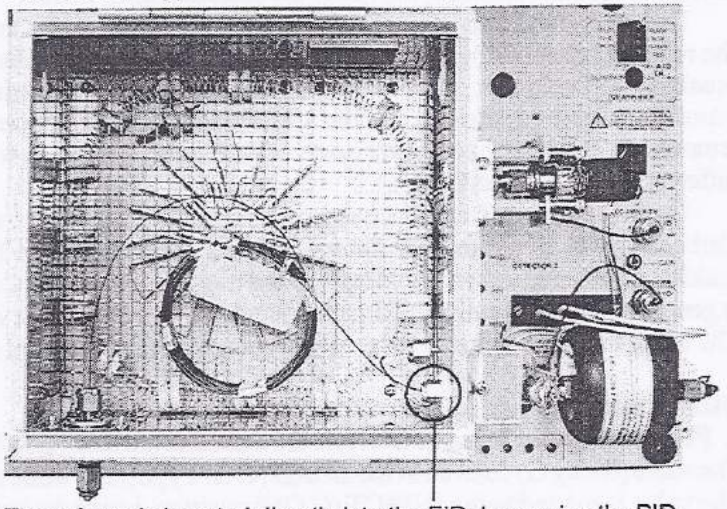
Direct Injection

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

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

2. Load or create a Temperature Program for the column oven. You can create an isothermal or ramped temperature program; deciding which to use depends on the sample being analyzed, and the goals of the analysis. There are several preset .tem files included with version 2.66 and higher of the PeakSimple software. If the analysis requires the column to be hotter than 150°C, it is best to disconnect the column from the PID detector. The PID represents a cold spot in which higher boiling analytes will become trapped, never making it to the much hotter (300°C) FID for detection. Also, when the column is heated over 150°C, stationary phase bleed will adhere to the PID lamp window. The higher boiling analytes and the column bleed will create a coating on the PID lamp window that will interfere with the analysis. The PID lamp window may be cleaned in the event of contaminant condensation, but the resulting change in the PID response usually requires detector recalibration. To bypass the PID, turn its lamp current OFF, then disconnect the column from the detector by loosening the swagelok-type nut from the bulkhead fitting in the column oven wall. Remove the tubing that connects the PID exit to the FID/DELCD by loosening that nut. Place the end of the column into the FID/DELCD bulkhead fitting instead and tighten it in place.

PID detector bypass for column oven temperatures above 150°C

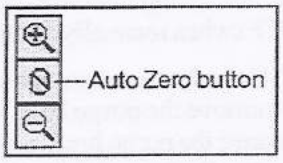


The column is inserted directly into the FID, bypassing the PID.

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

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

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



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

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

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.

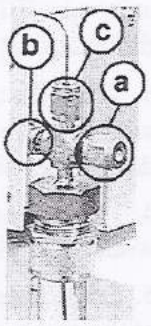
The SRI Purge & Trap concentrator is shipped with a blank trap and a Tenax™-GR trap installed, and a Carbosieve™ S-III packed trap for optional user installation. The Tenax-GR trap has a low affinity for water, making it a good adsorbent for the purge and trap technique. The Carbosieve has a high affinity 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.

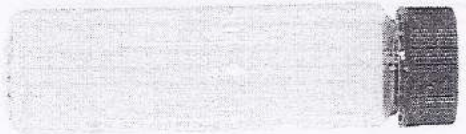
2. Load or create a temperature program for the column oven.

3. Perform **Detector Steps** 1-4, then proceed with the following steps.

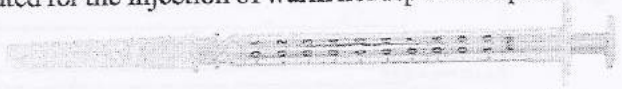
4. Fill a plastic medical syringe with the vial headspace. Inject the sample into the GC injection port, bypassing the Purge & Trap concentrator.

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

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



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



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

Detector Steps

1. With the black plastic lamp hood in place on the PID lamp, turn ON the PID lamp current with the flip switch on the GC's front control panel. Set the PID current to 70 (= 0.70ma) by adjusting the appropriate trimpot setpoint on the top edge of the GC's front control panel. (Each detector zone is labeled on the front control panel under DETECTOR PARAMETERS, with the corresponding trimpot setpoint directly above it.) The lamp should emit a violet-colored light visible down the center of the tube. Set the PID temperature to 150°C. Set the PID gain to LOW.

2. Turn on the air compressor using the switch on the GC's front control panel. NOTE: since most ambient air will not cause interference with the DELCD, the built-in air compressor is appropriate for most analytical situations. However, if you are doing analyses in a lab environment with low levels of halogenated compounds in the ambient air, they can cause the DELCD to lose sensitivity, and fluctuations in the level of organics in ambient air may cause additional baseline noise. To avoid this, use clean, dry tank air.

3. Set the FID hydrogen flow to 25mL/min, and the FID air flow to 250mL/min. The pressure required for each flow is printed on the right hand side of the GC chassis. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. Ensure that the flame is lit by holding the shiny surface of a chromed wrench to the tip of the collector electrode; when the flame is lit, you should be able to see condensation on the wrench. Set the FID gain to HIGH. If the peaks are more than 20 seconds wide at the base, use the HIGH FILTERED gain setting. If you wish to keep the ignitor ON to prevent flameout, set the ignitor voltage to -750 by adjusting the trimpot on the FLAME IGNITE zone.

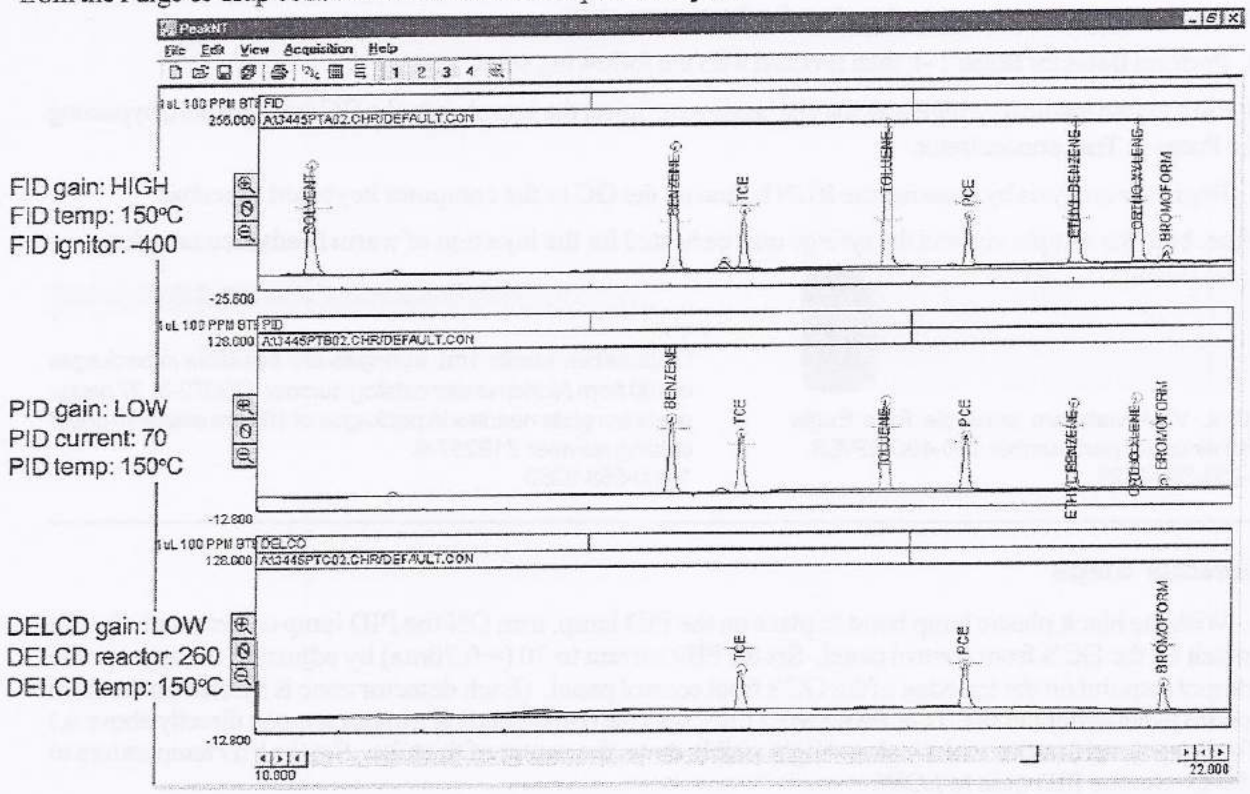
4. If a DELCD detector is installed, set the DELCD reactor temperature setpoint to 260 (=1000°C) by adjusting the appropriate trimpot. The DELCD will heat to around 254 and stabilize; the protruding end of the ceramic tube will glow bright red in the heat. Set the DELCD gain to LOW.

5. When the system has reached temperature and each detector is displaying a stable signal, begin the analysis by pressing the RUN button on the front of the GC or the spacebar on the computer keyboard.

POPULAR CONFIGURATION GCs
BTEX & Environmental

Expected Performance - Purge & Trap Concentrator

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



FID Results:

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

PID Results:

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

DELCD Results:

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

Sample: 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)

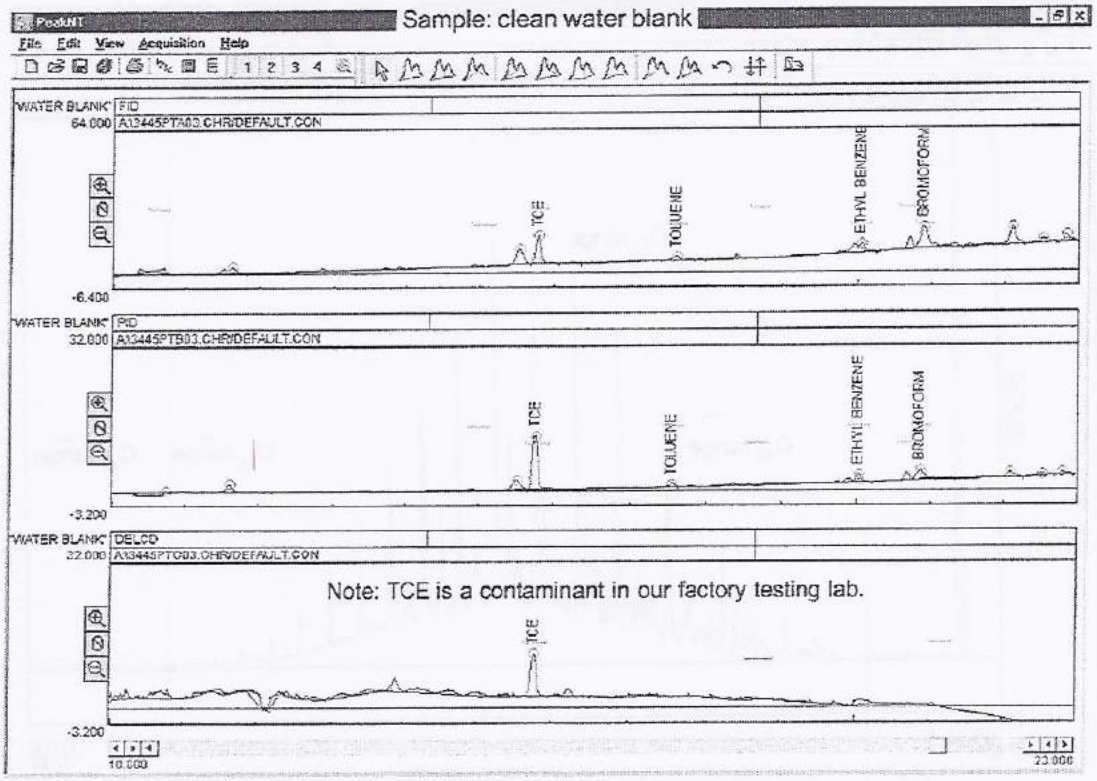
Initial	Hold	Ramp	Final
40°C	10.00	10.00	180°C

Events (5030.evt):

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

Expected Performance - Purge & Trap Concentrator

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



FID Results:

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

PID Results:

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

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340

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

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

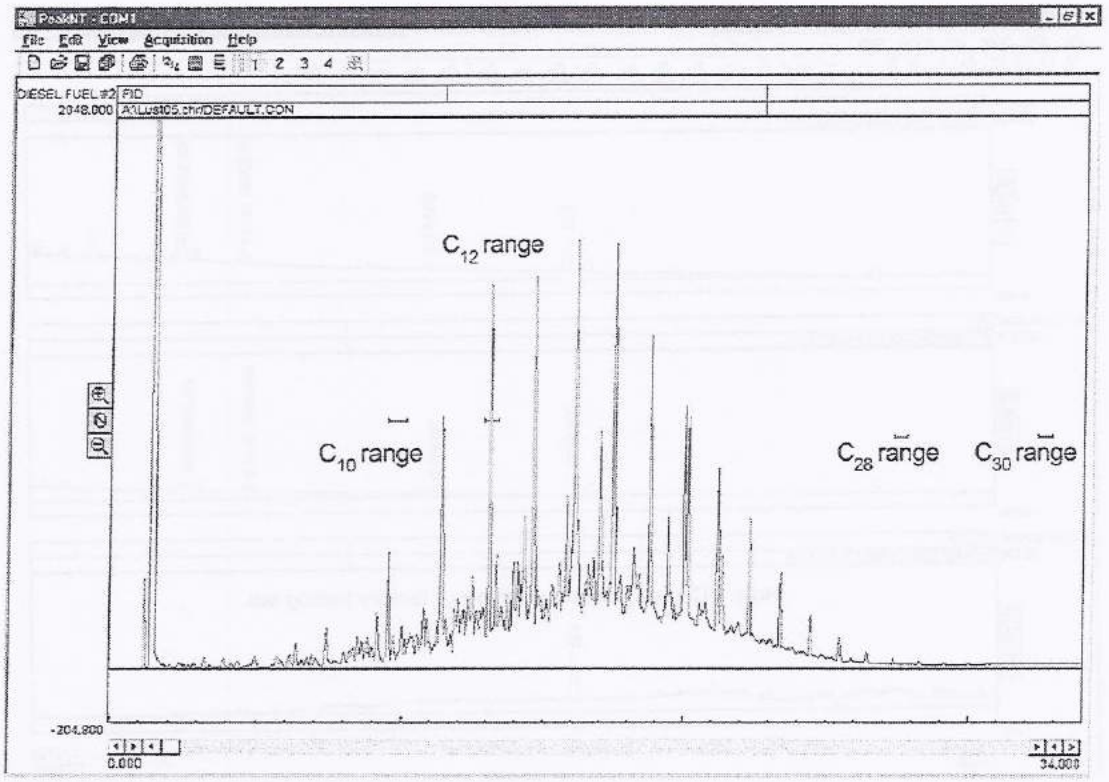
$$353x = 40ppb$$

$$x = 0.1133ppb$$

(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

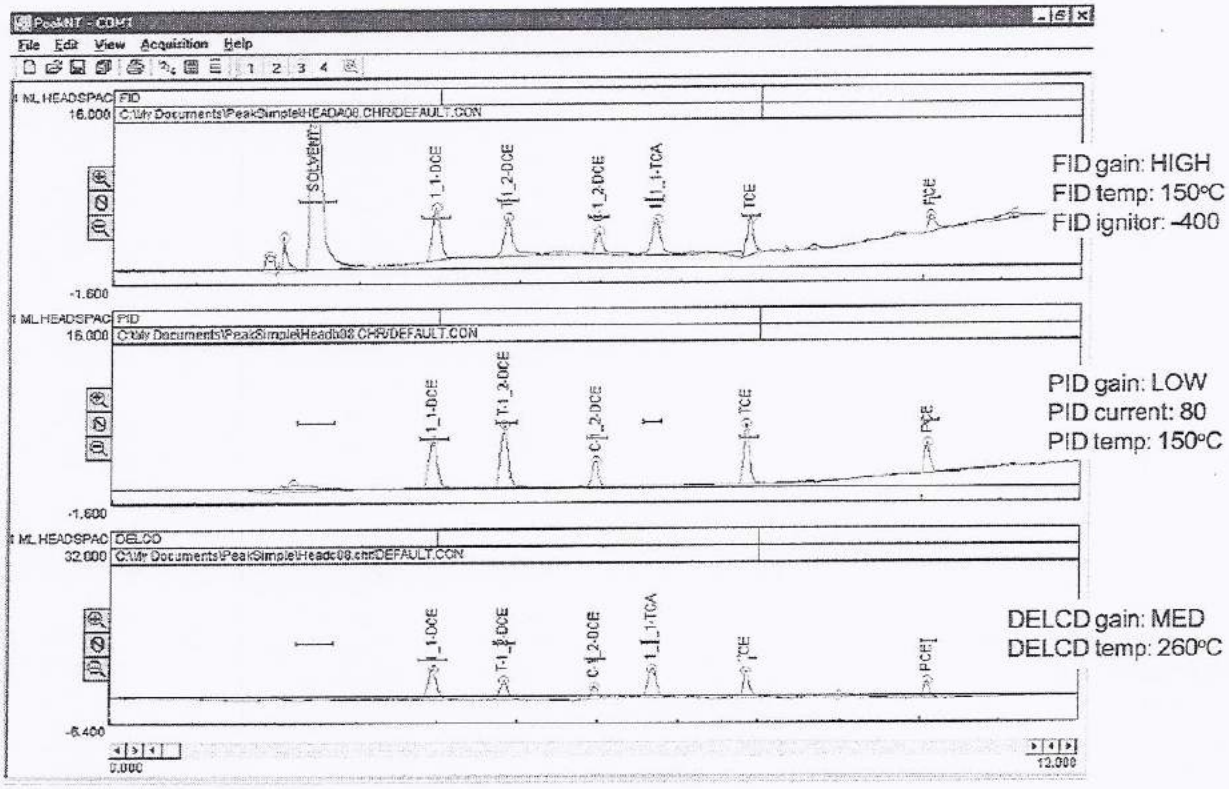
FID gain: HIGH
FID temp: 325°C
FID ignitor: -400

Temperature program:

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

Expected Performance - Manual Headspace Injection

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



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

Temperature program:

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

FID Results:

Component	Retention	Area
Solvent	2.416	290.1100
1_1-DCE	3.933	39.6100
T-1_2-DCE	4.833	34.3780
C-1_2-DCE	5.966	18.6020
1_1_1-TCA	6.663	29.6320
TCE	7.850	23.4490
PCE	10.083	10.7560
	Total	446.5370

PID Results:

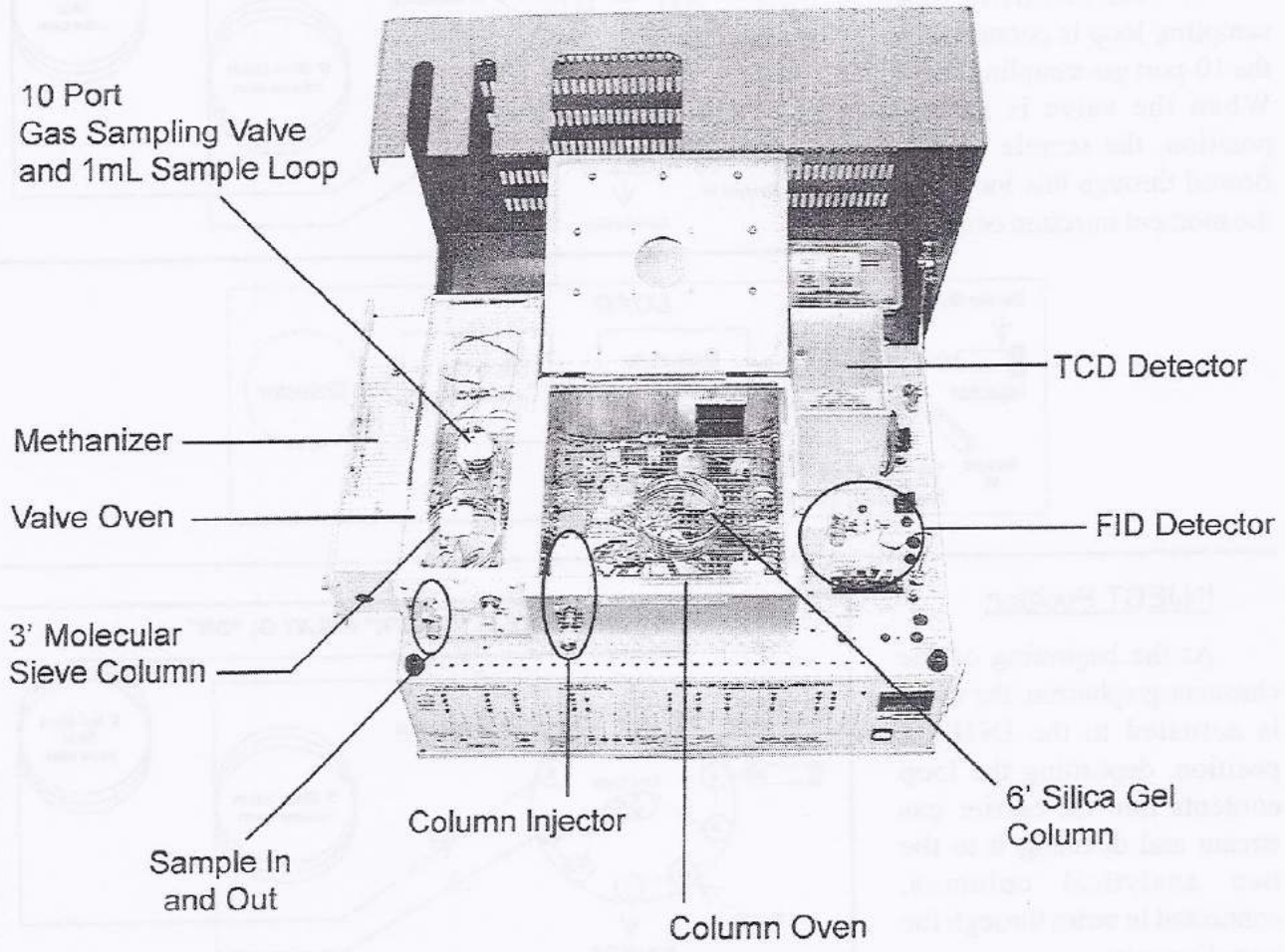
Component	Retention	Area
Solvent	2.183	22.7450
1_1-DCE	3.916	39.4070
T-1_2-DCE	4.800	45.0050
C-1_2-DCE	5.950	15.7380
TCE	7.816	33.7270
PCE	10.066	16.2780
	Total	172.9000

DELCD Results:

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

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.

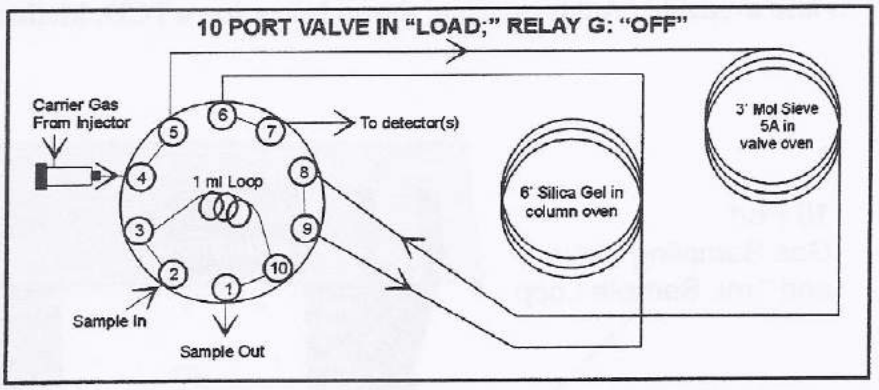
POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

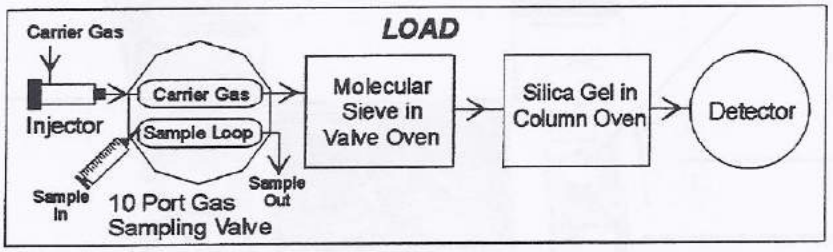
Theory of Operation
10 Port Gas Sampling Valve Plumbing Connections

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

LOAD Position

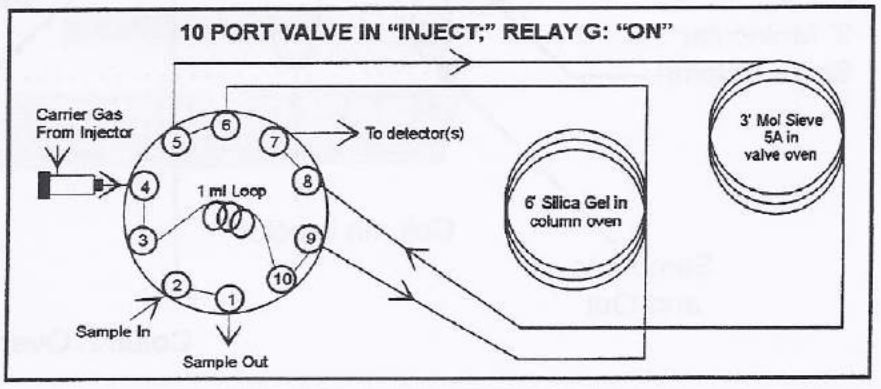
A one-milliliter gas sampling loop is connected to the 10-port gas sampling valve. When the valve is in load position, the sample may be flowed through this loop until the moment injection occurs.



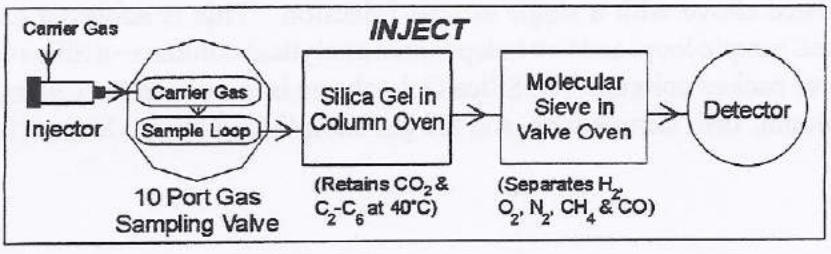


INJECT Position

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



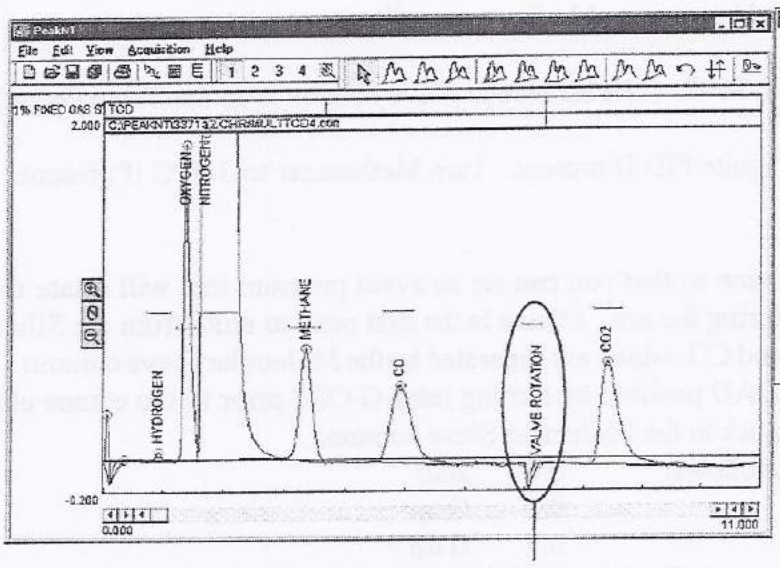
Column sequence is reversed while the flow direction remains the same



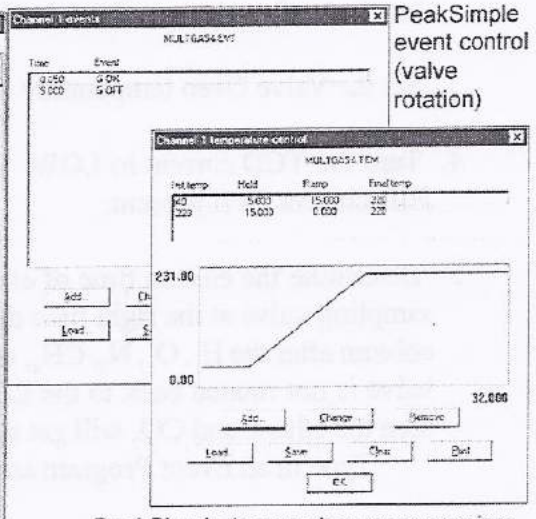
Theory of Operation
10 Port Gas Sampling Valve Plumbing Connections Continued

The sample is deposited first into the Silica Gel packed column, at 40°C in the column oven, where the ethane, propane, butanes, pentanes and carbon dioxide are retained. The remainder of the sample containing hydrogen or helium (whichever is not being used as a carrier), as well as oxygen, nitrogen, methane and any carbon monoxide, continues on to the Molecular Sieve column. During a chromatographic run with the sampling valve in the INJECT position, the hydrogen or helium, oxygen, nitrogen and methane components are the first to elute through the columns and into the detector. This is due to the Silica Gel column's long retention time at 40°C of C_2, CO_2 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



Gas sampling valve rotates back into the load position.

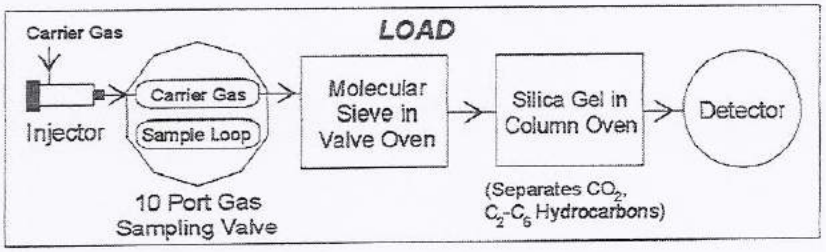


PeakSimple event control (valve rotation)

PeakSimple temperature programming:

Initial Temp	Hold	Ramp	Final Temp
40°C	5.00	15.00	220°C
220°C	15.00	0.00	220°C

This reverses the sequence of the columns prior to the detector, and sends the components preparing to elute from the Silica Gel packed column (ethane, propane, etc.) to the detector without passing them through the Molecular Sieve packed column. At the same time, the Silica Gel packed column is temperature ramped to promote the rapid elution of the remaining components.



POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

General Operating Procedure

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.

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

3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)

4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.

5. Determine the elution time of ethane so that you can set an event program that will rotate the gas sampling valve at the right time during the run. Ethane is the first peak to elute from the Silica Gel column after the H_2, O_2, N_2, CH_4, 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_2 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

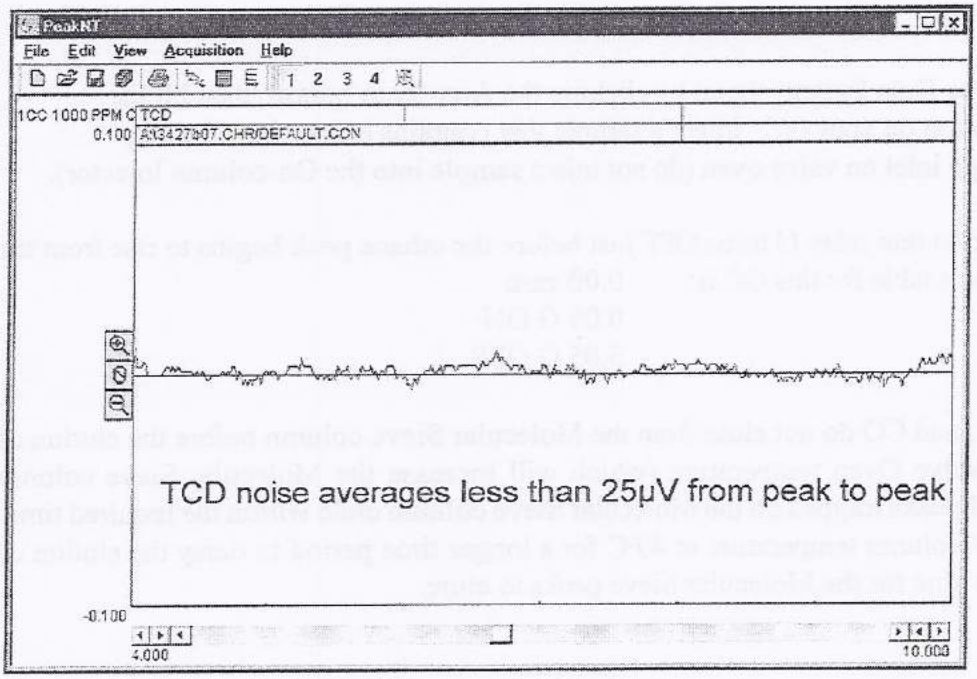
7. In PeakSimple, zero the Data System signal by clicking the Auto Zero button, then hit the spacebar or the run button on your GC. Inject a sample that contains ethane into the Gas Sampling Valve through inlet on valve oven (do not inject sample into the On-column Injector).

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 H_2, O_2, N_2, CH_4, and CO do not elute from the Molecular Sieve column before the elution of ethane, increase the Valve Oven temperature (which will increase the Molecular Sieve column temperature) so that all peaks trapped on the Molecular Sieve column elute within the required time. Or, hold the Silica Gel column temperature at 40°C for a longer time period to delay the elution of ethane, allowing more time for the Molecular Sieve peaks to elute.

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

Expected Performance



TCD noise run

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

Temperature Program:

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

Factory Test Analysis of Natural Gas Standard

Temperature program:

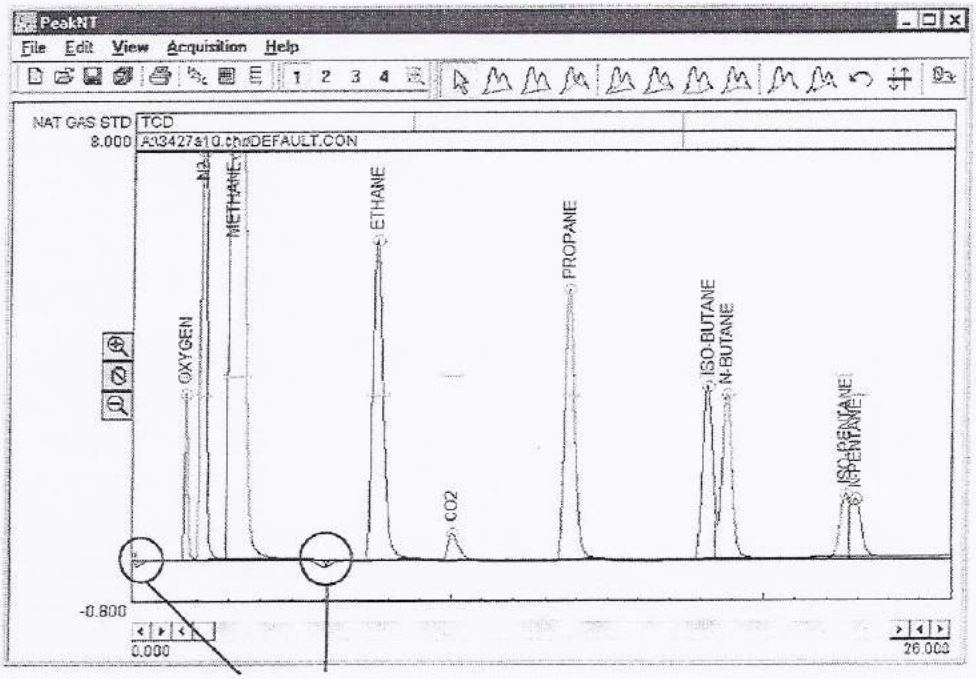
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

Events:

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

Results:

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



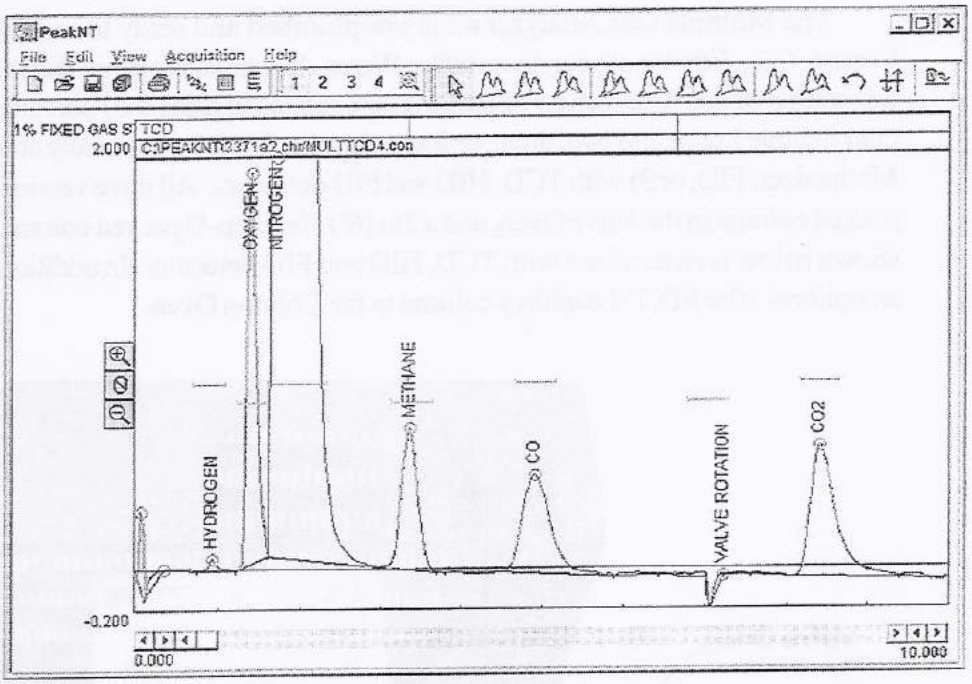
Expected Performance

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

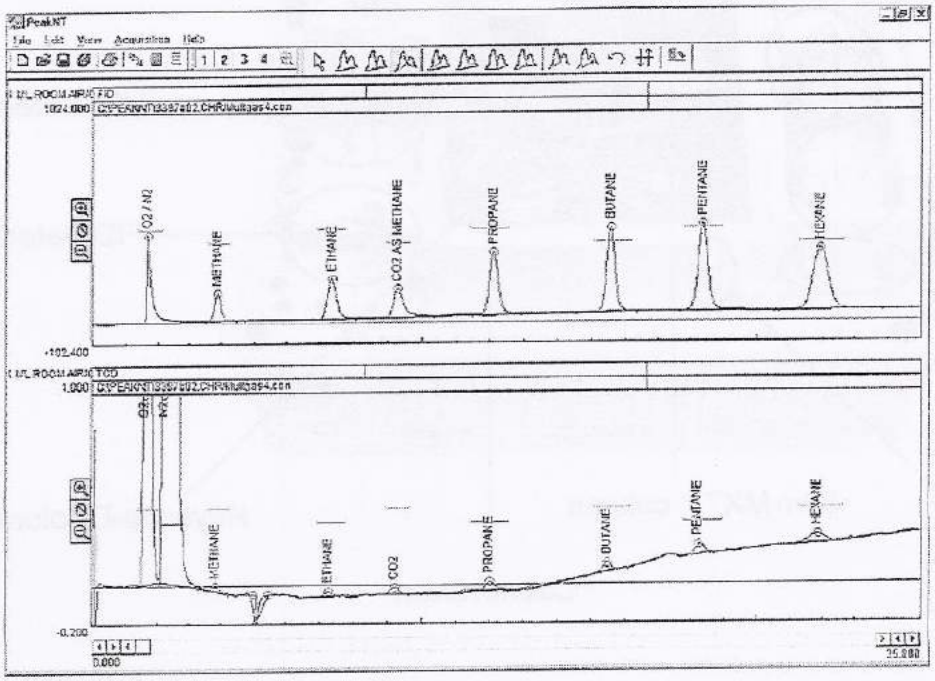
TCD current = LOW
TCD temp = 100°C
Valve Oven temp = 90°C

RESULTS:

Component	Retention	Area
Hydrogen	0.916	0.4710
Oxygen	1.383	14.9530
Nitrogen	1.700	1049.4940
Methane	3.333	9.4875
CO	4.900	11.4460
Valve Rotate	7.200	0.6460
CO_2	8.383	13.8000
	total	1100.2775



Factory Test Analysis of Room Air & C_1-C_6 Hydrocarbons on a dual-channel TCD-Methanizer-FID Multiple Gas Analyzer #1



FID Results:

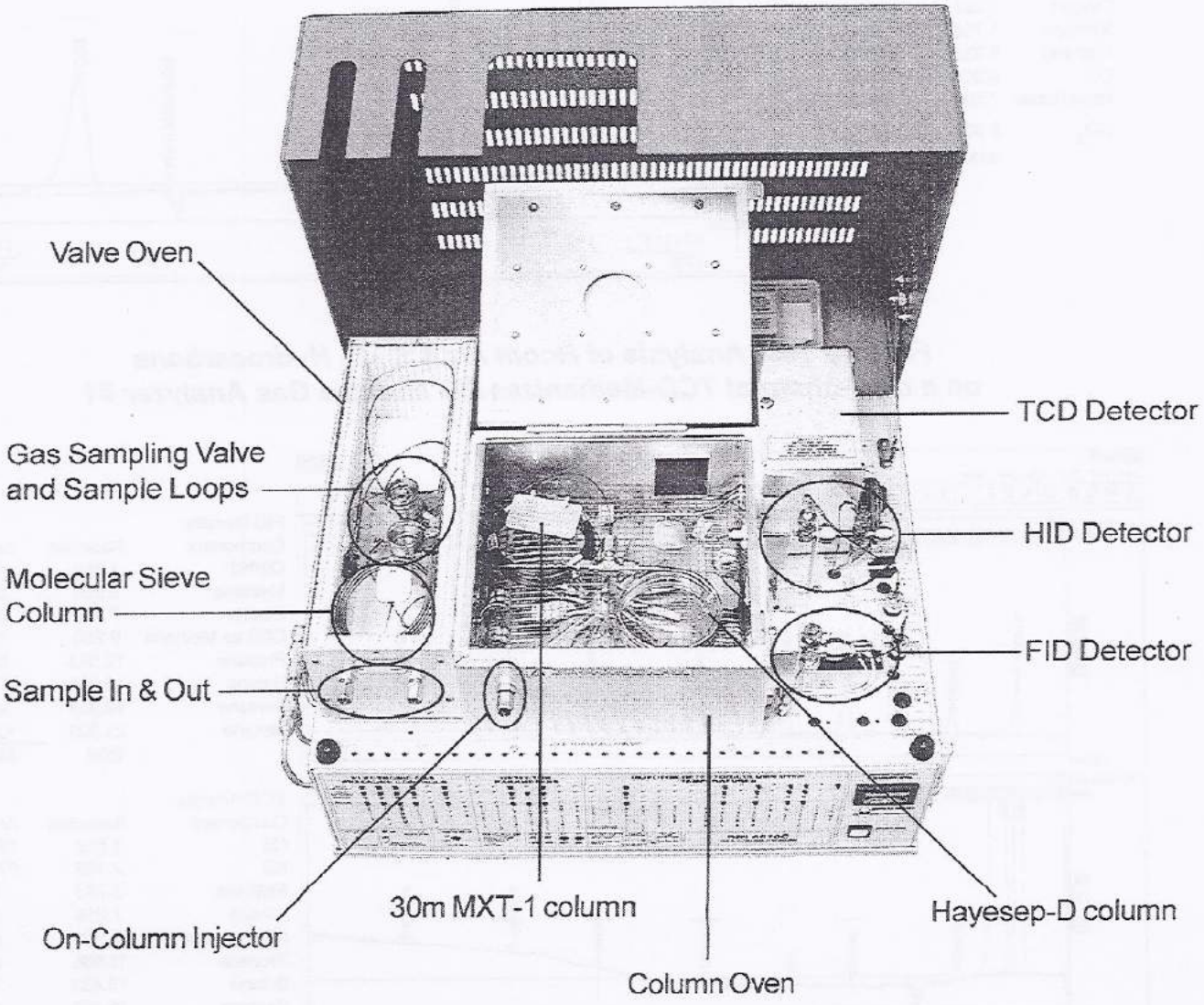
Component	Retention	Area
O2/N2	1.650	4731.2140
Methane	3.866	2008.6000
Ethane	7.316	3854.7300
CO2 as Methane	9.250	3142.1040
Propane	12.083	5379.8755
Butane	15.533	7326.4440
Pentane	18.333	9136.3340
Hexane	21.900	10408.3160
	Total	45987.6175

TCD Results:

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

System Overview

The Multiple Gas Analyzer #2 is pre-plumbed and ready to resolve H_2, He, O_2, N_2, Methane, CO, Ethane, CO_2, Ethylene/Acetylene, NOx, Water, Alcohols, Propane, Butanes, Pentanes, and C_6 through C_{20}. Separation of this wide variety of peaks is accomplished using a 10 port automated Gas Sampling Valve with dual Sample Loops and two, three, or four columns. It can be optionally configured with **1)** a TCD, **2)** a TCD, Methanizer, FID, or **3)** with TCD, HID and FID detectors. All three versions have a 1m (3') Molecular Sieve packed column in the Valve Oven, and a 2m (6') Hayesep-D packed column in the Column Oven. The model shown below is customized with TCD, HID and FID detectors. In addition to the Hayesep-D column, it has an optional 30m MXT-1 capillary column in the Column Oven.

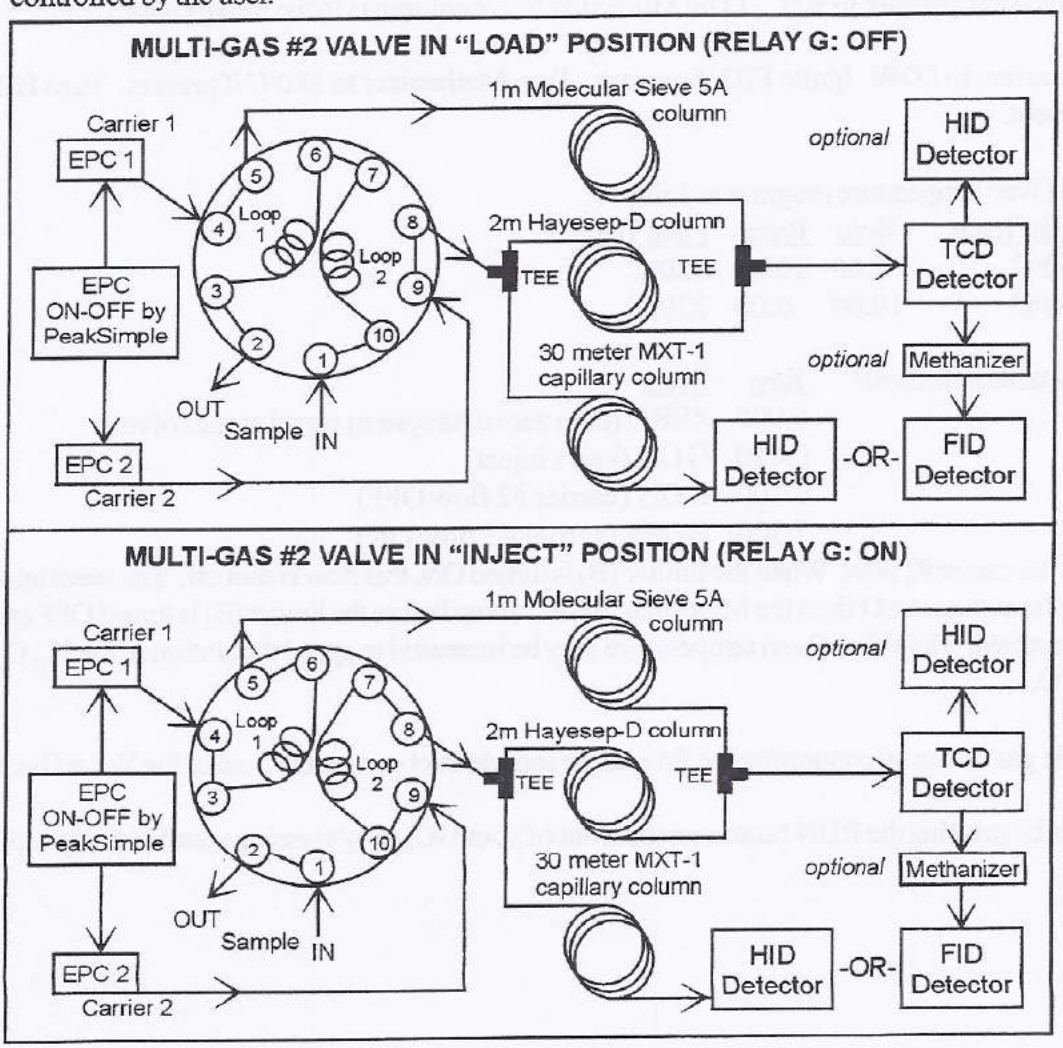


POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Theory of Operation

The Multiple Gas Analyzer #2 GC uses a single automated 10 port Gas Sampling Valve and multiple columns to separate a wide variety of peaks. The system achieves this by turning the carrier gas flow to each column on at different times during the run. This procedure allows the Molecular Sieve column in the Valve Oven to completely separate H_2, He, O_2, N_2, CH_4 and CO before the carrier flow to the Hayesep-D column in the Column Oven is turned on. The Hayesep-D column then separates all compounds in the C_1-C_6 range. An optional 30m MXT-1 capillary column in the Column Oven separates the remaining hydrocarbons out through C_{20}, using the same carrier gas flow as the Hayesep-D column and an FID or HID detector.

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



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

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

General Operating Procedure

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.

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

3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)

4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.

