RESTEK
Technical
Guide

A Guide to GC Setup

C O N T E N T S:

Gas chromatographs are shipped with an instrument manual that includes information on the pnuematics, electronics, computer hardware, and software installed in the new instrument. Unfortunately, practical instruction on how to set up the gas chromatograph is one area that many manuals do not adequately cover. This guide presents the basic steps in setting up a new GC and useful information on efficient and proper installation.

Next:Tools Required to Plumb a GC

Pre-Installation Instructions

The first, and perhaps most important, rule of setting up a new GC is to never throw anything away. All items are grouped and labeled by the manufacturer. Keeping all parts and manuals together will significantly reduce the chance of misplacing critical documentation or spare parts. It is a good idea to save the original box and packing materials in case the GC must be returned to the manufacturer. The GC oven fan is often bolted in place during transportation. The shipping bolt must be removed from the back of the GC before turning on.

Next, determine where to install the GC. Allocate sufficient bench space to permit installation of recorders, chromatography data systems, autosamplers, and other GC equipment. Two inches of space at the sides of the GC permits free air circulation. Allow two feet of access space at the rear of the GC for ease of service or future plumbing changes. Make the installation site accessible to easily change traps and connect gas lines.

Do not place the GC near a heating or air conditioning vent. Variations in room temperature can affect the heated zones of the GC. Chromatographically this problem is seen as retention time or baseline instability as the heating or AC units cycle on and off. A constant room temperature and a site free of hot or cold spots ensures optimum GC performance.

If the lab is not equipped with existing gas lines, set-up the GC in a location near the gas source. This will minimize the amount of tubing required to plumb new instruments. Continuous lengths of tubing between the cylinder and GC manifold should be used to eliminate the possibility of fitting leaks. If several GCs are being plumbed on the same carrier gas line, connecting tees should be easily accessible for leak checking and troubleshooting. (Do not hide tees or connectors in a ceiling or wall. This makes it difficult to periodically leak check!)

Determine the power requirements of the GC. If the power requirement is less than 15 amps, the instrument may be plugged into a 15 or 20 amp branch circuit. If the unit draws 15 amps or more, the GC power cord will have a 20 amp plug and must be plugged into a 20 amp circuit (a 20 amp plug looks similar to a standard three-prong plug, except that one prong is turned at a right angle towards the other one). If the plug on the GC doesn’t fit the outlets in your lab, consult a qualified electrician before proceeding!

Generally, only one GC should be plugged into a single 15 or 20 amp branch circuit. Plugging multiple GCs into the same electrical circuit may cause the circuit breaker to trip on occasions when two instruments are heating at the same time.

If possible, integrators or data systems should be plugged into the same outlet or circuit as the GC from which it is acquiring data. This will help to prevent ground loop currents from developing between the two instruments, which can contribute to baseline noise. To further reduce electrical noise, use high quality, shielded signal cables and keep the cables as short as possible.

Back:Pre-Installation Instructions Next:Choosing the Proper GC Carrier Gas

Tools Required to Plumb a GC

Once the instrument site is prepared, you are ready to consider what gases are needed for the GC. Items necessary to install a new GC include the following:

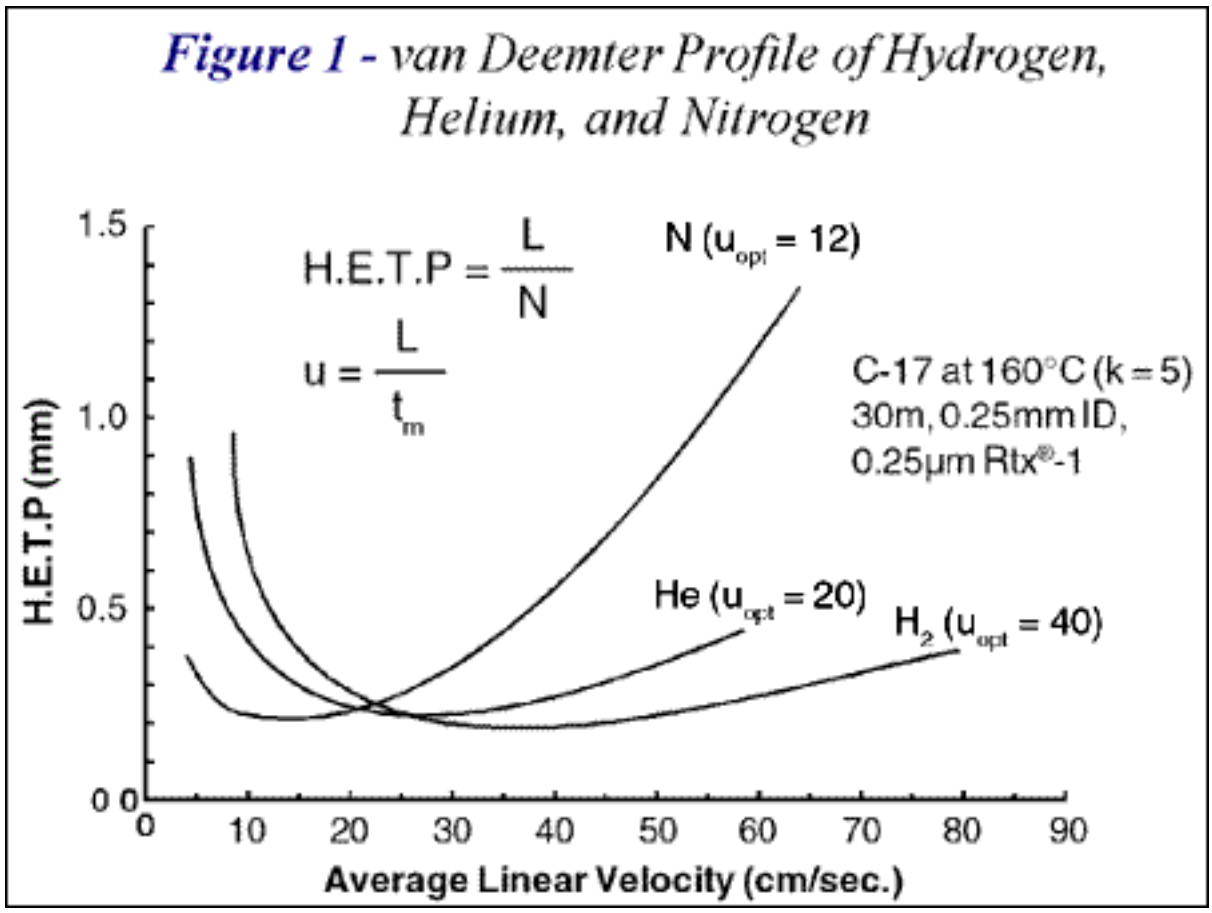
- Wrenches (1/8, 1/4, 7/16, 9/16, and 1/2-inch)
- Phillips & flat head screwdrivers
- Solvent rinsed & heat treated stainless steel tubing
- Hoke plug valves
- SS diaphragm regulators
- MINICYL regulators
- Ferrules
- Tubing cutter
- Tubing bender
- Reamer
- Files
- Replacement fittings
- Adjustable wrench
- PTFE tape
- Electronic leak detector
- Brass tees
- Swagelok® nuts & ferrules
- Pigtail fittings
- Traps
- Reducers
- Septa
- Deactivated sleeves
- O-rings
- Capillary column

Choosing the Proper GC Carrier Gas

Using the correct carrier and detector gases are an important factor in installing a new GC. The five gases commonly used as carrier gas and detector fuels in capillary gas chromatography are helium, hydrogen, nitrogen, argon-methane, and air. The types of gases necessary are partly determined by the detection system used. Factors to consider for each individual gas are discussed below.

Carrier Gas Choice

Carrier gases that exhibit a broad minimum on a van Deemter profile are essential in obtaining optimum performance. Volumetric flow through a capillary column is affected by temperature. When temperature programming from ambient to 300°C, the flow rate can decrease by 40 percent. A carrier gas that retains high efficiency over a wide range of flow rates and temperatures is essential in obtaining good resolution throughout a temperature programmed run. **Figure 1** shows the van Deemter profile for hydrogen, helium, and nitrogen carrier gases.



Hydrogen is the fastest carrier gas (u_{opt}), with an optimum linear velocity of 40cm/sec, and exhibits the flattest van Deemter profile. Helium is the next best choice, with an optimum linear velocity of uopt = 20cm/sec. Nitrogen's performance is inferior with capillary columns because of its slow linear velocity, uopt = 12cm/sec. Argon-methane has a slower optimum linear velocity than nitrogen and is not recommended for use as a carrier gas with capillary columns. Air is not recommended as a carrier gas because it can cause stationary phase oxidation.