5. Set the Column Oven temperature program as follows:

Initial Temp	Hold	Ramp	Final Temp
50°C	7.00	10.00	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)

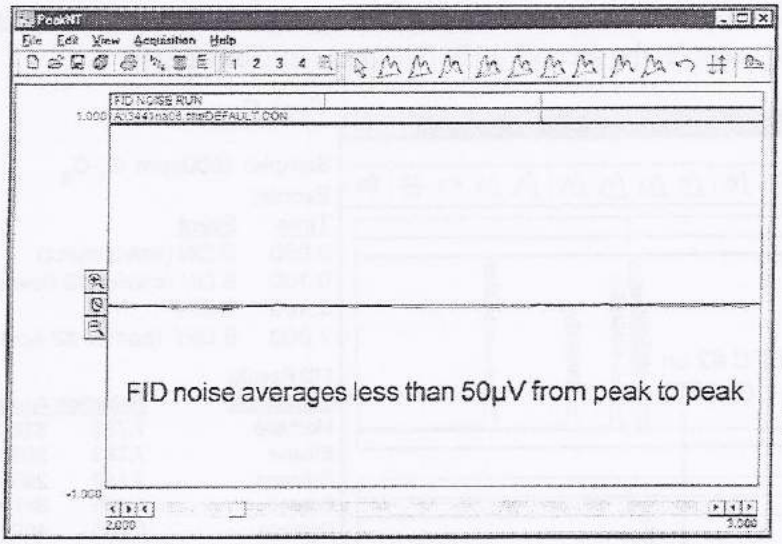
EPC #2 controls the carrier #2 flow. When the limiter (B) is turned ON, this flow is shut off. The event table should allow for the elution of CO from the Molecular Sieve column before the limiter (B) is turned OFF and carrier #2 flow restored. The Valve Oven temperature may be increased to speed the 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:

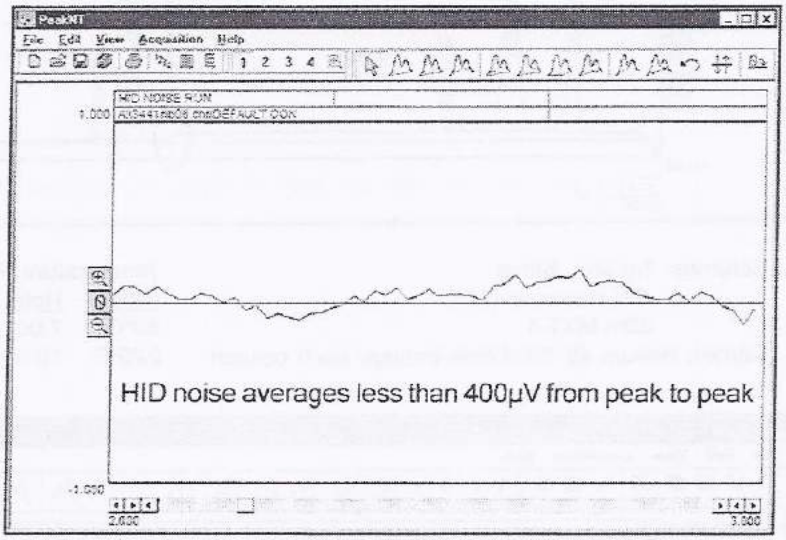
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

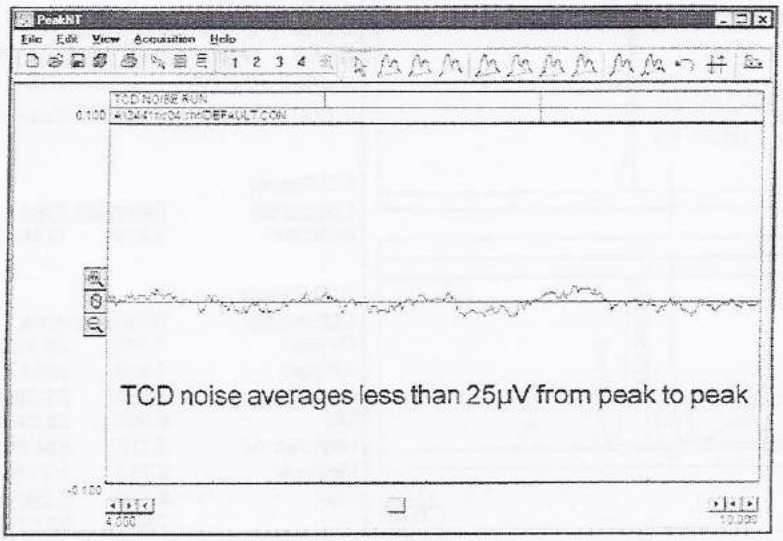
HID noise run

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	Hold	Ramp	Final
80°C	15.00	0.00	80°C





TCD noise run

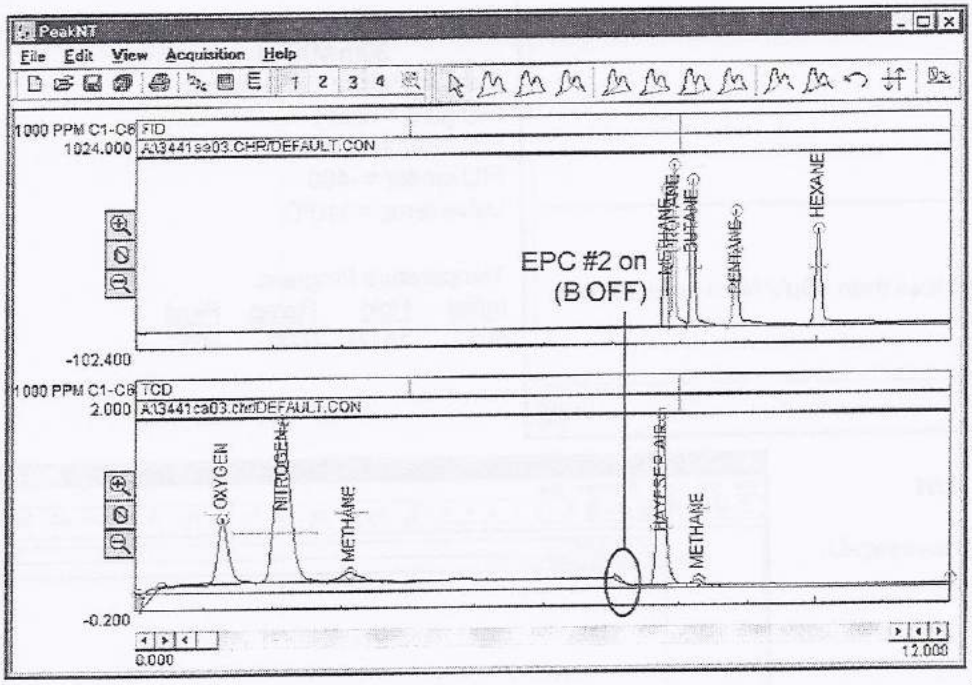
Columns: 1m Mol. Sieve, 2m Hayesep-D, 30m MXT-1
Carrier: Helium @ 10mL/min
TCD gain = LOW
TCD temp = 100°C
Valve temp = 110°C

Temperature Program:

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

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

Sample: 1000ppm C_1-C_6

Events:

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

FID Results:

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

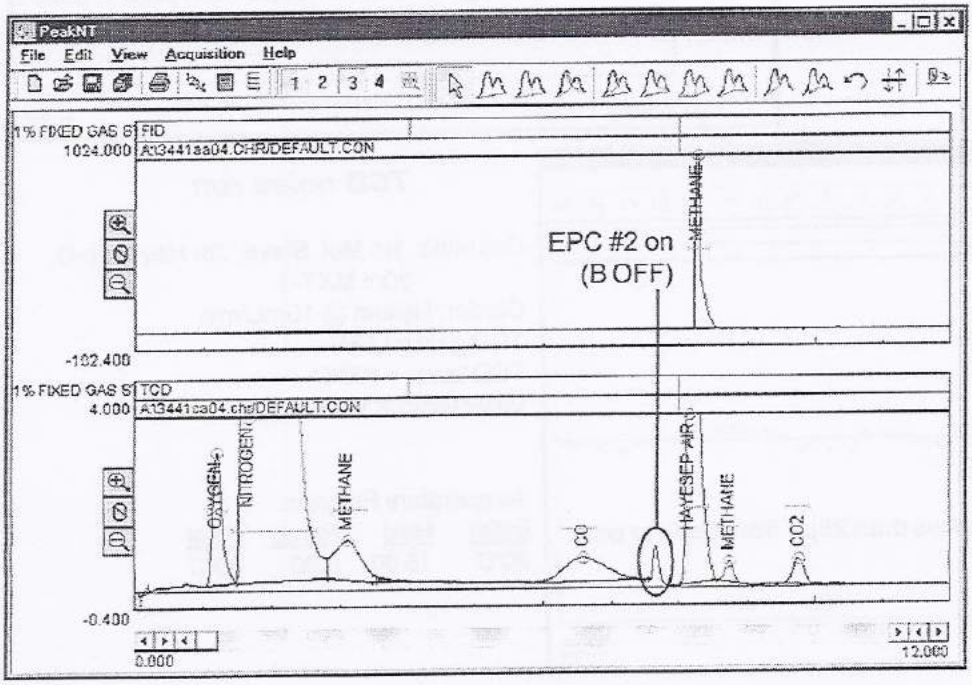
TCD Results:

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

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

Temperature Program:

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



Test Run #2

Sample: 1% fixed gas standard

Events:

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

FID Results:

Component	Retention	Area
Methane	8.233	12144.3770

TCD Results:

Component	Retention	Area
Oxygen	1.166	26.4920
Nitrogen	1.633	2251.7140
Methane	3.083	23.0975
CO	6.566	22.2440
Hayesep Air	8.116	524.2010
Methane	8.716	3.7730
CO2	9.750	6.3940
	Total	63.0115

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Expected Performance: HID & TCD Detectors

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

Test Run #1

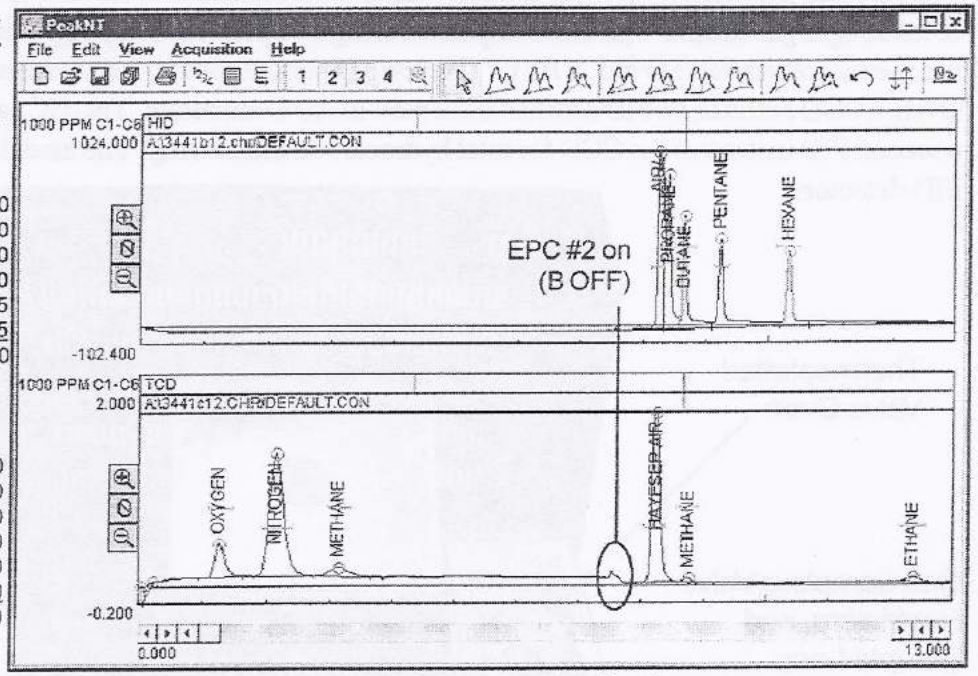
Sample: 1000ppm C_1-C_6

HID Results:

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

TCD Results:

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



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

Temperature Program:

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

Events:

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

Test Run #2

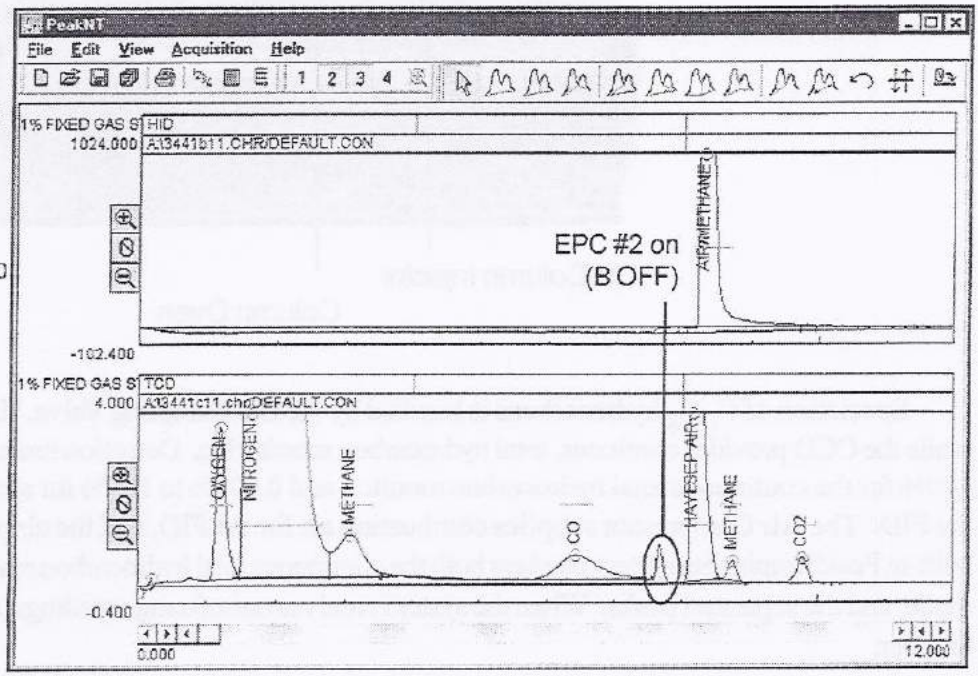
Sample: 1% fixed gas standard

FID Results:

Component	Retention	Area
Methane	8.266	44548.0540

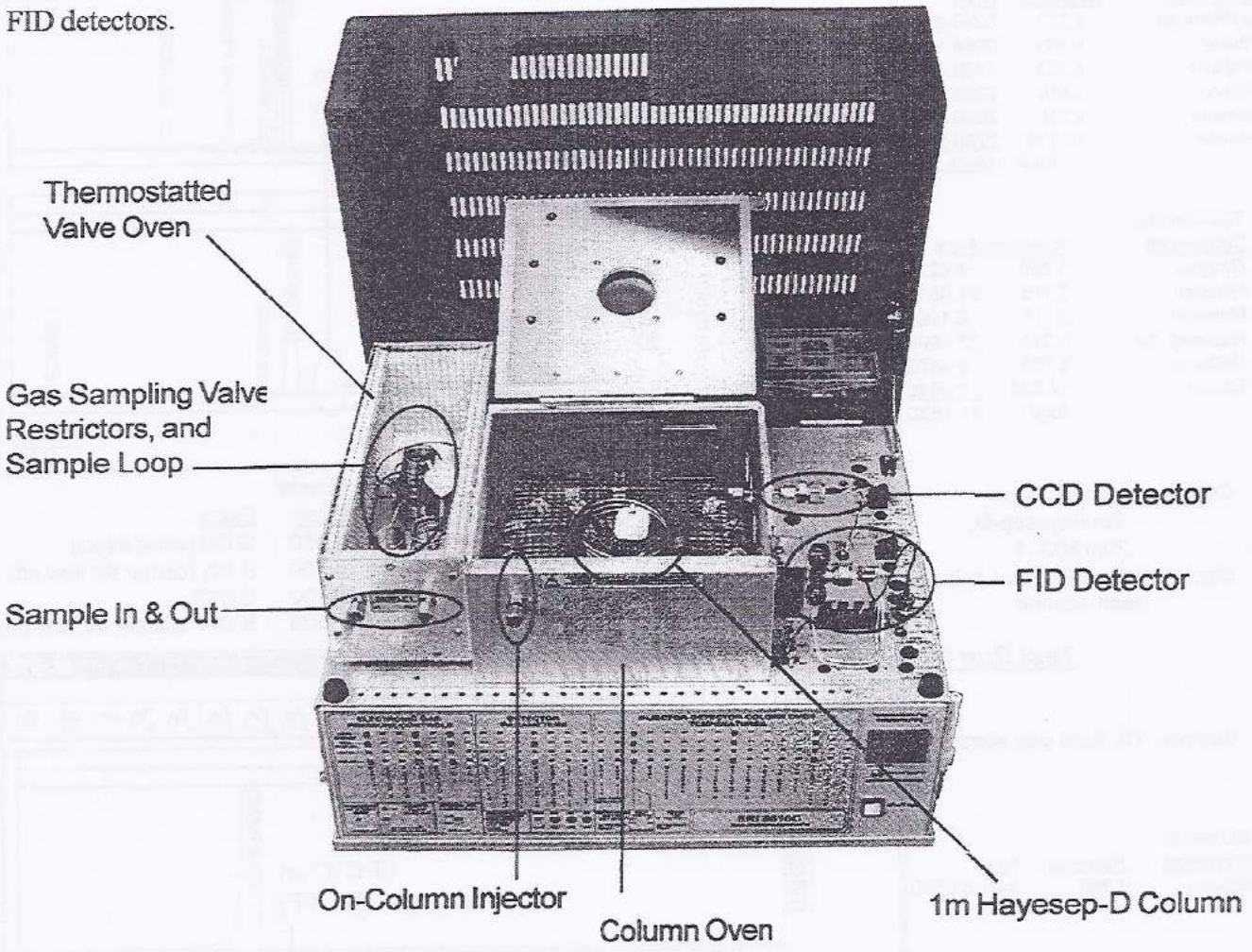
TCD Results:

Component	Retention	Area
Oxygen	1.166	31.0260
Nitrogen	1.616	2261.6430
Methane	3.050	12.6240
CO	6.400	22.4410
Hayesep Air	8.116	542.6790
Methane	8.716	3.5950
CO2	9.750	6.6920
	Total	2880.7000



System Overview

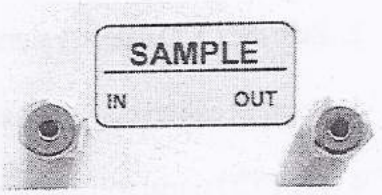
The Mud-Logging GC system is designed to provide a continuous reading of total hydrocarbons in a gas stream, while periodically performing a chromatographic separation of the sample to determine the composition of the sample gas stream. This is accomplished using a 10 port Gas Sampling Valve with a 25µL 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 FID detectors.

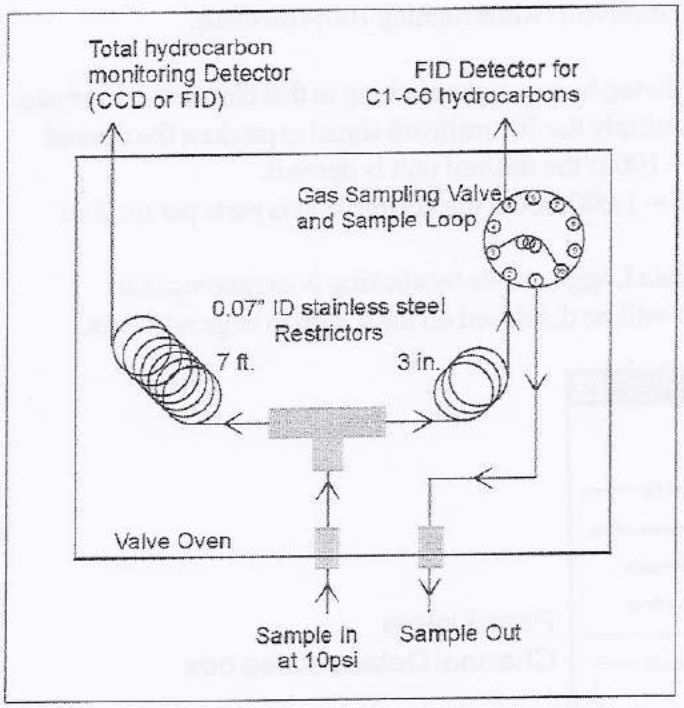


Speciation of C_1-C_6 hydrocarbons is handled by the Gas Sampling Valve, Hayesep-D column, and FID while the CCD provides contiuous, total hydrocarbon monitoring. Detection limits for this system are 0.1% to 100% for the continuous total hydrocarbon monitor, and 0.005% to 100% for speciated hydrocarbons 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.

Theory of Operation

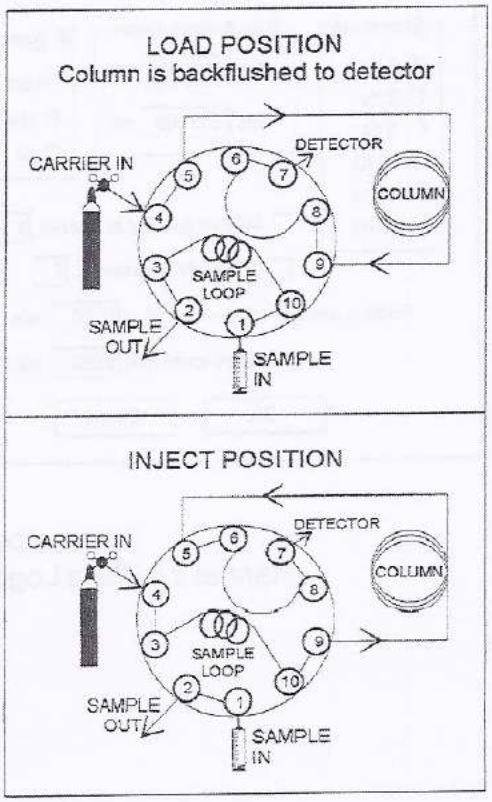
The sample gas stream is connected to a bulkhead fitting on the system's thermostatted Valve Oven where it flows through the sampling loop of the 10 port Gas Sampling Valve, and also to the CCD detector. The fitting labelled "Sample In" (pictured at right) on the front of the Valve Oven is the sample gas stream inlet. The user must regulate the pressure of the sample stream so that it enters this inlet at 10psi. The instrument is factory preset to deliver 5mL/min to the CCD at 10psi. The remainder of the flow, approximately 100mL/min, passes through the Sample Loop. This relatively high flow rate gets the sample from the sampling point into the GC with minimal delay.





10 Port Gas Sampling Valve

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.



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 25µL 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.

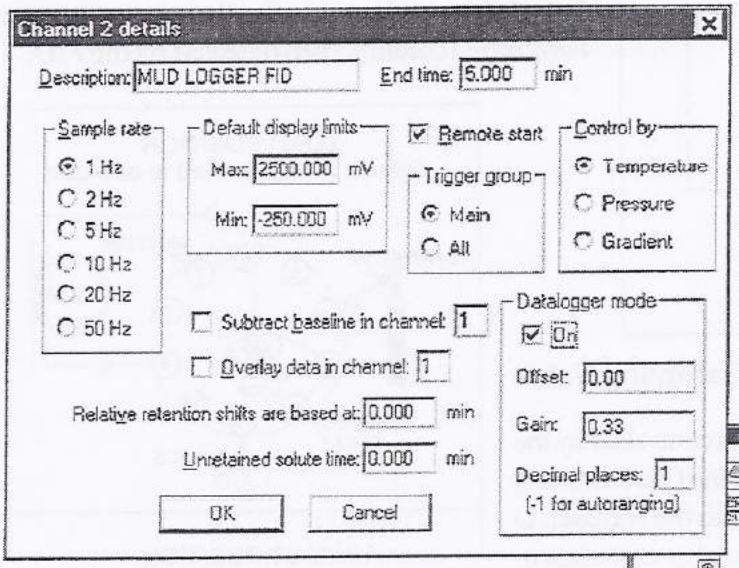
2. Zero the CCD detector signal using the Auto Zero button for its channel (typically channel 2).

3. Connect calibration gas standard to the sample inlet at 10psi. Calibration gas is typically 100% methane.

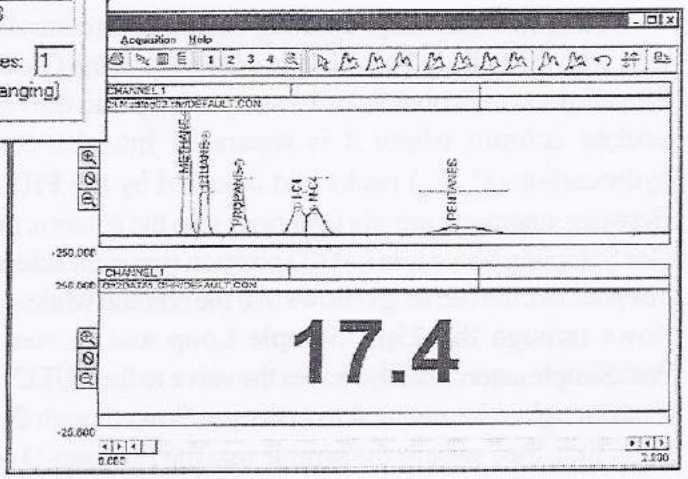
4. The CCD signal will increase approximately 300 millivolts while running 100% methane.

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

6. Also in the Channel Details dialog box, select Data Logger mode by clicking in the appropriate checkbox. The CCD signal times the gain factor will be displayed on the screen in large numbers.



PeakSimple
Channel Details dialog box



Chromatogram with
channel 2 in Data Logger mode

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

1. Connect the calibration gas standard to the sample inlet at 10psi.

2. Set the Valve Oven temperature to 90°C.

3. Ignite the FID.

4. Set an isothermal Column Oven temperature program as follows:

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

5. Type in an even table as follows:

Time	Event
0.00	Zero
0.050	G ON
1.5	G OFF

6. Set the FID gain to MEDIUM.

7. Start the analysis by hitting the spacebar on the computer keyboard.

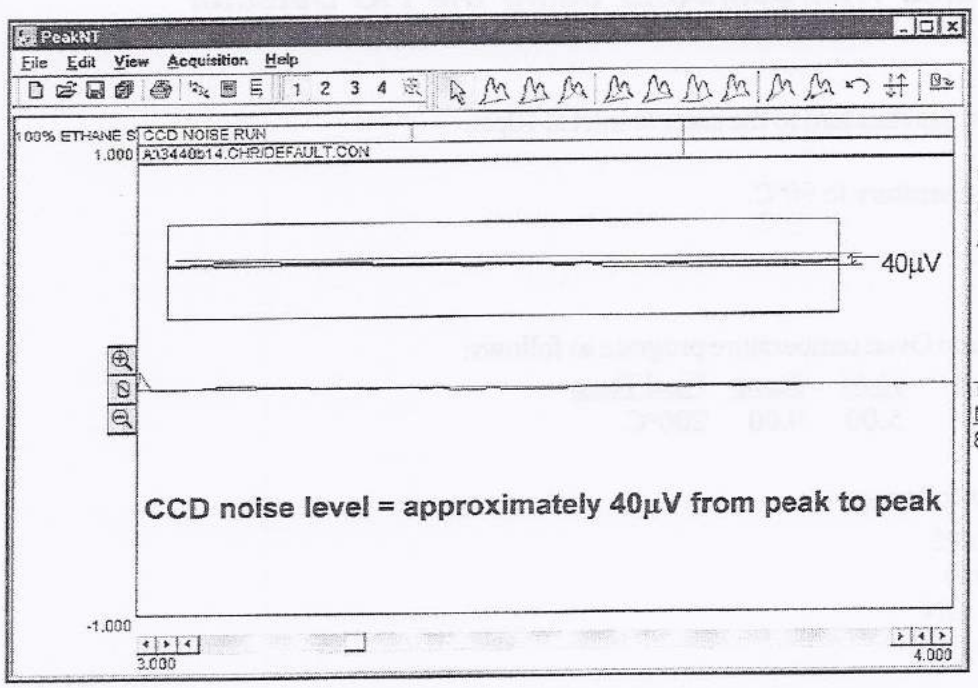
8. In PeakSimple, input the retention windows to identify the individual hydrocarbon components (methane, ethane, propane, butane, etc).

9. Calibrate the individual hydrocarbon peaks.

10. This instrument is plumbed for backflush. This gives the user the option to set the valve program to backflush the heavier hydrocarbons after the desired peaks have been separated. For instance, if your application required separation of hydrocabons up to C_5, you could set the valve to backflush after the elution of the C_5 component(s), and all the heavier hydrocarbons would together produce one large peak.

Expected Performance

CCD Noise



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

Temperature program:

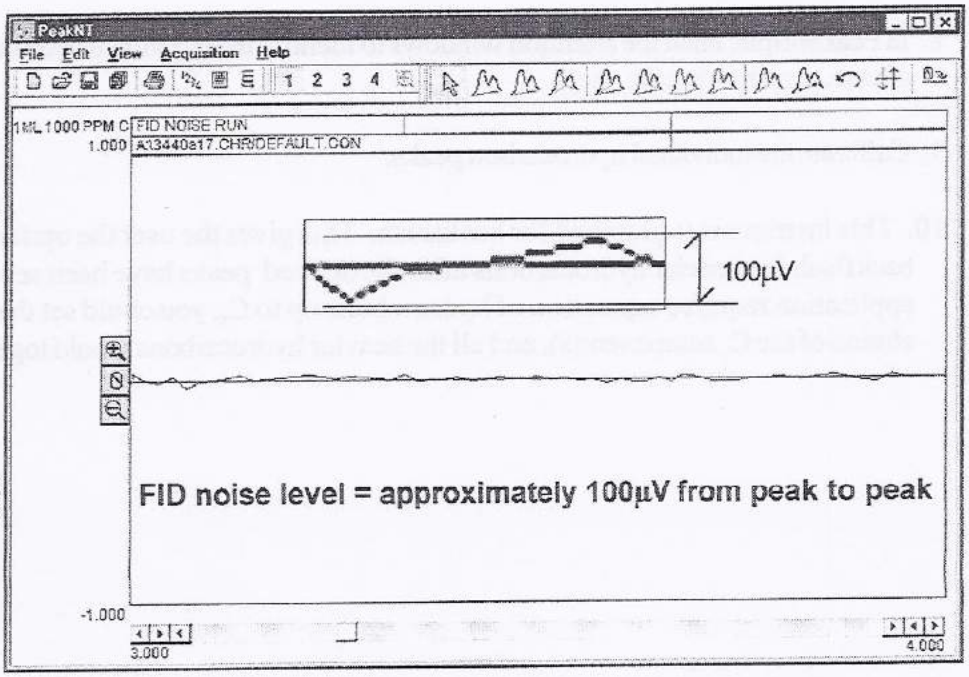
Initial	Hold	Ramp	Final
80°C	5.00	0.00	80°C

FID Noise

Column: 1m Hayesep-D
Carrier: Helium @ 10mL/min
FID gain = High
FID temp = 150°C
FID ignitor = -400
Valve temp = 90°C

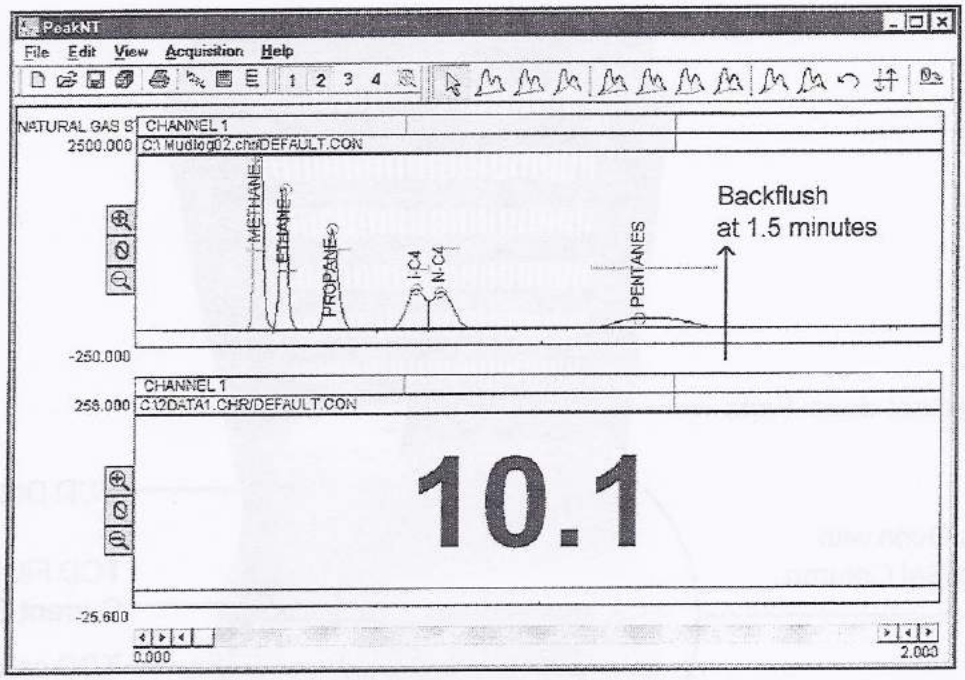
Temperature program:

Initial	Hold	Ramp	Final
80°C	5.00	0.00	80°C



Expected Performance

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



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

Temperature program:

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

Events:

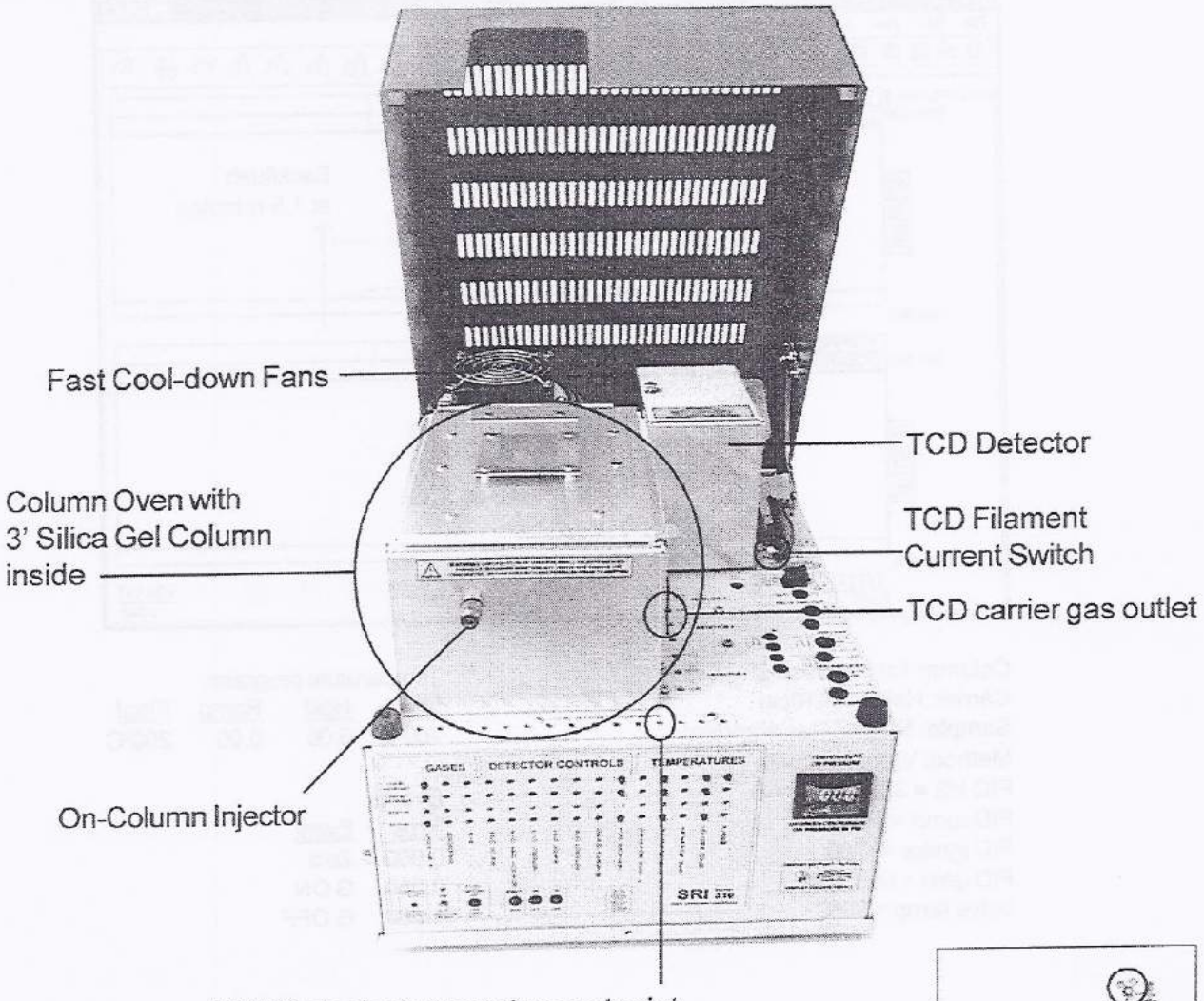
Time	Event
0.000	Zero
0.050	G ON
1.500	G OFF

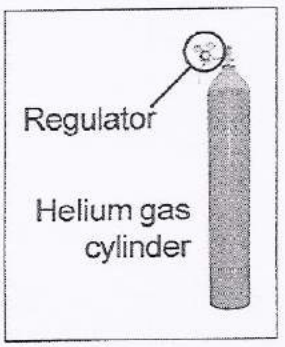
Results:

Component	Retention	Area
Methane	0.291	6664.1410
Ethane	0.366	2770.3785
Propane	0.483	2762.6450
i-C4	0.691	1754.0118
N-C4	0.750	1913.8415
Pentanes	1.241	1580.4310
	Total	17445.4488

System Overview

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





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

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

General Operating Procedure

1. Check to make sure that the TCD filament current is switched OFF. Plug in and turn on your GC. Allow the TCD detector oven to reach temperature (100°C) and stabilize. With the "Display Select" switch in the UP position, press on the TCD Temperature Actual button on the front control panel to read the TCD cell temperature.

2. The carrier gas head pressure is preset at the factory to 10mL/min for the Silica Gel column. Look on the right side of the GC for the carrier pressure that correlates to a flow of 10mL/min. Because different columns require different flow rates, the carrier head pressure may be adjusted by the user with the trimpot above the "CARRIER 1" buttons. For this GC, carrier cylinder pressure is normally set at 30psi, which is 10-20mL higher than the column head pressure. The column head pressure is the pressure developed by the carrier gas as it flows through the analytical column.

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

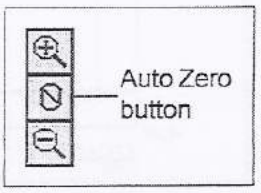
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column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

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

6. In PeakSimple, set an isothermal Column Oven temperature ramp program as follows:

Initial Temp.	Hold	Ramp	Final Temp.
80.00	7.00	0.00	80.00

7. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Hit the RUN button on your GC or hit the spacebar on your computer keyboard to begin the run. You may also open the Acquisition pull-down menu and select Run, but this gets difficult unless you have a partner, since your hands are occupied with the sample syringe.



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

Expected Performance

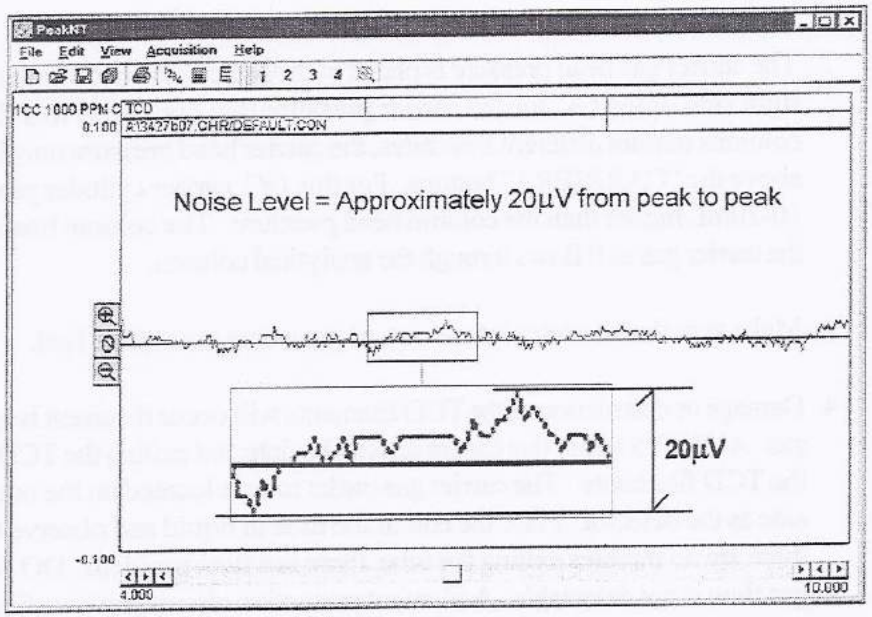
Every compound possesses some degree of thermal conductivity and therefore may be measured with a TCD detector. TCD detectors are most often used with helium as a carrier gas because of helium's high thermal conductivity, but other gases such as nitrogen, argon, or hydrogen may also be used as a carrier gas. A TCD detects all molecules in concentrations from 100% down to around 100ppm, and is especially useful for measuring inorganic gases like O_2, N_2, CO & CO_2.

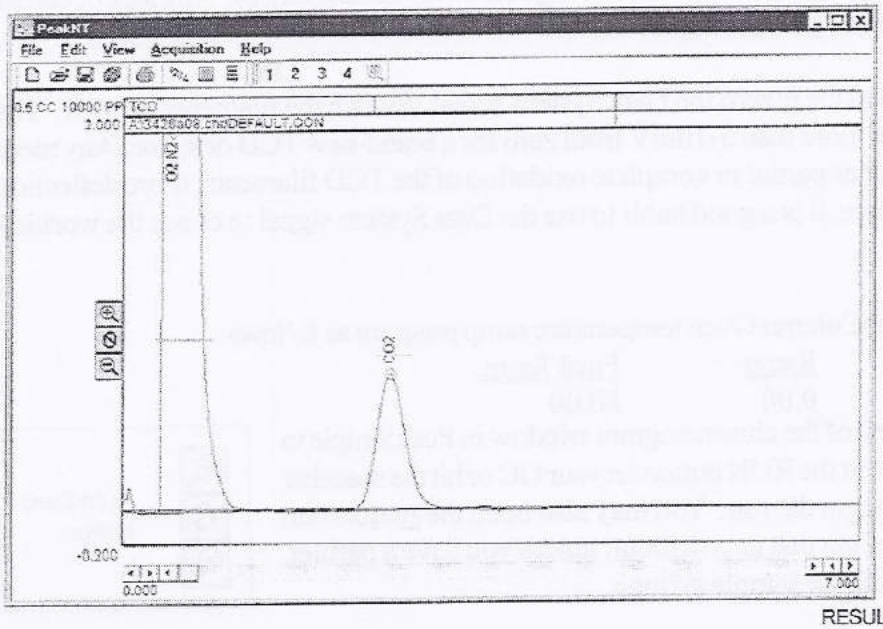
TCD Detector Noise

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 CO_2
TCD current = LOW
TCD Temp = 100°C

Temperature Program:

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

RESULTS:

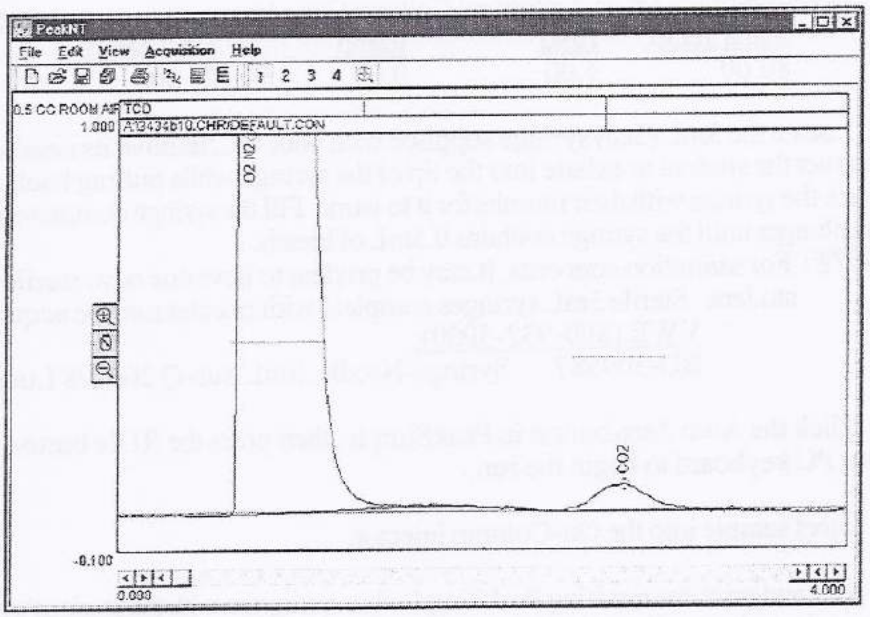
Component	Retention	Area
O2 N2	0.450	1252.9980
CO2	2.500	13.6460
Total		1266.6440

POPULAR CONFIGURATION GCs
Educational TCD

Expected Performance

TCD Room Air Analysis

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



Temperature Program:

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

Results:

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

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

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:
VWR (800-932-5000):
BD-309587 Syringe-Needle, 3mL Sub-Q 26G 5/8 Luer-lok™

4. Click the Auto Zero button in PeakSimple, then press the RUN button on your GC or the spacebar on your PC keyboard to begin the run.

5. Inject sample into the On-Column injector.

6. Save and print the resulting PeakSimple chromatogram with the student's name for the sample identification. Typical results are about 12-14 area counts per 1% of CO_2.

7. Repeat steps 2-5 for each student. Compare chromatograms to find the winner.

Example TCD Breath Analysis

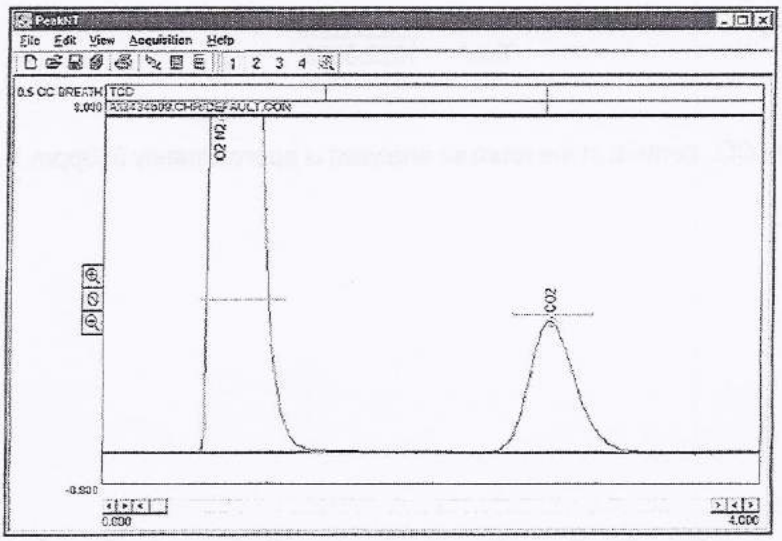
Column: 3' Silica Gel
Carrier: Helium at 10mL/min
Sample: 0.5cc human breath, direct injection
TCD current: LOW
TCD temperature: 100°C

Temperature Program:

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

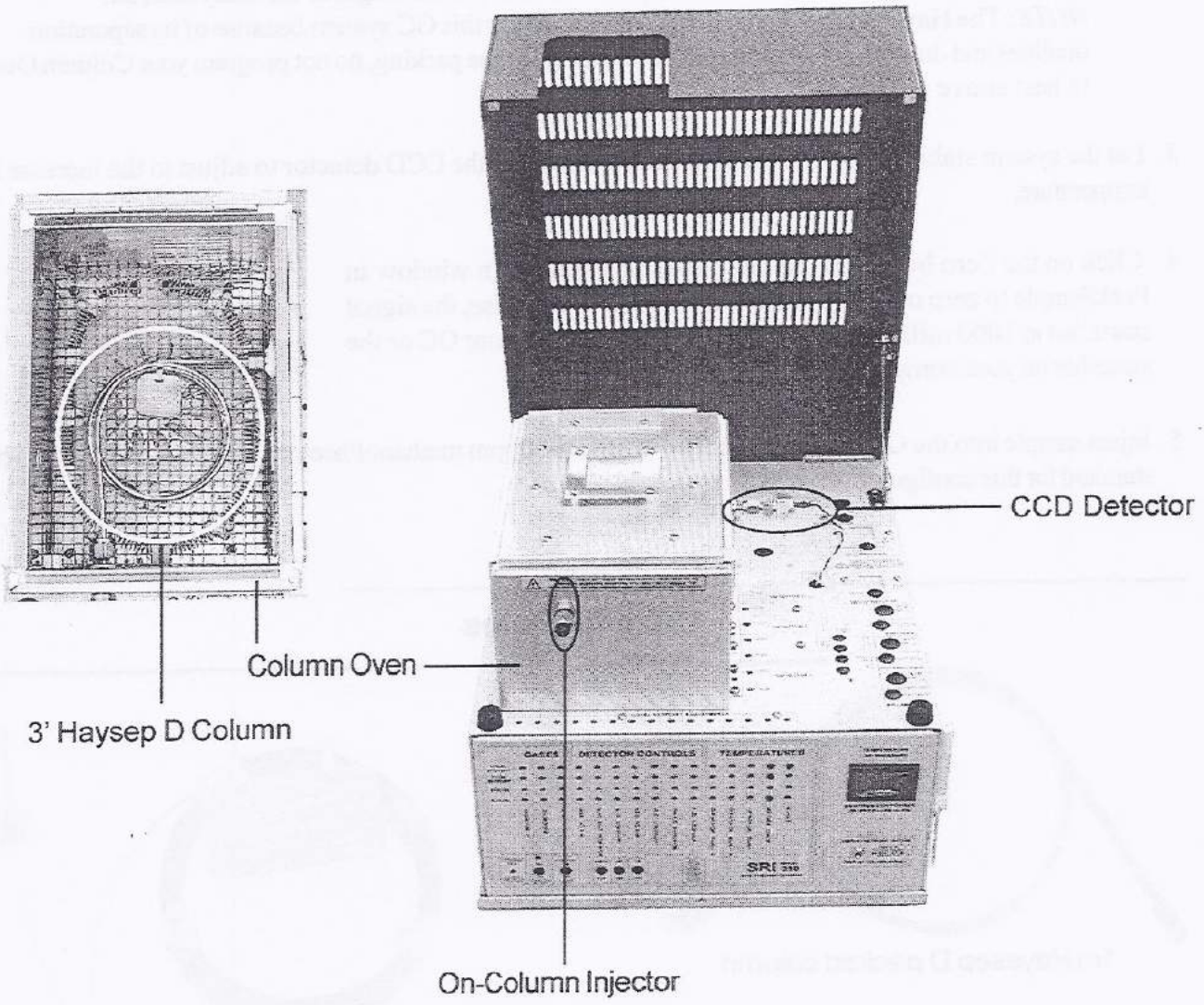
Results:

Component	Retention		Area
O_2 N_2	0.700		1379.4740
CO_2	2.700		61.9540
		Total	1441.4280



System Overview

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



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

General Operating Procedure

1. Connect your GC to your Windows PC with PeakSimple installed. Plug in your GC and turn its power on.

2. Set the Column Oven temperature to 130°C in PeakSimple as follows:

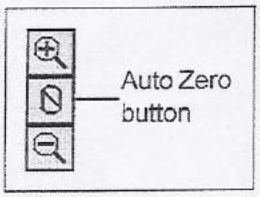
Initial Temp	Hold	Ramp	Final Temp
130.00	10.00	0.00	130.00

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

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

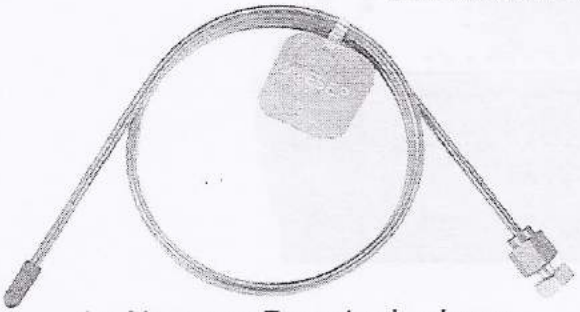
3. Let the system stabilize for at least 10 minutes, allowing the CCD detector to adjust to the increase in temperature.

4. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Otherwise, the signal starts out at 1000 millivolts. Press the RUN button on your GC or the spacebar on your computer keyboard to begin the run.



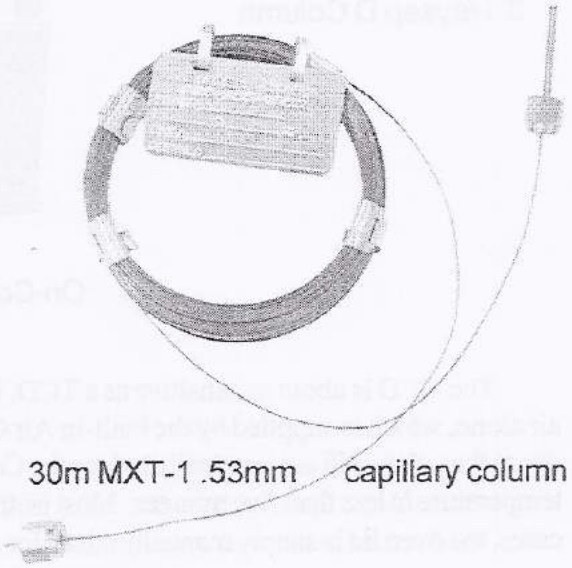
5. Inject sample into the On-Column Injector. A 1µL 1000ppm methanol/acetone sample is the factory test standard for this configuration.

Column Notes



1m Hayesep 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.



30m MXT-1 .53mm capillary column

Expected Performance

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

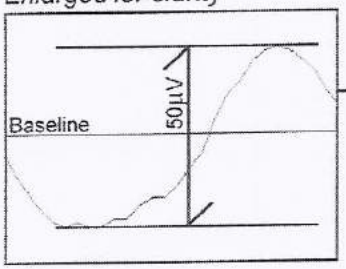
CCD Detector Noise Run

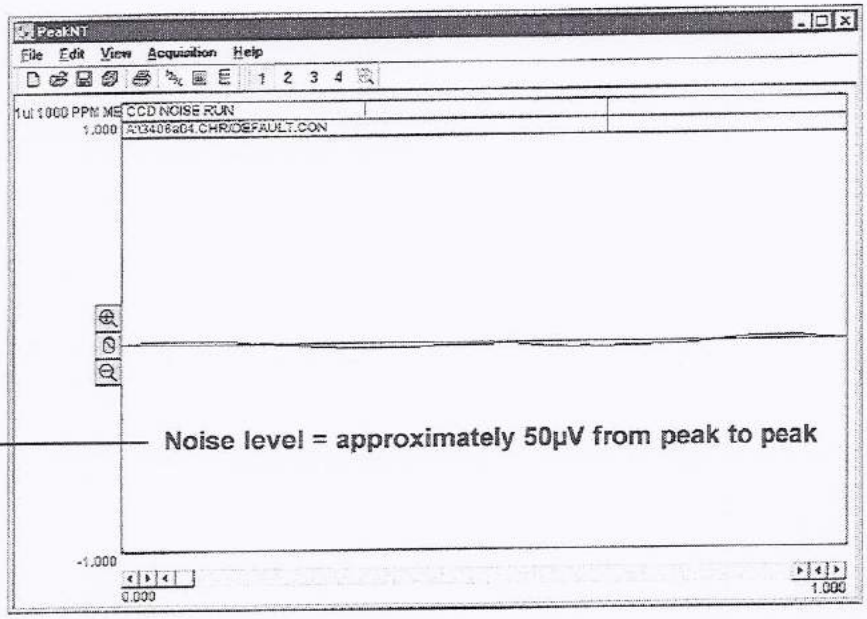
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:

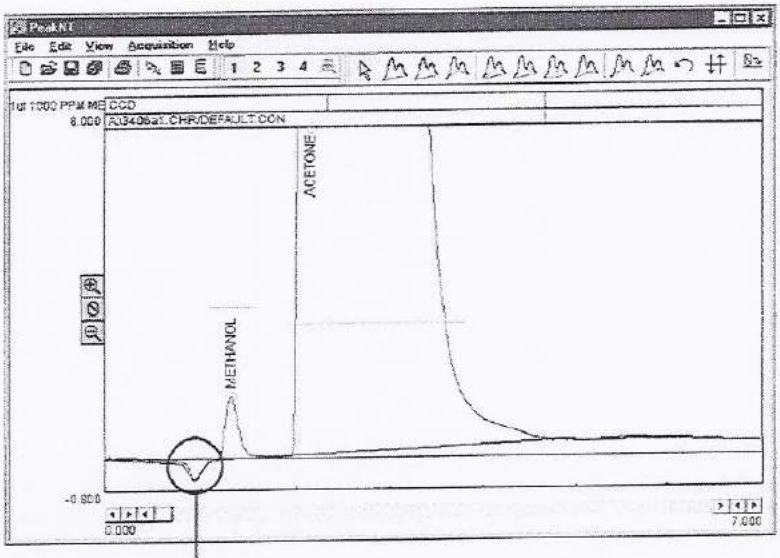
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

Enlarged for clarity





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



Negative water peak

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

Isothermal Temperature Program:

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

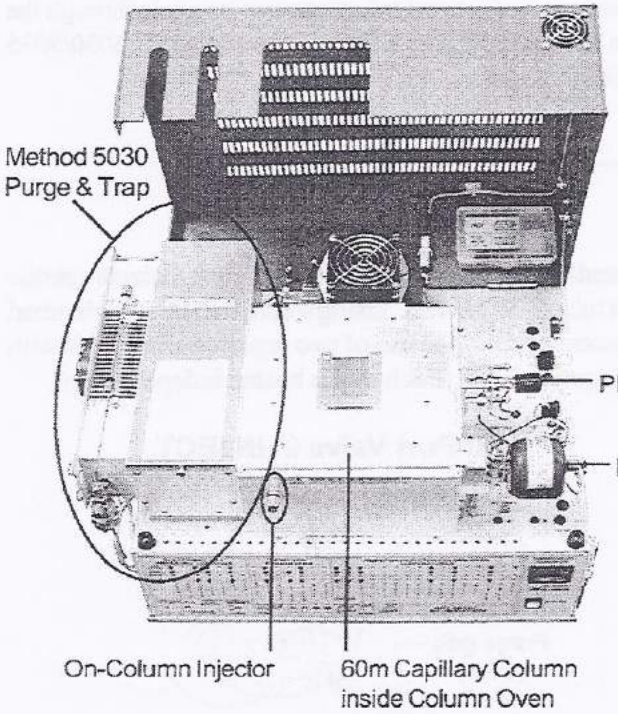
RESULTS:

Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
	Total	6958.5600

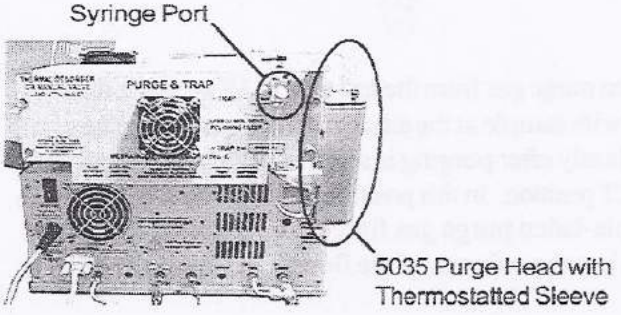
System Overview

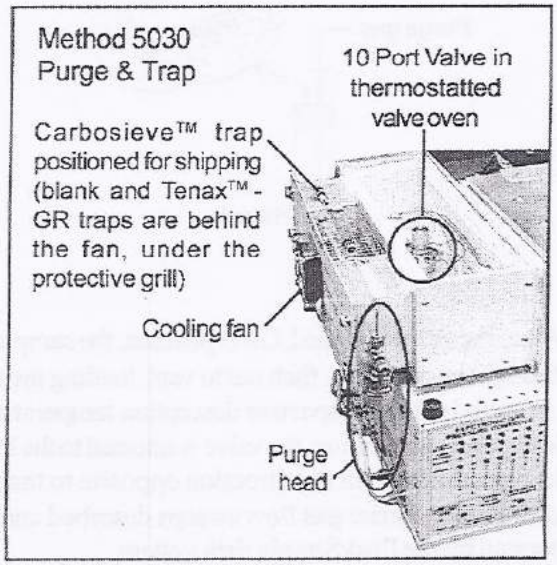
Your SRI Environmental GC is equipped with everything you need to generate certification quality data for EPA Methods 8010, 8015, 8021, and others. It is configured on the 8610C chassis, and includes a built-in Method 5030 or 5030/5035 compliant Purge & Trap for concentration of liquid and/or soil samples. Also included is an on-column injector for direct liquid injections. To detect commonly targeted pollutants, the Environmental GC uses a sensitive, non-destructive PID detector in series with a combination FID/DELCD detector. The PID detector responds to compounds whose ionization potential is below 10.6eV, including aromatics and chlorinated molecules with double carbon bonds. The FID detector responds to the hydrocarbons in the sample. The DELCD selectively detects the chlorinated and brominated compounds in the FID exhaust. Since the sample is pre-combusted in the FID flame, the DELCD is protected from contamination due to hydrocarbon overload. The PID is blind to certain compounds which can cause interference, such as methanol, and is recommended by the EPA. Peaks on the FID chromatogram that are obscured by the methanol peak are visible on the PID chromatogram. Benzene and carbon tetrachloride are common target analytes which co-elute. The FID responds to both. The PID responds only to benzene, while the DELCD responds only to carbon tetrachloride.

The BTEX GC is the same as the Environmental GC without the DELCD detector. Both systems have a "whisper quiet" internal air compressor and can be used with an H_2-50 hydrogen generator for tankless field operation.



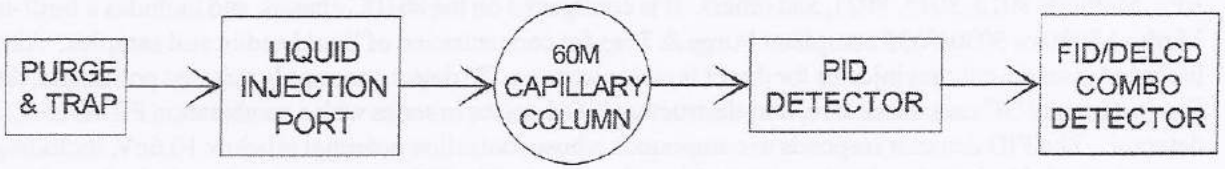
Method 5030/5035 Purge & Trap on an Environmental GC





POPULAR CONFIGURATION GCs
BTEX & Environmental

Theory of Operation

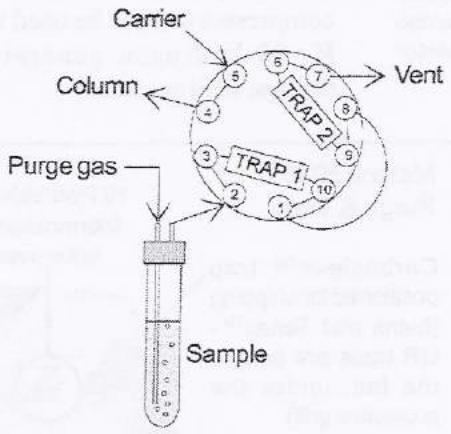


The versatile BTEX/Environmental GC systems can analyze gas, water, and soil samples. Four types of injection techniques can be used: purge and trap, direct liquid injection, TO-14 type gas sample concentration, and manual headspace injection. The Purge & Trap concentrator may be used for gas, liquid,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

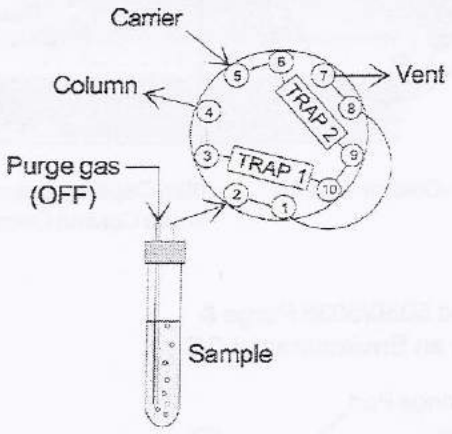
Purge & Trap Injection

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

10 Port Valve in LOAD



10 Port Valve in INJECT



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

Theory of Operation continued

Direct Injection

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

Gas Sample Concentration

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

Room Temperature Manual Headspace Injection

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

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

General Operating Procedures

EPA Style Purge & Trap Injection

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

1. The purge gas flow is controlled with an Electronic Pressure Controller (EPC). Set the purge flow (measurable at the trap vent at the rear of the purge and trap system); 40mL/min is a typical purge flow. The pressure required for 40mL/min through a single Tenax trap is printed on the right panel of the GC. ***NEVER use hydrogen as a purge gas***. SRI recommends helium purge gas.

2. TRAP 1 is in the lower position in the Purge & Trap, and TRAP 2 is in the upper position. The trap temperatures are factory set at 200°C for desorption. For adsorption temperatures, trap 1 is set at 30°C and trap 2 is set at 35°C. Trap heating will be controlled by the timed Event Table during the run. NOTE: the actual temperatures typically run 5°C over the setpoint. See the instructions in the Purge & Trap section of the manual for adjusting the trap adsorption temperature settings.

3. Load or create an Event Table that is appropriate to the sample to be analyzed, or that is designed for compliance with a particular EPA Method (such as **Epap&t1c.evt** for a single trap or **Epap&t2c.evt** for dual traps included in version 2.66 or higher of the PeakSimple software).

4. Load or create an appropriate Temperature Program for the column oven. **Epap&t.tem** is a typical Purge & Trap temperature program file provided with the PeakSimple software for your convenience. As a basic rule for good separation using the purge and trap injection technique, the column oven should be kept at 40°C for 10-12 minutes: 6 minutes while the sample is purging, plus 4-6 more minutes while the traps heat and the gas sampling valve (in the INJECT position) transfers the sample to the column.

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

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

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

General Operating Procedures continued

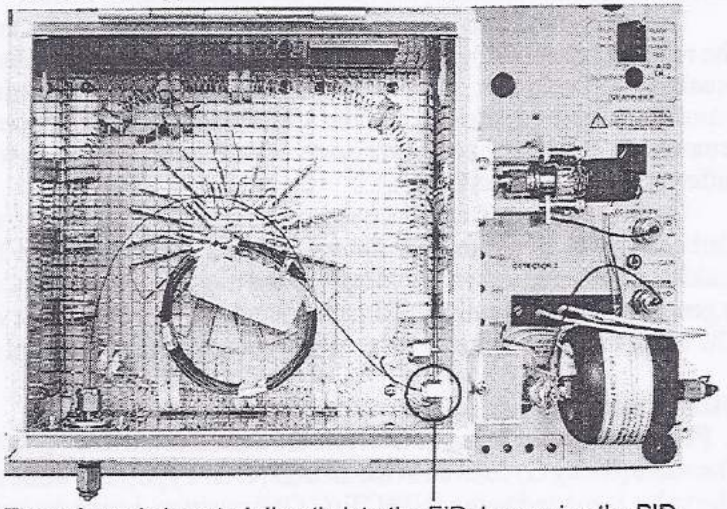
Direct Injection

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

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

2. Load or create a Temperature Program for the column oven. You can create an isothermal or ramped temperature program; deciding which to use depends on the sample being analyzed, and the goals of the analysis. There are several preset .tem files included with version 2.66 and higher of the PeakSimple software. If the analysis requires the column to be hotter than 150°C, it is best to disconnect the column from the PID detector. The PID represents a cold spot in which higher boiling analytes will become trapped, never making it to the much hotter (300°C) FID for detection. Also, when the column is heated over 150°C, stationary phase bleed will adhere to the PID lamp window. The higher boiling analytes and the column bleed will create a coating on the PID lamp window that will interfere with the analysis. The PID lamp window may be cleaned in the event of contaminant condensation, but the resulting change in the PID response usually requires detector recalibration. To bypass the PID, turn its lamp current OFF, then disconnect the column from the detector by loosening the swagelok-type nut from the bulkhead fitting in the column oven wall. Remove the tubing that connects the PID exit to the FID/DELCD by loosening that nut. Place the end of the column into the FID/DELCD bulkhead fitting instead and tighten it in place.

PID detector bypass for column oven temperatures above 150°C

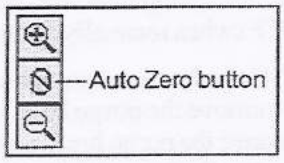


The column is inserted directly into the FID, bypassing the PID.

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

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

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



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

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

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.

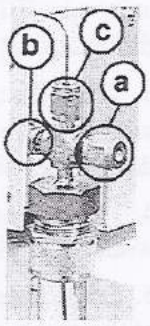
The SRI Purge & Trap concentrator is shipped with a blank trap and a Tenax™-GR trap installed, and a Carbosieve™ S-III packed trap for optional user installation. The Tenax-GR trap has a low affinity for water, making it a good adsorbent for the purge and trap technique. The Carbosieve has a high affinity 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.

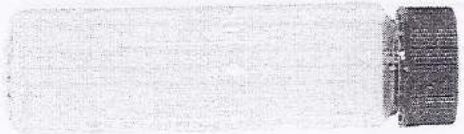
2. Load or create a temperature program for the column oven.

3. Perform **Detector Steps** 1-4, then proceed with the following steps.

4. Fill a plastic medical syringe with the vial headspace. Inject the sample into the GC injection port, bypassing the Purge & Trap concentrator.

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

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



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

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

Detector Steps

1. With the black plastic lamp hood in place on the PID lamp, turn ON the PID lamp current with the flip switch on the GC's front control panel. Set the PID current to 70 (= 0.70ma) by adjusting the appropriate trimpot setpoint on the top edge of the GC's front control panel. (Each detector zone is labeled on the front control panel under DETECTOR PARAMETERS, with the corresponding trimpot setpoint directly above it.) The lamp should emit a violet-colored light visible down the center of the tube. Set the PID temperature to 150°C. Set the PID gain to LOW.

2. Turn on the air compressor using the switch on the GC's front control panel. NOTE: since most ambient air will not cause interference with the DELCD, the built-in air compressor is appropriate for most analytical situations. However, if you are doing analyses in a lab environment with low levels of halogenated compounds in the ambient air, they can cause the DELCD to lose sensitivity, and fluctuations in the level of organics in ambient air may cause additional baseline noise. To avoid this, use clean, dry tank air.

3. Set the FID hydrogen flow to 25mL/min, and the FID air flow to 250mL/min. The pressure required for each flow is printed on the right hand side of the GC chassis. Ignite the FID by holding up the ignitor switch for a couple of seconds until you hear a small POP. Ensure that the flame is lit by holding the shiny surface of a chromed wrench to the tip of the collector electrode; when the flame is lit, you should be able to see condensation on the wrench. Set the FID gain to HIGH. If the peaks are more than 20 seconds wide at the base, use the HIGH FILTERED gain setting. If you wish to keep the ignitor ON to prevent flameout, set the ignitor voltage to -750 by adjusting the trimpot on the FLAME IGNITE zone.

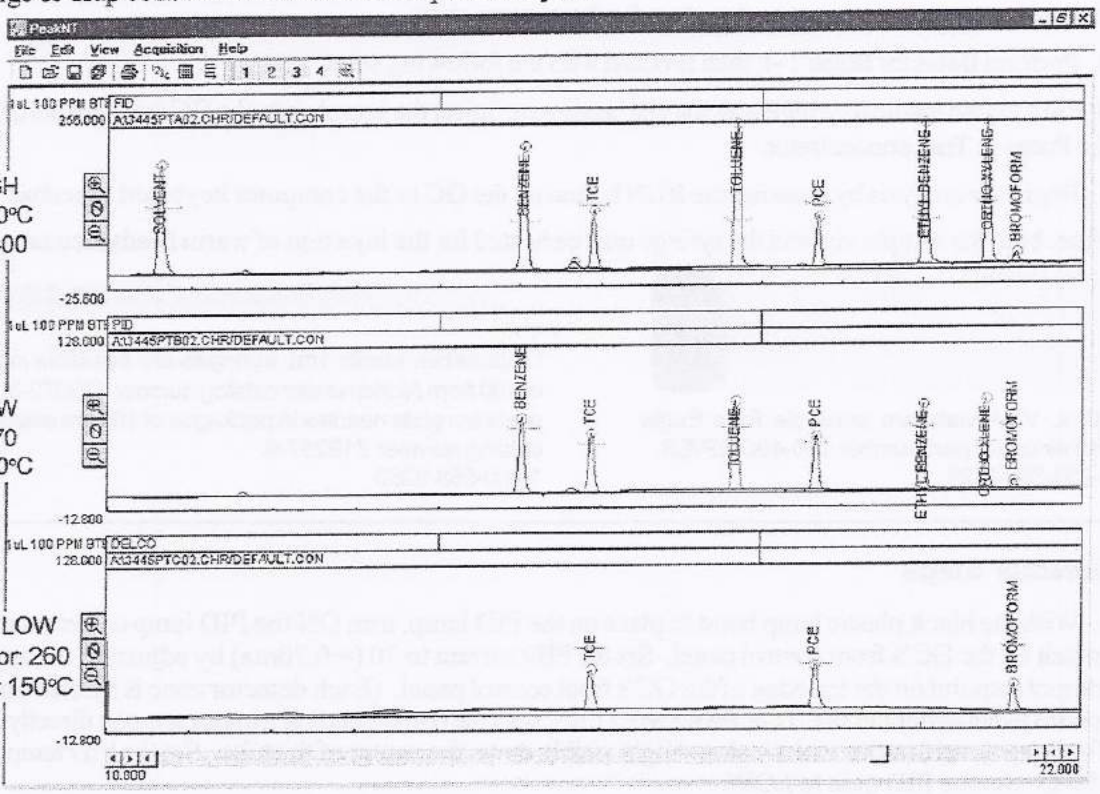
4. If a DELCD detector is installed, set the DELCD reactor temperature setpoint to 260 (=1000°C) by adjusting the appropriate trimpot. The DELCD will heat to around 254 and stabilize; the protruding end of the ceramic tube will glow bright red in the heat. Set the DELCD gain to LOW.

5. When the system has reached temperature and each detector is displaying a stable signal, begin the analysis by pressing the RUN button on the front of the GC or the spacebar on the computer keyboard.

POPULAR CONFIGURATION GCs
BTEX & Environmental

Expected Performance - Purge & Trap Concentrator

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



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

PID gain: LOW
PID current: 70
PID temp: 150°C

DELCD gain: LOW
DELCD reactor: 260
DELCD temp: 150°C

FID Results:

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

PID Results:

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

DELCD Results:

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

Events (5030.evt):

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

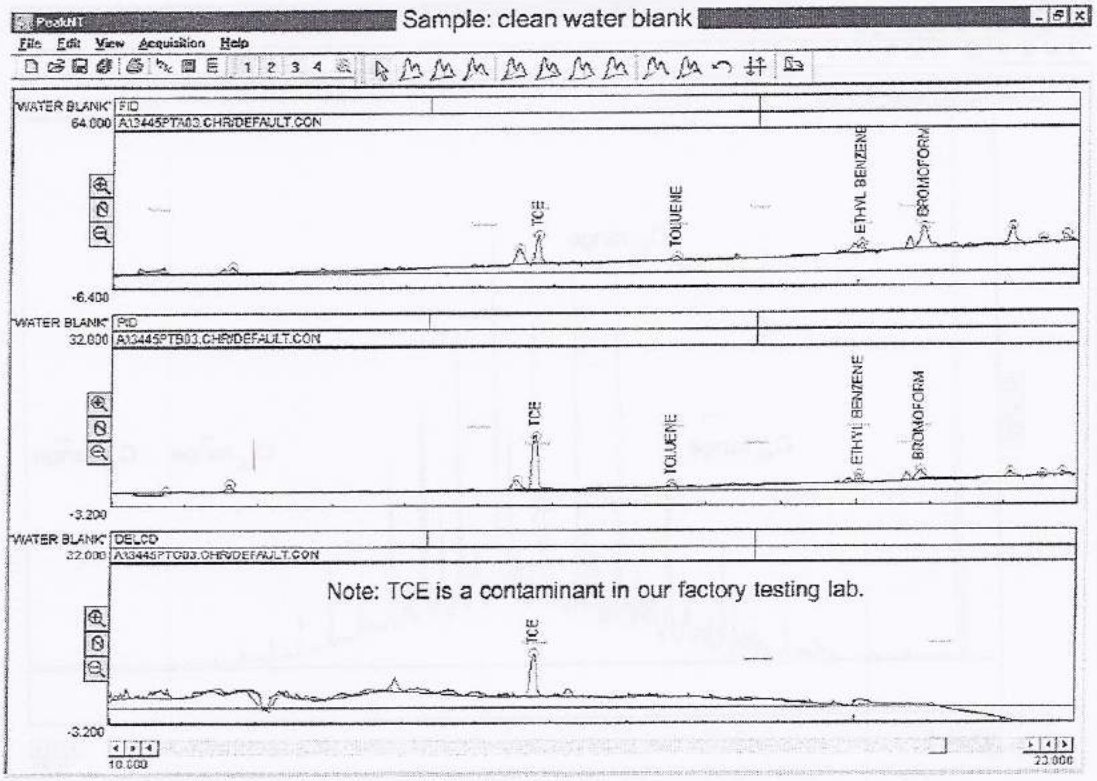
Sample: 1µL 100ppm BTEX Plus standard dissolved in 10mL of water to yield 10ppb of each analyte
Method: 5030 P&T injection
Column: 60m MXT-VOL
Carrier: Helium @ 10mL/min

Temperature Program:
(Epap&t.tem)

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

Expected Performance - Purge & Trap Concentrator

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



FID Results:

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

PID Results:

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

DELCD Results:

Component	Retention	Area
TCE	15.750	46.0340

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

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

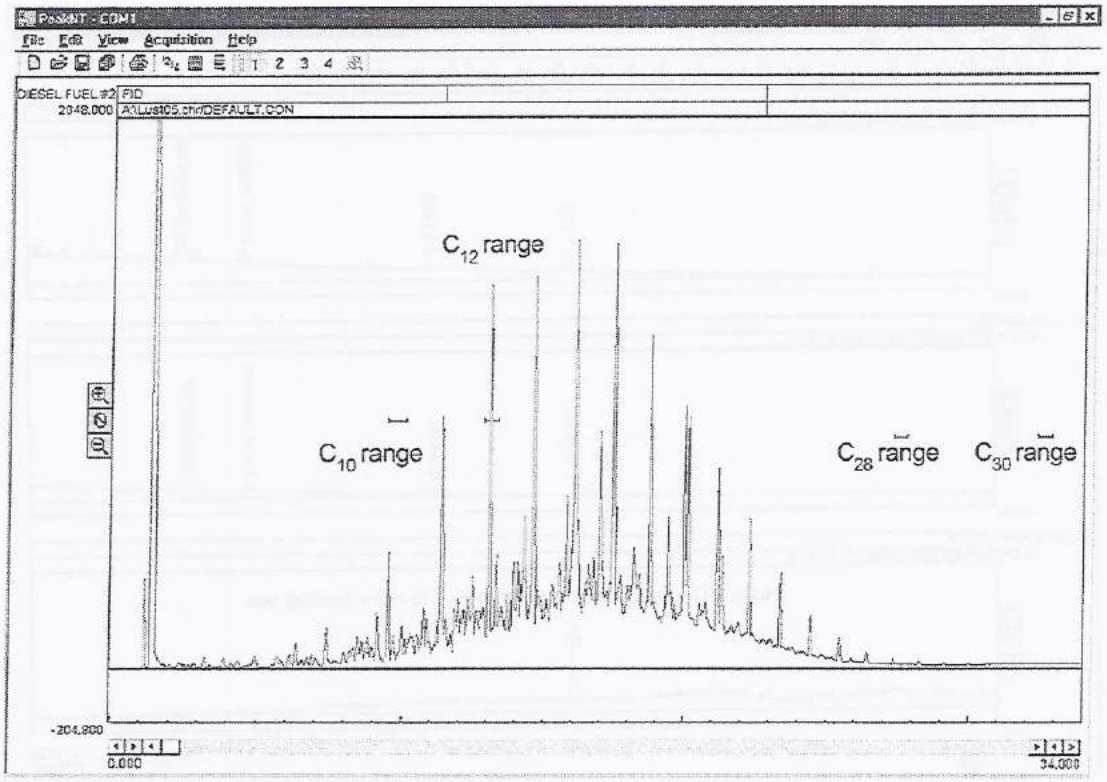
$$353x = 40ppb$$

$$x = 0.1133ppb$$

(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

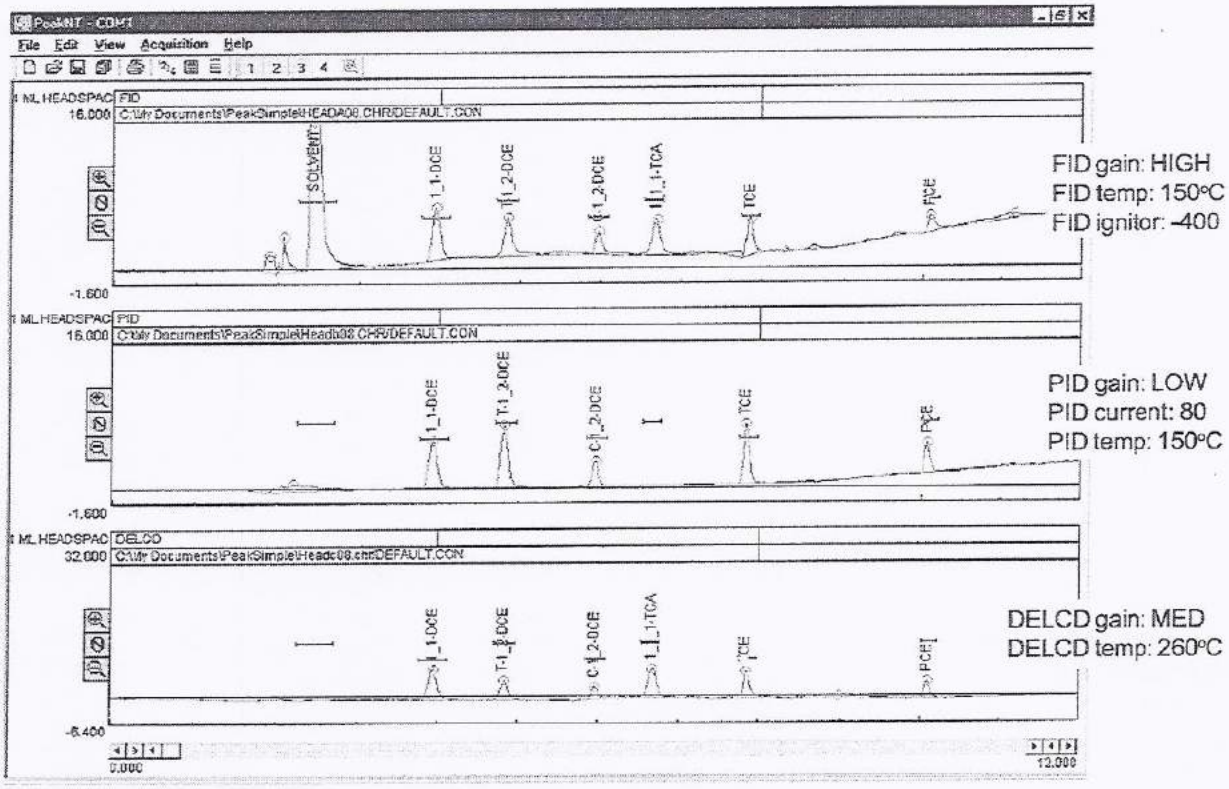
FID gain: HIGH
FID temp: 325°C
FID ignitor: -400

Temperature program:

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

Expected Performance - Manual Headspace Injection

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



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

Temperature program:

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

FID Results:

Component	Retention	Area
Solvent	2.416	290.1100
1_1-DCE	3.933	39.6100
T-1_2-DCE	4.833	34.3780
C-1_2-DCE	5.966	18.6020
1_1_1-TCA	6.663	29.6320
TCE	7.850	23.4490
PCE	10.083	10.7560
	Total	446.5370

PID Results:

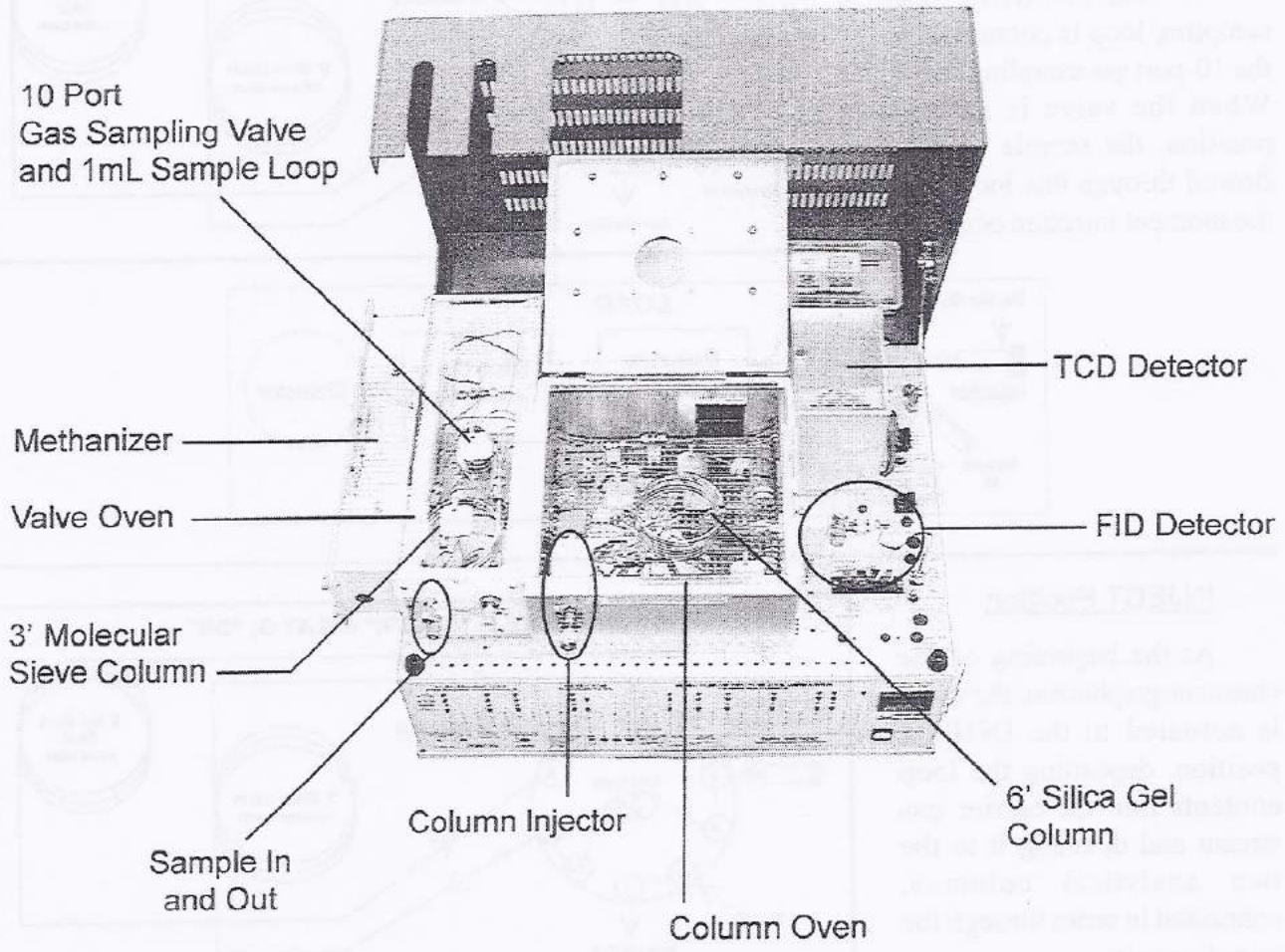
Component	Retention	Area
Solvent	2.183	22.7450
1_1-DCE	3.916	39.4070
T-1_2-DCE	4.800	45.0050
C-1_2-DCE	5.950	15.7380
TCE	7.816	33.7270
PCE	10.066	16.2780
	Total	172.9000

DELCD Results:

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

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.

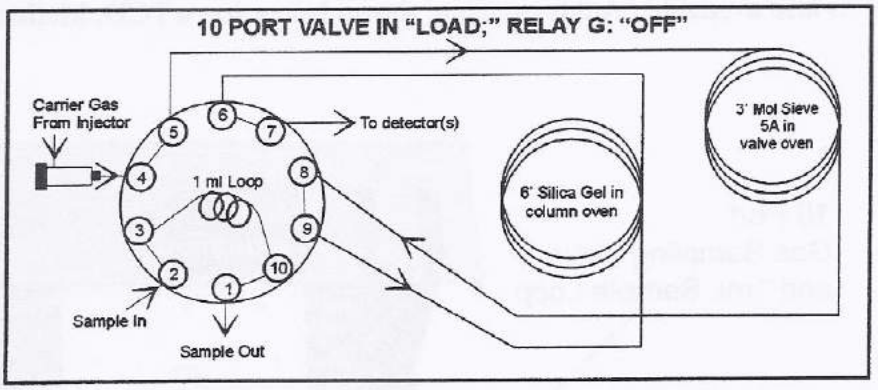
POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

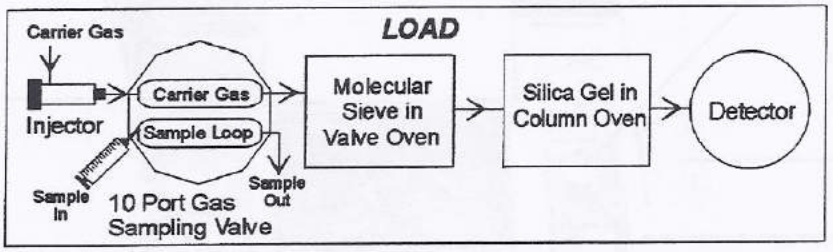
Theory of Operation
10 Port Gas Sampling Valve Plumbing Connections

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

LOAD Position

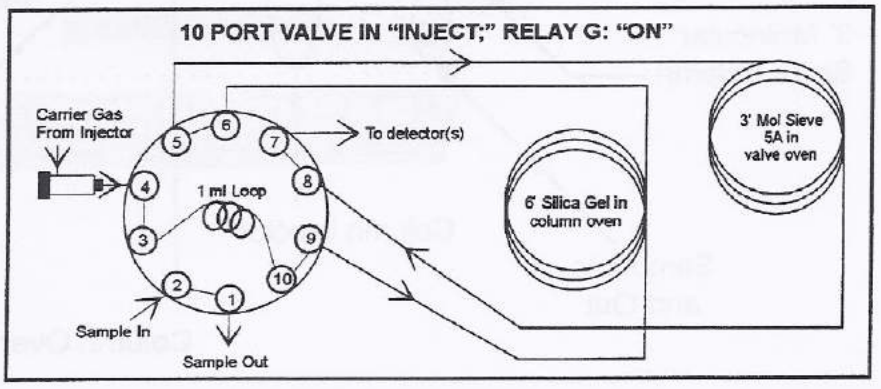
A one-milliliter gas sampling loop is connected to the 10-port gas sampling valve. When the valve is in load position, the sample may be flowed through this loop until the moment injection occurs.



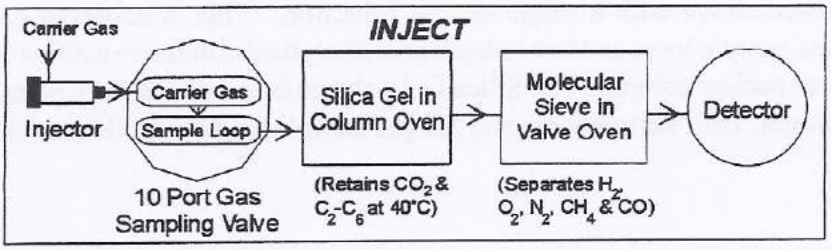


INJECT Position

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



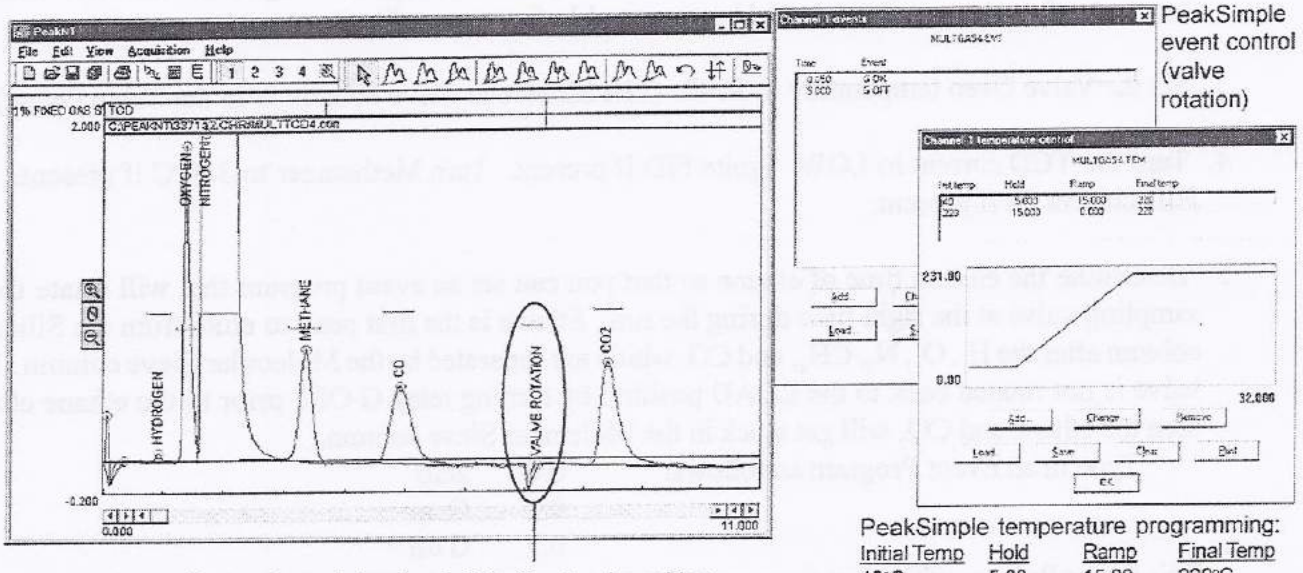
Column sequence is reversed while the flow direction remains the same



Theory of Operation
10 Port Gas Sampling Valve Plumbing Connections Continued

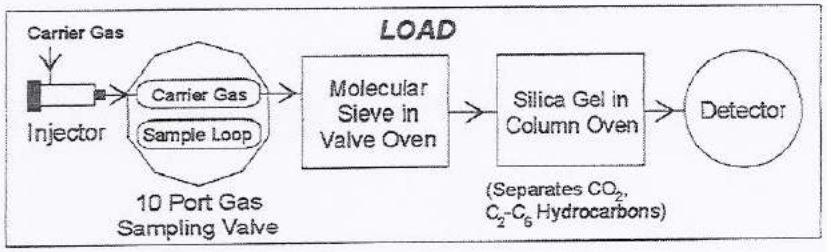
The sample is deposited first into the Silica Gel packed column, at 40°C in the column oven, where the ethane, propane, butanes, pentanes and carbon dioxide are retained. The remainder of the sample containing hydrogen or helium (whichever is not being used as a carrier), as well as oxygen, nitrogen, methane and any carbon monoxide, continues on to the Molecular Sieve column. During a chromatographic run with the sampling valve in the INJECT position, the hydrogen or helium, oxygen, nitrogen and methane components are the first to elute through the columns and into the detector. This is due to the Silica Gel column's long retention time at 40°C of C_2, CO_2 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



Gas sampling valve rotates back into the load position.

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

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.

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

3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)

4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.

5. Determine the elution time of ethane so that you can set an event program that will rotate the gas sampling valve at the right time during the run. Ethane is the first peak to elute from the Silica Gel column after the H_2, O_2, N_2, CH_4, 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_2 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

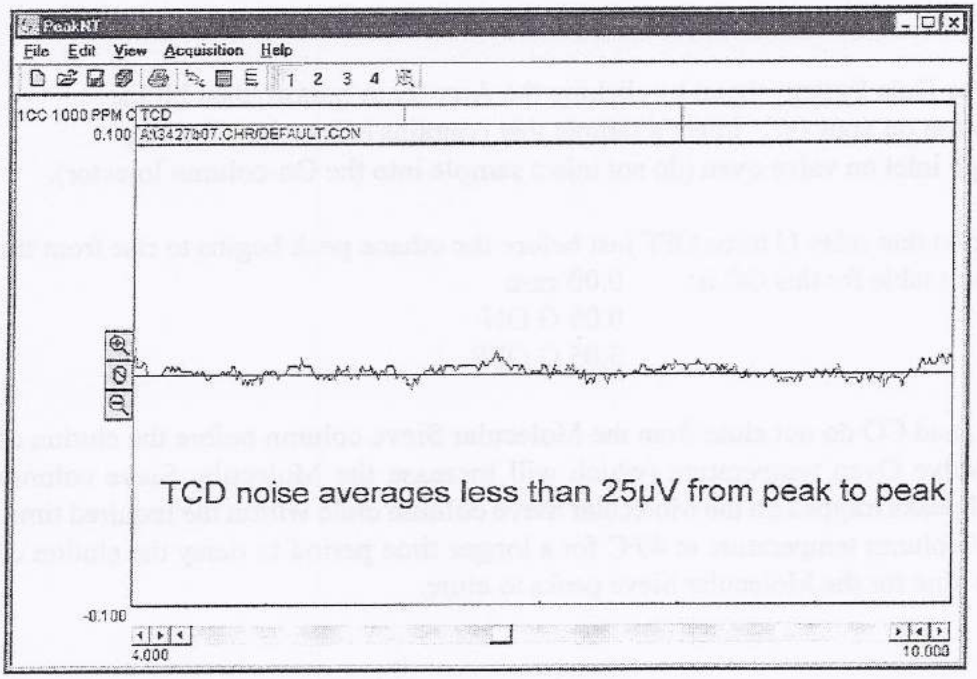
7. In PeakSimple, zero the Data System signal by clicking the Auto Zero button, then hit the spacebar or the run button on your GC. Inject a sample that contains ethane into the Gas Sampling Valve through inlet on valve oven (do not inject sample into the On-column Injector).

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 H_2, O_2, N_2, CH_4, and CO do not elute from the Molecular Sieve column before the elution of ethane, increase the Valve Oven temperature (which will increase the Molecular Sieve column temperature) so that all peaks trapped on the Molecular Sieve column elute within the required time. Or, hold the Silica Gel column temperature at 40°C for a longer time period to delay the elution of ethane, allowing more time for the Molecular Sieve peaks to elute.

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #1

Expected Performance



TCD noise run

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

Temperature Program:

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

Factory Test Analysis of Natural Gas Standard

Temperature program:

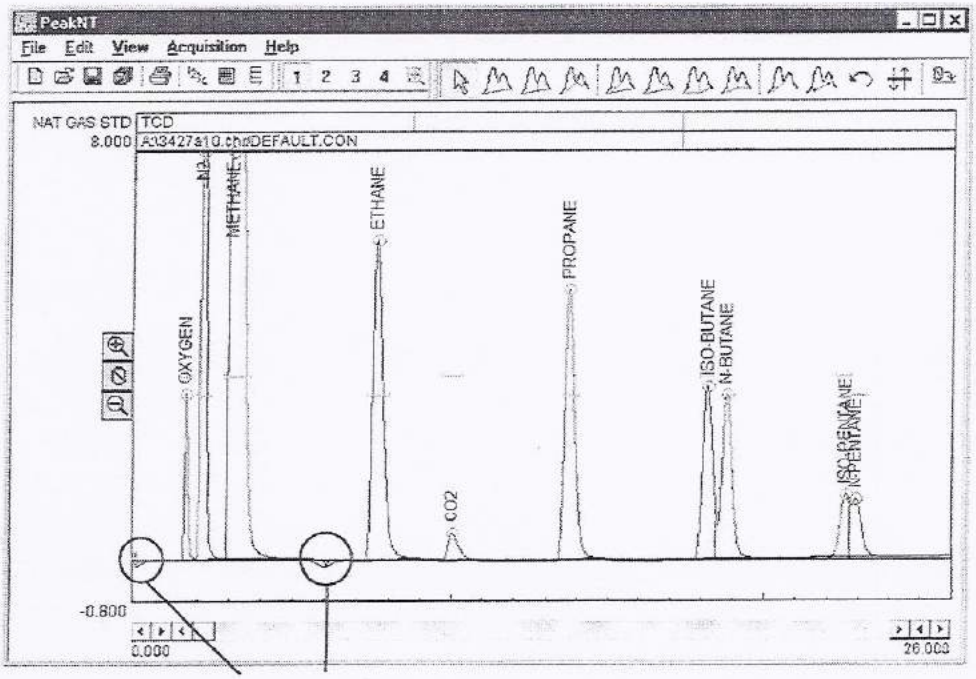
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

Events:

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

Results:

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



Valve rotations

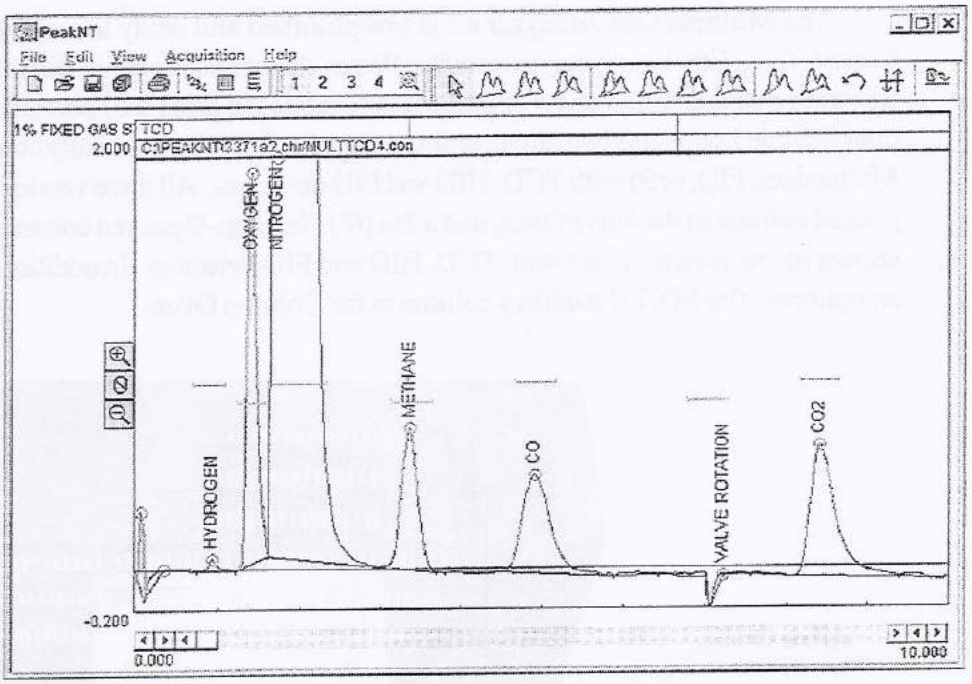
Expected Performance

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

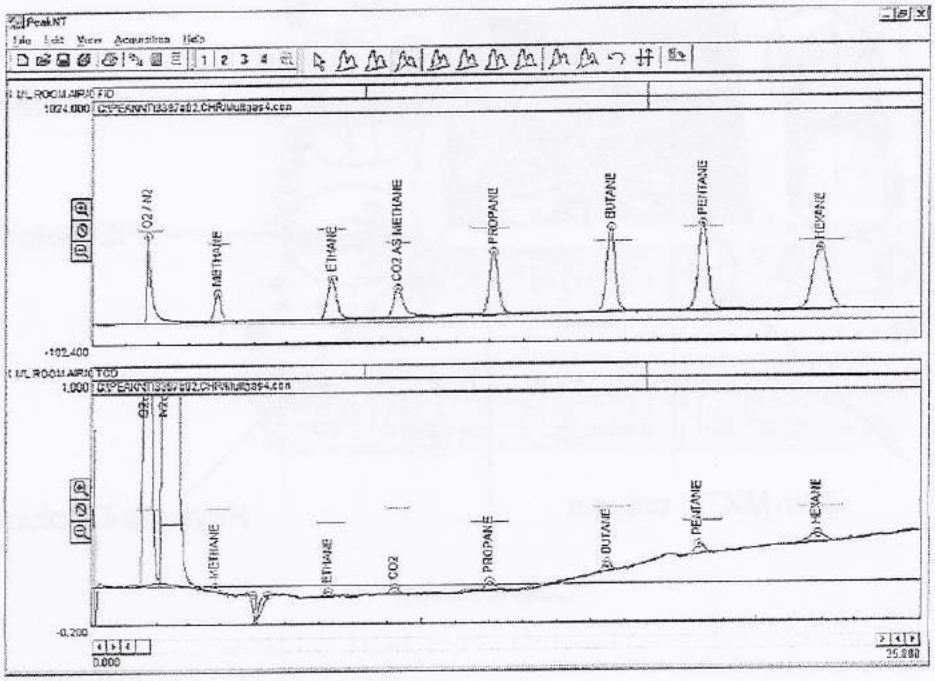
TCD current = LOW
TCD temp = 100°C
Valve Oven temp = 90°C

RESULTS:

Component	Retention	Area
Hydrogen	0.916	0.4710
Oxygen	1.383	14.9530
Nitrogen	1.700	1049.4940
Methane	3.333	9.4875
CO	4.900	11.4460
Valve Rotate	7.200	0.6460
CO_2	8.383	13.8000
	total	1100.2775



Factory Test Analysis of Room Air & C_1-C_6 Hydrocarbons on a dual-channel TCD-Methanizer-FID Multiple Gas Analyzer #1



FID Results:

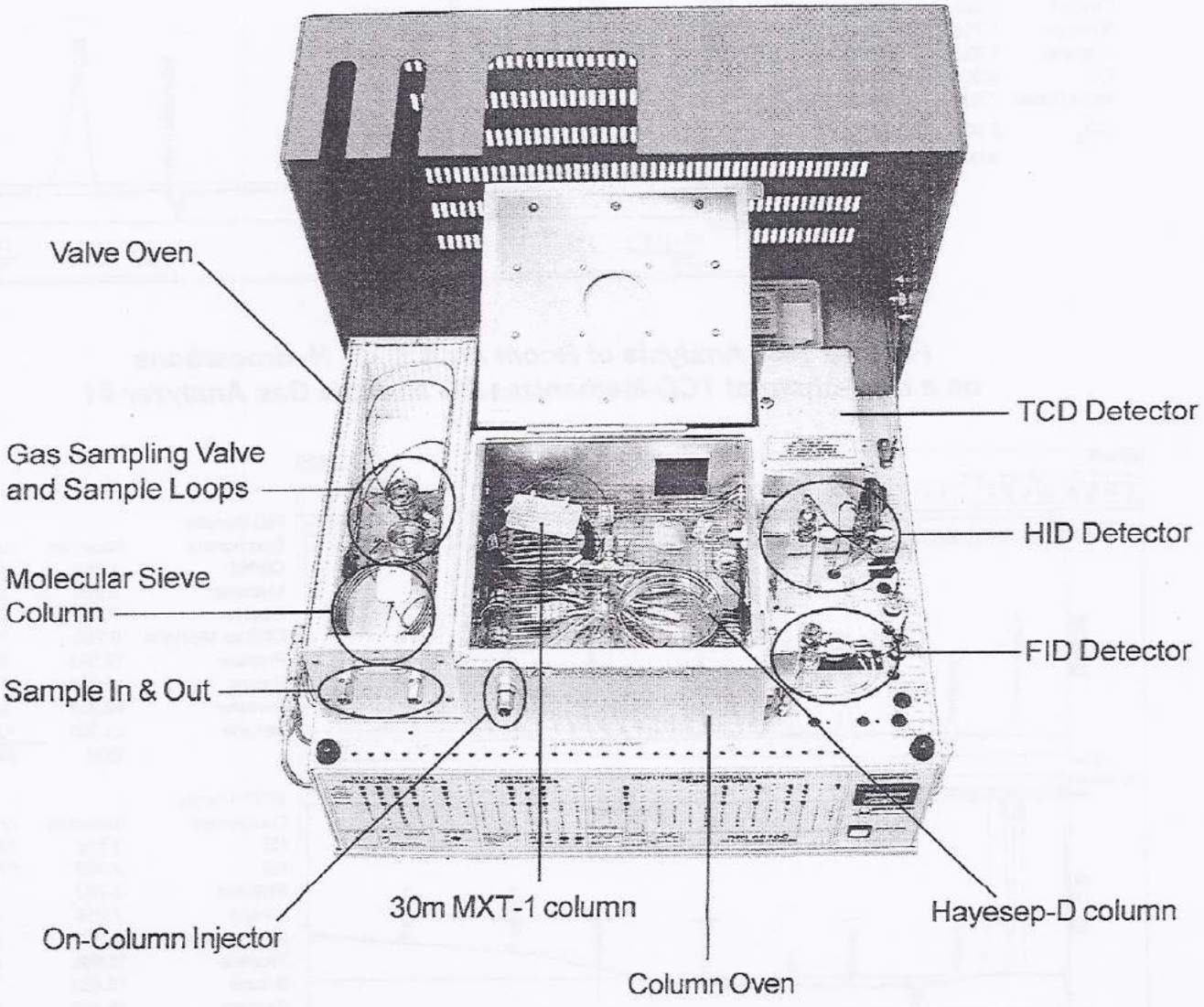
Component	Retention	Area
O2/N2	1.650	4731.2140
Methane	3.866	2008.6000
Ethane	7.316	3854.7300
CO2 as Methane	9.250	3142.1040
Propane	12.083	5379.8755
Butane	15.533	7326.4440
Pentane	18.333	9136.3340
Hexane	21.900	10408.3160
	Total	45987.6175

TCD Results:

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

System Overview

The Multiple Gas Analyzer #2 is pre-plumbed and ready to resolve H_2, He, O_2, N_2, Methane, CO, Ethane, CO_2, Ethylene/Acetylene, NOx, Water, Alcohols, Propane, Butanes, Pentanes, and C_6 through C_{20}. Separation of this wide variety of peaks is accomplished using a 10 port automated Gas Sampling Valve with dual Sample Loops and two, three, or four columns. It can be optionally configured with **1)** a TCD, **2)** a TCD, Methanizer, FID, or **3)** with TCD, HID and FID detectors. All three versions have a 1m (3') Molecular Sieve packed column in the Valve Oven, and a 2m (6') Hayesep-D packed column in the Column Oven. The model shown below is customized with TCD, HID and FID detectors. In addition to the Hayesep-D column, it has an optional 30m MXT-1 capillary column in the Column Oven.