With hydrogen and helium as carrier gases, the minimum H.E.T.P. values can be maintained over a broader range of linear velocities than with nitrogen, and high linear velocities can be used without sacrificing efficiency. Nitrogen is beneficial only when analyzing highly volatile gases under narrow temperature ranges where increasing stationary phase interaction is desirable. Otherwise, the use of N_2 results in longer analysis times and a loss of resolution for compounds analyzed on a wide temperature range.

Exert Caution when Using Hydrogen as a Carrier Gas

Hydrogen is explosive when concentrations exceed 4% in air. Proper safety precautions should be utilized to prevent an explosion within the column oven. Most gas chromatographs are designed with spring loaded doors, perforated or corrugated metal column ovens, and back pressure/flow controlled pneumatics to minimize the hazards when using hydrogen carrier gas. Additional precautions include:

- Frequently checking for leaks using an electronic leak detector (Restek's Leak Detective™, cat.# 21607, 110 volts/cat.# 21609, 220 volts).

- Using electronic sensors that shut down the carrier gas flow in the event of pressure loss.
- Minimizing the amount of carrier gas that could be expelled in the column oven if a leak were to occur by installing a flow controller (needle valve) prior to the carrier inlet bulkhead fitting to throttle the flow of gas (for head pressure controlled systems only) as shown in **Figure 2**.

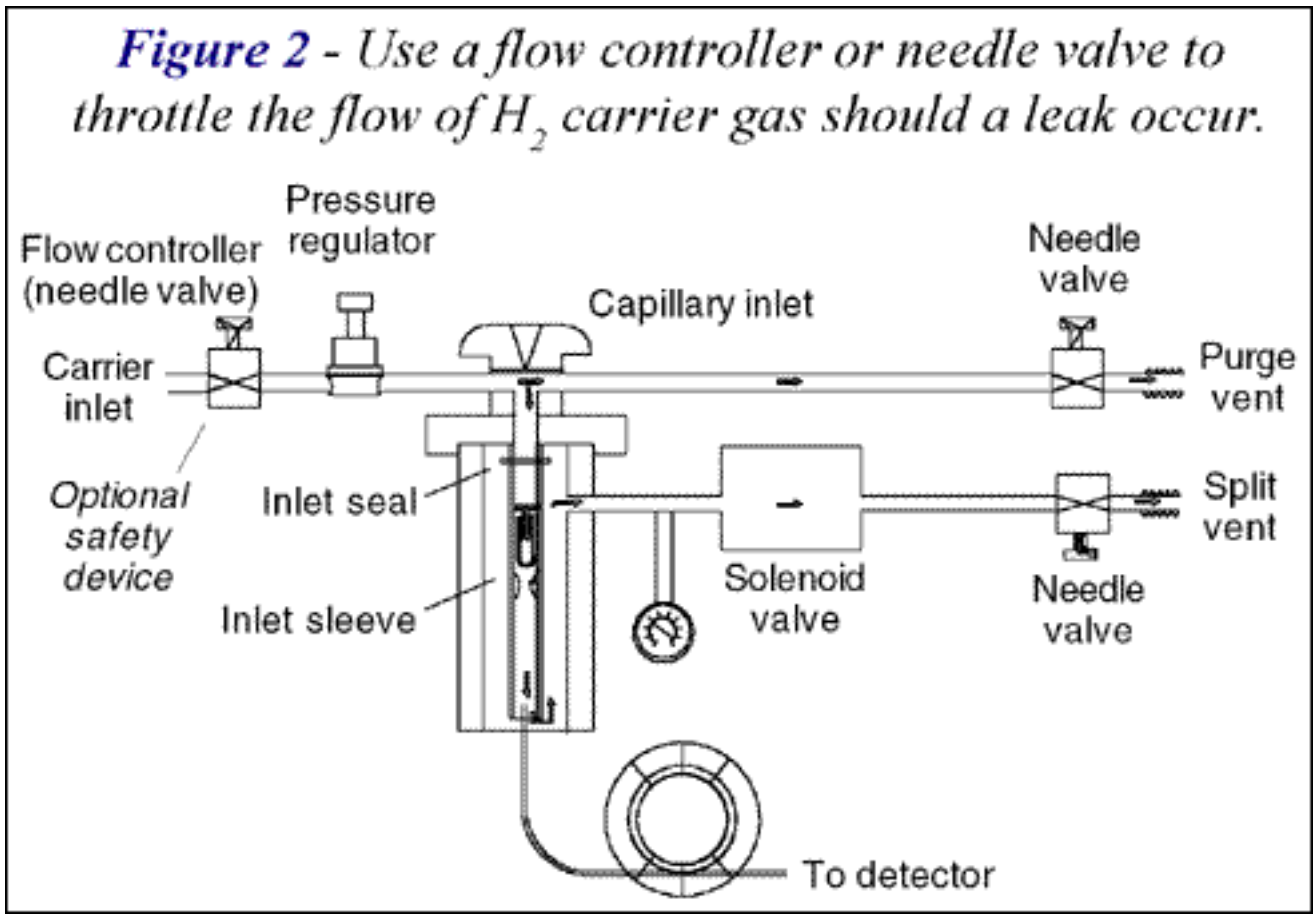


***Figure 2** - Use a flow controller or needle valve to throttle the flow of H_2 carrier gas should a leak occur.*

Fully open the flow controller (needle valve) and obtain the proper column head pressure, split vent flow, and septum purge flow rates. Decrease the needle valve flow rate until the head pressure gauge begins to drop (throttle point). Next, increase the flow controller (needle valve) setting so that the right amount of flow is available to the system. Should a leak occur, the flow controller will throttle the flow, preventing a large amount of hydrogen from entering the oven.

Make-up and Detector Fuel Gases

Choosing the correct make-up and detector gases will depend on both the detector and application. Most GC detectors operate best with a total gas flow of approximately 30ml/min. to ensure high sensitivity and excellent peak symmetry. Refer to your GC manual for optimum flow rates on different instruments. Carrier gas flows for capillary columns range from 0.5 to 10ml/min. which are well below the range where most detectors exhibit optimal performance. To minimize detector dead volume, make-up gas is often added at the exit end of the column to increase the total flow entering the detector. Make-up gas helps to efficiently sweep detector dead volume thereby enhancing detector sensitivity. Make-up gas can be

added directly to the hydrogen flame gas for flame ionization detectors (FID), nitrogen phosphorous detectors (NPD), and flame photometric detectors (FPD) or added to the column effluent by an adaptor fitting. However, GCs such as PerkinElmer and Fisons do not require make-up gas.

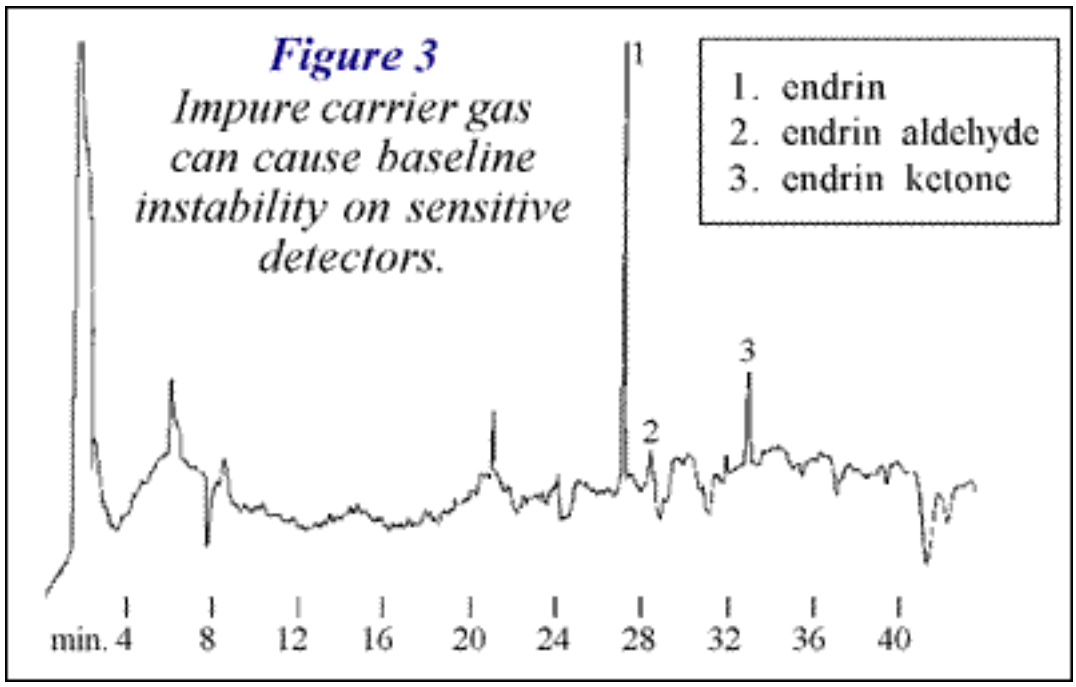
Combustion type detectors (FID, NPD, FPD) use three gases: make-up, hydrogen (fuel gas), and air (combustion/oxidizing gas). For non-combustion detectors, such as the thermal conductivity detector (TCD), electron capture (ECD), and photo ionization detector (PID), only carrier and make-up gases are required. In the case of the electrolytic conductivity detector (ELCD), the make-up gas is hydrogen, as a reaction gas in the halogen and nitrogen mode or air in the sulfur mode. **Table I** shows recommended gases for various detectors.