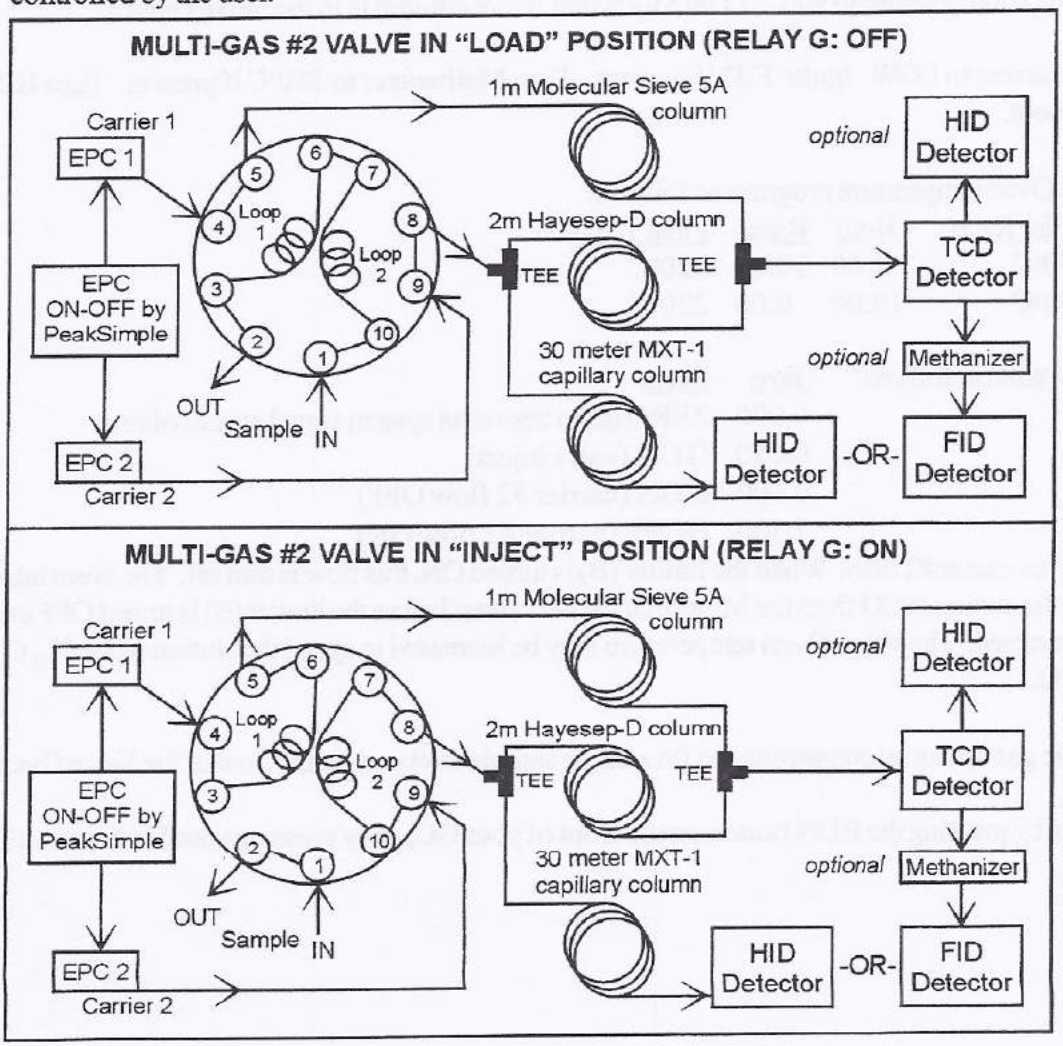


POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Theory of Operation

The Multiple Gas Analyzer #2 GC uses a single automated 10 port Gas Sampling Valve and multiple columns to separate a wide variety of peaks. The system achieves this by turning the carrier gas flow to each column on at different times during the run. This procedure allows the Molecular Sieve column in the Valve Oven to completely separate H_2, He, O_2, N_2, CH_4 and CO before the carrier flow to the Hayesep-D column in the Column Oven is turned on. The Hayesep-D column then separates all compounds in the C_1-C_6 range. An optional 30m MXT-1 capillary column in the Column Oven separates the remaining hydrocarbons out through C_{20}, using the same carrier gas flow as the Hayesep-D column and an FID or HID detector.

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



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

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

General Operating Procedure

1. Set the gas cylinder pressure 15-20psi higher than the head pressure (helium carrier). The carrier head pressure used to generate the test chromatograms at the factory is printed on the right side of your GC. Typical head pressure for a Multi-Gas instrument operating at 20mL/min is about 20psi.

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

3. Set the Valve Oven temperature to 90°C. (The Molecular Sieve column is in the Valve Oven.)

4. Turn the TCD current to LOW. Ignite FID if present. Turn Methanizer to 380°C if present. Turn HID current on if present.

5. Set the Column Oven temperature program as follows:

Initial Temp	Hold	Ramp	Final Temp
50°C	7.00	10.00	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)

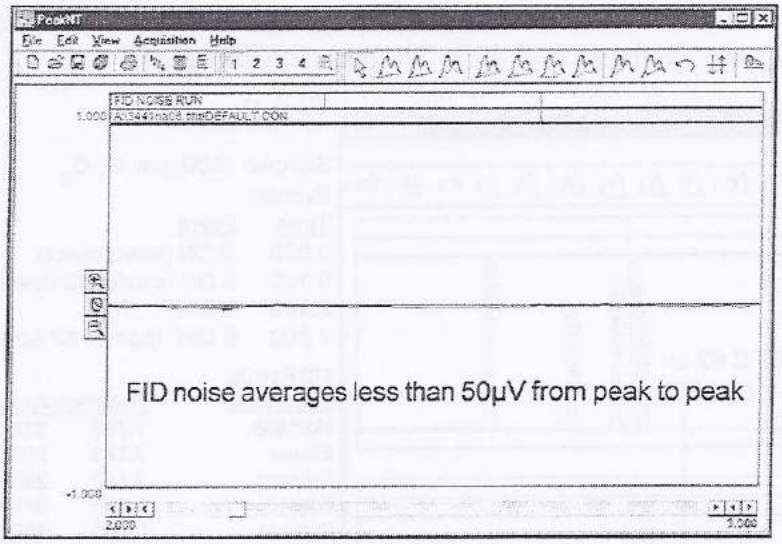
EPC #2 controls the carrier #2 flow. When the limiter (B) is turned ON, this flow is shut off. The event table should allow for the elution of CO from the Molecular Sieve column before the limiter (B) is turned OFF and carrier #2 flow restored. The Valve Oven temperature may be increased to speed the 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:

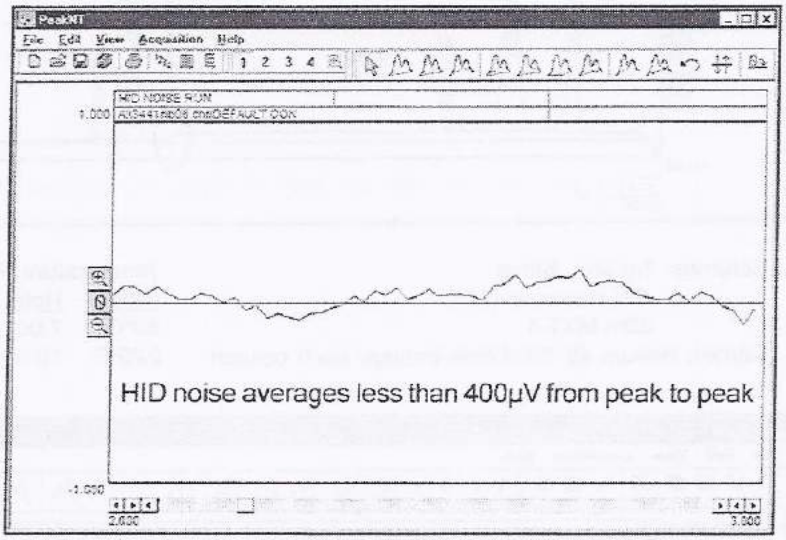
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

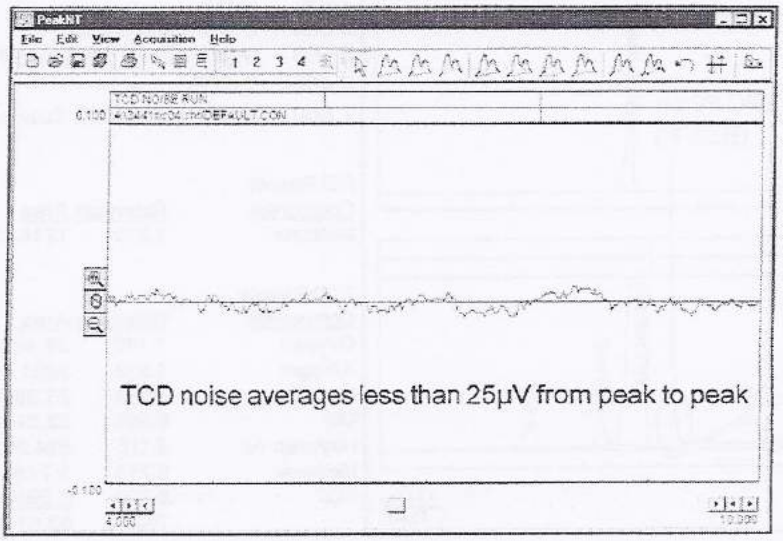
HID noise run

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	Hold	Ramp	Final
80°C	15.00	0.00	80°C





TCD noise run

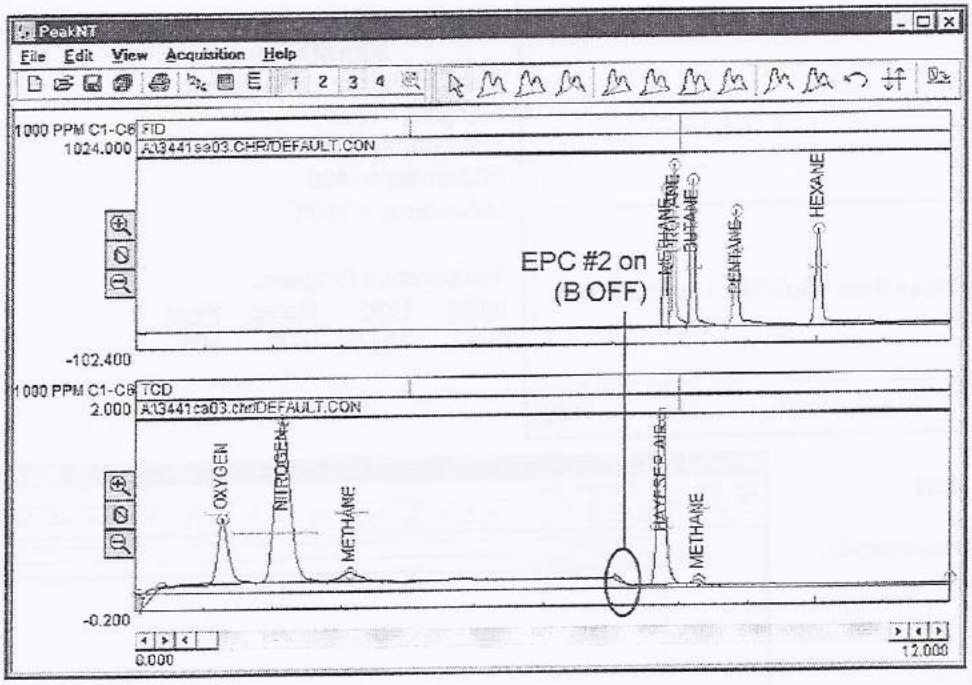
Columns: 1m Mol. Sieve, 2m Hayesep-D,
30m MXT-1
Carrier: Helium @ 10mL/min
TCD gain = LOW
TCD temp = 100°C
Valve temp = 110°C

Temperature Program:

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

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

Sample: 1000ppm C_1-C_6

Events:

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

FID Results:

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

TCD Results:

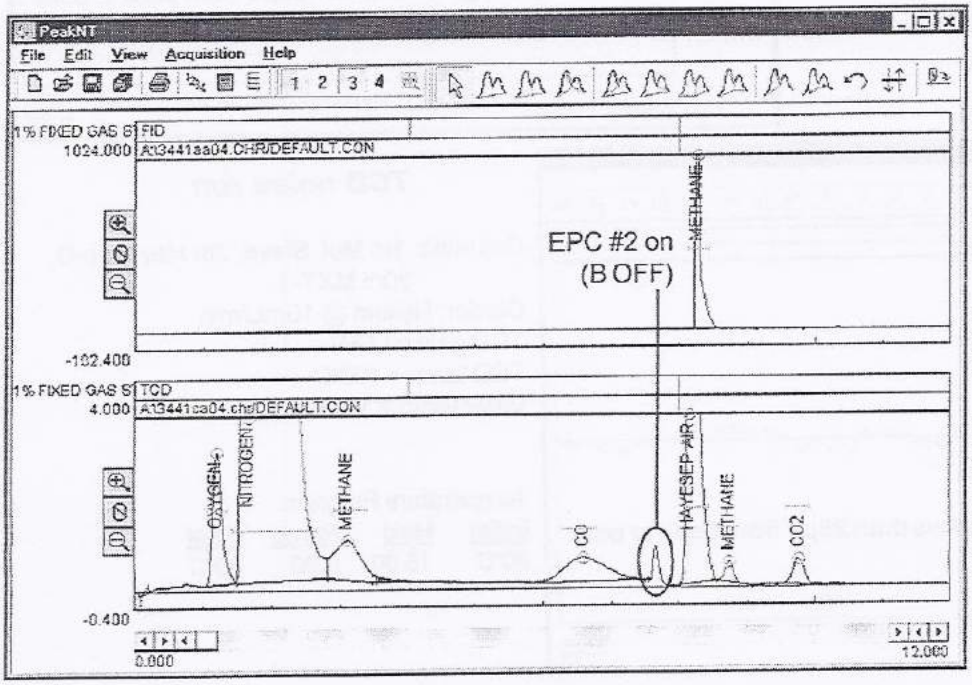
Component	Retention	Area
Oxygen	1.250	7.9800
Nitrogen	2.116	27.9765
Methane	3.116	2.0210
Hayesep Air	7.716	23.5150
Methane	8.250	0.4950
Ethane	12.133	1.0240
	Total	63.0115

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

Carrier: Helium @ 10mL/min through each column

Temperature Program:

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



Test Run #2

Sample: 1% fixed gas standard

Events:

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

FID Results:

Component	Retention	Area
Methane	8.233	12144.3770

TCD Results:

Component	Retention	Area
Oxygen	1.166	26.4920
Nitrogen	1.633	2251.7140
Methane	3.083	23.0975
CO	6.566	22.2440
Hayesep Air	8.116	524.2010
Methane	8.716	3.7730
CO2	9.750	6.3940
	Total	63.0115

POPULAR CONFIGURATION GCs
Multiple Gas Analyzer #2

Expected Performance: HID & TCD Detectors

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

Test Run #1

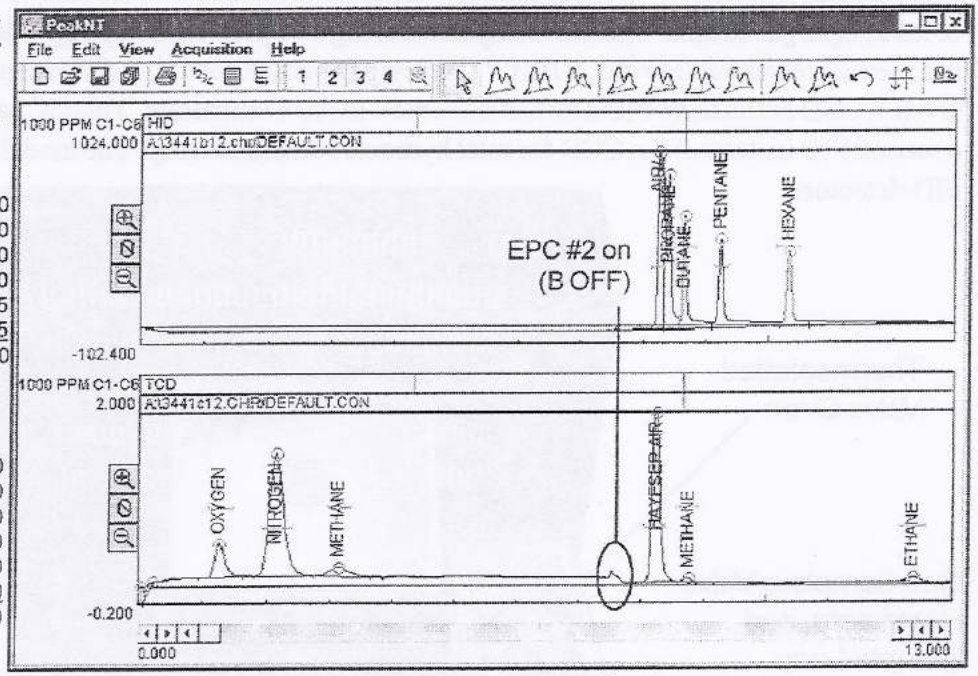
Sample: 1000ppm C_1-C_6

HID Results:

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

TCD Results:

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



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

Temperature Program:

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

Events:

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

Test Run #2

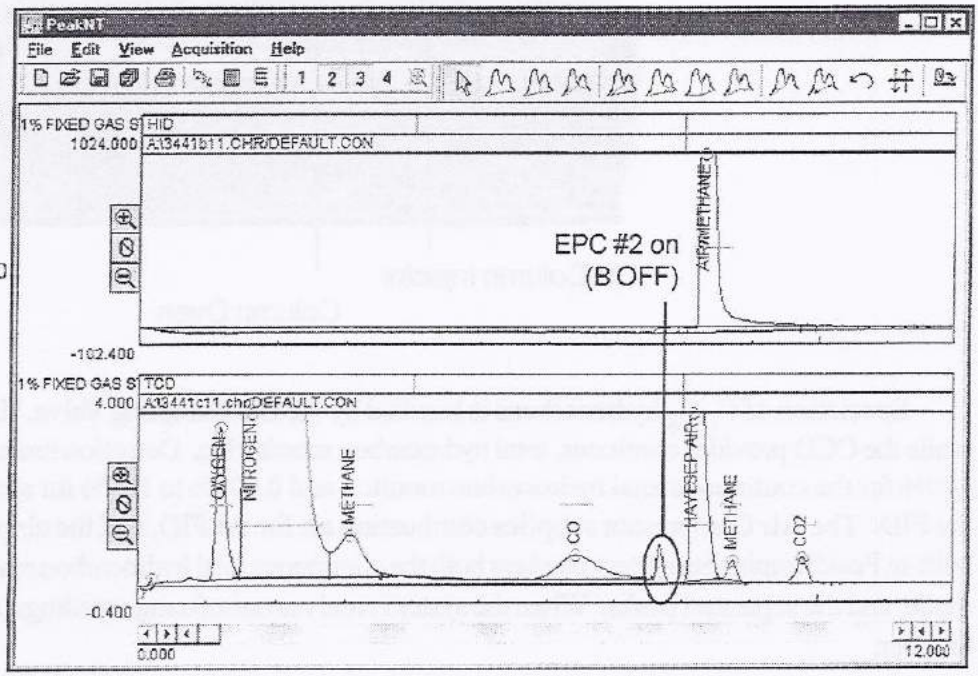
Sample: 1% fixed gas standard

FID Results:

Component	Retention	Area
Methane	8.266	44548.0540

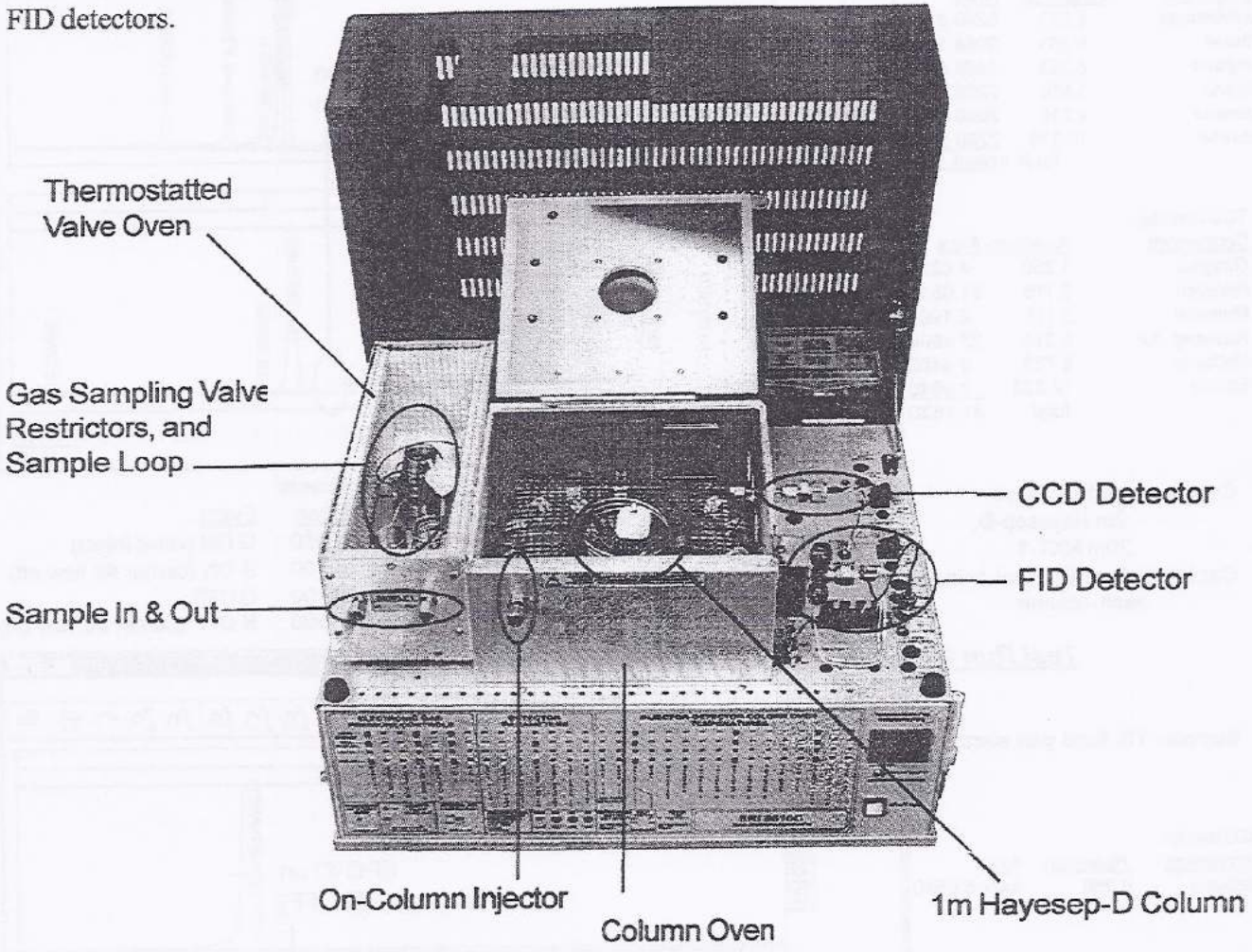
TCD Results:

Component	Retention	Area
Oxygen	1.166	31.0260
Nitrogen	1.616	2261.6430
Methane	3.050	12.6240
CO	6.400	22.4410
Hayesep Air	8.116	542.6790
Methane	8.716	3.5950
CO2	9.750	6.6920
	Total	2880.7000



System Overview

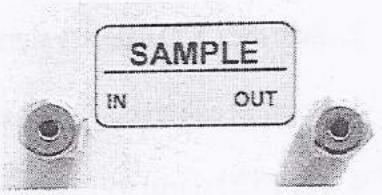
The Mud-Logging GC system is designed to provide a continuous reading of total hydrocarbons in a gas stream, while periodically performing a chromatographic separation of the sample to determine the composition of the sample gas stream. This is accomplished using a 10 port Gas Sampling Valve with a 25µL 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 FID detectors.

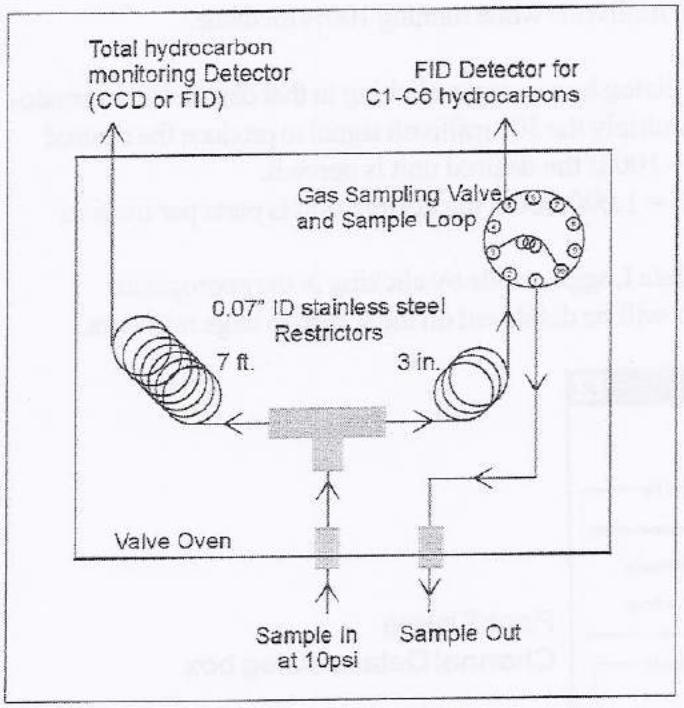


Speciation of C_1-C_6 hydrocarbons is handled by the Gas Sampling Valve, Hayesep-D column, and FID while the CCD provides contiuous, total hydrocarbon monitoring. Detection limits for this system are 0.1% to 100% for the continuous total hydrocarbon monitor, and 0.005% to 100% for speciated hydrocarbons 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.

Theory of Operation

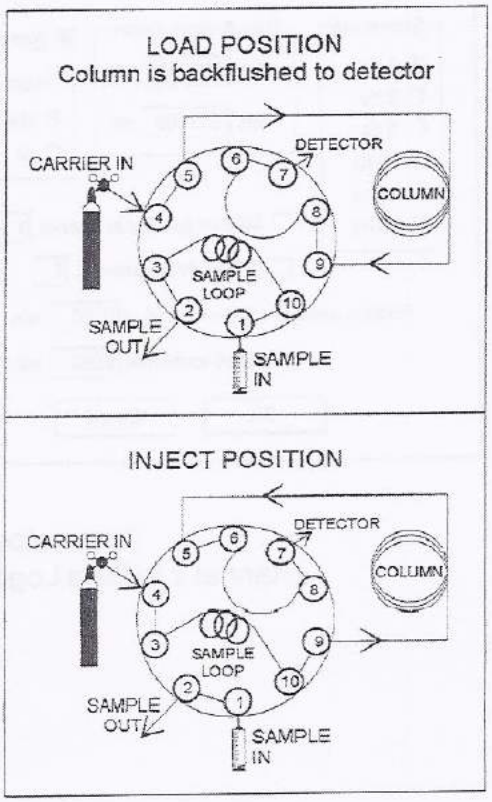
The sample gas stream is connected to a bulkhead fitting on the system's thermostatted Valve Oven where it flows through the sampling loop of the 10 port Gas Sampling Valve, and also to the CCD detector. The fitting labelled "Sample In" (pictured at right) on the front of the Valve Oven is the sample gas stream inlet. The user must regulate the pressure of the sample stream so that it enters this inlet at 10psi. The instrument is factory preset to deliver 5mL/min to the CCD at 10psi. The remainder of the flow, approximately 100mL/min, passes through the Sample Loop. This relatively high flow rate gets the sample from the sampling point into the GC with minimal delay.





10 Port Gas Sampling Valve

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.



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 25µL 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.

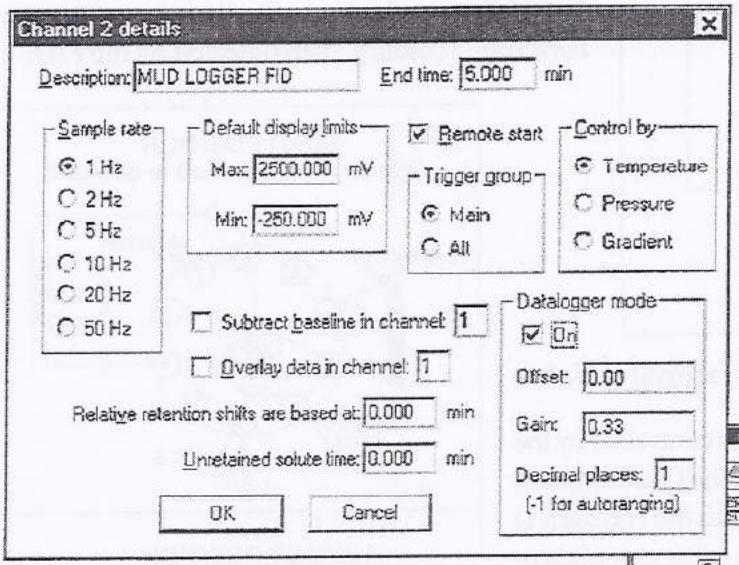
2. Zero the CCD detector signal using the Auto Zero button for its channel (typically channel 2).

3. Connect calibration gas standard to the sample inlet at 10psi. Calibration gas is typically 100% methane.

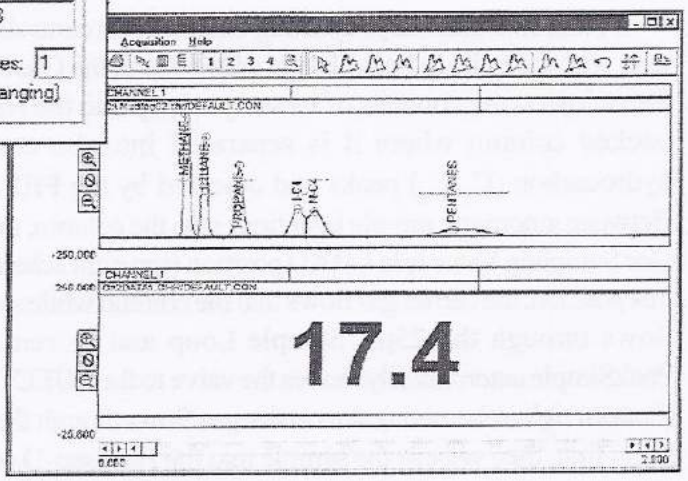
4. The CCD signal will increase approximately 300 millivolts while running 100% methane.

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

6. Also in the Channel Details dialog box, select Data Logger mode by clicking in the appropriate checkbox. The CCD signal times the gain factor will be displayed on the screen in large numbers.



PeakSimple
Channel Details dialog box



Chromatogram with
channel 2 in Data Logger mode

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

1. Connect the calibration gas standard to the sample inlet at 10psi.

2. Set the Valve Oven temperature to 90°C.

3. Ignite the FID.

4. Set an isothermal Column Oven temperature program as follows:

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

5. Type in an even table as follows:

Time	Event
0.00	Zero
0.050	G ON
1.5	G OFF

6. Set the FID gain to MEDIUM.

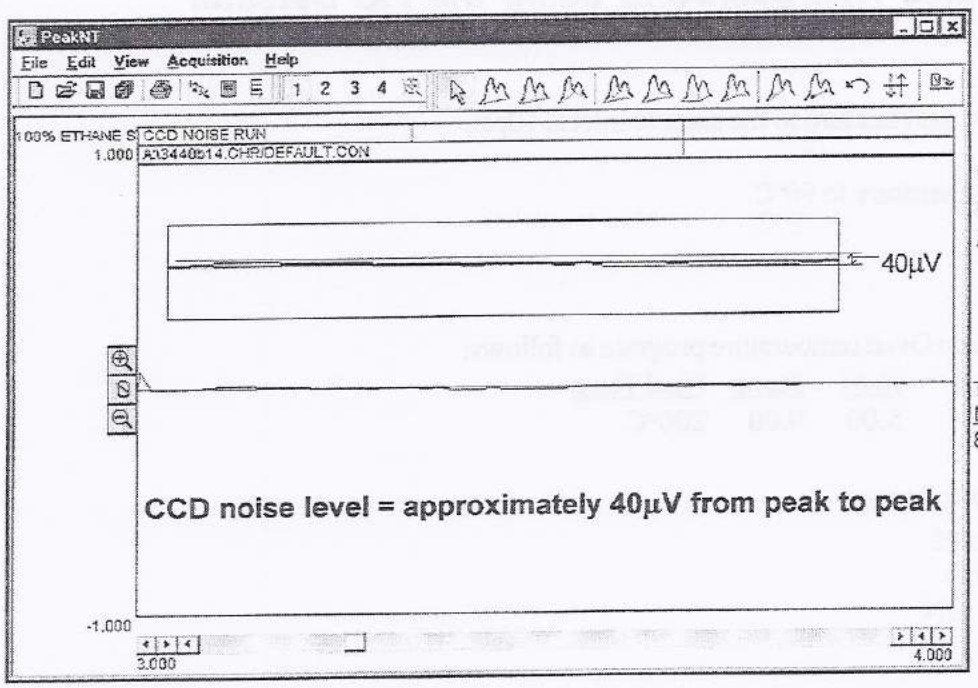
7. Start the analysis by hitting the spacebar on the computer keyboard.

8. In PeakSimple, input the retention windows to identify the individual hydrocarbon components (methane, ethane, propane, butane, etc).

9. Calibrate the individual hydrocarbon peaks.

10. This instrument is plumbed for backflush. This gives the user the option to set the valve program to backflush the heavier hydrocarbons after the desired peaks have been separated. For instance, if your application required separation of hydrocabons up to C_5, you could set the valve to backflush after the elution of the C_5 component(s), and all the heavier hydrocarbons would together produce one large peak.

Expected Performance



CCD Noise

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

Temperature program:

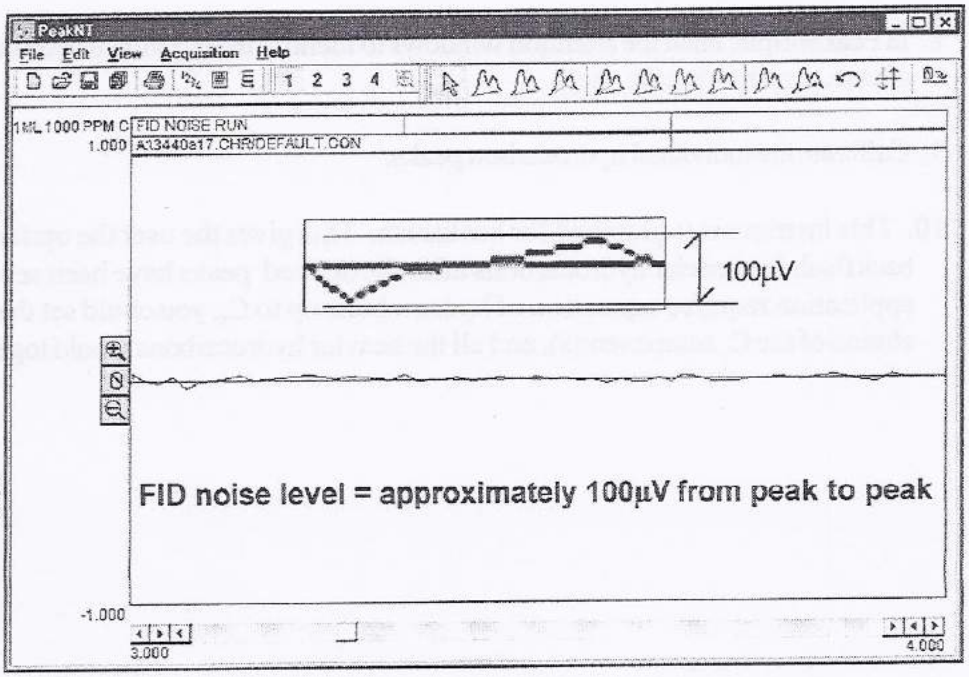
Initial	Hold	Ramp	Final
80°C	5.00	0.00	80°C

FID Noise

Column: 1m Hayesep-D
Carrier: Helium @ 10mL/min
FID gain = High
FID temp = 150°C
FID ignitor = -400
Valve temp = 90°C

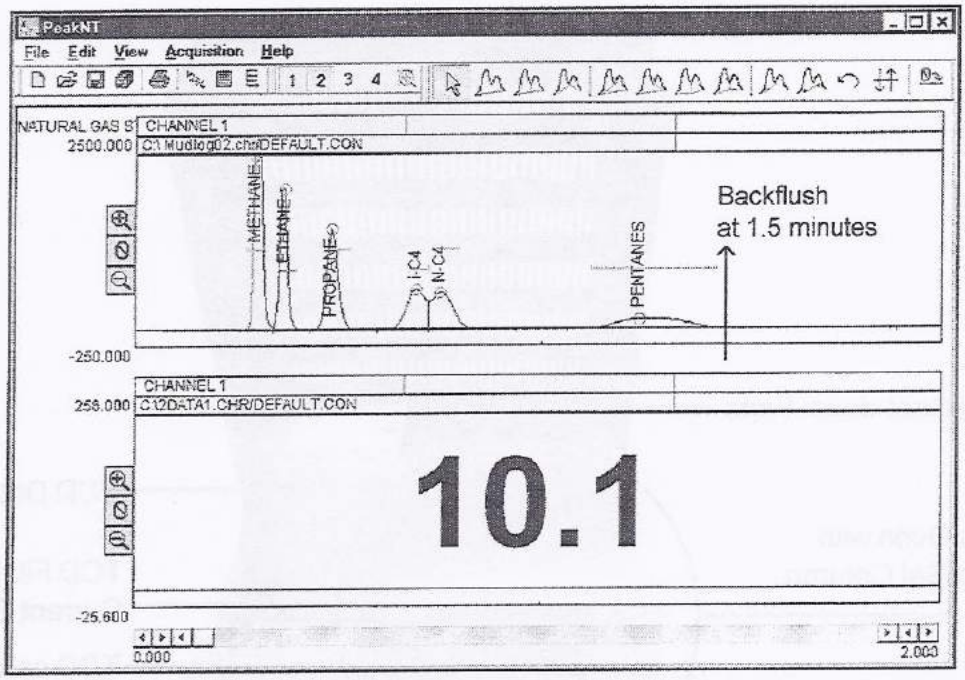
Temperature program:

Initial	Hold	Ramp	Final
80°C	5.00	0.00	80°C



Expected Performance

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



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

Temperature program:

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

Events:

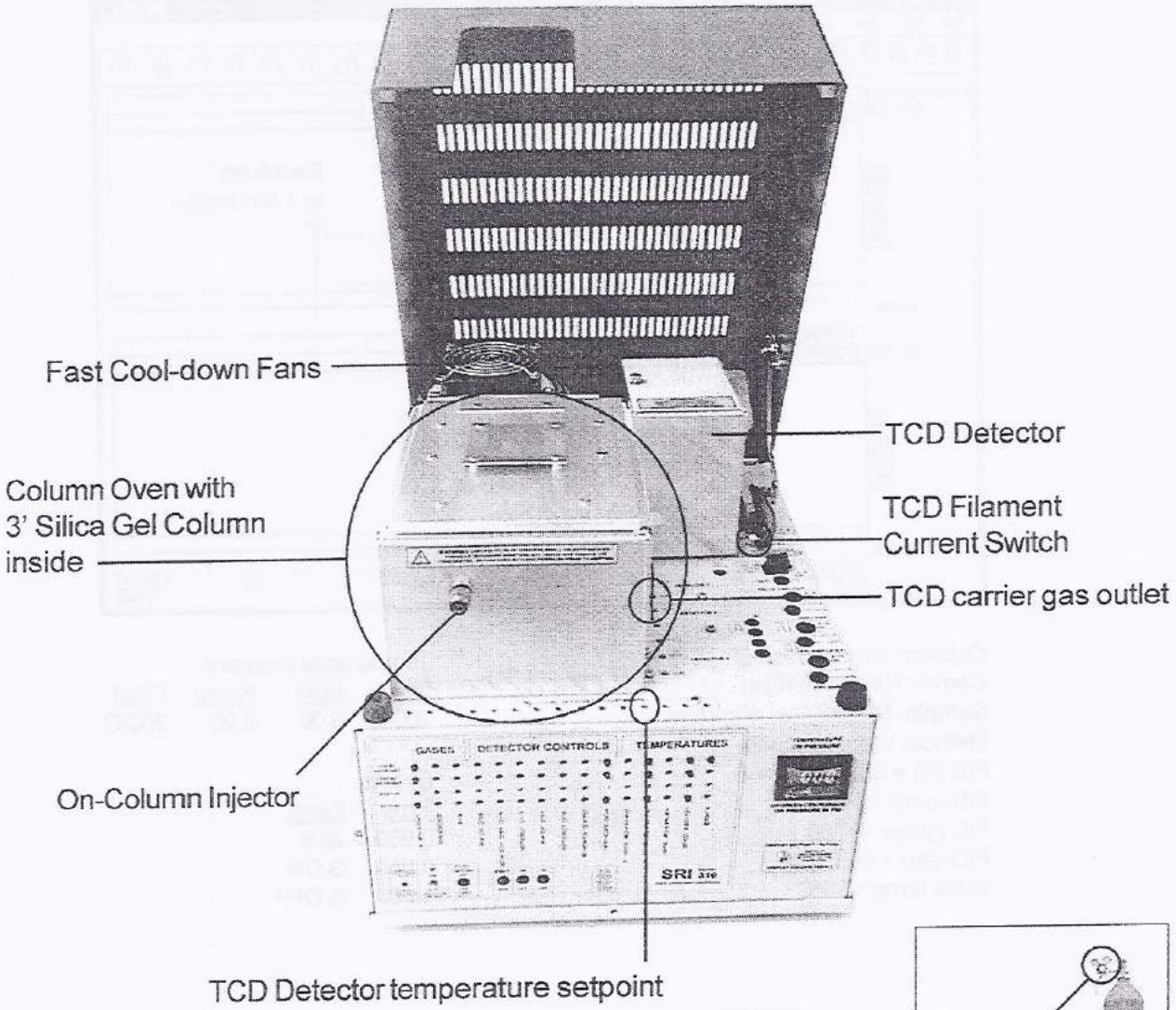
Time	Event
0.000	Zero
0.050	G ON
1.500	G OFF

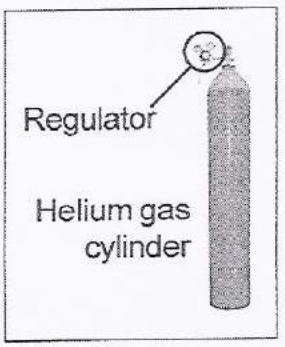
Results:

Component	Retention	Area
Methane	0.291	6664.1410
Ethane	0.366	2770.3785
Propane	0.483	2762.6450
i-C4	0.691	1754.0118
N-C4	0.750	1913.8415
Pentanes	1.241	1580.4310
	Total	17445.4488

System Overview

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





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

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

General Operating Procedure

1. Check to make sure that the TCD filament current is switched OFF. Plug in and turn on your GC. Allow the TCD detector oven to reach temperature (100°C) and stabilize. With the "Display Select" switch in the UP position, press on the TCD Temperature Actual button on the front control panel to read the TCD cell temperature.

2. The carrier gas head pressure is preset at the factory to 10mL/min for the Silica Gel column. Look on the right side of the GC for the carrier pressure that correlates to a flow of 10mL/min. Because different columns require different flow rates, the carrier head pressure may be adjusted by the user with the trimpot above the "CARRIER 1" buttons. For this GC, carrier cylinder pressure is normally set at 30psi, which is 10-20mL higher than the column head pressure. The column head pressure is the pressure developed by the carrier gas as it flows through the analytical column.

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

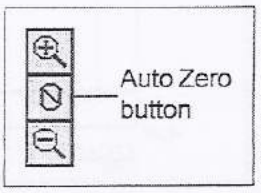
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column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

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

6. In PeakSimple, set an isothermal Column Oven temperature ramp program as follows:

Initial Temp.	Hold	Ramp	Final Temp.
80.00	7.00	0.00	80.00

7. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Hit the RUN button on your GC or hit the spacebar on your computer keyboard to begin the run. You may also open the Acquisition pull-down menu and select Run, but this gets difficult unless you have a partner, since your hands are occupied with the sample syringe.



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

Expected Performance

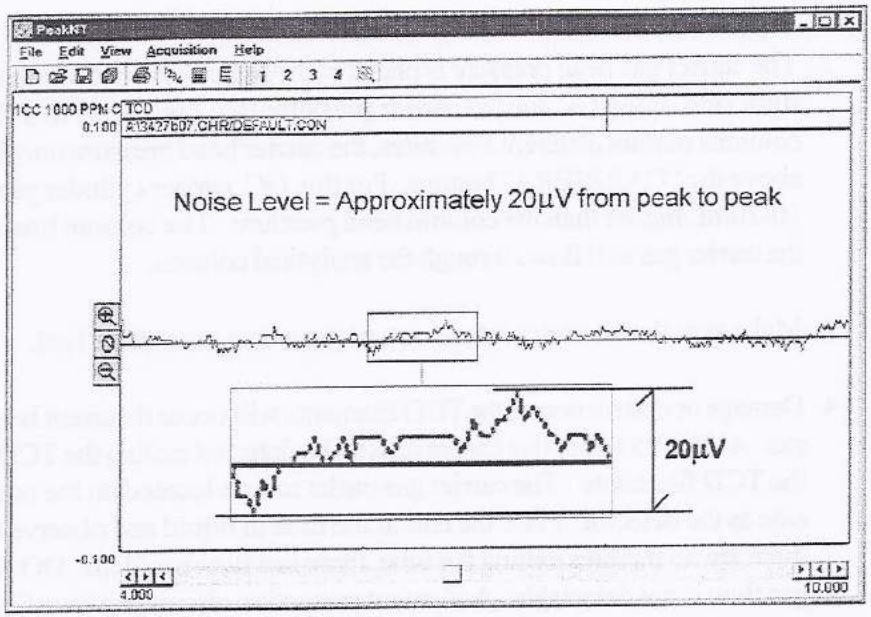
Every compound possesses some degree of thermal conductivity and therefore may be measured with a TCD detector. TCD detectors are most often used with helium as a carrier gas because of helium's high thermal conductivity, but other gases such as nitrogen, argon, or hydrogen may also be used as a carrier gas. A TCD detects all molecules in concentrations from 100% down to around 100ppm, and is especially useful for measuring inorganic gases like O_2, N_2, CO & CO_2.

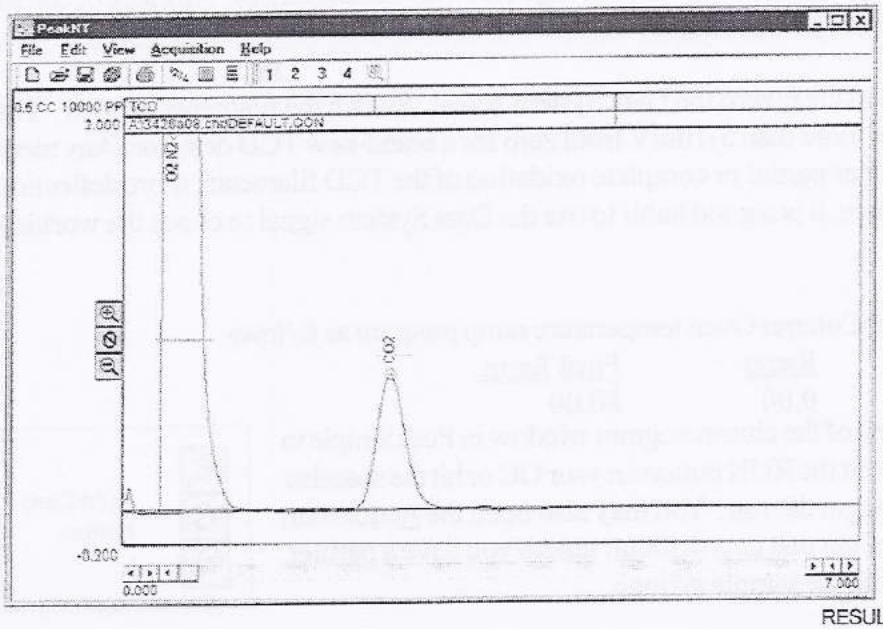
TCD Detector Noise

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 CO_2
TCD current = LOW
TCD Temp = 100°C

Temperature Program:

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

RESULTS:

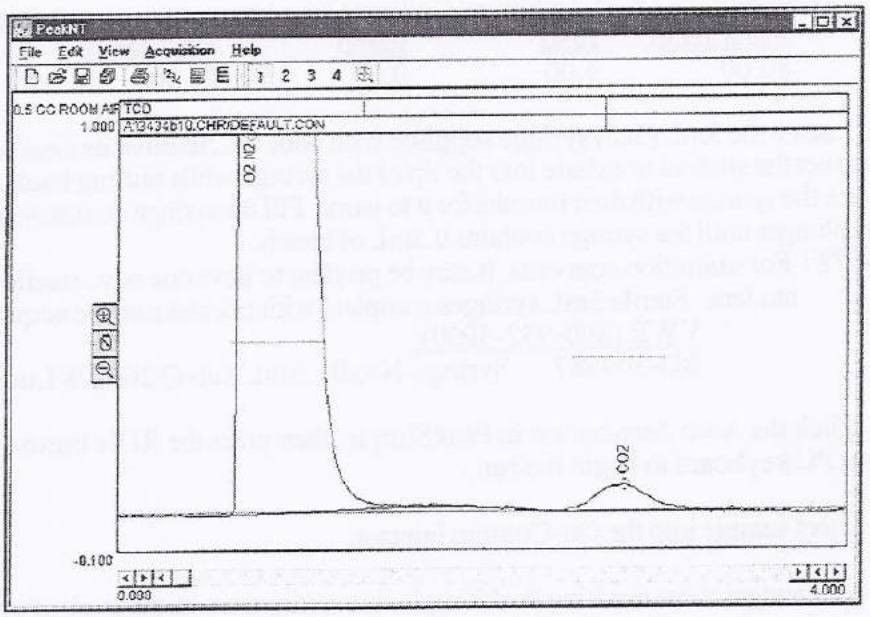
Component	Retention	Area
O2 N2	0.450	1252.9980
CO2	2.500	13.6460
Total		1266.6440

POPULAR CONFIGURATION GCs
Educational TCD

Expected Performance

TCD Room Air Analysis

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



Temperature Program:

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

Results:

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

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

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:

VWR (800-932-5000):
BD-309587 Syringe-Needle, 3mL Sub-Q 26G 5/8 Luer-lok™

4. Click the Auto Zero button in PeakSimple, then press the RUN button on your GC or the spacebar on your PC keyboard to begin the run.