Table I: Carrier gases and detector fuel gases for use with various GC detectors.

Detector:		TCD	ECD	FID	NPD	FPD	ELCD	PID
Carrier Gases	He	•	•	•	•	•	•	•
	H_2	•	--	•	--	•	•	•
	N_2	•	•	•	•	•	--	•
Combustion/ Reaction Gases	H_2	--	--	•	•	•	•	--
	Air	--	--	•	•	•	--	--
Make-up Gases	N_2	•	•	•	•	•	--	•
	He	•	--	•	•	•	--	•
	$ArCH_2$	--	•	--	--	--	--	--

Recommended Gas Purities

Gas purity is very important. The expense of using high purity gases in combination with carrier gas purifiers will be offset by longer column lifetime and less GC maintenance. Carrier gas should contain less than 1ppm of oxygen, moisture, or other trace contaminants to prevent column degradation, increase column lifetime, and decrease stationary phase bleed. Carrier gas impurities can also contribute to detector noise. **Figure 3** illustrates O_2 contamination on a sensitive ECD and shows how an impure carrier gas can affect detector performance. Contaminants such as trace hydrocarbons can be detected by an FID during a temperature programmed run, causing ghost peaks to appear. Make-up and fuel gases should be contaminant-free to reduce baseline fluctuations and excessive detector noise.