5. Inject sample into the On-Column injector.

6. Save and print the resulting PeakSimple chromatogram with the student's name for the sample identification. Typical results are about 12-14 area counts per 1% of CO_2.

7. Repeat steps 2-5 for each student. Compare chromatograms to find the winner.

Example TCD Breath Analysis

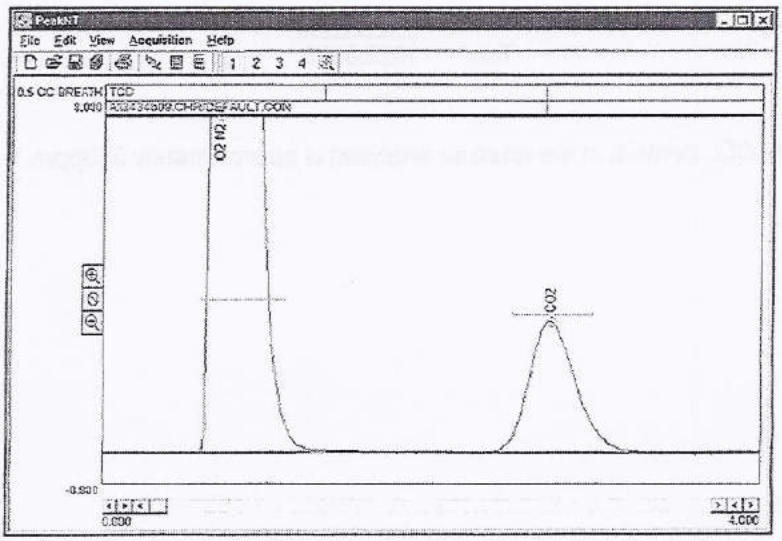
Column: 3' Silica Gel
Carrier: Helium at 10mL/min
Sample: 0.5cc human breath, direct injection
TCD current: LOW
TCD temperature: 100°C

Temperature Program:

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

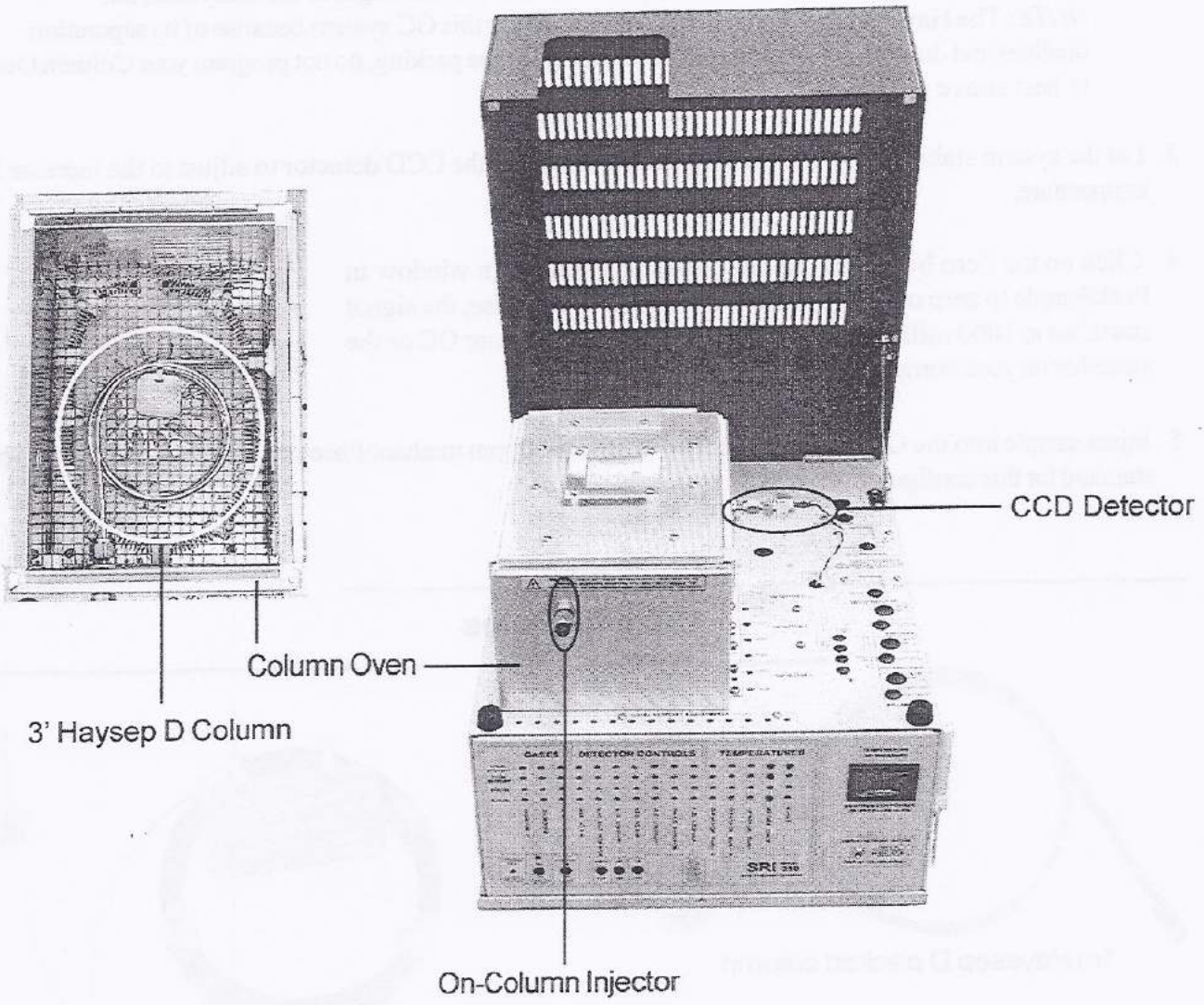
Results:

Component	Retention		Area
O_2 N_2	0.700		1379.4740
CO_2	2.700		61.9540
		Total	1441.4280



System Overview

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



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

General Operating Procedure

1. Connect your GC to your Windows PC with PeakSimple installed. Plug in your GC and turn its power on.

2. Set the Column Oven temperature to 130°C in PeakSimple as follows:

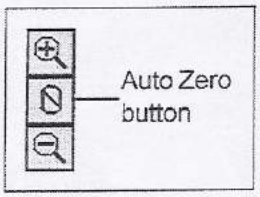
Initial Temp	Hold	Ramp	Final Temp
130.00	10.00	0.00	130.00

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

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

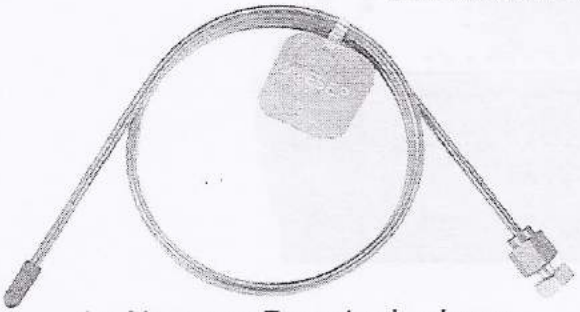
3. Let the system stabilize for at least 10 minutes, allowing the CCD detector to adjust to the increase in temperature.

4. Click on the Zero button to the left of the chromatogram window in PeakSimple to zero out the Data System signal. Otherwise, the signal starts out at 1000 millivolts. Press the RUN button on your GC or the spacebar on your computer keyboard to begin the run.



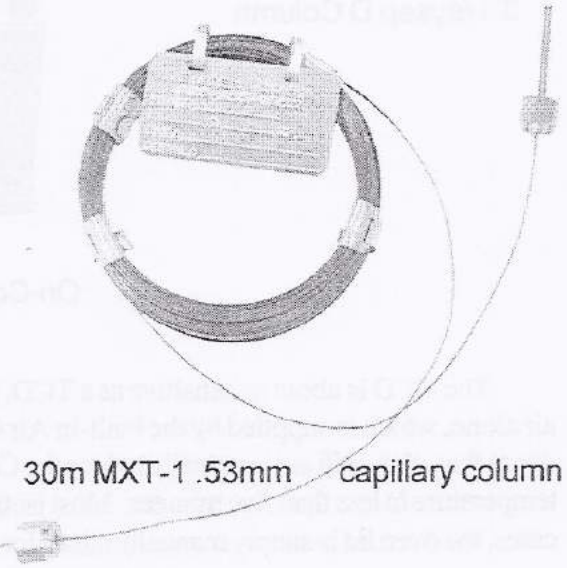
5. Inject sample into the On-Column Injector. A 1µL 1000ppm methanol/acetone sample is the factory test standard for this configuration.

Column Notes



1m Hayesep 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.



30m MXT-1 .53mm capillary column

Expected Performance

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

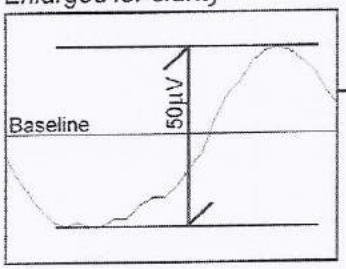
CCD Detector Noise Run

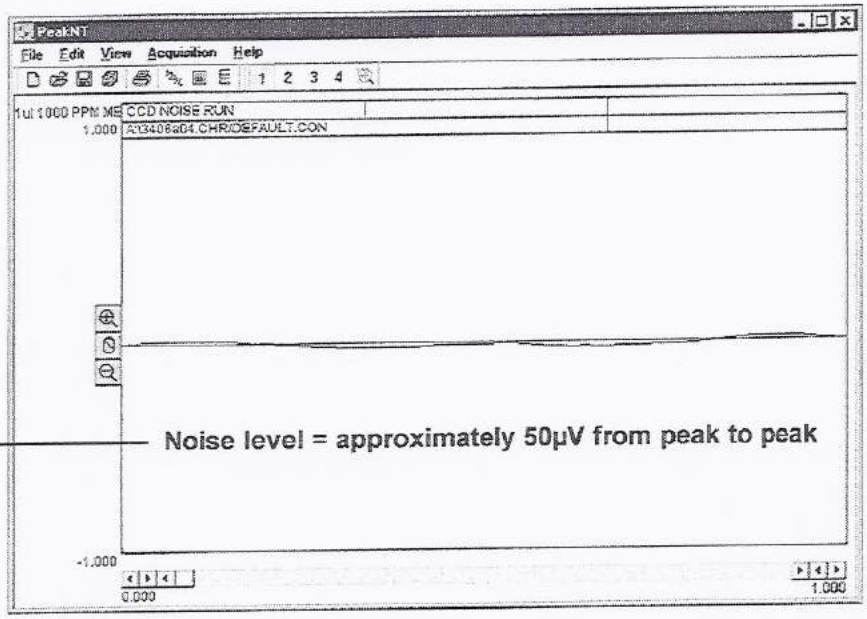
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:

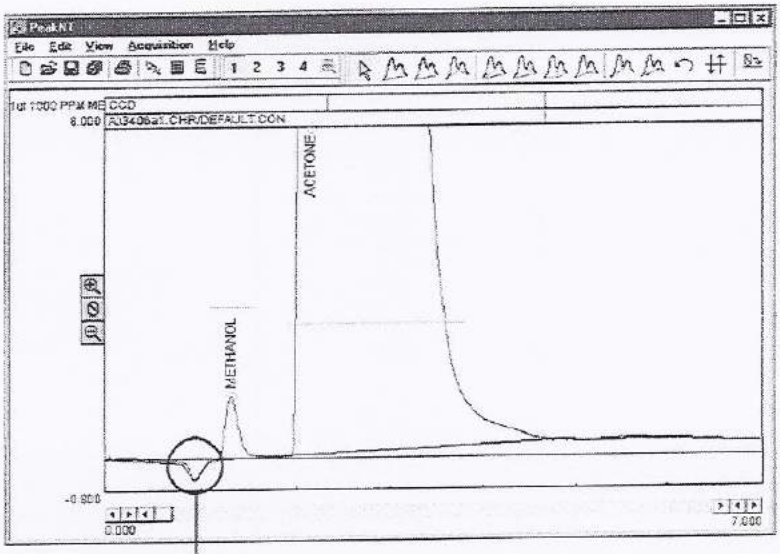
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C

Enlarged for clarity





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



Negative water peak

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

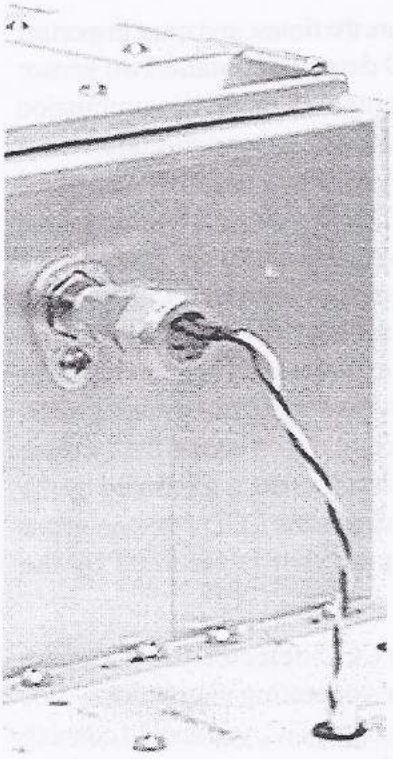
Isothermal Temperature Program:

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

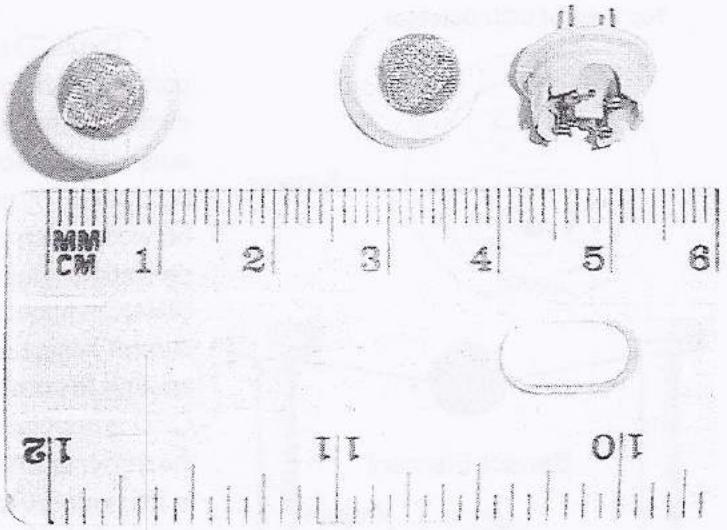
RESULTS:

Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
	Total	6958.5600

Overview

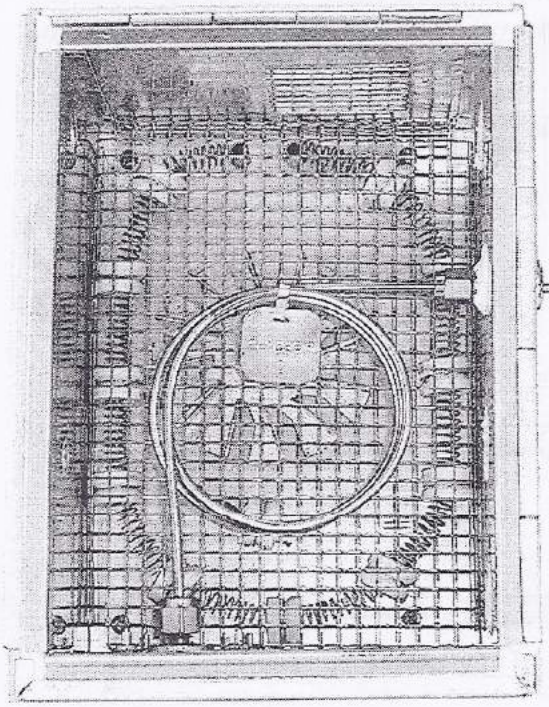


CCD on Column Oven



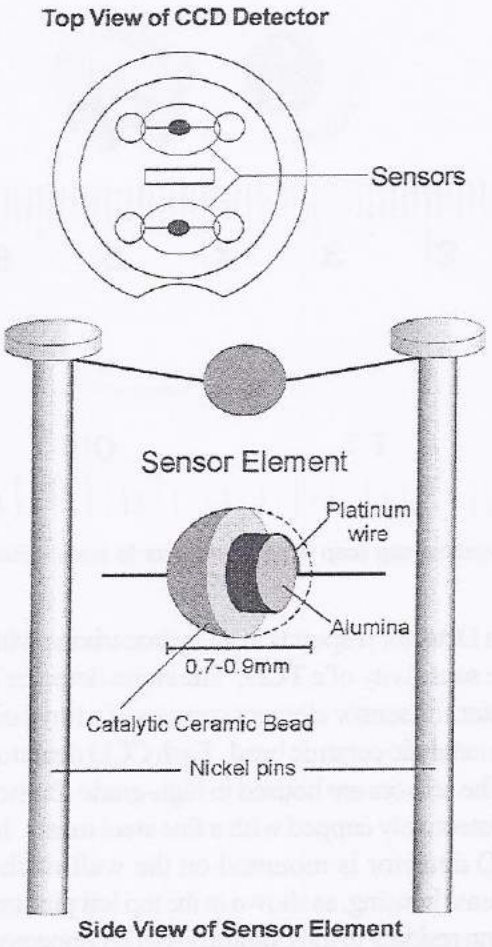
CCD Detector and protective cap (cap is removed prior to installation)

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



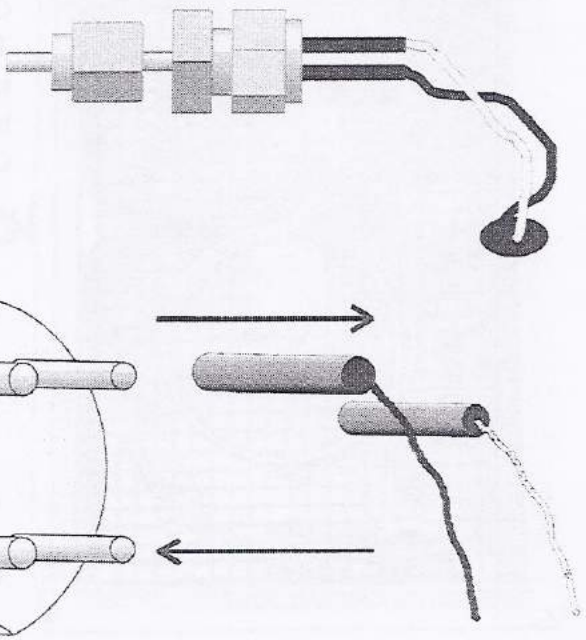
The CCD detector can also be used as a hydrocarbon monitor in non-chromatographic applications where the CCD senses the total hydrocarbon content of a flowing air stream, or as a hydrogen/hydrocarbon leak detector.

Theory of Operation



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

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



Expected Performance

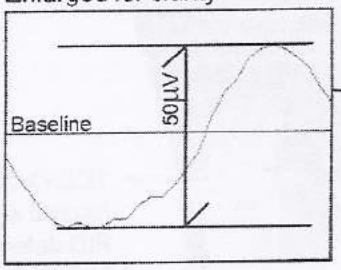
CCD Detector Noise Run

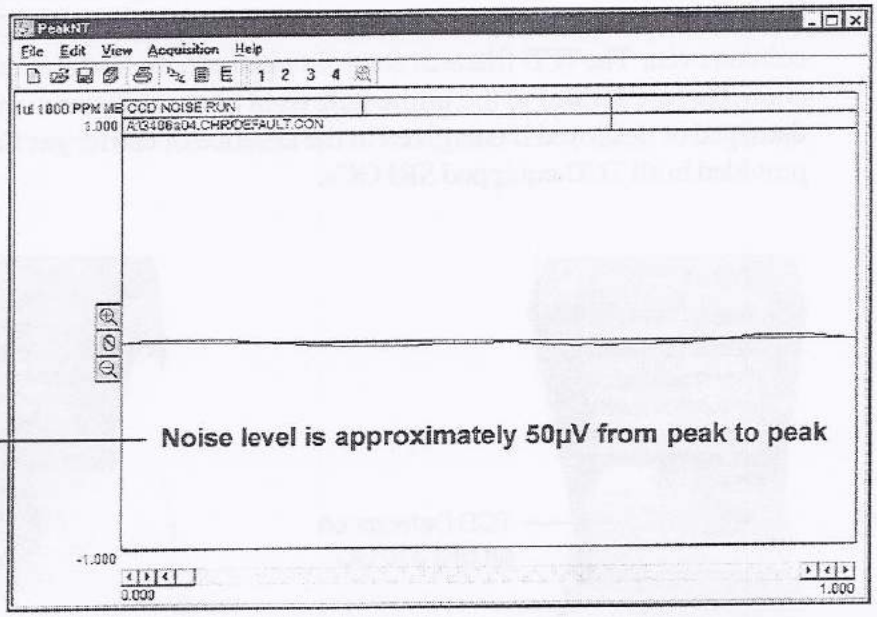
Column = 1m Hayesep D
Flow = 37mL/min

Isothermal Temperature Program:

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

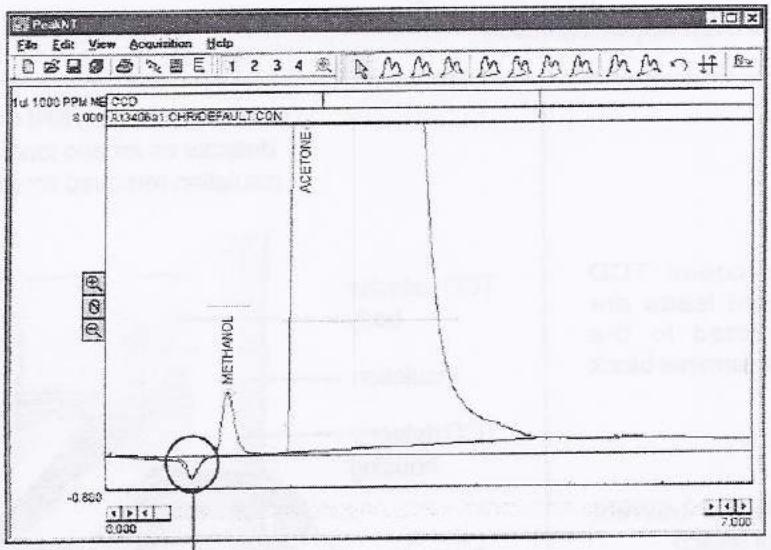
Enlarged for clarity





Noise level is approximately 50µV from peak to peak

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



Negative water peak

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

Isothermal Temperature Program:

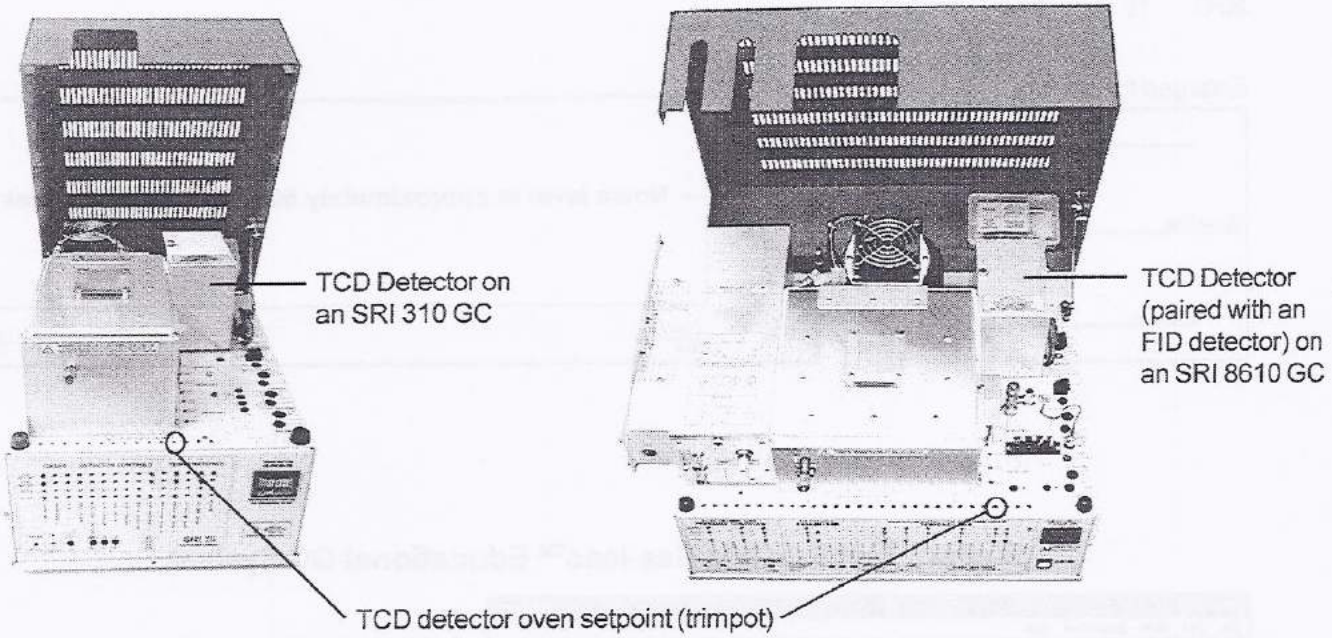
Initial Temp	Hold	Ramp	Final Temp
130°C	10.00	0.00	130°C

RESULTS:

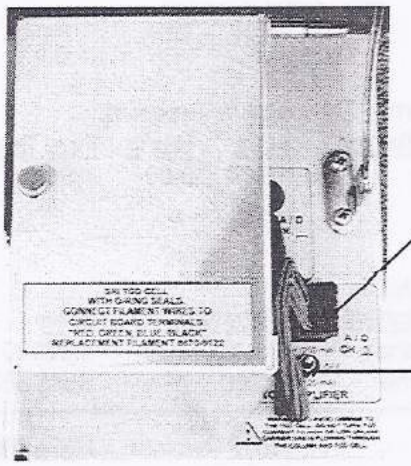
Component	Retention	Area
Methanol	0.816	13.2030
Acetone	2.000	6945.3570
	Total	6958.5600

Overview

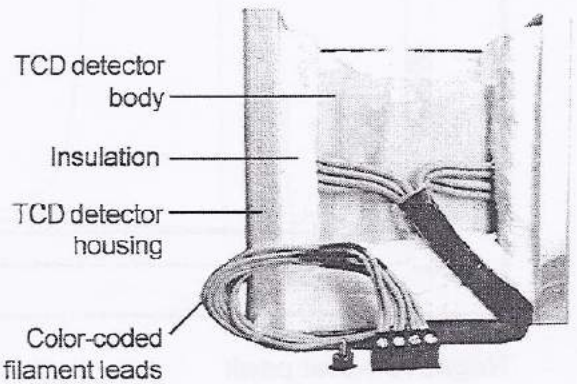
The Thermal Conductivity Detector (TCD) is the most universal detector available. Depending on the compound, the TCD responds with a detection range of 0.01% to 100% (100-1,000,000ppm). The SRI TCD consists of four filaments housed in a stainless steel detector block. The TCD detector block is installed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



TCD Detector on an SRI GC



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

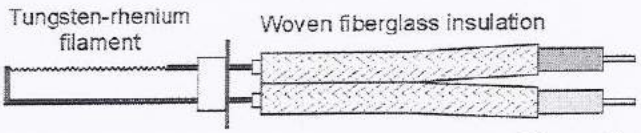


DETECTORS
Thermal Conductivity Detector - TCD

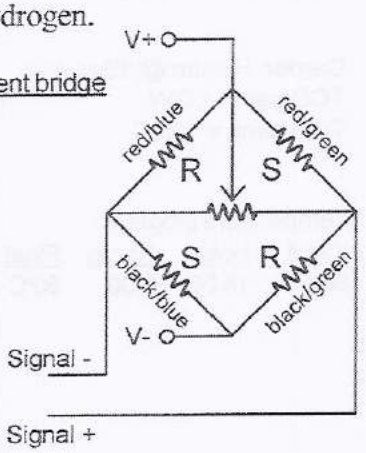
Theory of Operation

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

One of four TCD filaments

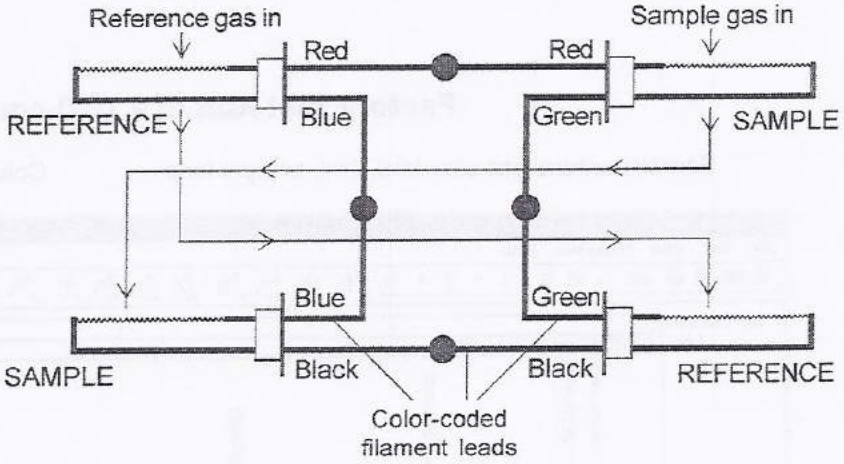


TCD filament bridge



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

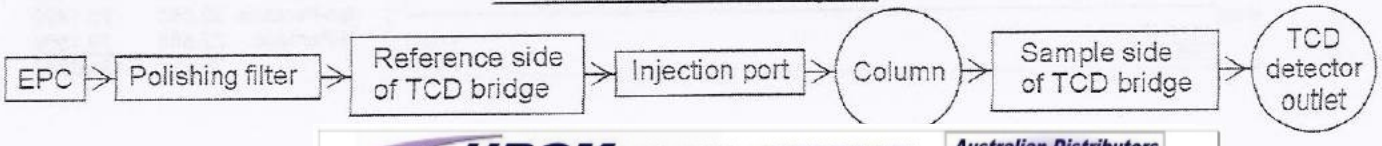
Simplification of filament interconnection



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

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

TCD carrier gas flow diagram



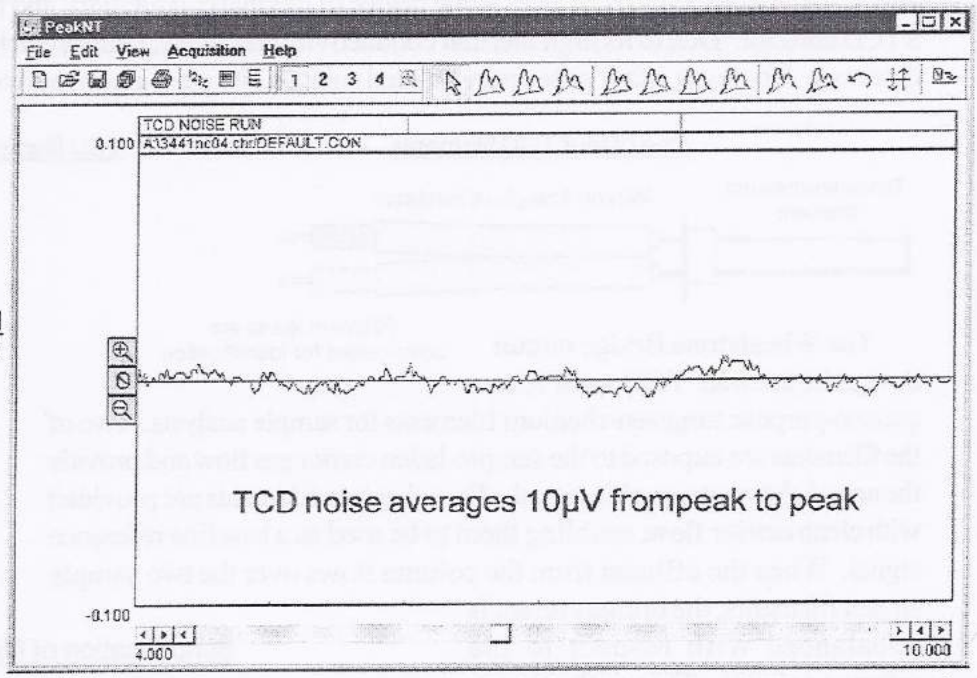
Expected Performance

TCD Noise Run

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

Temperature program:

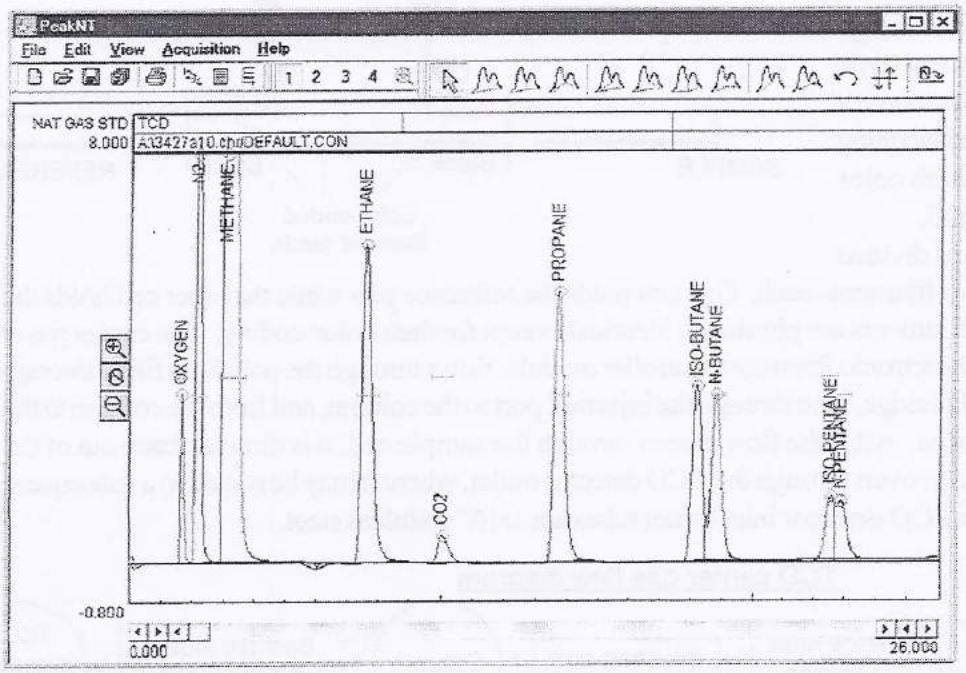
Initial	Hold	Ramp	Final
80°C	15.00	0.00	80°C



Factory Test Run of a TCD-equipped SRI GC

Sample: natural gas standard, 1mL sample loop

Columns: 1m Molecular Sieve, 2m Silica Gel



Events:

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

Temperature program:

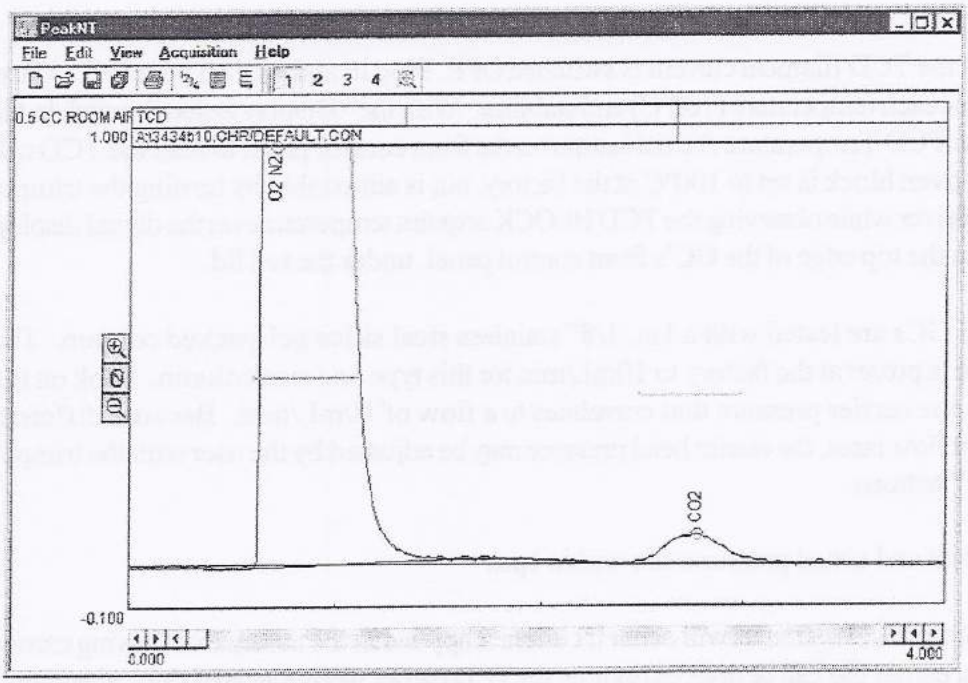
Initial	Hold	Ramp	Final
40°C	5.00	10.00	220°C
220°C	16.00	0.00	220°C

Results:

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

DETECTORS
Thermal Conductivity Detector - TCD

Expected Performance



TCD Room Air Analysis

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

Temperature Program:

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

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

Results:

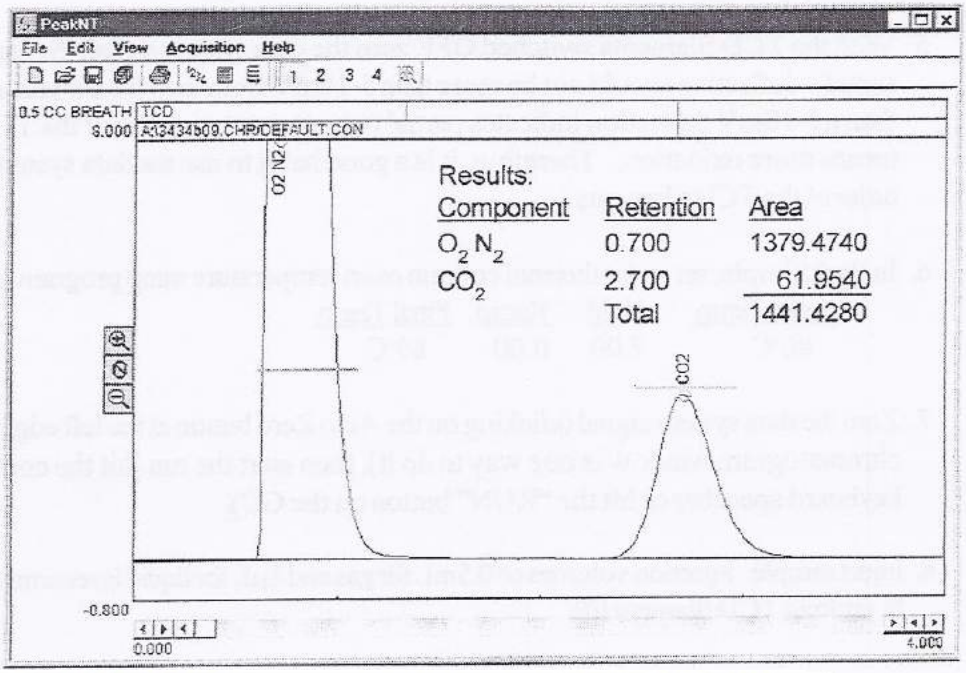
Component	Retention	Area
O_2 N_2	0.716	1021.3830
CO_2	2.766	1.5060
	Total	1022.8890

TCD Breath Analysis

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

Temperature Program:

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



General Operating Procedure

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

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

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

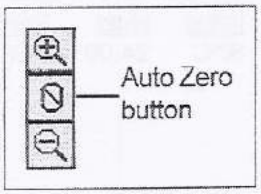
4. Damage or destruction of the TCD filaments will occur if current is applied in the absence of flowing carrier gas. ALWAYS verify that carrier gas can be detected exiting the TCD carrier gas outlet BEFORE energizing the TCD filaments. The carrier gas outlet tube is located on the outside of the Column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

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

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

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

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

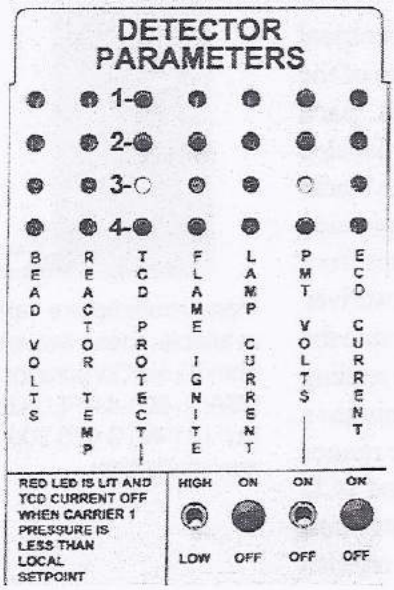


8. Inject sample. Injection volumes of 0.5mL for gas and 1µL for liquid is recommended to prolong TCD filament life.

TCD Filament Protection Circuit

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

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



1- LOCAL SETPOINT button
2- TOTAL SETPOINT button
3- STATUS LED
4- ACTUAL button

Bright red LED display

LED panel displays control data corresponding to the button pressed

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

1- Pressing the LOCAL SETPOINT button displays the filament cut-off setpoint value (factory set at 3psi) in the bright red LED display in the upper right corner of the GC's front control panel. If the carrier gas pressure reaches or falls below this value, the filament current will immediately be interrupted.

2- Pressing the TOTAL SETPOINT button displays the carrier gas pressure present in the GC system. Under normal operation, this value will be well above the 3psi cut-off setpoint.

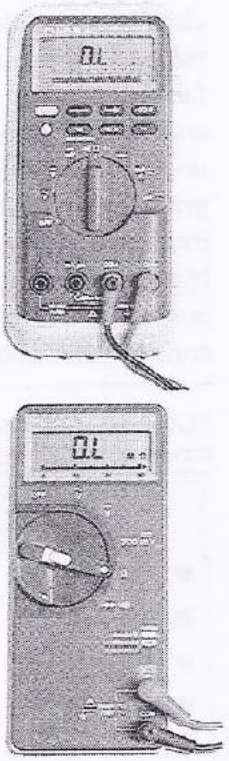
3- The STATUS LED glows bright red only when the TCD protection circuit has been activated.

4- Pressing the ACTUAL button displays the voltage present across one half of the TCD bridge. A value of 3.5 to 4.5 volts is typical when using high current; low current will display 2.5-3.5 volts (note: the LED displays 4 volts as "400," 3.5 as "350," etc.). Any value lower than these indicates a potential problem in the TCD detector bridge.

TCD Troubleshooting

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

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



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

SRI TCD detector replacement parts

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

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

Replacing the TCD Filaments

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

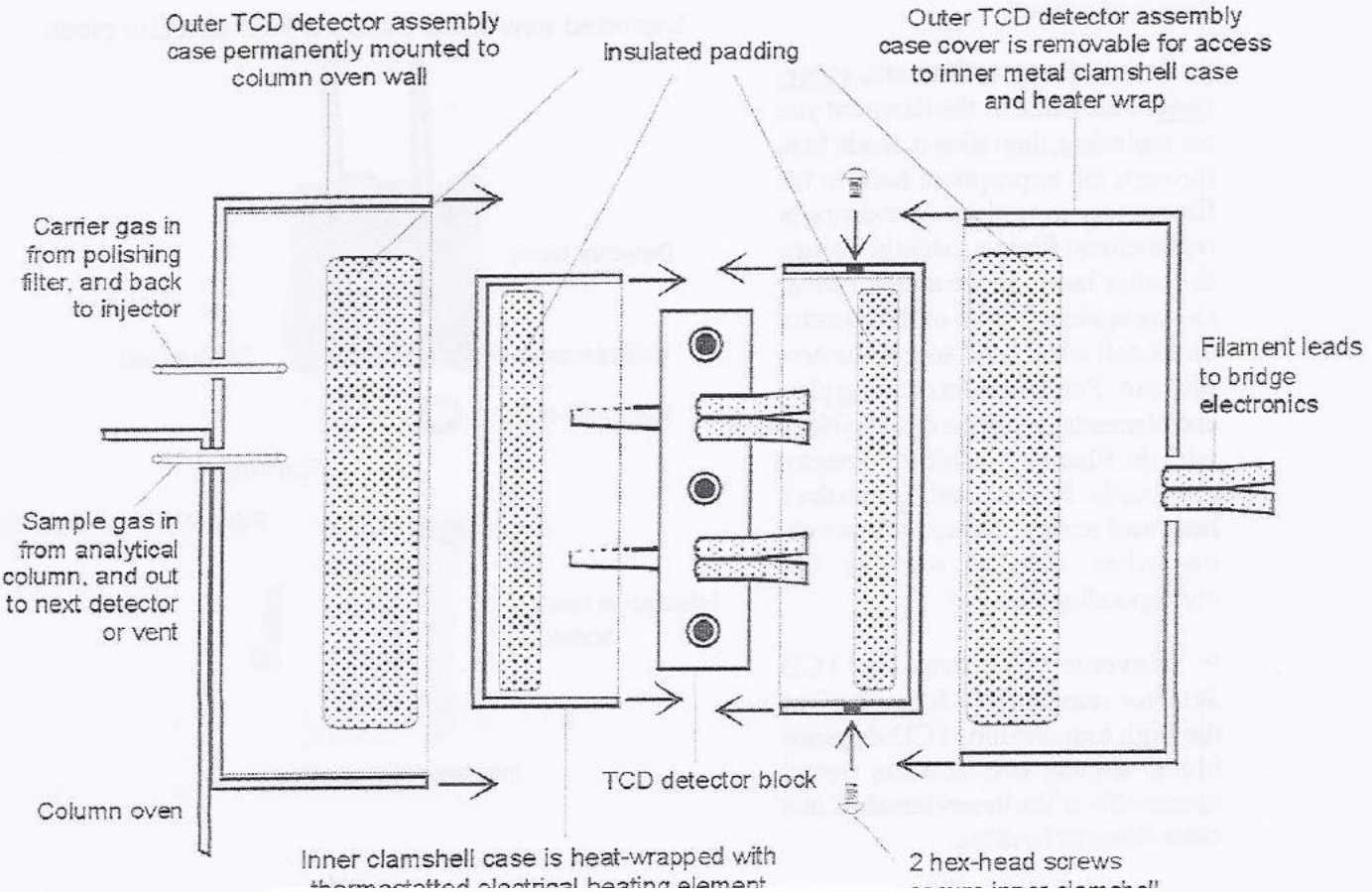
A. Standard TCD detector block access

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

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

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

Exploded view of the standard TCD detector assembly



Replacing the TCD Filaments continued

(Standard TCD detector block access continued)

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

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

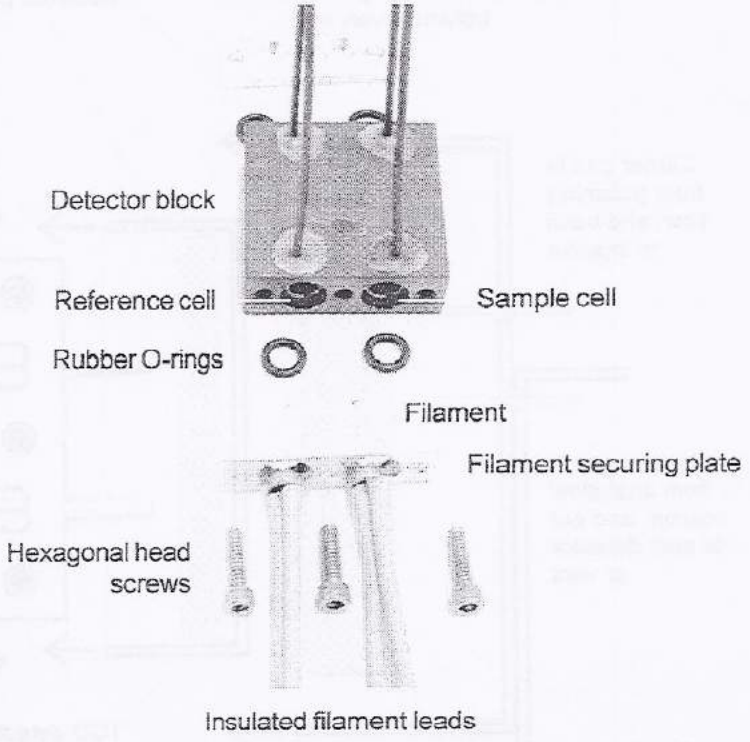
6. The TCD filaments are secured in the detector block by two plates, each of which is held in place with three hexagonal head screws. Holding the detector block with one hand, use an Allen wrench to unscrew and remove the hexagonal head screws from one of the filament securing plates. Then, slide the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

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

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

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

Exploded view of the standard TCD detector block



Replacing the TCD Filaments continued

B. High temperature TCD detector block access

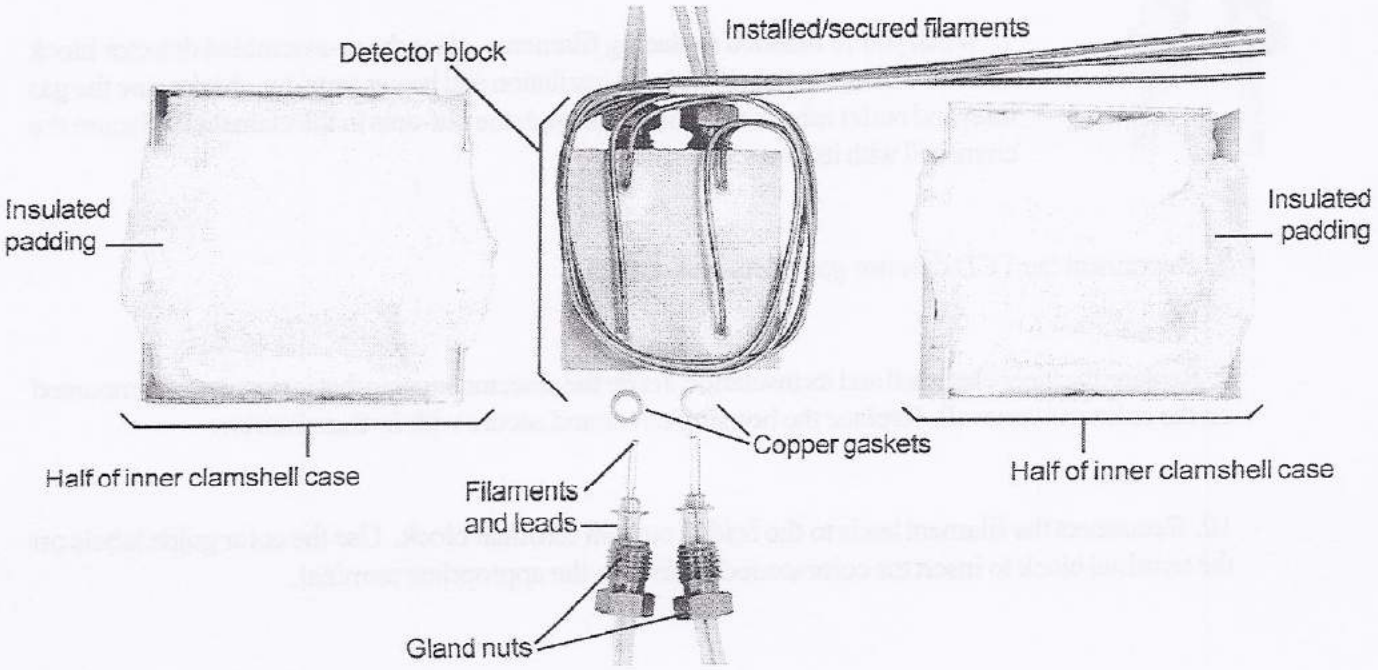
The high temperature TCD assembly is the same as the standard: outer housing around an inner clamshell case. The high temp detector block uses gland nuts and copper gaskets to secure the four filaments in its two cells. Instead of the heater rope, it employs a heating cartridge, which is inside the inner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

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

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

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

Exploded view of high temperature TCD detector block and inner clamshell

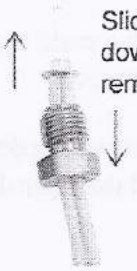


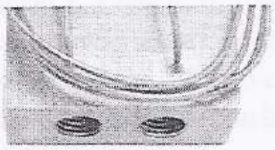
Replacing the TCD Filaments continued

(High temperature TCD detector block access continued)

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

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





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

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

8. Reconnect the TCD detector gas inlets and outlets.

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

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

Chapter: TCD Detector

Topic: Thermal Conductivities of common gases

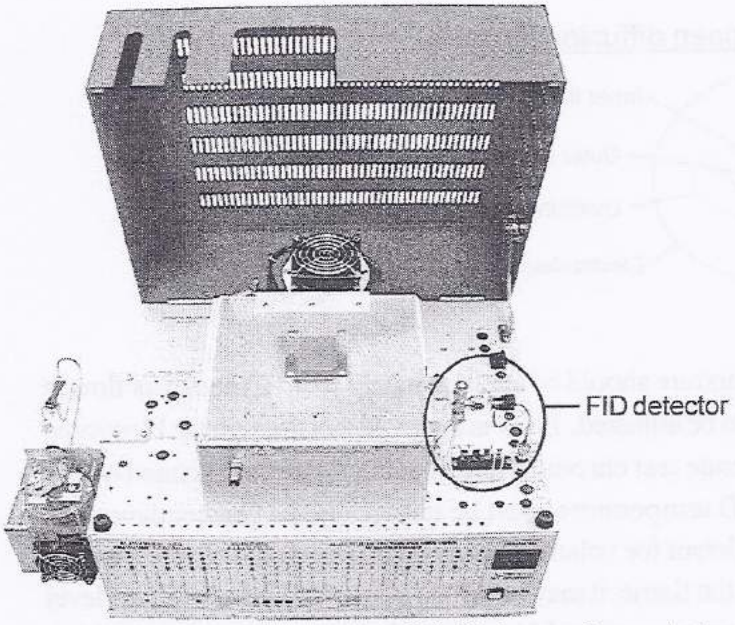
As illustrated by the table below, Helium and Hydrogen have the highest thermal conductivities of any gases. The TCD detector responds to the difference between the thermal conductivity of the carrier gas and the analyte peak. The greater the difference, the better the sensitivity. For this reason, Nitrogen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

THERMAL CONDUCTIVITIES OF SOME COMMON GASES

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

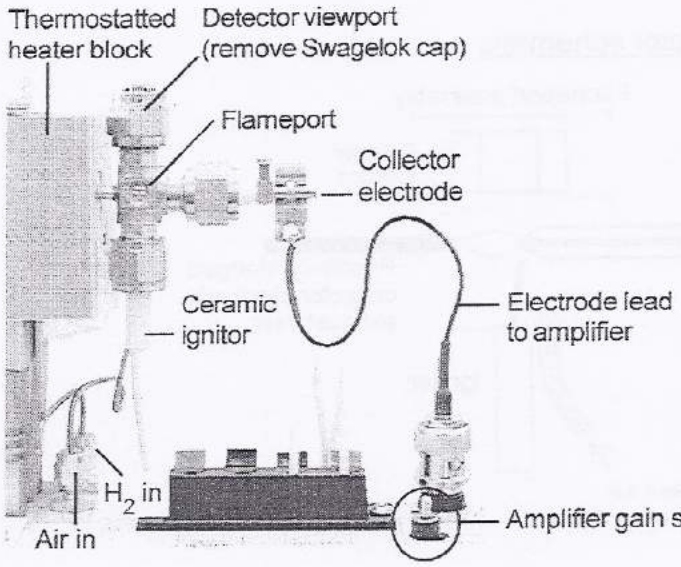
Overview

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



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

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

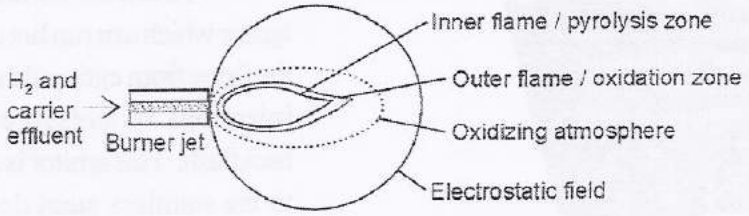


DETECTORS
FID - Flame Ionization Detector

Theory of Operation

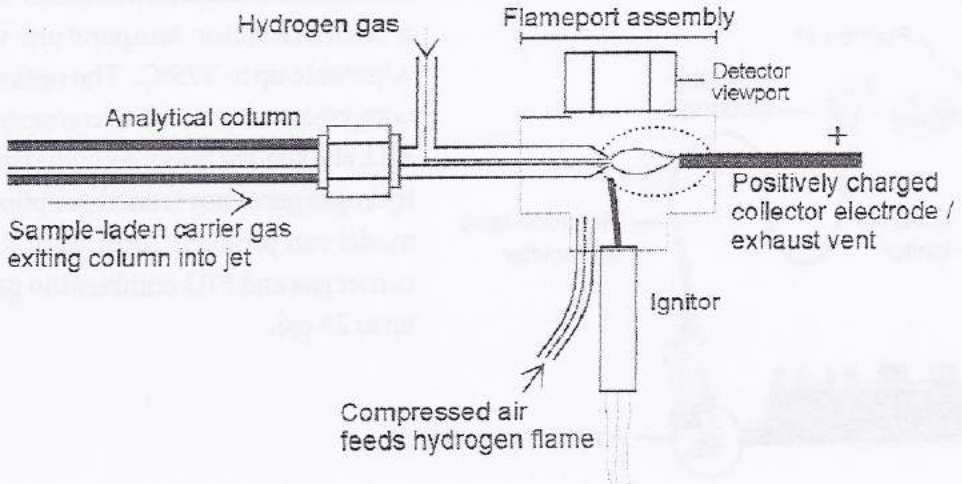
In the SRI FID, the carrier gas effluent from the GC column is mixed with hydrogen, then routed through an unbreakable stainless steel jet. The hydrogen mix supports a diffusion flame at the jet's tip which ionizes the analyte molecules. Positive and negative ions are produced as each sample component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

The FID hydrogen diffusion flame



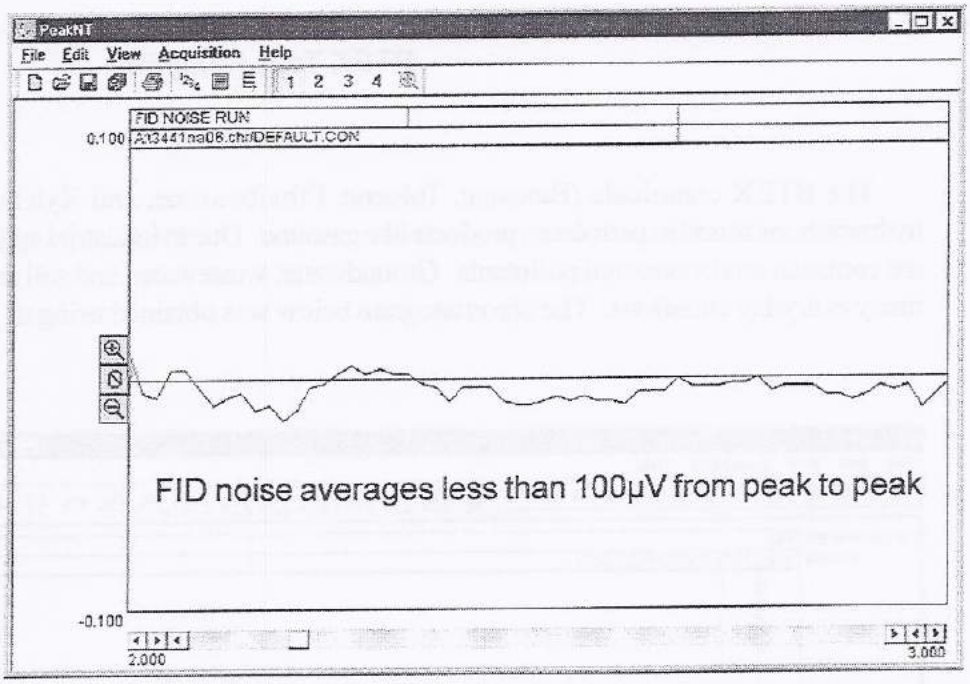
The ratio of air to hydrogen in the combustion mixture should be approximately 10:1. If the carrier flow is higher than normal, the combustion ratio may need to be adjusted. Flow is user adjusted through the Electronic Pressure Controllers (EPC); the rates used to generate test chromatograms at the factory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

FID detector schematic



Expected Performance

FID noise run

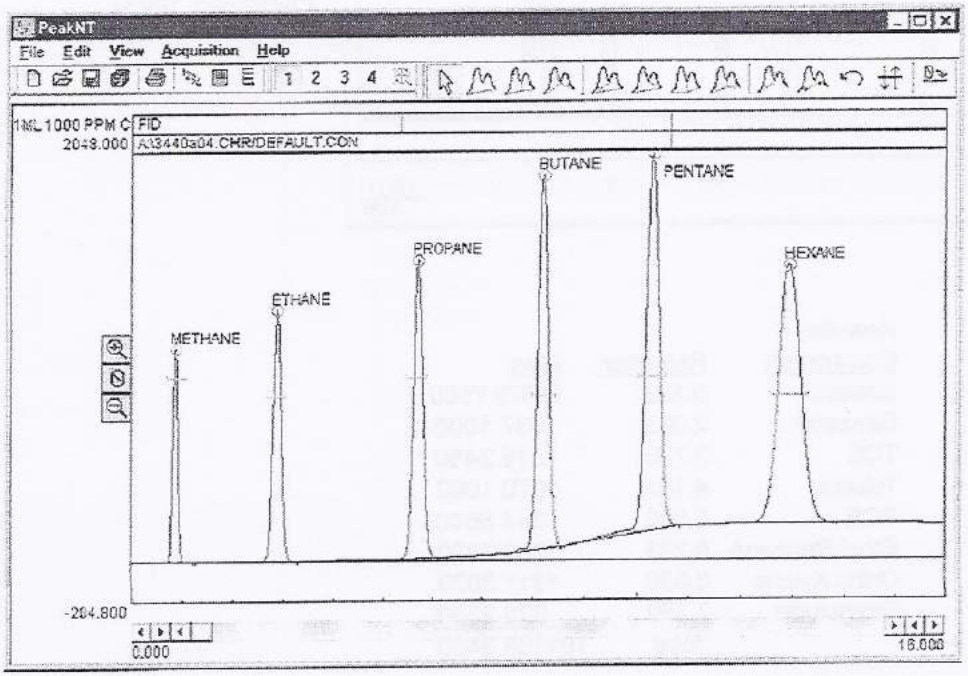


Column: 15m MXT-1
Carrier: Helium @ 10mL/min
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Temperature program:

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

C_1-C_6 Hydrocarbon Test Analysis



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

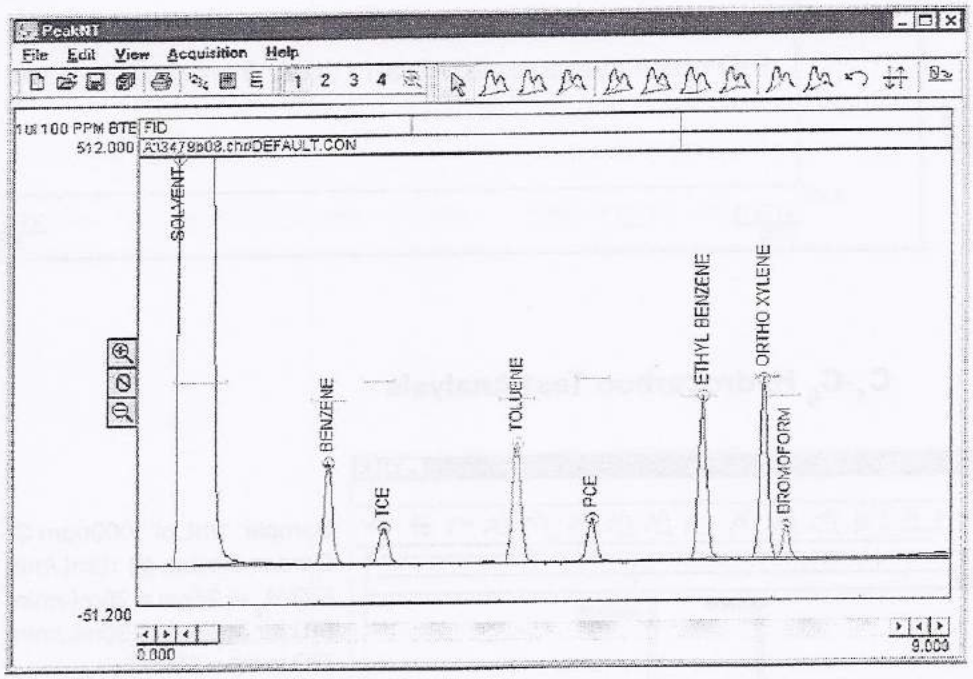
Results:

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

Expected Performance

BTEX Test Analysis

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



1µL 100ppm BTEX sample

15m MXT-VOL capillary column

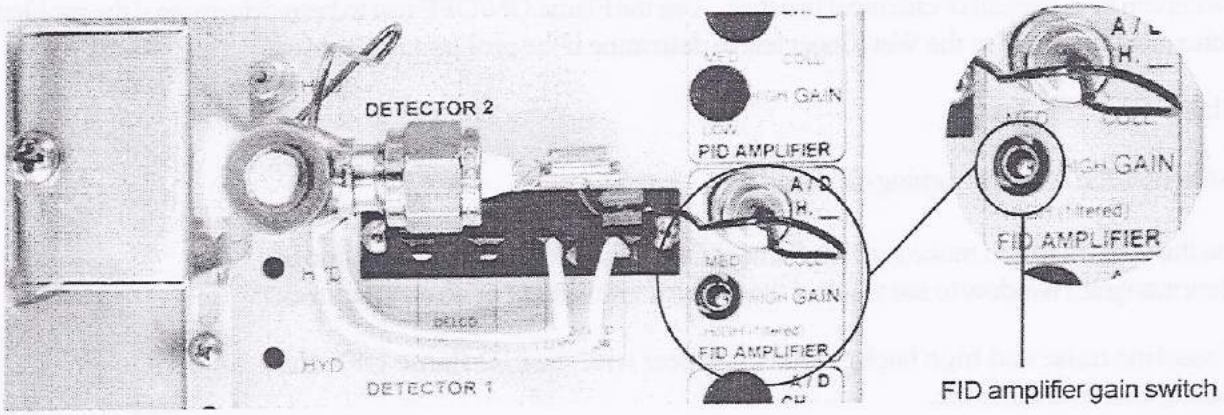
FID gain = HIGH
FID temp = 150°C
FID ignitor = -400

Results:

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

General Operating Procedure

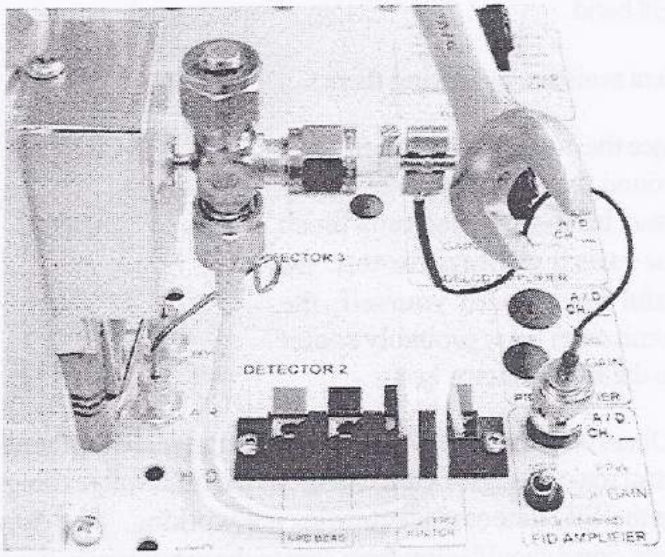
1. Set the FID amplifier gain switch to HIGH for most hydrocarbon applications. If peaks of interest go off the scale (greater than 5000mV), set the gain to MEDIUM. When peaks of interest are 20 seconds wide or more at the base and extra noise immunity is desired, set the gain switch to HIGH (filtered).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



FID amplifier gain switch

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

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



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

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

FID Troubleshooting

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

A. Flame ON/OFF Test

1. Extinguish the flame by turning off the air.

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

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

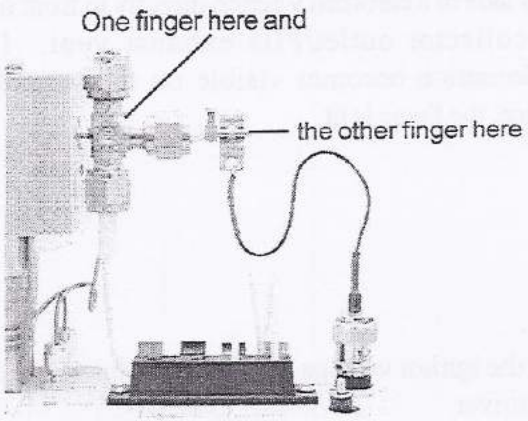
4. Isolate the column by capping off the column entrance to the detector with a swagelok-type cap or a nut and septum. Turn the air back on and light the FID flame. If the detector noise is similar to the background that was observed with the flame OFF, the column is suspect.

B. Wet Finger Test

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

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

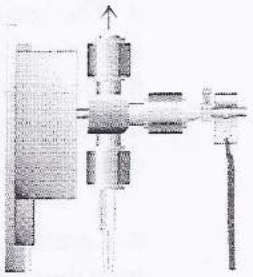
3. Place one finger on the collector electrode, and place the other on bare metal (like the FID detector body or the column oven lid) to ground the collector. Make your contact brief--you need only brush these parts to perform the test. Be careful not to burn yourself; the column oven lid is probably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



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

Cleaning the FID

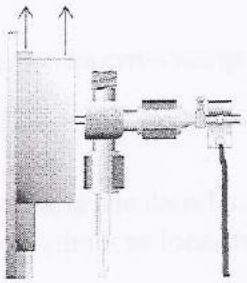
The FID detector rarely requires cleaning or servicing. It may develop a film or coating of combustion desposits in the flameport with extended use. Use the FID detector viewport to check for visible deposits. If you're experiencing problems with your FID detector, try cleaning it, even if you can't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



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

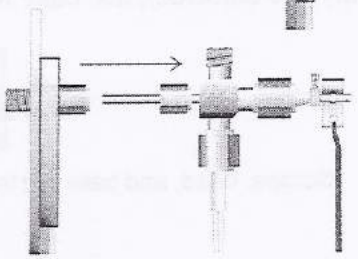
2. Remove flameport assembly from the heater block

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

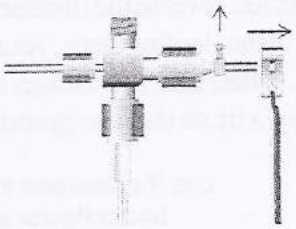


   b. Using a philps head screwdriver, remove the screw on the top of the FID's heater block and pull the aluminum cover up and off.

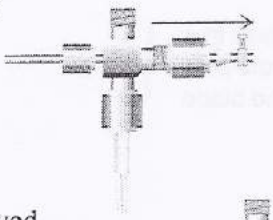
   c. Gently pull off the white insulation to reveal the detector's bulkhead fitting on the column oven wall. Loosen this fitting to disconnect the flameport.



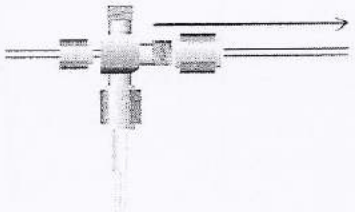
3. Remove the collector electrode



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



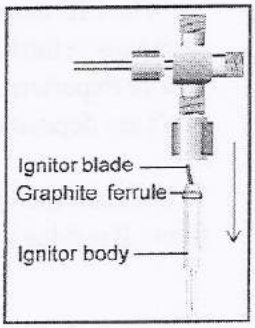
   b. Loosen and remove the nut and ferrule that hold the collector electrode in the flameport body.



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

Cleaning the FID continued

4. Remove the FID ignitor element

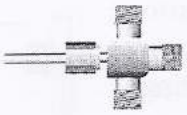


a. The ignitor element is brittle and will break when stressed, so handle the ignitor carefully, mindful of any torque on the blades. While holding the ignitor by the ceramic body with one hand, loosen the 1/4" swagelok-type nut that holds it in place. There is a graphite ferrule inside this nut that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

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

FID ignitor removed from the flameport assembly

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



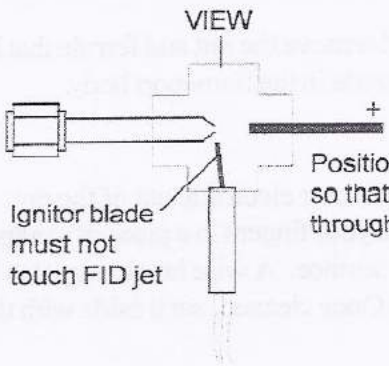
Scrape, rinse, and bake out the FID flameport interior

6. Re-assembly

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

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

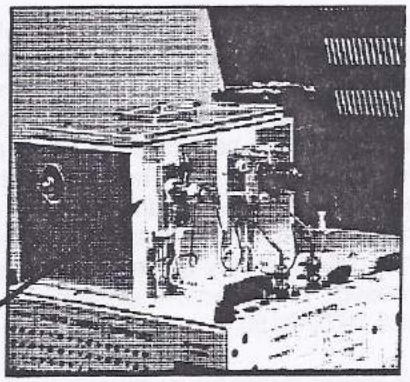
Use the viewport to correctly position the FID ignitor and collector electrode inside the flameport



Chapter: FID DETECTOR

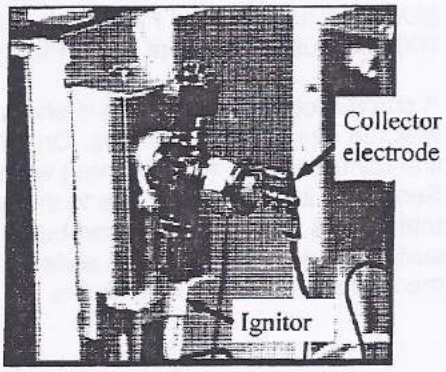
Topic: Operation of FID detector without hydrogen (FLID mode)

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

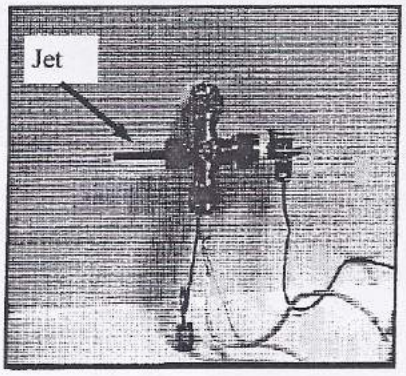


The FID detector is normally located on the right hand side of the column oven.

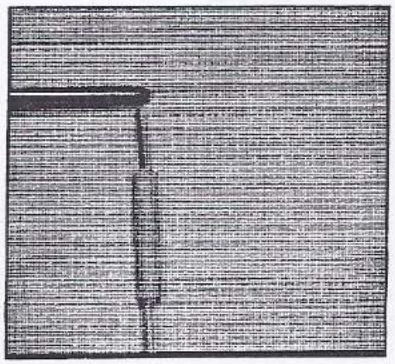
The FID normally requires a flow of 20-30 ml/min of hydrogen and 200-300 ml/min of air to support a hydrogen flame at the tip if the jet. The heat of the flame ionizes the analyte molecules, and the negative ions allow a small electric current to flow between the collector electrode and the grounded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



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



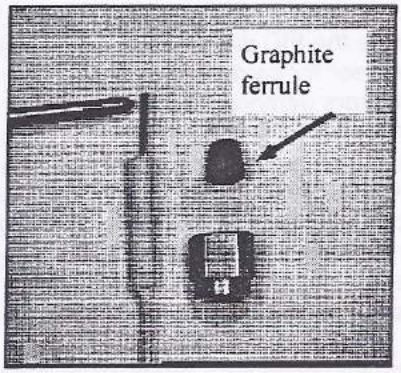
Inside the FID detector body, the ignitor is normally positioned just below and behind the tip of the jet. Notice that the ignitor blade is tilted at a 15 degree angle from the ceramic tube in which it is fabricated. In normal FID operation, the ignitor is positioned below and behind the jet so it will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



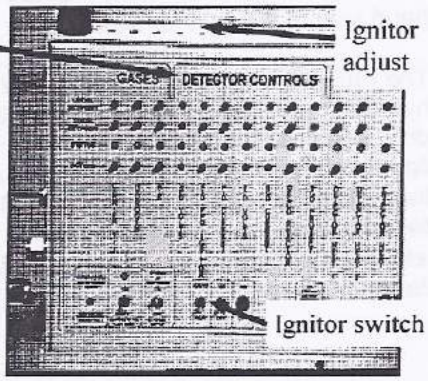
Chapter: FID DETECTOR

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

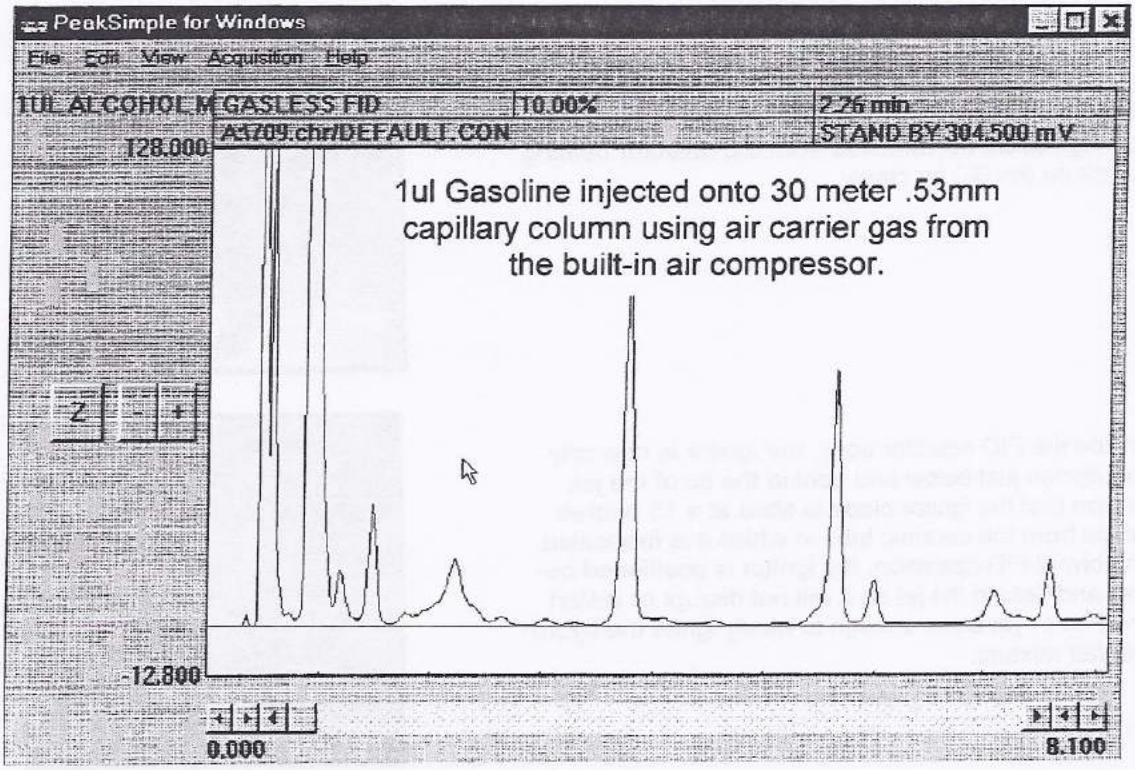
In the FLID mode, the ignitor itself provides the heat necessary to ionize the sample molecules. Accordingly, the ignitor needs to be positioned directly in front of the jet. The slight angle of the ignitor allows the ignitor tip to be located 1-2 mm in front and slightly above the jet. The ignitor is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



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



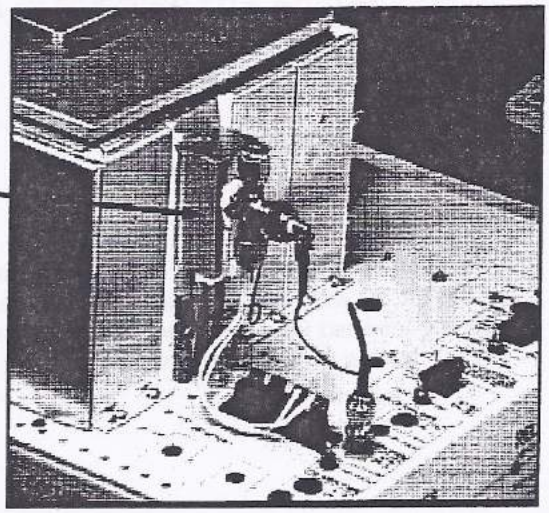
A chromatogram of gasoline is shown below which was run using the FLID mode. Only the larger gasoline components (> 1000 ppm) were detected. Sensitivity is exponential due to the temperature rise that occurs when the peak combusts on the ignitor surface. Large peaks which elute quickly may cool the ignitor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



Chapter: Detectors

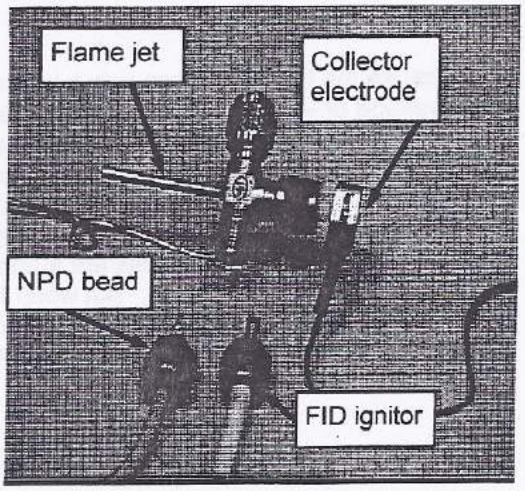
Topic: Converting from FID to NPD mode

To convert the FID detector to NPD detector:

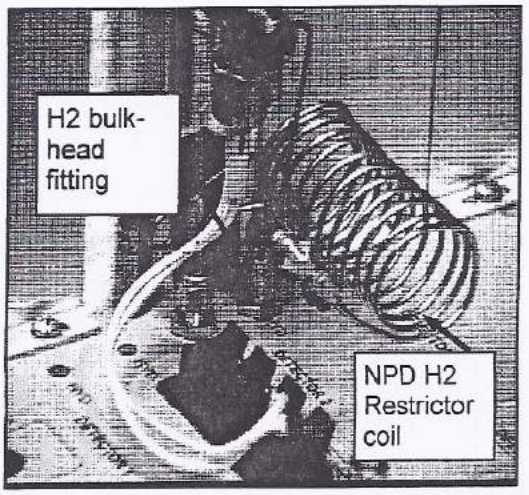


1) The FID and NPD detectors are almost identical. The detector body is mounted on a heated aluminum block on the right hand side of the GC oven.

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



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

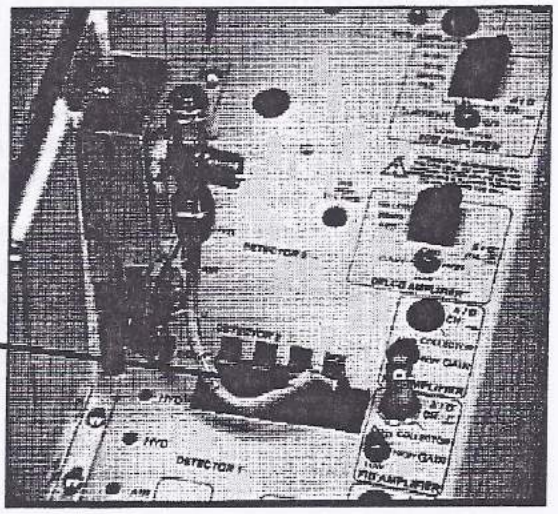


3) The gas flows to the NPD detector are different than the FID gas flows. The NPD hydrogen flow is normally about 3 ml/min while the FID hydrogen flow is about 25ml/min. To obtain this lower H2 flow rate, an additional restrictor coil is attached to the hydrogen bulkhead fitting immediately below the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

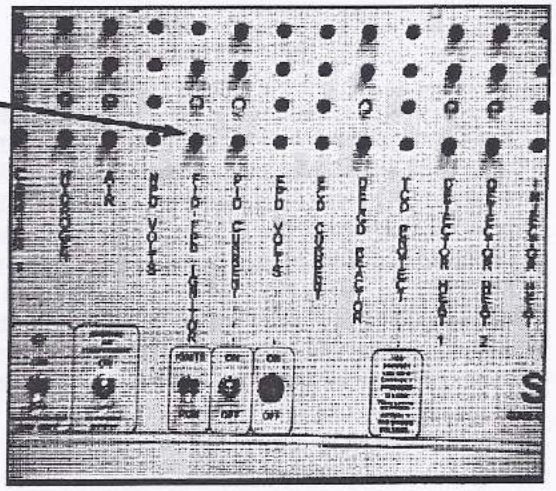
Chapter: Detectors

Topic: Converting from FID to NPD mode

To convert the FID detector to NPD detector:



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



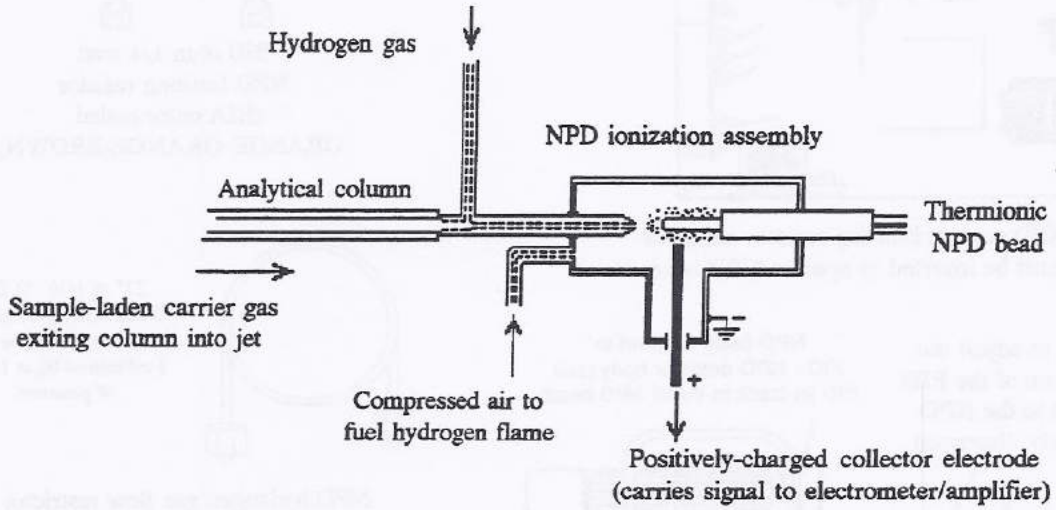
5) Because the NPD bead can only tolerate a maximum voltage of –4.50 volts, be careful not to set the FID volts setpoint higher than –4.50. Be especially careful not to flip the FID ignite switch to the up position, as this will apply 10 volts to the NPD bead and burn it out. When an NPD detector is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

Chapter: DETECTORS

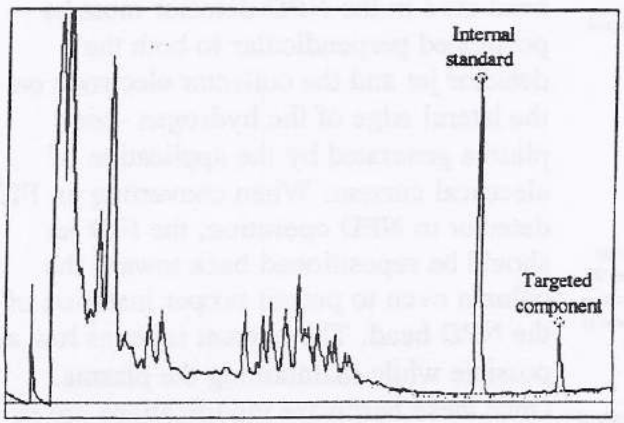
Topic: NPD - Theory of Operation and General Information

The Nitrogen / Phosphorus Detector, or NPD, as it is commonly referred to, is specified for the analysis of organic compounds containing nitrogen and/or phosphorus. The NPD detector is ideal for the analysis of pesticides and herbicides such as Parathion®, which contains nitrogen, phosphorus and sulfur. Minimum detectable quantities are in the 10 picogram range for this compound.

In principle, when organic compounds containing either nitrogen or phosphorus are introduced into a hydrogen plasma induced around a salt, charged particles or ions are generated, either by thermal ionization or oxidation. In the case of the NPD detector, ionization occurs as each sample component is eluted into the hydrogen plasma glowing around the NPD bead which is located directly at the exit of the analytical column. Ions freed in the plasma are then collected by a positively-charged collector electrode immediately adjacent to the gas plasma, and the electrical current produced by the passing of each component is carried to the electrometer/amplifier for processing and routing to the data system. The SRI NPD design exhibits extremely linear response.



BASIC NITROGEN/PHOSPHORUS DETECTOR DIAGRAM



5 ppm concentration of target component in nitrogen

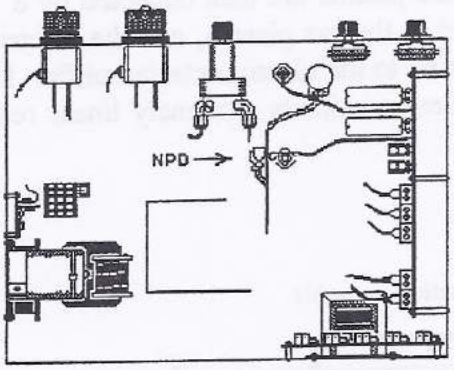
The illustration at left shows a 5ppm concentration of a proprietary gasoline additive in nitrogen. The operation of the NPD detector was adjusted to permit display of the gasoline peaks immediately preceding the internal standard and target peak. If the detector response was optimized, the selectivity of the detector would have eliminated the gasoline peaks from the chromatogram. In this case, it was desireable to show the gasoline peaks, and the NPD H_2 gas and bead were adjusted according.

Chapter: DETECTORS

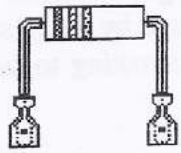
Topic: Preparation of The Nitrogen-Phosphorus Detector For Operation

The majority of gas chromatographs manufactured by SRI Instruments that offer the Nitrogen-Phosphorus Detector option are equipped with a flame ionization detector as standard equipment. The Nitrogen-Phosphorus Detector is configured for use by a conversion (hardware modification) to the FID system in the field. The modification of the FID detector is minimal and may be performed in a matter of minutes. Reversion back to the standard FID detector configuration may be performed as needed without any major interruption to the throughput of analyses through the chromatograph.

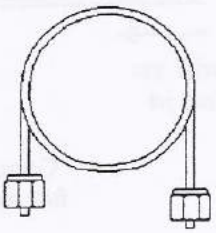
In some specific cases, the gas chromatograph is equipped with a permanent configuration of the nitrogen-phosphorus detector (NPD). On such models, the hardware modifications indicated on this page are not applicable, as the detector is already configured and not convertible.



Location of NPD current limiting resistor inside of GC chassis (must be inserted to operate NPD bead).

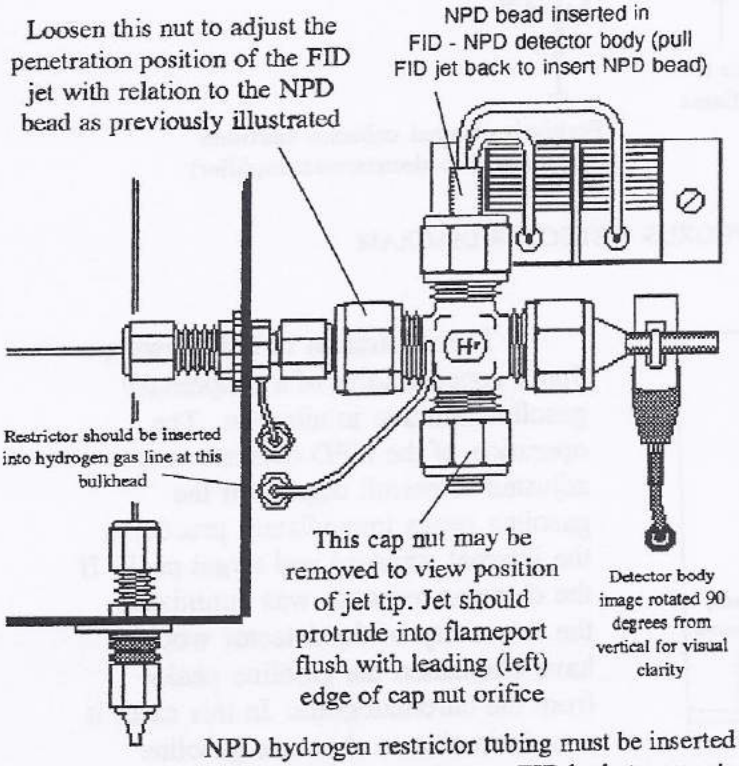


330 ohm 1/4 watt
NPD limiting resistor
(EIA color coded
ORANGE-ORANGE-BROWN)



21" of 1/16" O.D.,
0.005" I.D. S.S. tubing
Flow should be
3 ml/min of H_2 at 10 psi
of pressure.

NPD hydrogen gas flow restrictor
(for reducing hydrogen gas flow
to proper level for NPD operation)

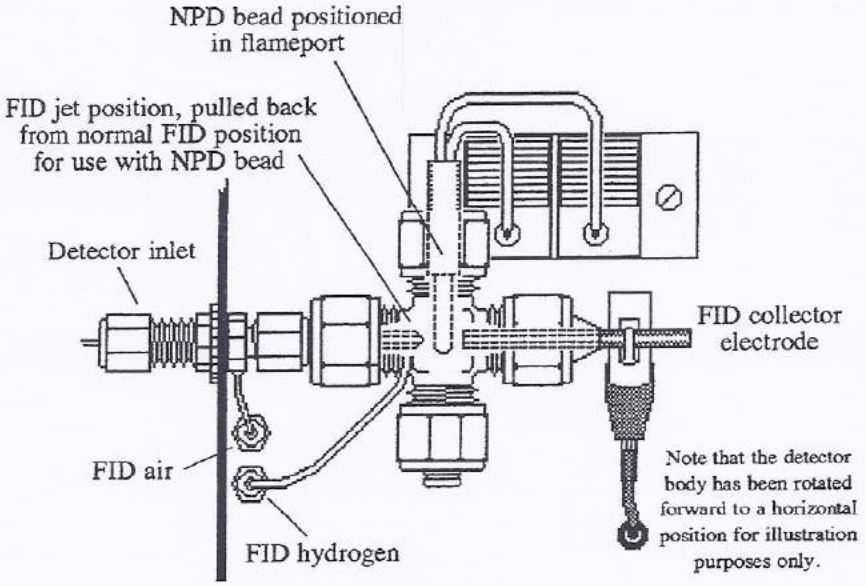


NPD hydrogen restrictor tubing must be inserted into hydrogen supply line to FID body to operate NPD detector

The thermionically-heated alkali bead used in the NPD detector must be positioned perpendicular to both the detector jet and the collector electrode on the lateral edge of the hydrogen - air plasma generated by the application of electrical current. When converting an FID detector to NPD operation, the FID jet should be repositioned back toward the column oven to permit proper insertion of the NPD bead. The current is set as low as possible while maintaining the plasma. Once these hardware modifications are in place as indicated, adjust the NPD detector as instructed on the previous page.

Chapter: DETECTORS

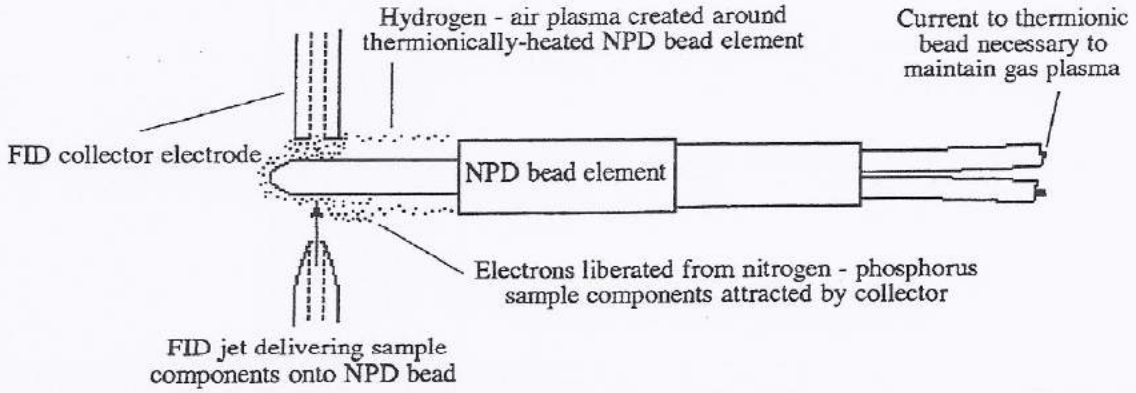
Topic: Operation Of The Nitrogen - Phosphorus (NPD) Detector



NPD detector assembly (modified FID detector)

The SRI design FID detector converts quickly into a nitrogen - phosphorus detector by the simple replacement of the FID ignitor element with the electrically-heated thermionic NPD bead element. The hydrogen flow is then manually reduced to between 1 and 7 ml. per minute by the installation of a flow restrictor prior to the FID detector hydrogen bulkhead fitting (between 1 and 3 ml. per minute is optimum). With the heated NPD bead installed directly in the carrier gas flow exiting the FID jet orifice, the sample components are directed toward the area of the bead, where any molecule containing a nitrogen or phosphorus group is prompted to release an electron in the hydrogen-air plasma generated by the bead. The liberated electron is attracted to the charged collector electrode and it creates a current that is delivered to the FID electrometer for processing. Note that the FID jet should be relocated (pulled away) from the NPD bead and collector electrode, as illustrated, for proper operation. Return the FID jet to its original position when returning to FID operation.

The nitrogen - phosphorus bead is extremely selective, providing 10,000 times higher response to nitrogen - phosphorus compounds than to hydrocarbons. When using the NPD detection method, nitrogen is the carrier gas of preference although helium carrier gas may be used. The bead must be operated with the minimum current required to maintain the hydrogen-air plasma. If more current than necessary is applied, the detector will show greater sensitivity, but the life and subsequent sensitivity of the NPD bead will be greatly reduced. The NPD should only be operated with the 330 ohm NPD current-limiting resistor in place in the circuit. AT NO TIME should the NPD voltage be operated above 4.4 volts. Nitrogen - phosphorus compounds increase the current in the plasma as they collide with the surface of the thermionic heated alkali metal bead.

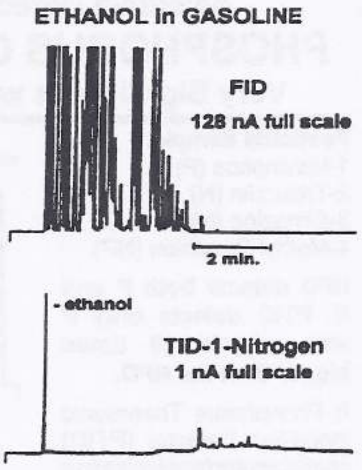


In order to operate the NPD detector, verify that the hydrogen flow rate is in the vicinity of 3 ml per minute by using a bubble flow meter capable of accurate measurement in that range. The hydrogen flow rate is normally maintained at 20 to 25 ml per minute. The air flow must be adjusted to approximately 80 ml per minute. This is much lower than the 250 - 300 ml per minute flow rate that is typically used for flame ionization detector operation.

Oxygenate Selective Detection for GC
ETHANOL in GASOLINE

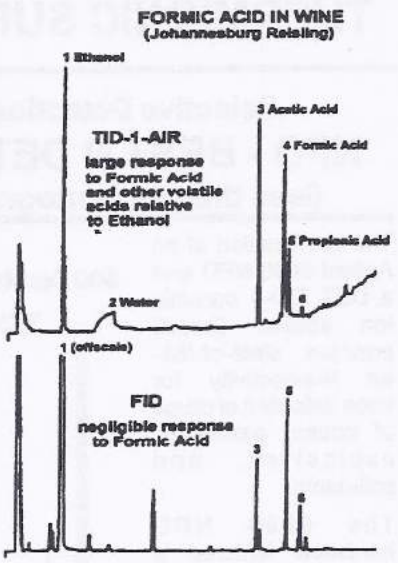
Negative Ionization on a ceramic TID-1 surface detects Oxygenates with good selectivity vs. Hydrocarbons.

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



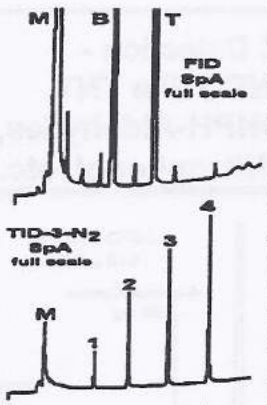
Oxygenate Selective Detection for GC
CARBOXYLIC ACIDS

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



Selective Detection for GC
TRIHALOMETHANES

TID-3 surface catalyzed negative ionization process

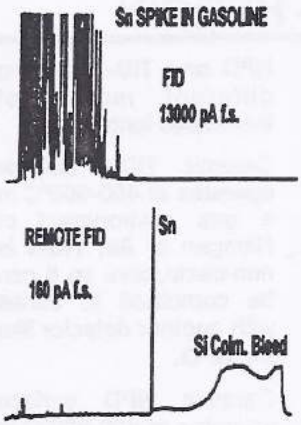


Volatile HALOGENATES detected with a sensitivity of 1 pg/sec, selectivity of 100,000:1 vs. hydrocarbons, and linear response exceeding a range of 10,000 in sample weight.

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

Sample analyzed:
640 pg each: 1=$CHCl_3$ 2=$CHCl_2Br$ 3=$CHClBr_2$ 4=$CHBr_3$
47,000 pg each: B=benzene T=toluene
2,500,000 pg: M=methanol Solvent: water

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



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

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

Sample:
12 ppm tetrabutyltin in gasoline

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

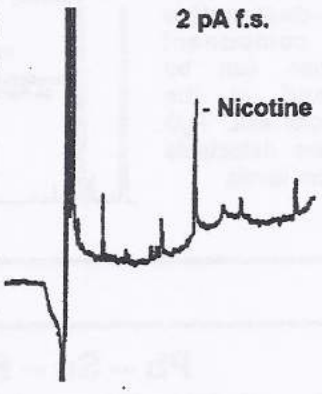
CHEMICAL DETECTION by DET

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

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

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

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

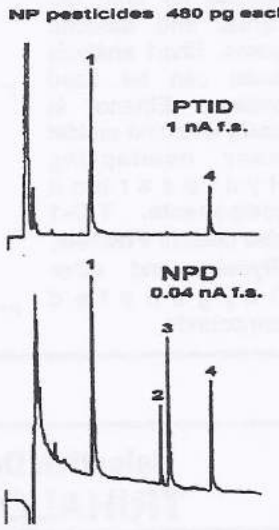


Selective Detection for GC
PHOSPHORUS COMPOUNDS
Very Big Signals with a New PTID

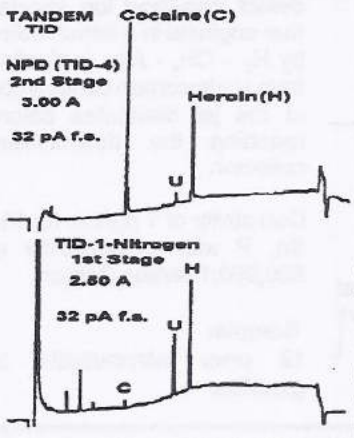
Pesticide Sample:
1-Mevinphos (P)
2-Trifluralin (N)
3-Simazine (N)
4-Methyl Parathion (NP)

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

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



Tandem Thermionic Detection for GC
COCAINE - HEROIN



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

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

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

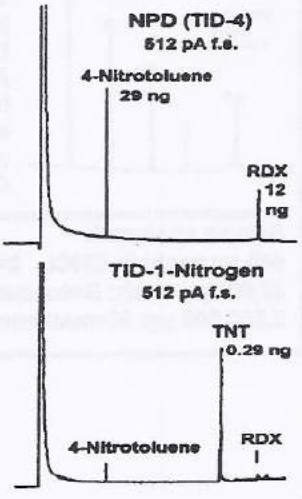
Sample analyzed: NPD detects both Cocaine (C) and Heroin (H). TID-1 detects Heroin and Heroin impurity (U) Tandem combination gives simultaneous TID-1 and NPD signals for each sample injection.

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

Unique **TID-1** surface ionization provides better selectivity than ECD and NPD, and needs only Air or N_2 as the detector gas with no requirement for high purity.

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

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



DETector Engineering & Technology, inc.

Journal of Chromatographic Science, Vol. 24, February 1986

Invited Paper

Recent Advances in Thermionic Ionization Detection for Gas Chromatography

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Abstract

Thermionic ionization detectors are most widely used for the specific detection of nitrogen-phosphorus compounds in gas chromatography. The operating mechanism of these detectors is a surface ionization process in which the key parameters are the work function of the thermionic emission surface, the temperature of the thermionic surface, and the composition of the gas environment in the immediate vicinity of the thermionic surface. By systematic variations of each of these three key parameters, the technique of thermionic ionization detection has been greatly expanded to encompass a number of different modes of response, all of which use similar detector hardware and electronic components.

Introduction

Thermionic ionization detectors (TID) are best known in gas chromatography (GC) for their application to the specific detection of nitrogen (N) or phosphorus (P) compounds. All modern TIDs are essentially derivations of a basic design first described by Kolb and Bischoff (1) in 1974. The main component in this type of detector is an electrically-heated thermionic emission source in the form of a bead or cylinder which is usually composed of an alkali-metal compound impregnating a glass or ceramic matrix. In the TID, the thermionic source is positioned so that sample compounds may impinge upon its surface, and any ionization produced is measured by an adjacent collector electrode. Kolb and Bischoff were the first to report that a thermionic source comprised of a Rb-silicate glass bead produced very specific NP responses when the bead was operated at high temperatures in a gas environment of dilute H_2 in air.

Since the original work of Kolb and Bischoff, there have been continuing developments in NP detectors, with much emphasis on improved methods of construction and composition of the thermionic emission sources. The most important development, however, has been the recognition in recent years that the operation mechanism of a TID is a surface ionization process (2) rather than the gas phase ionization process originally proposed by Kolb et al. (1,3). Once it was clear that a surface ionization process was operative, it was possible to identify three key operating parameters which control the ionization produced. These parameters are: the electronic work function of the thermionic emission surface which is determined by the chemical composition of the surface; the temperature of the thermionic surface; and the chemical composition of the gas environment immediately surrounding the thermionic surface.

The identification of these parameters has led to a clearer understanding of the complex chemistry active in NP detection, and has provided an important guide for expanding the applications of thermionic ionization techniques. Through systematic variations in each of the key parameters, many different modes of detector response have been achieved (3-6). Hence, the technique of thermionic ionization detection now correctly refers to a number of GC detector responses which are related through the use of many common hardware and electronic components. This article reviews the present state of development of the members of this unique group of detectors.

Types of Thermionic Emission Sources

All commercially available TIDs use thermionic emission sources formed according to one of the following four general methods:

(A) homogeneous alkali-glass bead formed on a loop of bare platinum wire (1);
(B) alkali salt activator coated on a ceramic cylinder core containing an embedded heater coil (7);
(C) homogeneous alkali-ceramic bead formed on a coil of nichrome heater wire (2,8,9);
(D) multiple layers of cylindrically-shaped ceramic coatings, with a non-corrosive, electrically-conducting sub-layer of Ni-ceramic completely covering a loop of nichrome wire, and a surface layer comprised of alkali and/or other additives in a ceramic matrix (5,10).

Thermionic sources representing all four categories cited above have been used in NP detectors available from different manufacturers. Generally, those sources formed from ceramic materials provide greater flexibility for varying the chemical composition of the source. This is because the ceramic compositions are formulated and coated from a slurry at room temperature (9), whereas the glass compositions are formed in

a process that proceeds through a molten glass state (11).

The detailed chemical compositions of thermionic emission sources are usually regarded as confidential proprietary information by the manufacturer. Since the first alkali-glass bead reported by Kolb and Bischoff used Rb as the alkali compound, there existed for many years a belief that Rb was an essential component for optimum NP responses. However, in recent years, NP detectors with state-of-the-art performance specifications have been reported in which Cs rather than Rb is used as the alkali component (5). Also, another recent report (12) has described an NP detector which uses a LaB_6/SiO_2 bead and no alkali additive. In accordance with a mechanism of surface ionization prevailing in the TID, the most important characteristic of the thermionic emission surface is its electronic work function (i.e., the amount of energy required to emit a unit of electrical charge from the surface). Alkali-metal compounds have been especially successful additives because they lower the work function of the glass or ceramic matrix, thereby facilitating the emission of charged particles from the heated thermionic surface. The mathematical relationships between work function, surface temperature, and thermionic emission current have been discussed (2,12).

The development of multiple-layered, ceramic-coated thermionic emission sources has allowed examination of coatings of many different chemical compositions without the risk of materials in the surface layer corroding the heater wire. In the search for expanded applications for thermionic ionization techniques, the basic task is to define a specific match of a thermionic source type with an operating gas environment and a range of operating source temperatures. To date, three different chemical compositions of thermionic sources have been shown (5,6) to have useful applications in differing modes of thermionic detection. These source compositions are shown in Table I. Data obtained using these three types of thermionic emission sources are presented in the following sections.

Modes of Response

Schematic illustrations of four different versions of thermionic ionization detection equipment are shown in Figures 1 through 4. Common components in each version are as follows:

(A) an electrically-heated, thermionic/catalytic source constructed of multiple layers of ceramic coatings;
(B) a cylindrical collector electrode surrounding the cylindrically shaped thermionic source;
(C) a source power supply that provides heating current to heat the source to typical temperatures of 400° to 800°C, and a bias voltage to polarize the source structure at a negative voltage with respect to the collector;
(D) an electrometer that measures negative ionization currents arriving at the collector electrode.

The TID hardware usually mounts onto an FID-type detector base that is resident on a GC, so that two different detector gases may be supplied in addition to the GC effluent. Therefore, changes in the modes of detector response that correspond to the schematics of Figures 1 through 4 are accomplished by changes in the type of thermionic source, changes in the composition of gases supplied to the detector, or by changes in the operating temperature of the thermionic surface.

Most of the TIDs available commercially function by the collection of negative ionization rather than positive ionization. In the discussion that follows, it will be shown that the concepts of negative ion chemistry provide a logical pattern for correlating the responses of the different modes of thermionic detection.

TID-1-N_2: Nitro/electronegative specific response

The simplest mode of thermionic detection is represented by the schematic in Figure 1. In this mode, the low work function thermionic source designated by the TID-1 nomenclature is operated in a detector gas environment of N_2. Because the detector gases are inert, sample compounds interact directly with the TID-1 surface, which is typically heated to temperatures in the range of 400° to 600°C. The ionization process in this case is direct transfer of negative charge from the TID-1 surface to

Table I. Thermionic Source Surface Layers

Source type*	Additive	Work function
TID-1	High concentration Cs	Low
TID-2	Low concentration Cs/Sr	Medium
CFID	Nickel	High

*Nomenclature adopted from Patterson (5).

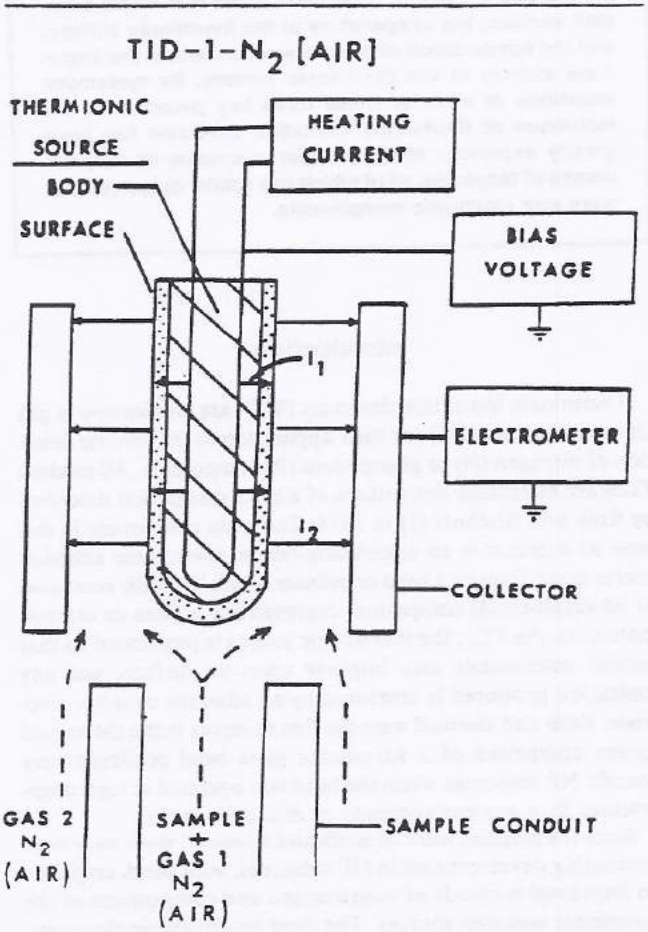

Figure 1. Schematic illustration of the detection configuration for the thermionic ionization modes TID-1-N_2 and TID-1-air. Detector gas 1=FID-H_2 inlet line; detector gas 2=FID-air inlet line.

the sample molecule. Consequently, this mode of response provides exceptionally high specificity and sensitivity to many compounds containing the nitro (NO_2) functional group (5,13), as well as to certain other electronegative compounds (e.g., pentachlorophenol, diazepam, and methaqualone). This mode of detection is very sensitive to the detailed electronegativity of the sample's molecular structure, as has been illustrated by the observation that a larger signal is obtained for the 2,4-isomer of dinitrotoluene in comparison to the 2,6-isomer (5). The TID-1-N_2 mode is superior to a conventional NP detector or an electron capture detector (ECD) for detection of trace level nitro-compounds such as nitro-PAH, nitro-explosives, nitro-pesticides (e.g., parathion, methyl parathion), nitro-drugs, nitro-derivatives. For many nitro-compounds, the specificity vs. hydrocarbons is an astonishing 10^6, and detectivity is in the 0.1- to 1.0-pg range (5).

TID-1-air: Halogen/nitro specific response

When the TID-1 thermionic source is operated in an oxygen-containing gas environment rather than one of N_2, specific responses to halogenated compounds are enhanced while responses to nitro-compounds are decreased somewhat (14). The TID-1-air mode of detection is generally not as sensitive as an ECD or Hall detector for chlorinated compounds, but it provides halogen specificity at higher concentrations where ECD and Hall are saturated. Typical specificity is 10^4 and detectivity is 0.1 to 1.0 ng. This is an especially simple mode of detection for ethylene dibromide (EDB) in the headspace vapors of food products.

TID-2-H_2/air: Nitrogen/phosphorus specific response

The schematic illustration of Figure 2 represents the situation that prevails in an NP detection mode. For this mode, H_2 and air gases are supplied to the detector, and a thermionic source of moderate work function (i.e., TID-2) is operated hot enough (600° to 800°C) to cause thermal/chemical decomposition of the H_2 and O_2 gases. A critical parameter in this NP mode is the restriction of the H_2 to low flows (e.g., 3 to 6 ml/min) which are not sufficient to maintain a self-sustaining flame at the sample conduit (i.e., jet structure) depicted in Figure 2. Instead, a flame-like gaseous boundary layer is created in the immediate vicinity of the hot thermionic source. Since this boundary layer is very reactive chemically, sample compounds are decomposed by the active gas phase chemistry, and electronegative products of decomposition are selectively ionized by surface ionization on the thermionic source. N or P compounds are ionized with especially high specificity by this process. An essential condition for the onset of NP detection is that the thermionic source must be hot enough to "ignite" the boundary layer chemistry. Under these conditions, a thermionic source of moderate work function provides the optimum compromise of sample response signal vs. detector background signal. A low work function therm-

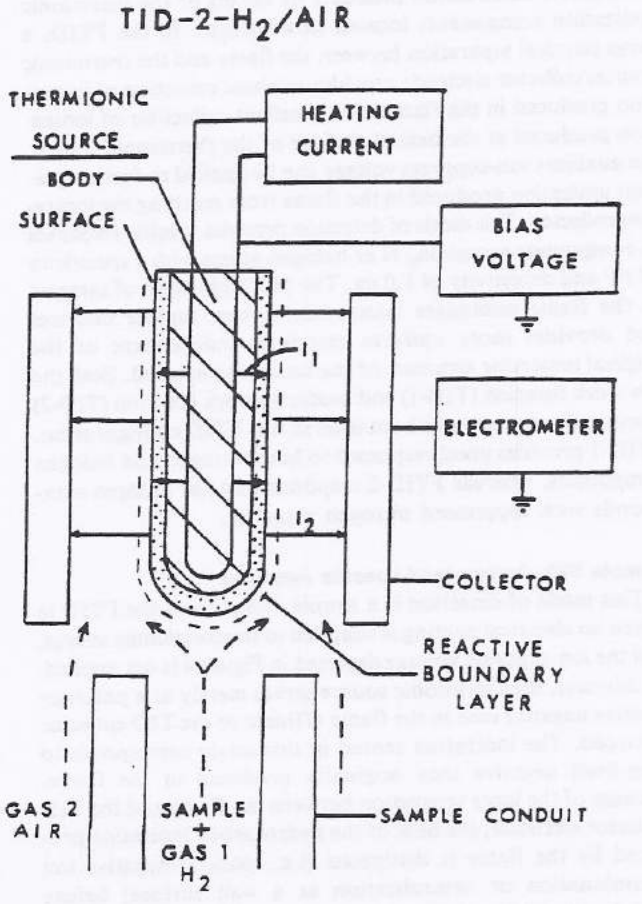

Figure 2. Schematic illustration of the detection configuration for the TID-2-H_2/air or NP mode of thermionic ionization.

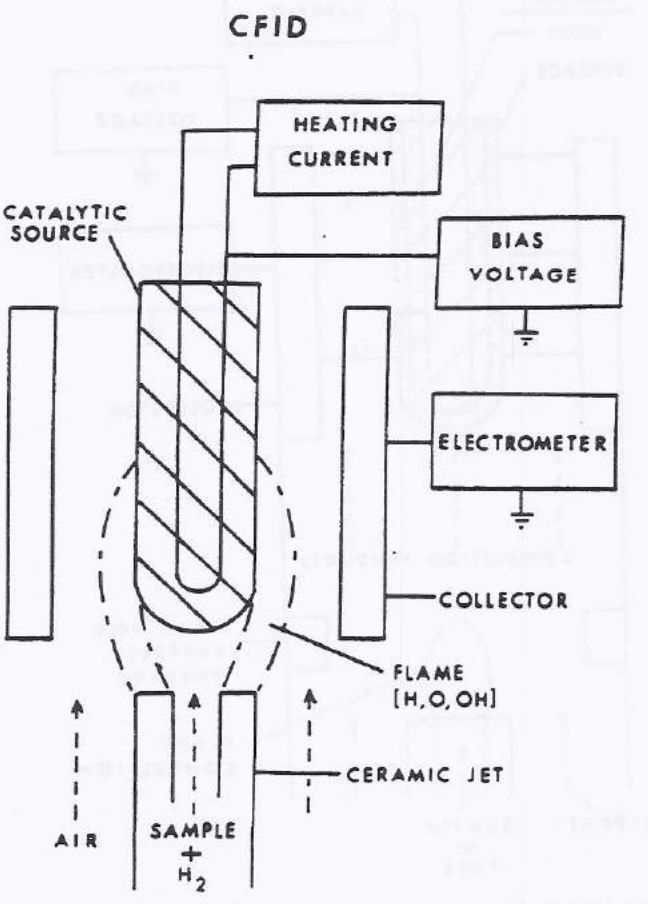

Figure 3. Schematic illustration of the detection configuration for the catalytic flame ionization detector (CFID) mode of response.

ionic source (e.g., TID-1) operated under these conditions would produce an overwhelmingly large background signal. Conversely, a high work function thermionic source (e.g., catalytic flame ionization detection) operated under these conditions would produce smaller NP signals and less specificity than the moderate work function thermionic source. Typical performance specifications for NP detectors are detectivities in the range of 1 to 10 pg, and specificity with respect to hydrocarbons in the range of 10^4 to 10^5.

CFID: Universal response to all organics

Figure 3 depicts a mode of response achieved when the H_2 flow to the detector is sufficient to produce a true self-sustaining flame burning at the jet structure. This mode of operation has been designated (5) catalytic flame ionization detection (CFID) because of its close similarity to a conventional FID. The CFID is essentially an FID which has been modified by inserting an electrically-heated catalytic source comprised of a Ni-impregnated ceramic into the center of the active flame region. In this detection mode, the catalytic source structure serves the threefold function of flame ignitor, flame polarizer, and catalytic combustion modifier/thermionic surface ionizer. In the CFID, two types of ionization processes are active: gas phase ionization processes identical to those which occur in a conventional FID, and surface ionization processes at the catalytic source structure which especially enhance the ionization efficiency of many heteroatom compounds (especially halogenated and phosphorus compounds).

Like a conventional FID, the magnitude of the gas phase ionization is determined primarily by the magnitudes of H_2 and air flows and the size of the jet orifice, so that additional electrical heating of the CFID source has little effect on the gas phase ionization. However, the magnitudes of ionization produced by surface processes at the CFID source is strongly dependent on the electrical heating of the source. Hence, in many cases, response factors for heteroatom compounds can be enhanced to be comparable to hydrocarbons by a judicious selection of source heating current. For this CFID mode of detection, the thermionic/catalytic source of highest work function is most suitable because the flame heat would otherwise cause an excessive thermionic emission background signal. The CFID provides detectivities in the 10- to 100-pg range for most organic compounds.

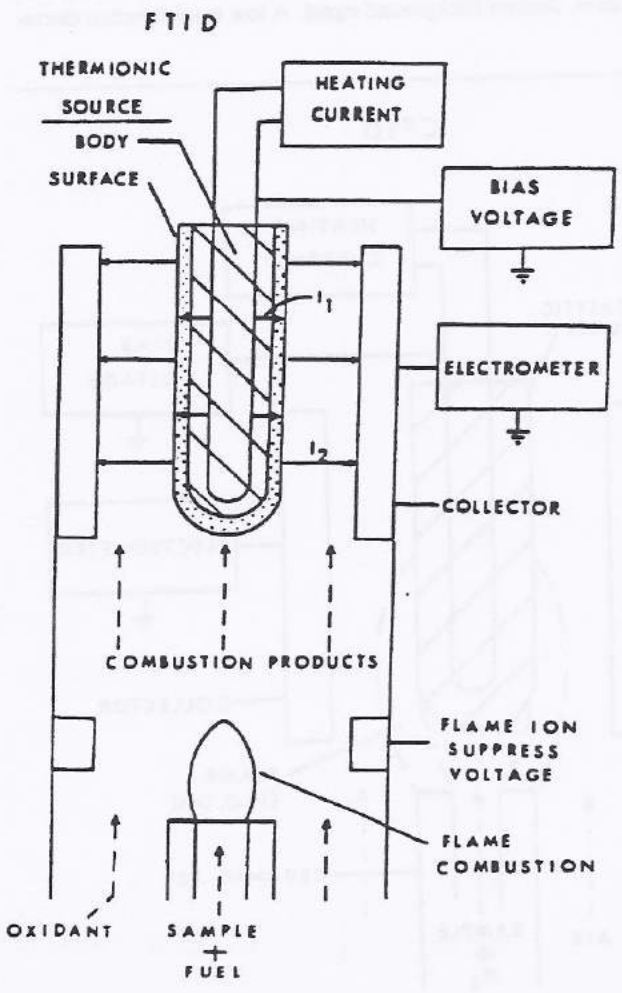

Figure 4. Schematic illustration of the detection configuration for the flame thermionic ionization detector (FTID) mode of response.

FTID: Nitrogen/halogen specific response

Figure 4 depicts a further detection mode in which the thermionic source and collector electrode structure are positioned well downstream of the active region of a self-sustaining flame. The basic concept (6) of this flame thermionic ionization detection (FTID) mode is to burn sample compounds in a self-sustaining H_2/air flame at a flame jet, and to selectively re-ionize electronegative combustion products by means of the thermionic ionization components located downstream. In the FTID, a large physical separation between the flame and the thermionic source/collector electrode provides minimal collection of ionization produced in the flame, but excellent collection of ionization produced at the heated surface of the thermionic source. An auxiliary ion-suppress voltage can be applied to further prevent ionization produced in the flame from reaching the ionization collector. This mode of detection provides specific responses to compounds containing N or halogen atoms with a specificity of 10^3 and detectivity of 1.0 ng. The precombustion of samples in the flame minimizes interferences from sample matrices and provides more uniform responses independent of the original molecular structure of the sample compound. Both the low work function (TID-1) and moderate work function (TID-2) thermionic sources have been used in this FTID configuration. FTID-1 provides good responses to both nitrogen and halogen compounds, whereas FTID-2 responds best for halogen compounds with suppressed nitrogen response.

Remote FID: Organo-lead specific response

This mode of detection is a simple variation of the FTID in which no electrical heating is supplied to the thermionic source, and the ion-suppress voltage depicted in Figure 4 is not applied. In this case, the thermionic source serves merely as a polarizer to drive negative ions in the flame effluent to the TID collector electrode. The ionization sensed in this mode corresponds to long-lived negative ions originally produced in the flame. Because of the large separation between the flame and the TID collector electrode, the bulk of the hydrocarbon ionization produced by the flame is dissipated (i.e., positive-negative ion recombination or neutralization at a wall surface) before reaching the TID collector. However, certain heteroatom compounds appear to combust to negative ion products which are especially stable and long-lived. The outstanding demonstration

of this effect is the specific detection of lead compounds in gasoline. Since the thermionic source is not electrically-heated and is not used as a source of surface ionization, any type of thermionic source may be employed in this mode.

Experimental

The applications data presented in this report were obtained with equipment previously described (5,6,14). All the data were obtained using a Model 3740 gas chromatograph (Varian Associates) equipped with either a TID/CFID detector assembly (TID-1-N_2, TID-1-air, TID-2-H_2/air, and CFID modes) or an FTID/TID detector assembly (FTID and remote FID modes) (Detector Engineering and Technology). The TID-1, TID-2, and CFID thermionic emission sources were also manufactured by Detector Engineering and Technology, as was the Model 4000 detector current supply. Negative ionization signals from the detector were measured using the differential electrometer (Varian) in a negative polarity configuration.

The detector assemblies mounted onto the FID-base on the GC, so that different detector gas environments were implemented by plumbing in the appropriate gas through the two gas lines that normally supply H_2 and air to an FID. Typical gas flows supplied for different modes of detection are detailed in Table II.

All the data presented in this report were obtained using glass columns, 6 ft × ¼ in. × 2 mm i.d., packed with either 3% SP-2250, 3% SP-2100, or 1% SP-1240 DA on 100/120 Supelcoport (Supelco) or 80/100 Chromosorb 102. The GC carrier gas in all cases was N_2 at a flow rate of 30 ml/min. For complex samples like gasoline, the chromatographic separation was intentionally very poor in order to produce a challenging detector environment to demonstrate specificity of response in the simultaneous presence of many overlapping compounds.

Sample mixtures that were analyzed included a TSD test sample (ng levels of azobenzene, methyl parathion, and malathion) (Varian); Base-Neutral 1 sample (Supelco); phenol mix (Supelco); DCMA PCB mixture (Supelco); and nitroaromatic mixture (Supelco) diluted in reagent-grade benzene. Gasoline, cologne, and diesel fuel samples were analyzed by direct injection of 0.5 to 1.0 µl amounts onto the column. Other samples chromatographed were a 1% v/v each mixture of acetone and carbon tetrachloride in water; and an 11% v/v each mixture of methylene chloride, n-C_6, benzene, i-C_8, toluene, n-C_8, *p*-Xylene, n-C_{10}, and n-C_{12}.

Table II. Typical Gas Flows Supplied for Each Mode of Detection

Mode	Gas 1	Gas 2
TID-1-N_2	10 ml/min N_2	60 ml/min N_2
TID-1-air	10 ml/min air	60 ml/min air
TID-2-H_2/air	3 ml/min H_2	60 ml/min air
CFID	25 ml/min H_2	200 ml/min air
FTID	20 ml/min H_2	200 ml/min air
Remote FID	20 ml/min H_2	200 ml/min air

Applications*

Specially formulated test samples are often employed to demonstrate the specificity and sensitivity of NP detectors. The data in Figure 5 correspond to such a test sample comprised of 2.2 ng each of azobenzene (N) and methyl parathion (N,P), 4.4 ng of malathion (P), and 4400 ng of n-C_{17} in a solvent of iso-octane. The data illustrate clearly the substantial differences in response between two different modes of thermionic detection, and the very high specificity of the TID-1-N_2 mode for sensing the NO_2 group in methyl parathion. The ionization signals in both chromatograms are very large, indicating detectivities in the pg and sub-pg range.

Figure 6 shows another comparison of the TID-1-N_2 mode vs. the NP mode of detection for a sample consisting of 15 pg amounts of the 2,4- and 2,6-isomers of dinitrotoluene in a relatively impure, reagent grade benzene solvent. For many nitro-compounds, the TID-1-N_2 mode provides substantial improvements in specificity and sensitivity in comparison to an NP mode. However, the NP mode (i.e., TID-2-H_2/air) provides more universal detection for all N-compounds. In Figure 6, the differing magnitudes of TID-1-N_2 signals for the two dinitrotoluene isomers also demonstrate that the TID-1-N_2 signals are very sensitive to the detailed electronegative character of the sample's molecular structure.

Figure 7 shows the differing responses of six modes of thermionic detection in the analysis of base neutral compounds of concern as water pollutants. The CFID provides universal response to all compounds in this sample with a relatively uniform sensitivity of 0.01 coul/gC. The TID-1-N_2 mode of opera-

*Data presented are from References 6 and 14.

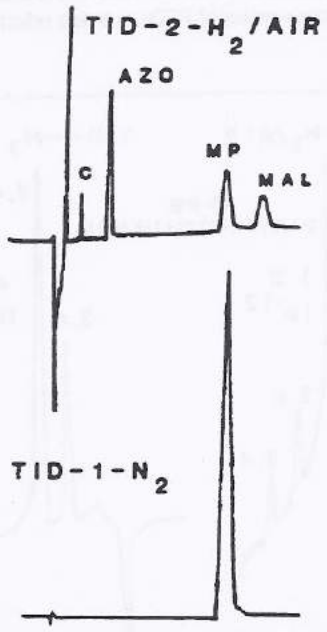

Figure 5. Chromatograms showing two modes of response to a test sample containing a large concentration of a hydrocarbon (C) and trace levels of azobenzene (AZO), methyl parathion (MP), and malathion (MAL). Both chromatograms were recorded at the same sensitivity of 128 × 10^{-11} amps/mV. Column: SP-2250, isothermal at 210°C.

tion provides very specific responses only to a nitro-compound (2,6-dinitrotoluene) and a chlorinated compound (3,3'-dichlorobenzidene). The lack of significant TID-1-N_2 response to nitrobenzene, bis(2-chloroethyl)ether, bis(2-chloroisopropyl)ether, or 4-bromophenyl-phenyl-ether demonstrates that the TID-1-N_2 response depends on how the electronegative functionalities are bound up in the molecular structure of the sample compound. When the detector gas environment of the TID-1 source is changed from N_2 to O_2, the TID-1-O_2 mode provides enhanced relative responses to the chlorinated compounds and a diminished relative response to the nitro-compound. The TID-1-O_2 mode continues to provide good discrimination in favor of chlorinated compounds with respect to hydrocarbons, while exhibiting some low level responses to phthalate compounds. The TID-2-H_2/air mode responds to all the N-compounds, with some small interferences from chlorinated compounds. The FTID-1 mode uses a TID-1 source and provides responses to all the nitrogen and halogen compounds in the sample. The FTID-2 mode uses a TID-2 source which produces responses to the halogenated compounds but suppressed responses to nitrogen compounds in comparison to FTID-1. This set of six chromatograms provides a good illustration of how the detector response can be varied through simple changes in the composition of the detector gas environment.

Figure 8 shows chromatograms of a sample mixture consisting of 75 ng each of 2-chlorophenol, 2-nitrophenol, phenol, 2,4-dimethlyphenol, and 2,4 dichlorophenol; 225 ng each of 2,4,6-trichlorophenol, and 2,4-dinitrophenol; and 375 ng each of 4-chloro-*m*-cresol, 4,6-dinitro-*o*-cresol, pentachlorophenol, and 4-nitrophenol. The CFID provides a relatively uniform response of 0.004 coul/gC for all these compounds. (Note: The flame tip orifice for these CFID data was 0.062 in. instead of 0.031 in., which is normally used. The smaller orifice usually provides improved sensitivities of approximately 0.01 coul/gC.)

The CFID data provide a good illustration of a principal difference in the responses of a CFID vs. a conventional FID. It is well known that conventional FIDs provide relatively uniform response to many hydrocarbon compounds. However, when heteroatoms, such as O, Cl, or P, are present in the sample compounds, the FID response is frequently significantly lower than its response to hydrocarbons. In contrast, the CFID appears to yield more uniform response to all organic compounds ir-

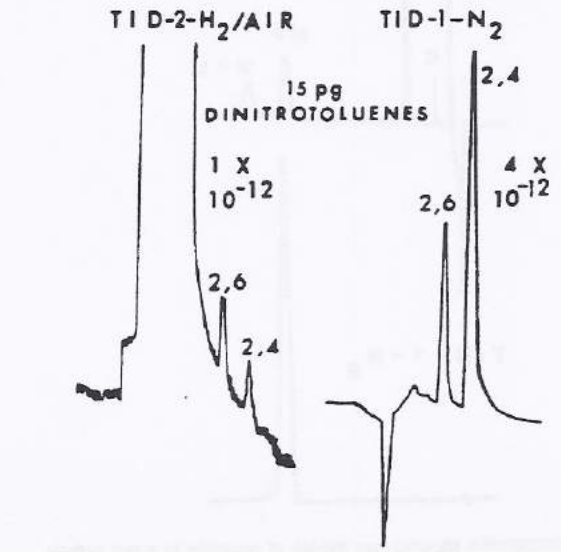

Figure 6. Chromatograms comparing the responses of the TID-1-N_2 mode and the TID-2-H_2/air (NP) mode to traces of dinitrotoluenes. Column: SP-2100, 160° to 200°C at 10°/min.

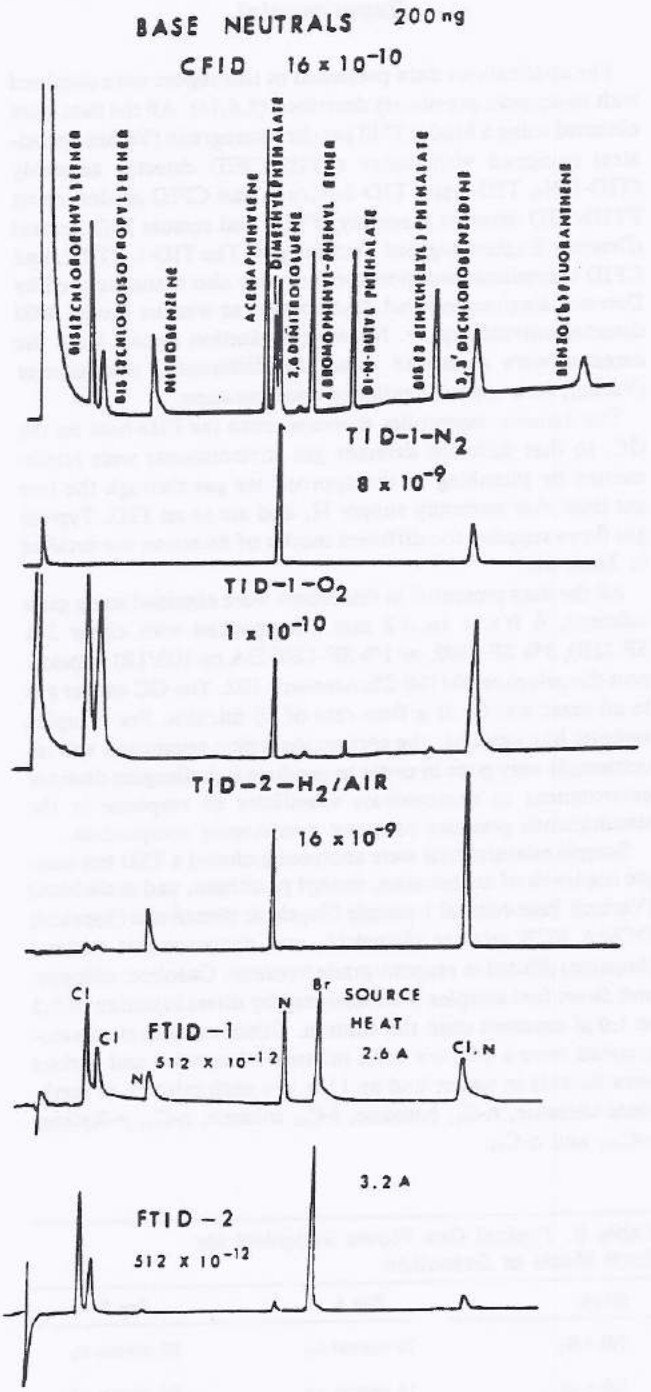

Figure 7. Chromatograms showing six different modes of detector response to a sample of base neutral compounds. In increasing order of retention time, the components of the sample are bis(2-chloroethyl)ether; bis(2-chloroisopropyl)ether; nitrobenzene; acenaphthylene; dimethylphthalate; 2,6-dinitrotoluene; 4-bromophenyl-phenyl ether; di-*n*-butylphthalate; bis(2-ethylhexyl)phthalate; 3,3'-dichlorobenzidine; and benzo(*b*)fluoranthene. Column: SP-2250; 100°C, held for 4 min, then 100° to 270°C at 16°/min.

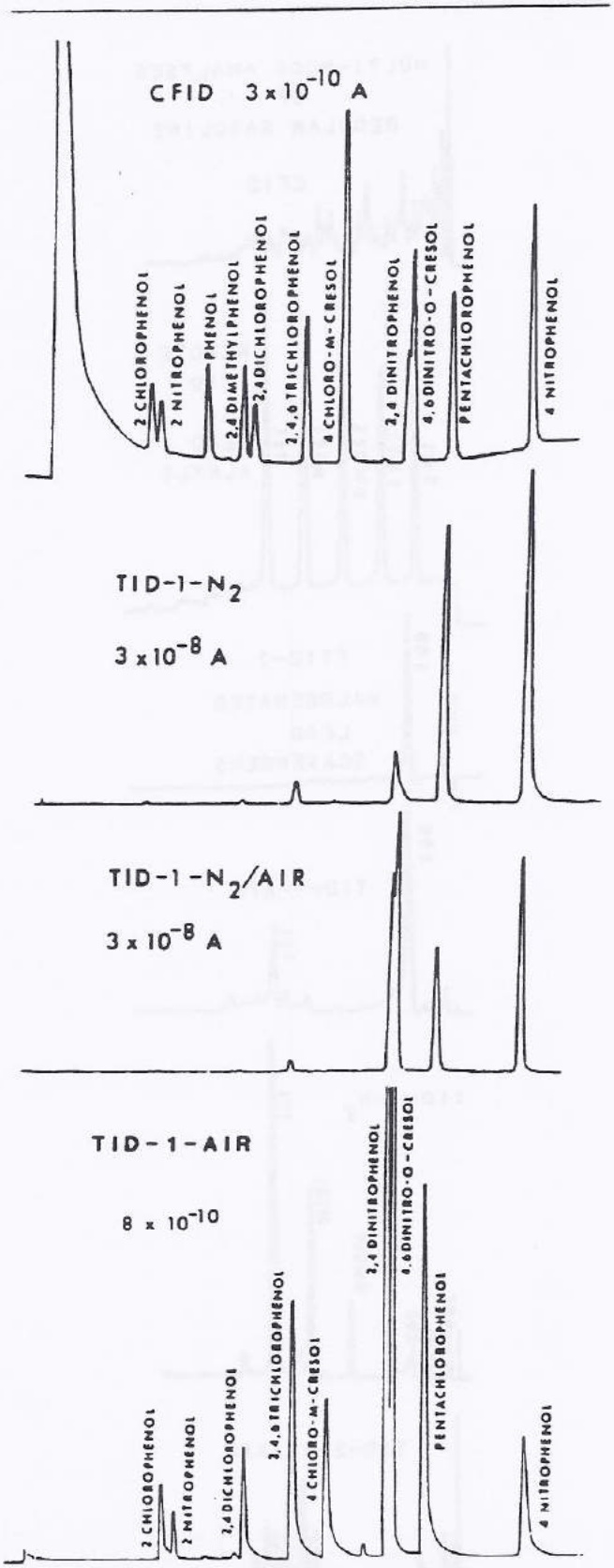

Figure 8. Chromatograms showing different modes of detector response to phenol sample containing 2-chlorophenol; 2-nitrophenol; phenol; 2,4-dimethylphenol; 2,4-dichlorophenol; 2,4,6-trichlorophenol; 4-chloro-*m*-cresol; 2,4-dinitrophenol; 4,6-dinitro-*o*-cresol; pentachlorophenol; and 4-nitrophenol in order of increasing retention time. Column: SP-1240, 100° to 210°C at 8°/min.

respective of whether they are hydrocarbon or heteroatom compounds. This is the result of the additional ionization process that occurs at the surface of the catalytic CFID source.

As anticipated, the TID-1-N_2 mode in Figure 8 provides high specificity and sensitivity to certain nitro- and polychlorinated-phenols. The responses to pentachlorophenol and 4-nitrophenol are more than 100 times larger than the CFID responses to these compounds. Comparing the responses of 4-nitrophenol and 2-nitrophenol again illustrates a significantly greater TID-1-N_2 response for the isomer with the nitro group located at the 4-position in the molecule.

In the third chromatogram in Figure 8, the low work function thermionic source (TID-1) was operated in a detector gas environment comprised of approximately equal flows of N_2 and air. This illustrates that the composition of the gas environment is an additional parameter which can be used to suppress the response to certain compounds while enhancing the response to others.

The bottom chromatogram in Figure 8 shows the analysis of the phenol sample for the case where both detector gases 1 and 2 are air. Responses are now obtained for all the chlorinated and nitro-phenols, with the dinitro-compounds continuing to give the dominant responses. The responses to 2-nitrophenol and 4-nitrophenol are now comparable, in contrast to the TID-1-N_2 response in which there was significant preference for the nitro group in the 4-location vs. the 2-location.

Figure 9 demonstrates the high specificity for lead alkyls in

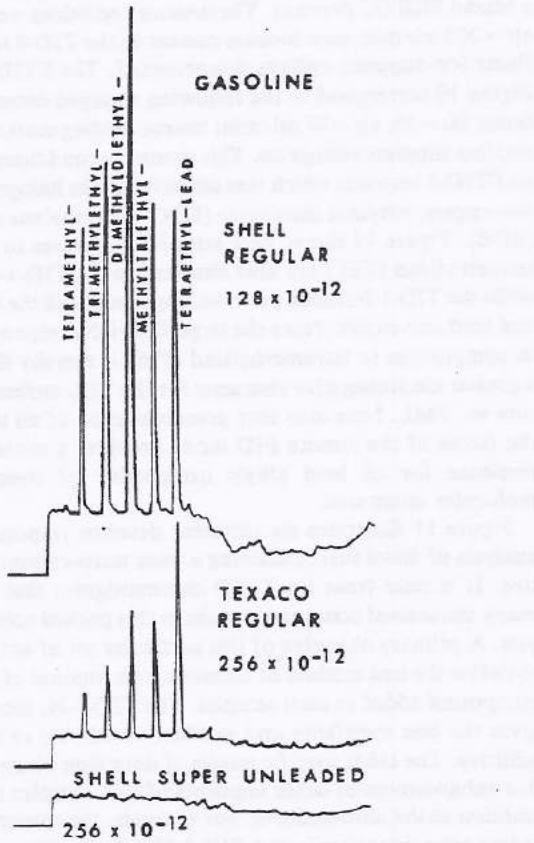

Figure 9. Chromatograms illustrating the specific detection of lead alkyls in gasoline using the remote FID. Data correspond to samples randomly obtained from local gasoline stations. Column: SP-2100, 50° to 230°C at 15°/min.

gasoline provided by the remote FID mode of detection. Previously, GC/atomic absorption spectroscopy (AAS) instrumentation has been described for the specific detection of lead alkyls in gasoline or other samples (15). By comparison with AAS, the remote FID provides excellent sensitivity and specificity, and is considerably simpler and less expensive to operate. The data shown in Figure 9 were obtained by direct injections of 0.8-μl amounts of the gasolines onto a temperature-programmed, packed column.

The remote FID mode of detection has some configuration similarities to a hydrogen atmosphere flame ionization detector (HAFID), which has also been reported to provide specific detection of lead alkyls in gasoline (16). Both the remote FID and HAFID use a collector electrode well removed from the flame jet structure. However, the signals in the remote FID are largest with an oxygen-rich flame, whereas the HAFID requires a hydrogen-rich atmosphere doped with silane.

Gasoline is a readily available, complex mixture of organic compounds which is especially well suited to demonstrating the different responses obtained in the family of thermionic detection modes. Figure 10 shows multiple modes of analysis of a sample of regular gasoline. The chromatographic separation was performed on a packed column to purposely create the demanding situation in which many overlapping compounds are present in the detector volume at the same time. This situation is shown to be the case by the CFID response. When the same gasoline sample is chromatographed using the remote FID mode, selective responses are obtained only for the five lead alkyls. The remote FID data in Figure 10 were obtained with an FTID detector assembly and a TID-2 source mounted on a Model 3700 GC (Varian). The detector conditions were $H_2 = 30$, air = 200 ml/min; zero heating current to the TID-2 source; and flame ion-suppress voltage disconnected. The FTID-2 data in Figure 10 correspond to the following changed detector conditions; $H_2 = 30$, air = 80 ml/min; source heating current = 3.2 A; and ion-suppress voltage on. This change in conditions produced an FTID-2 response which was selective for the halogenated lead scavengers, ethylene dichloride (EDC) and ethylene dibromide (EDB). Figure 10 shows that selective responses to EDB and tetraethyllead (TEL) are also obtained in the TID-1-air mode, while the TID-1-N_2 mode provides responses to all the lead alkyls and lead scavengers. Note the large TID-1-N_2 response to TEL in comparison to tetramethyllead (TML), thereby illustrating a greater electronegative character for the TEL molecular structure vs. TML. Note also that precombustion of all samples in the flame of the remote FID mode provides a more uniform response for all lead alkyls irrespective of their original molecular structures.

Figure 11 illustrates six different detector responses in the analysis of diesel fuel containing a trace nitro-compound additive. It is clear from the CFID chromatogram that there are many unresolved component peaks in this packed column analysis. A primary objective of this particular set of analyses was to define the best method of measuring the amount of the nitro-compound added to such samples. The TID-1-N_2 mode clearly gives the best specificity and excellent sensitivity to the nitro-additive. The other specific modes of detection illustrate selective enhancement of other segments of this complex sample in addition to the nitro-additive. For example, the cluster of peaks at late retention times in the TID-2-H_2/air chromatogram undoubtedly corresponds to other N-compounds in the sample. The two FTID-1 chromatograms illustrate that the H_2-air mixture ratio is a further means of significantly altering the FTID re-

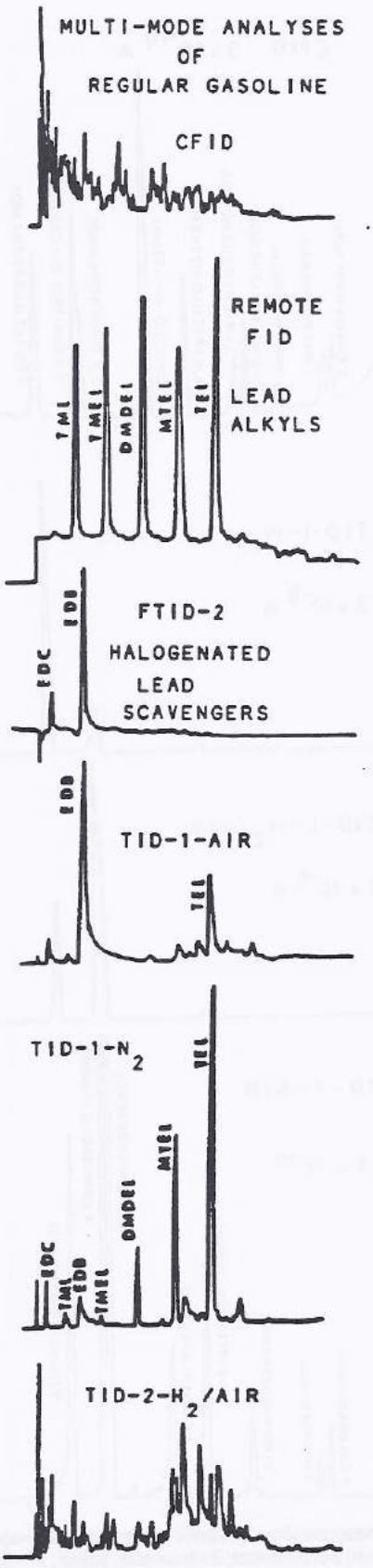

Figure 10. Chromatograms showing multiple modes of detector response to a sample of regular leaded gasoline. Column and program same as in Figure 9.

sponse. The mixture of 20 ml/min H_2, 100 ml/min air represents an oxygen-rich flame, while the mixture of 35 ml/min H_2, 70 ml/min air is a hydrogen-rich flame. Generally, stoichiometric or oxygen-rich flames are the most useful for the FTID.

Figure 12 shows multiple mode analyses of a commercial brand of cologne. These sets of chromatograms illustrate the advantageous use of the specific detection modes to enhance responses for trace fragrance components in colognes. The TID-1-N_2 mode is especially useful because it is non-destructive. Therefore, TID-1-N_2 emits exhaust gases that are characterized by distinctly different fragrances that change with time as the various segments of the chromatogram elute. For the cologne shown, as well as for other brands that have been examined, the set of chromatograms obtained from the different modes of thermionic detection provide a characteristic fingerprint that distinguishes one brand from another.

All the modes of detection described in this article measure negative ionization currents. The magnitudes of these currents are very dependent on the electronegative character of the chemical species adjacent to the heated thermionic source.

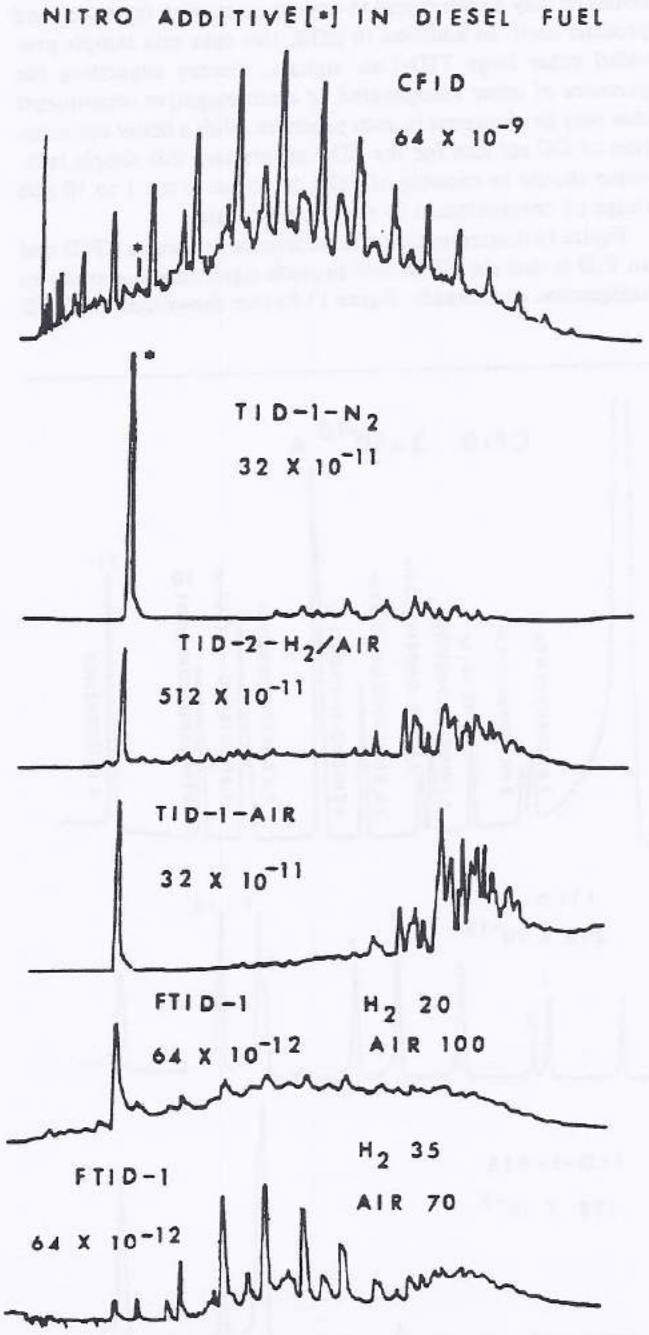

Figure 11. Chromatograms showing different detector responses to a sample of diesel fuel containing a trace nitro-compound additive identified by the astersk. Column SP-2100, 90° to 270°C at 10°/min.

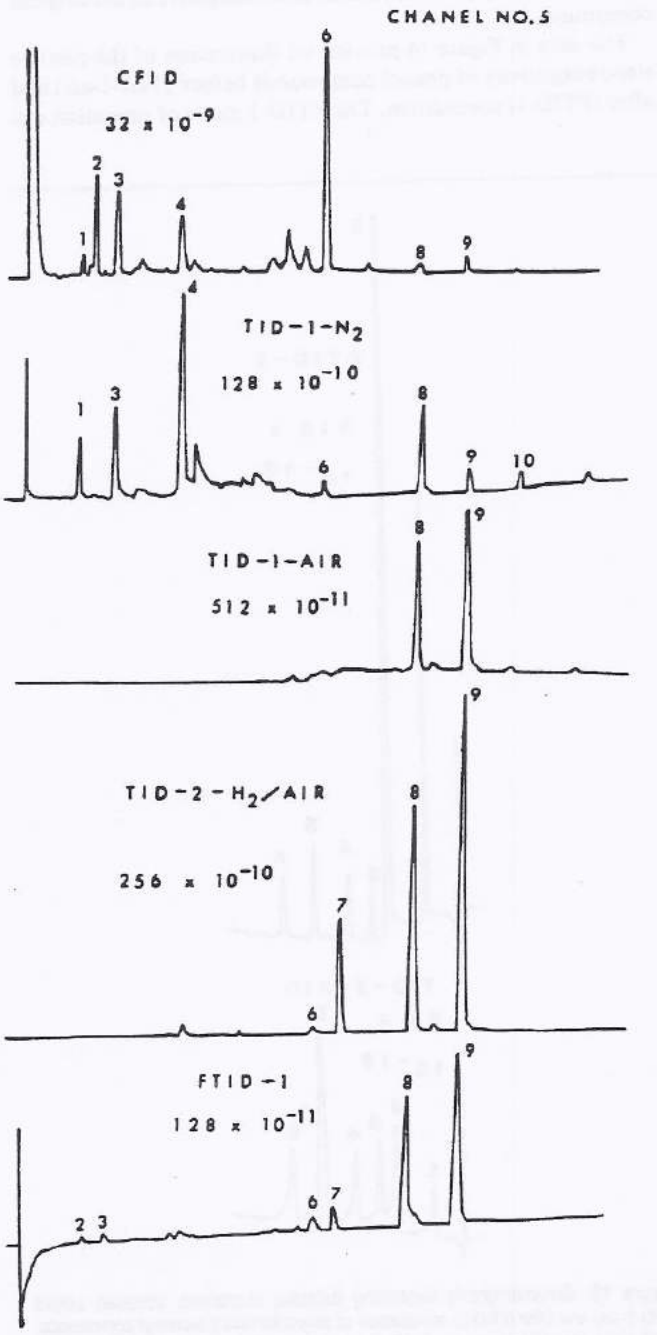

Figure 12. Chromatograms showing multiple modes of detector response to a brand of cologne randomly obtained. Column: SP-2100, 100° to 270°C at 16°/min.

Figure 13 illustrates how the response to a group of polychlorinated biphenyl (PCB) compounds changes with precombustion of the compounds. The data obtained with a TID-2 source in an air environment exhibit large differences in response between the different PCB compounds, similar to the known characteristics of an ECD. The FTID-2 data show that the precombustion of the PCBs yield negative ionization currents, which are larger in magnitude as well as more uniform per Cl atom. In this case of PCBs, precombustion improves the detectability by producing chemical species more electronegative than the original compounds. For other compound types, precombustion sometimes causes the opposite effect of producing combustion products which are not as electronegative as the original compound.

The data in Figure 14 provide an illustration of the relative electronegativity of phenol compounds before (TID-1-air) and after (FTID-1) combustion. The FTID-1 mode of operation can be easily converted to the TID-1-air mode by simply turning off the H_2 fuel to the FTID flame. Figure 14 shows that the FTID-1 mode produces a much more uniform response for all the chloro- and nitrophenols of this sample, but the absolute magnitudes of FTID response for the dinitrophenols, pentachlorophenol, and 4-nitrophenol are substantially lower than the TID-1-air response to these compounds.

Figure 15 illustrates the use of the TID-1-air mode to detect EDB in food products. A simple headspace technique was used for the data. One of the advantages of the TID-1-air mode is that it is insensitive to the large air component in the injection headspace vapors. Hence, all chromatographic peaks that are obtained may be attributed to vapors emanating from the food product itself. In addition to EDB, this cake mix sample provided other large TID-1-air signals, thereby suggesting the presence of other halogenated or electronegative constituents that may be of interest in such products. With a better optimization of GC column for the EDB separation, this simple technique should be capable of EDB detection in the 1 to 10 ppb range of concentration in the food product.

Figure 16 illustrates that one difference between a CFID and an FID is that the CFID will provide significant responses to halogenated compounds. Figure 17 further shows that the CFID

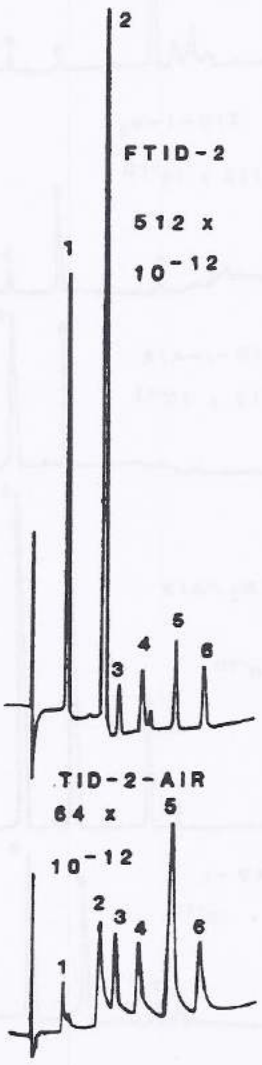

Figure 13. Chromatograms illustrating detector responses obtained before (TID-2-air) and after (FTID-2) combustion of polychlorinated biphenyl compounds. PCB peak identifications: 1=1000 ng 2-chloro-, 2=1000 ng 3,3'-dichloro-, 3=100 ng 2,4,5-trichloro-, 4=100 ng 2,2',4,4'-tetrachloro-, 5=100 ng 2,3',4,5', 6-pentachloro-, 6=100 ng 2,2',3,3',6,6'-hexachlorobiphenyl. Column: SP-2250, 200° to 270°C at 10°/min.

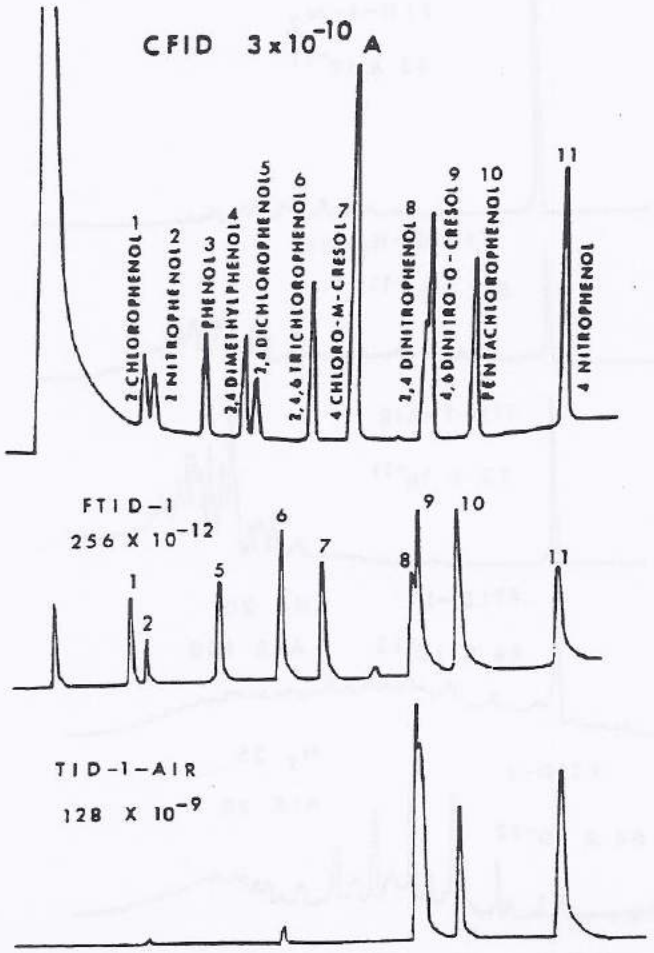

Figure 14. Chromatograms illustrating detector responses obtained before (TID-1-air) and after (FTID-1) combustion of phenol compounds. Sample, column, and program were the same as in Figure 8.

response to halogenated compounds can be selectively enhanced by increasing the source heating current, while the CFID responses to hydrocarbons remain unchanged. Consequently, by judiciously adjusting the source heating current, the CFID response to halogenated and some other heteroatom compounds can be tuned to yield about the same response factor as obtained for hydrocarbons.

Summary

The preceding data have demonstrated that thermionic ionization techniques and equipment have applications in gas chromatography that go well beyond the usual NP detection, such as that of TID-2-H_2/air. To achieve the best possible signal-to-noise ratio and specificity for each mode of detection, the specific chemical composition of the thermionic emission source needs to be matched with the temperature and gas phase environment in which the source is operated.

From their extensive use in NP detection, thermionic ionization detectors are known to often exhibit decreasing sensitivity with increasing operating time as a result of depletion of the thermionic source activity. Consequently, the thermionic source usually needs to be replaced at periodic intervals. Of the different modes of detection described in this report, the NP mode is the most demanding with regard to the operating life of the thermionic source. Generally, in modes of detection (e.g., TID-1-N_2) where the operating temperature is lower and the gas environment is less reactive, the thermionic sources maintain their responses over longer periods of time. For all modes of detection, a practical guideline for achieving the longest possible source lifetime is to operate the thermionic source just hot enough to achieve the response required.

The developments of thermionic techniques in GC have also spawned applications of the technology in liquid chromatography detection (17), thin layer chromatography (18), and mass spectrometry ion sources (19). Since there remain to be studied many different combinations of thermionic source compositions, thermionic source temperatures, and gas environment compositions, it is probable that the technology will continue to evolve in coming years.

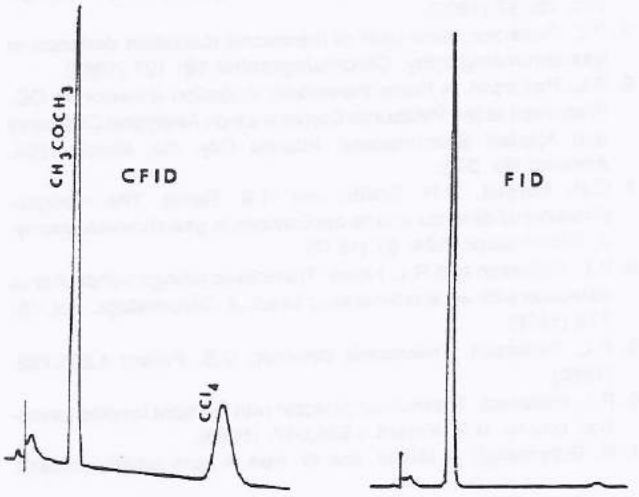

Figure 16. Comparison of CFID and FID responses to a water sample containing acetone and carbon tetrachloride. Column: Chromosorb 102, isothermal 120°C.

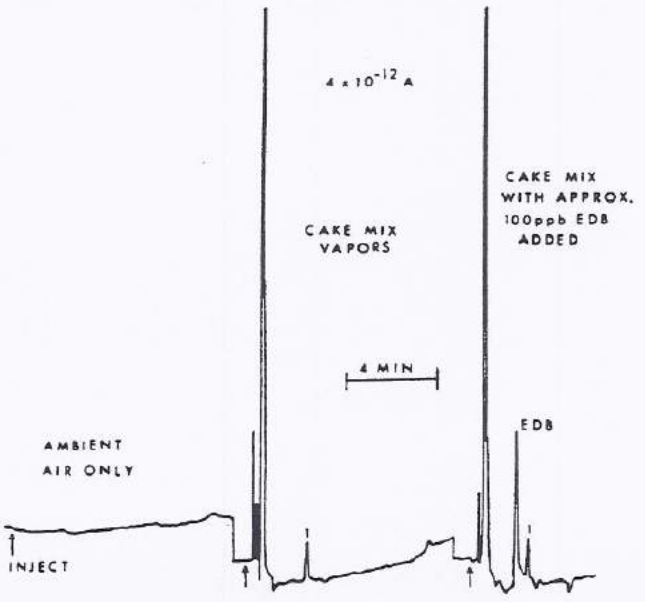

Figure 15. Example of TID-1-air application to determine EDB in cake mix. Sample: headspace vapors from 0.7 gm Duncan Hines Deluxe Yellow Cake Mix in a sealed 2-ml vial. Sampling procedure: heat sealed sample vial to 120°C; extract 200 µl headspace vapor with gas-tight syringe; inject into GC and start column oven temperature program. Column: SP-2100, 40° to 110°C at 10°/min.

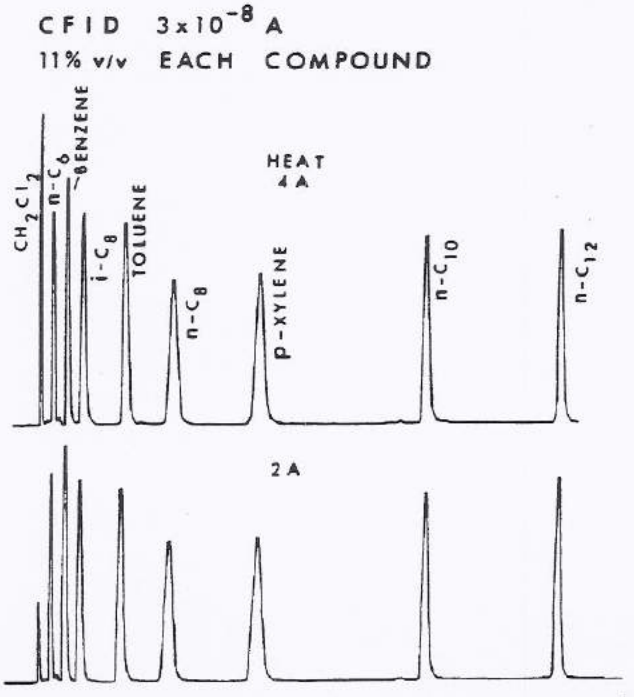

Figure 17. Comparison of CFID responses to sample of methylene chloride and various hydrocarbons at two different magnitudes of heating current to the catalytic source. Column: SP-2100, 40° to 180°C at 10°/min.

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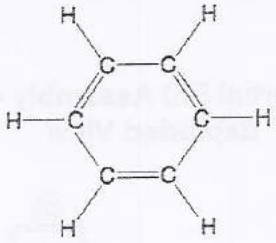
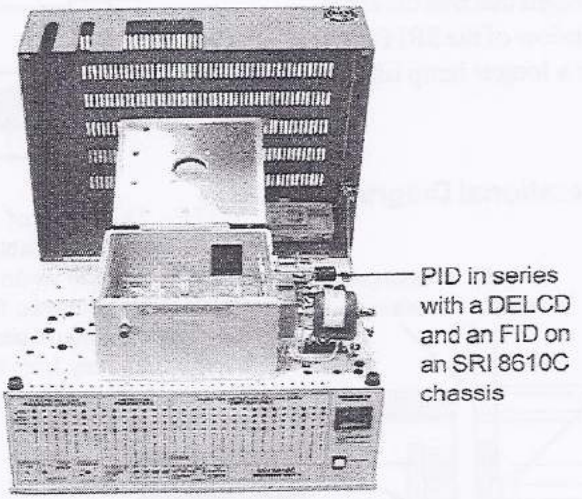
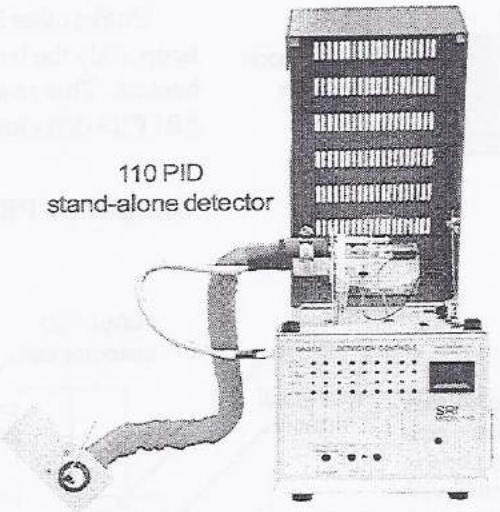


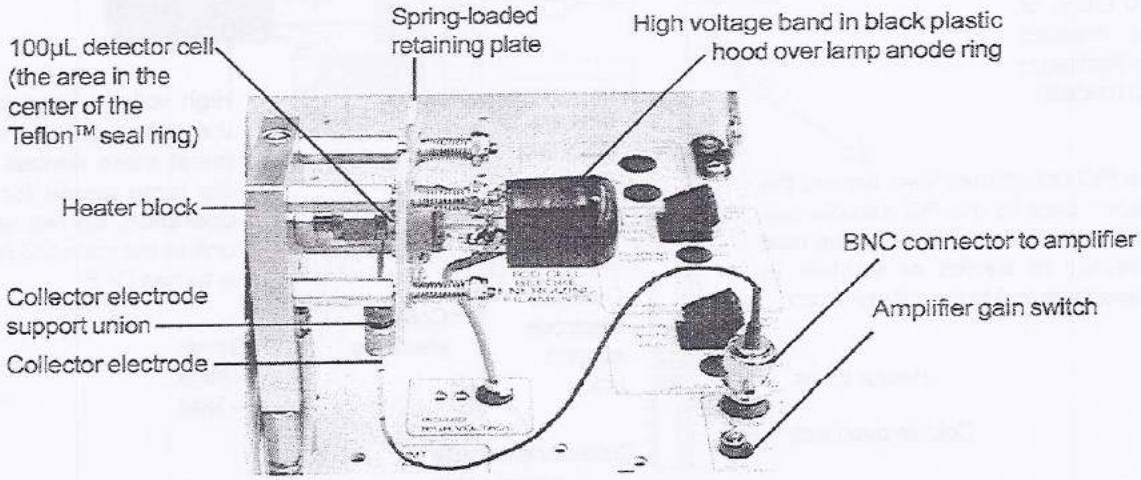
Benzene

Overview

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

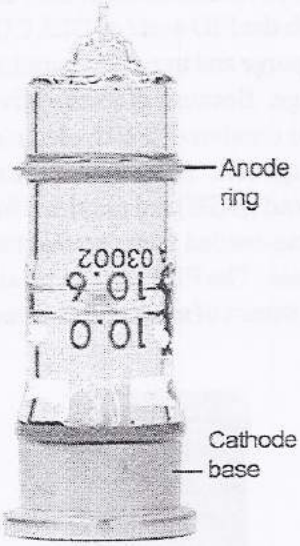

PID in series with a DELCD and an FID on an SRI 8610C chassis


110 PID stand-alone detector



Theory of Operation

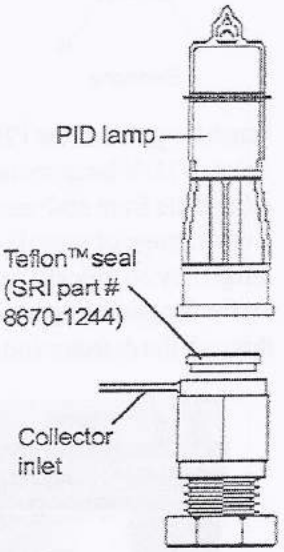
10.6eV PID Lamp (SRI Part # 8670-1242)



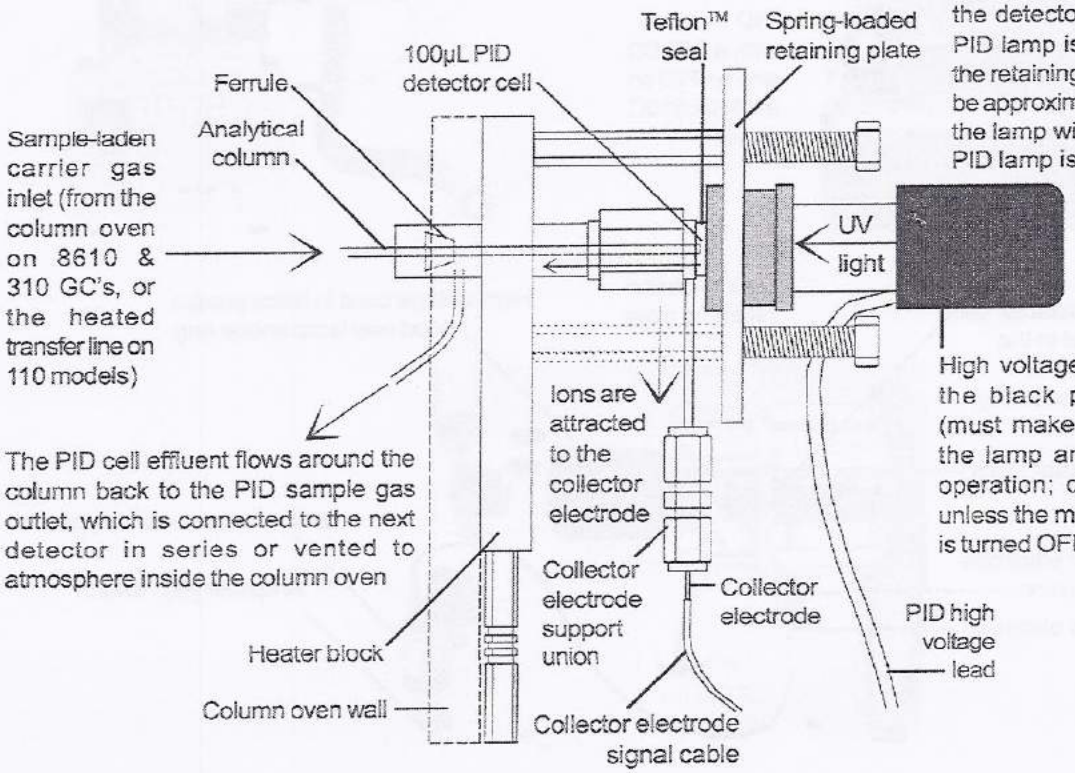
The SRI PID design uses a 10.6eV lamp with a high voltage power supply. Sample laden carrier gas flows from the analytical column into the PID sample inlet, where it is streamed through a 100µL flow-through cell. When sample molecules flow into the cell, they are bombarded by the UV light beam. Molecules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

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

Partial PID Assembly - Exploded View



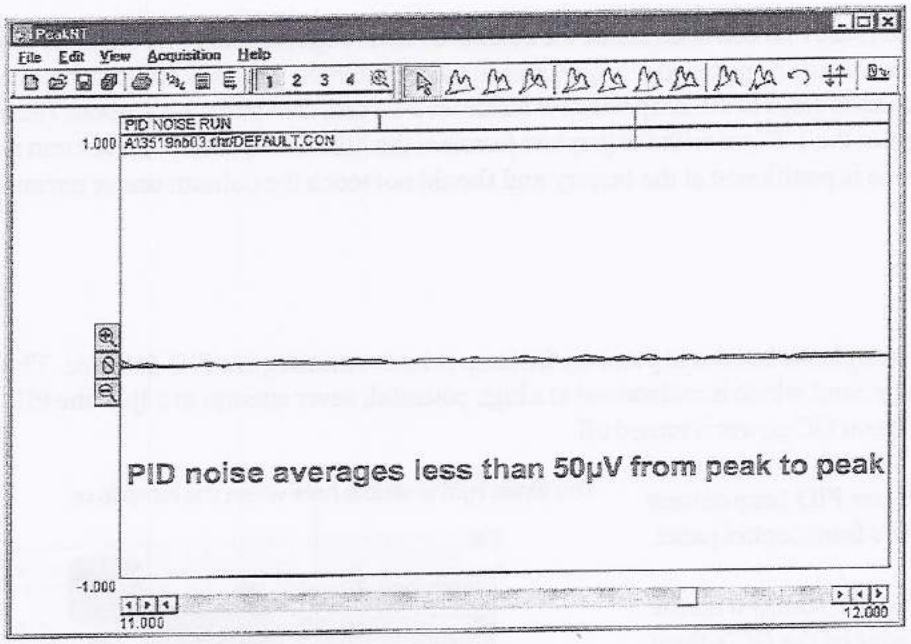
Simplified PID Operational Diagram



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

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

Expected Performance



PID Noise Run

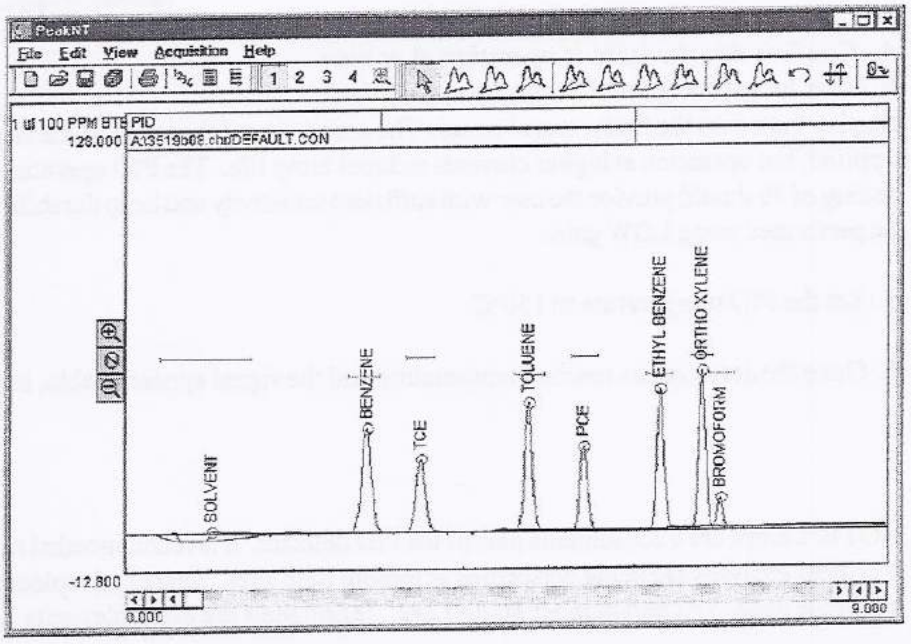
Column: 15m MXT-VOL
Carrier: Helium @ 10mL/min
PID gain: LOW
PID temp: 150°C
PID current: 70

PID BTEX Analysis (in series with FID and DELCD)

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

Temperature program:

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



Results:

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

General Operating Procedure

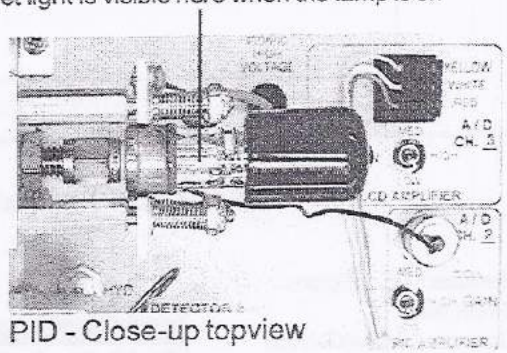
The capillary column enters the PID cell from inside the column oven through the bulkhead fitting in the insulated oven wall. The column may be installed with the lamp in place. Insert the capillary column into the PID detector inlet until the column stops at the lamp window inside the PID cell, then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

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

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

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

The violet light is visible here when the lamp is on



PID - Close-up topview

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

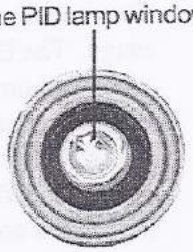
5. Set the PID temperature to 150°C.

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

NOTE: Lamps are a consumable part of the PID detector. It is recommended to have a spare lamp available if critical analyses are being performed at remote field sites. Spare and replacement 10.6eV PID lamps are available under SRI part number 8670-1242. Teflon seals are available under SRI part number 8670-1244.

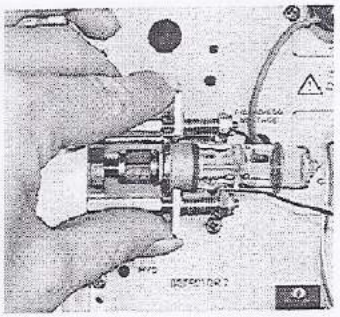
Troubleshooting and Maintenance
Cleaning the PID Lamp

The PID lamp window



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

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



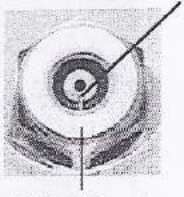
2. Disconnect the high-voltage band from the lamp anode by removing the black plastic hood.

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

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

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

The collector electrode protrudes into the cell

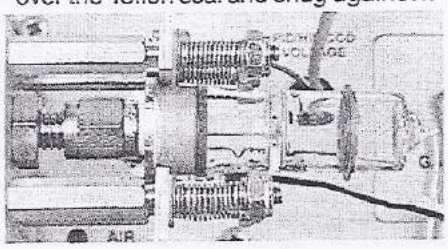


Teflon™ seal

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

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

Make sure the lamp window is centered over the Teflon seal and snug against it

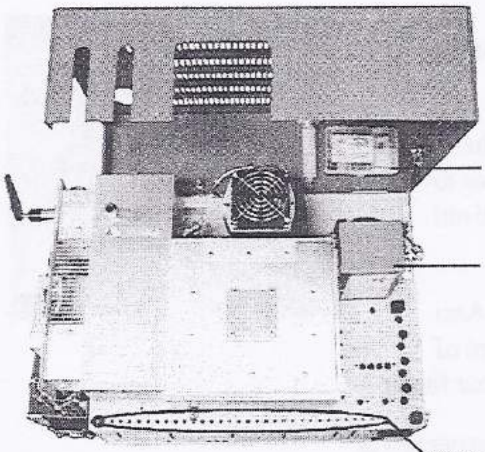


8. Open the spring-loaded retainer plate and replace the PID lamp snug against the seal. The lamp window has a slightly larger diameter than the seal; try to center it against the seal. Replace the high voltage band / black plastic lamp hood.

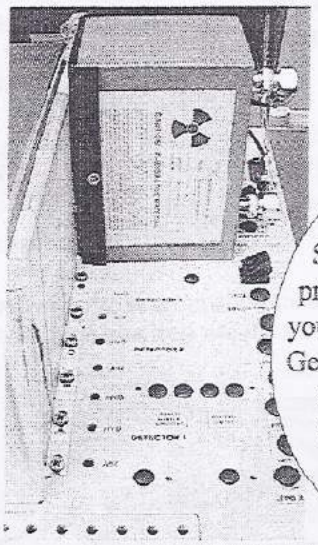
9. Recalibrate the PID detector before returning it to service.

OVERVIEW

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

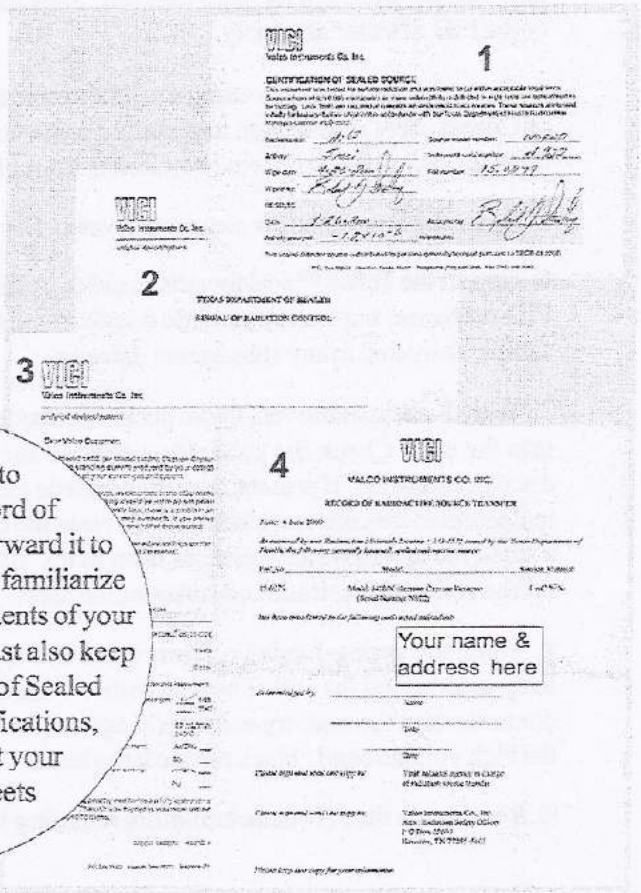


ECD-equipped SRI GC



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

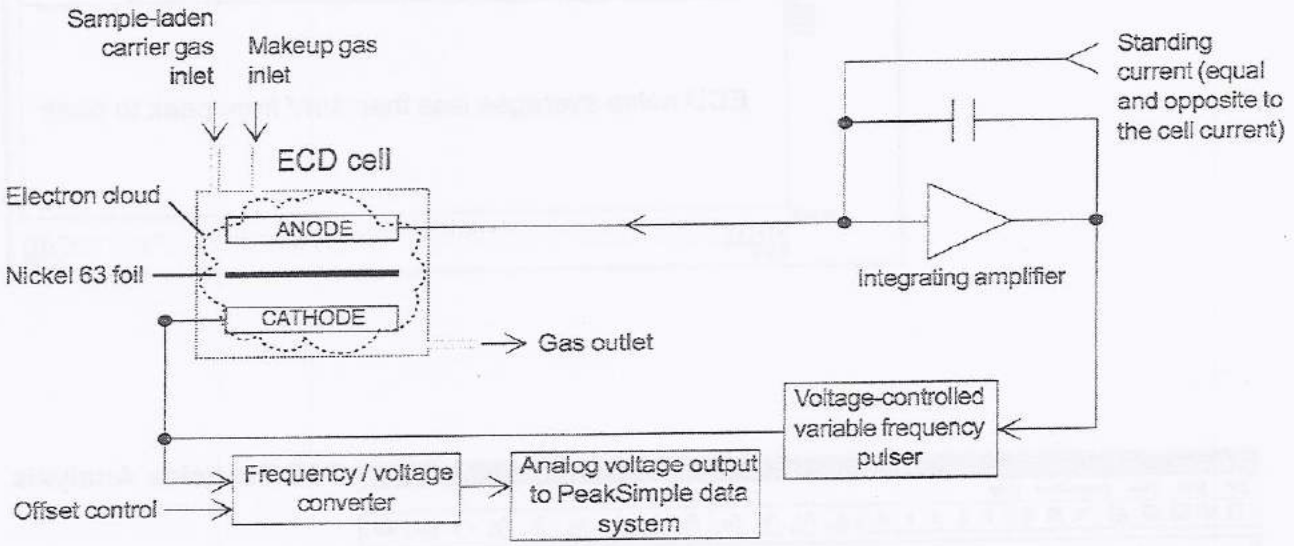
It is imperative to complete the Record of Source Transfer and forward it to proper authorities, and to familiarize yourself with the requirements of your General License. You must also keep on file the Cerification of Sealed Source and Test Specifications, which are proof that your ECD detector meets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



Theory of Operation

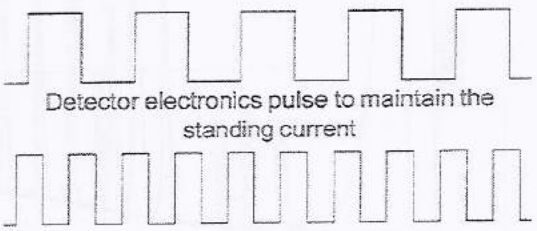
The radioactive Nickel 63 sealed inside the ECD detector emits electrons (beta particles) which collide with and ionize the make-up gas molecules (either nitrogen or P5). This reaction forms a stable cloud of free electrons in the ECD detector cell. The ECD electronics work to maintain a constant current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

ECD Detector Operational Diagram



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

Example Pulse Trains



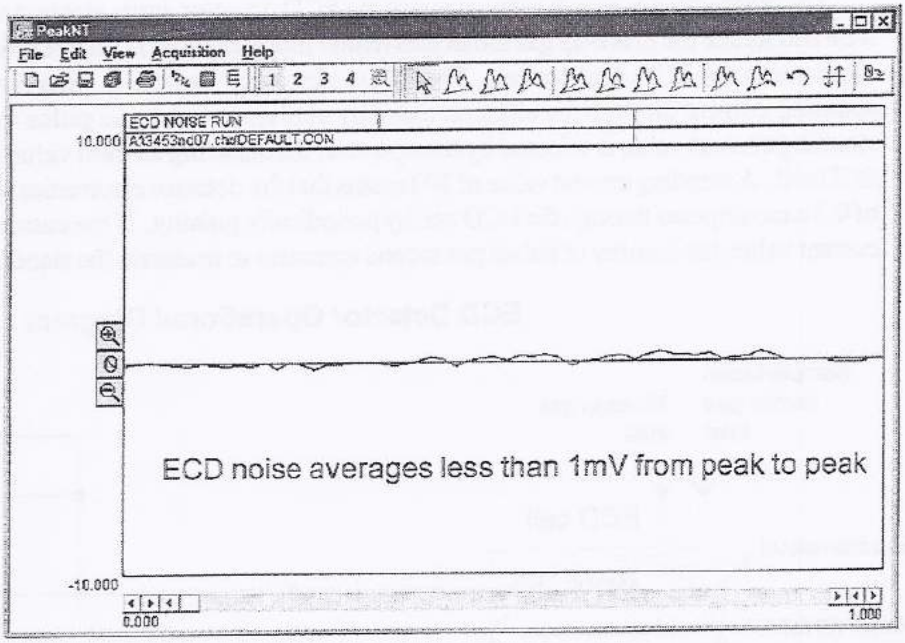
Detector electronics pulse to maintain the standing current

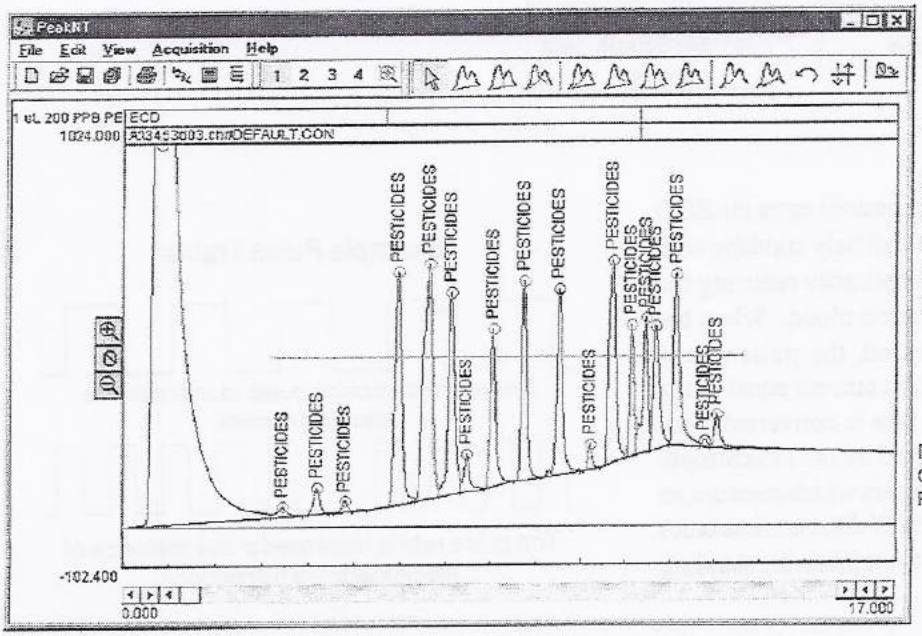
The pulse rate is increased in the presence of electronegative compounds

Expected Performance

ECD Noise Run

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





ECD Pesticide Analysis

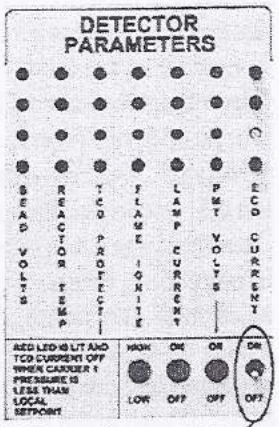
Sample: 1µL 200ppb chlorinated pesticides
Column: 15m MXT-5 capillary
Carrier: Helium @ 10mL/min
Makeup: Nitrogen @ 60mL/min
ECD temp: 250°C
ECD standing current: 300

Results:

Component	Retention	Area
Pesticides	2.850	45792.4350

General Operating Procedure

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



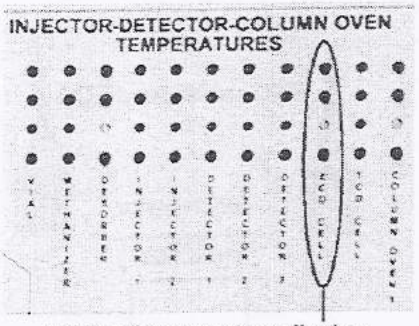
ECD standing current ON/OFF switch

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

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

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

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



ECD cell temperature display

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

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

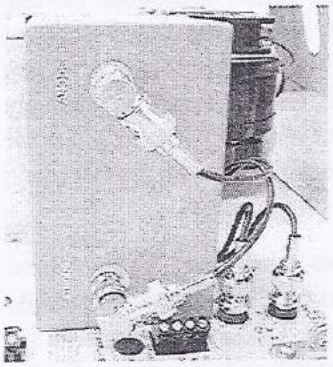
6. You may need to adjust the ECD standing current using its trimpot setpoint. The trimpot setpoints are located on the top edge of the front control panel, directly above the display push-buttons for each controlled zone. Remember, increasing the standing current increases the ECD's sensitivity and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

ECD Troubleshooting

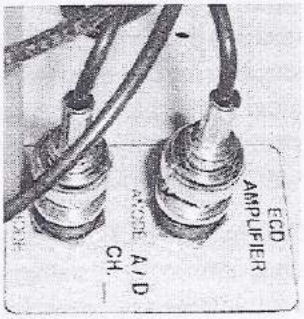
If you are experiencing baseline offset and noise problems withyour ECD detector, try the following two diagnostic tests:

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

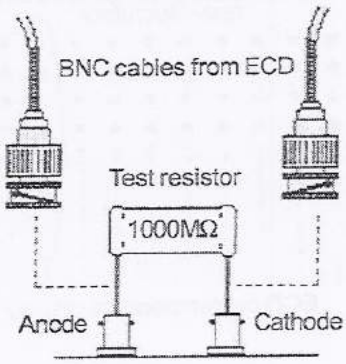
The ECD anode and cathode are connected to the ECD amplifier via BNC cables



Anode and cathode BNC connectors

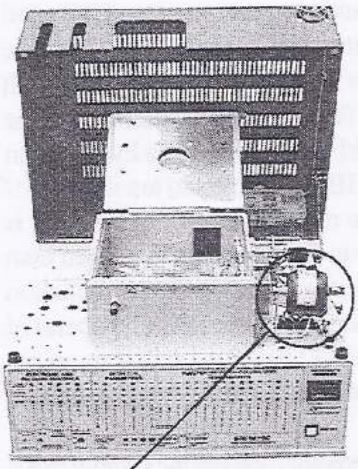


ECD electronics test



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

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



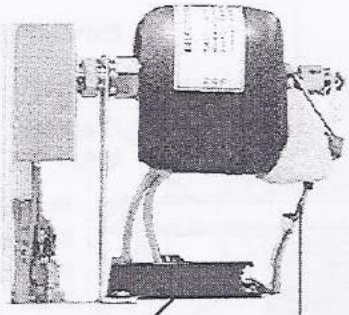
DELCD on an 8610C GC

Overview

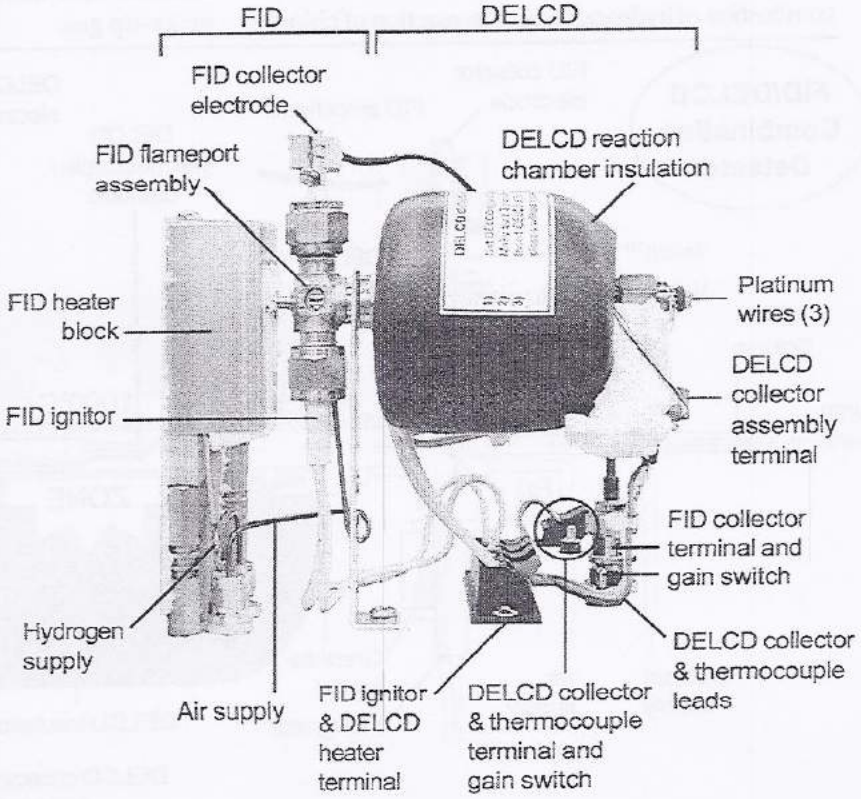
The Dry Electrolytic Conductivity detector, or DELCD, is selective to chlorinated and brominated molecules. It differs from the traditional wet ELCD in that it does not use a solvent electrolyte, and the reaction products are detected in the gaseous phase. The SRI DELCD is available alone or in combination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

DELCD - À la carte

Close-up of the same DELCD detector



FID / DELCD Combo Detector



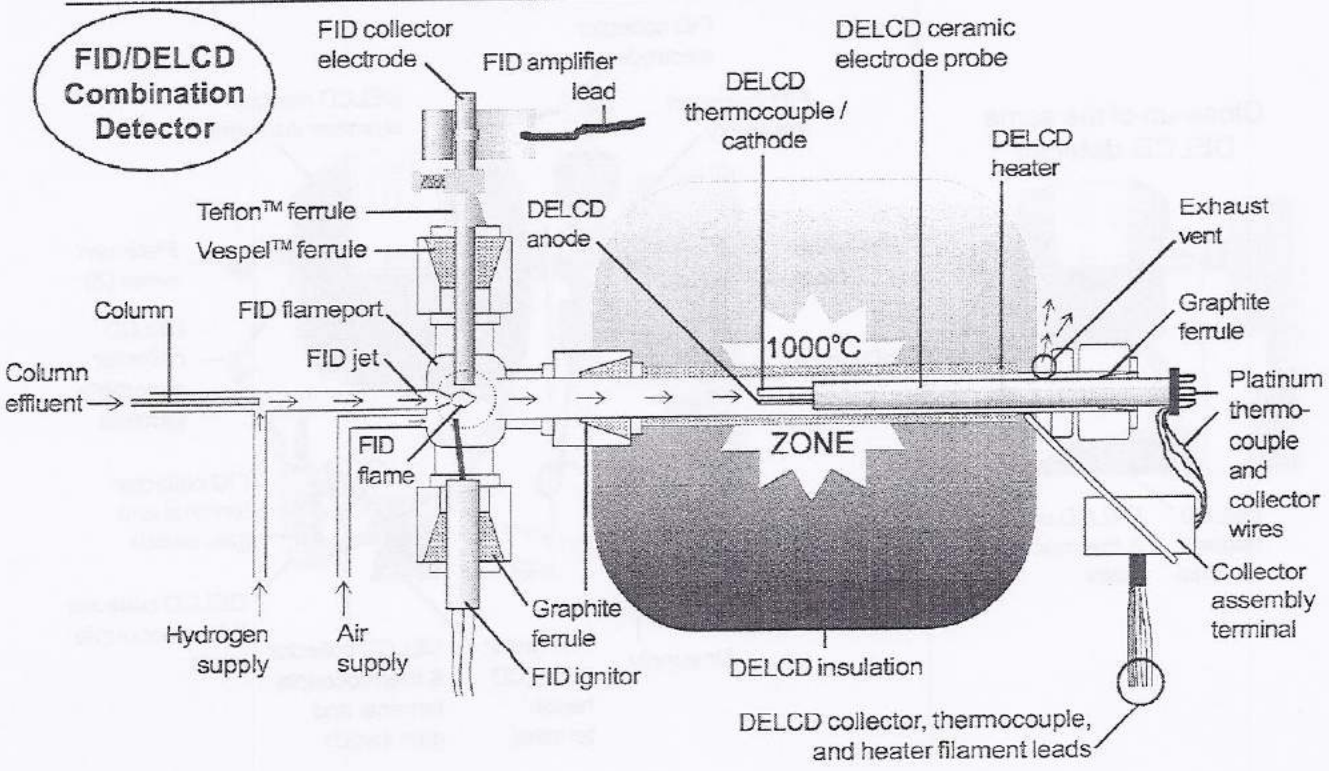
Dry Electrolytic Conductivity Detector - DELCD

Theory of Operation

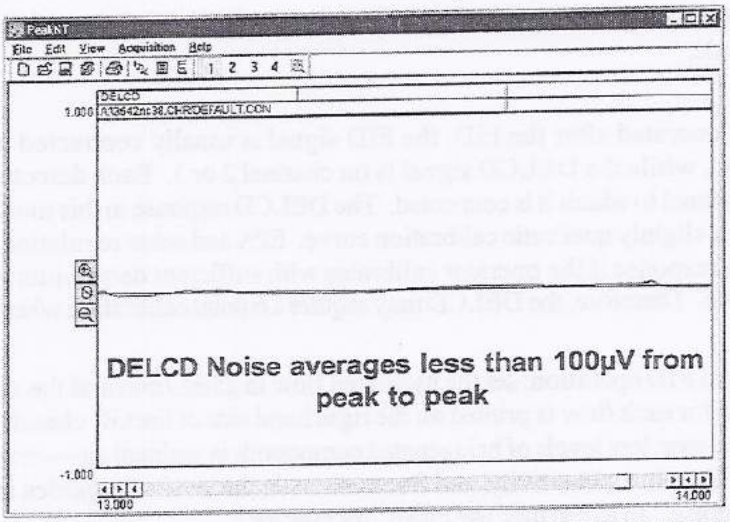
The DELCD consists of a small ceramic tube—the DELCD reactor—heated to 1000°C. Inside the reactor, a platinum thermocouple measures the detector temperature, and a nichrome collector electrode measures the conductivity of the gases flowing through the DELCD. The detector response is dependent upon its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

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

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



Expected Performance



DELCD Noise Run

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

Temperature program:

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

FID / DELCD Combo Test Run

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

Temperature program:

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

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

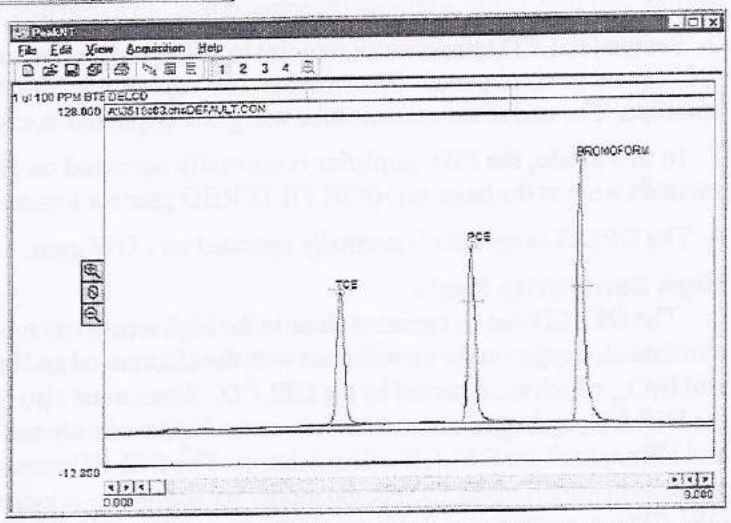
DELCD Results:

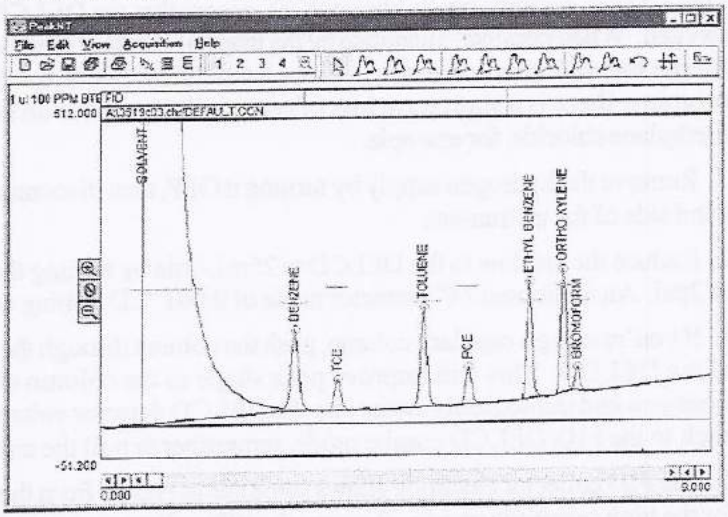
Component	Retention	Area
TCE	3.483	463.5080
PCE	5.416	532.2900
Bromoform	7.016	759.6650
	Total	1755.4630

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

FID Results:

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





DETECTORS
Dry Electrolytic Conductivity Detector - DELCD

General Operating Procedure

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

Combo Mode

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

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

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

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

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

High Sensitivity Mode

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

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

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

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

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

General Operating Procedure continued

FID/DELCD - FID Only

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

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

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

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

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

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

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

DELCD Only

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

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

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

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

Troubleshooting and Maintenance

Installing the Spare DELCD Cell

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

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

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

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

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

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

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