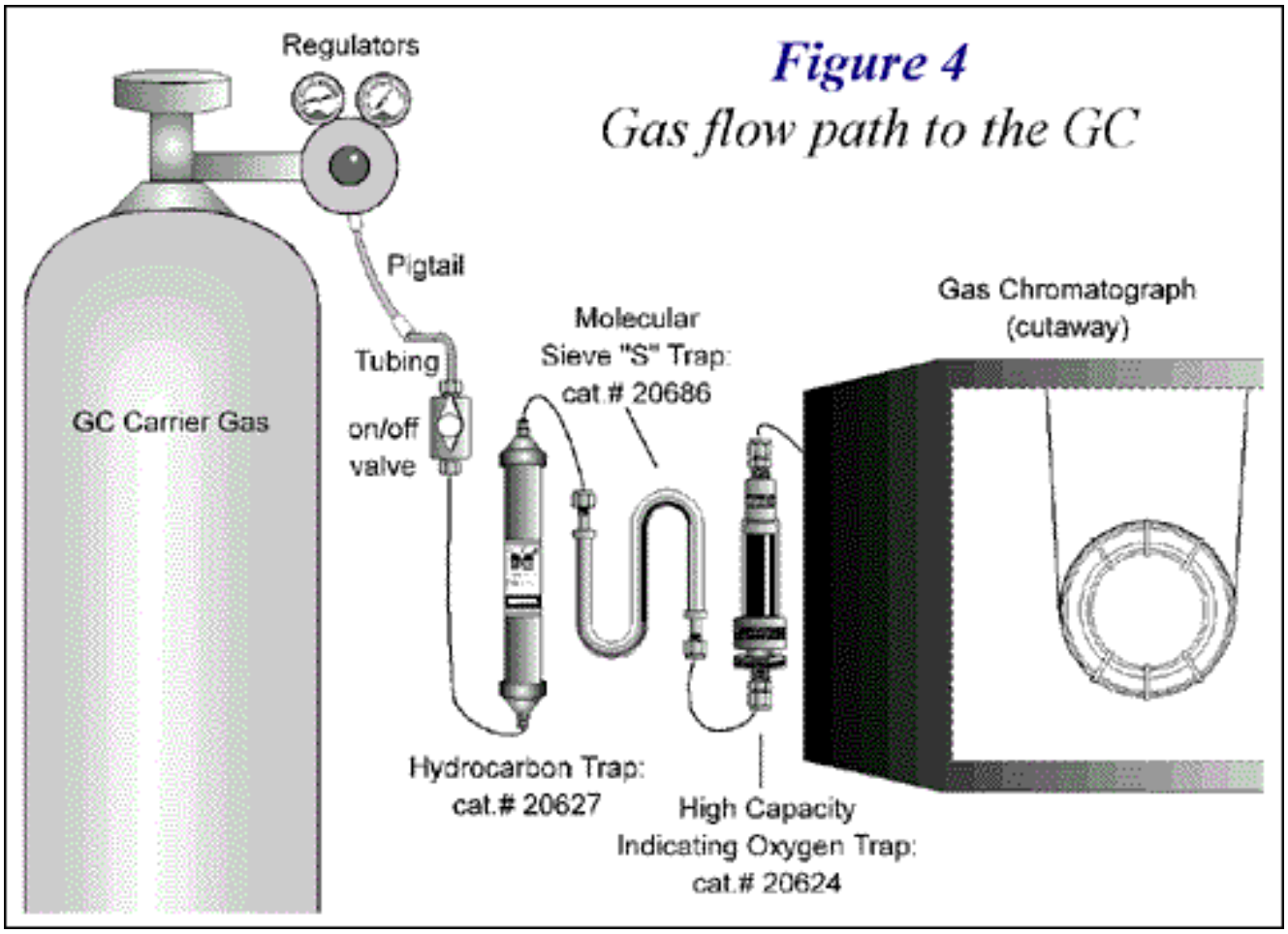


Back:Choosing the Proper GC Carrier Gas Next:GC Gas Purification

Plumbing Gases to the GC

Gas Sources | Hints for Properly Handling Gas Cylinders | Gas Generators | Regulators | Flexible Pigtails | Tubing | Tubing Cutting and Bending | Tubing Benders | Fittings | Valves

Once the proper carrier and detector gases are selected, they must be connected to the instrument. The gas flow path travels through regulators, pigtails, tubing, valves, and traps (**Figure 4**). Each component in the flow path will be discussed in sequence with helpful hints on their proper use.



Gas Sources

Gases are most often supplied to the instrument by gas cylinders. Begin installation at the cylinder and work towards the GC. Cylinders are under very high pressure and dropping one could result in an extremely dangerous situation. All cylinders (full or empty) should be securely chained to a wall or bench for safety. Any spare cylinders should also be chained to a

wall in their storage location with the valve cap intact. It is advisable to have back-up cylinders on all gas lines to avoid any interruption of flow. This is particularly important for carrier gas. Should you lose carrier flow while the column is being heated, irreversible column damage may occur. Two-stage pressure regulators are used with gas cylinders to reduce the pressure of a gas supplied from a high pressure source to a desired working pressure. For safety reasons, when removing a regulator from a cylinder, always position yourself so that the regulator is pointing away from you.

It is common for a newly installed gas cylinder to shut down unpredictably (within the first 24 hours) if the main valve is not completely opened when it is installed. As the cylinder pressure decreases, the force against the valve seat decreases, allowing it to close. Always make sure cylinder valves are completely open when installing new tanks and completely closed before removing the regulator.

As a general rule, change a cylinder when the pressure regulator indicates that there is 200-300psi remaining in the cylinder. As the cylinder pressure drops, the concentration of impurities such as moisture and hydrocarbons increase. Therefore, column damage or premature purifier consumption will occur if you attempt to "save money" by using all the gas left in a cylinder. In addition, if the cylinder pressure drops below the supply pressure required by the GC, retention times and detector sensitivities can slowly change and affect the validity of your data.

Hints for Properly Handling Gas Cylinders

Observe safe laboratory practice in the transportation, storage, and usage of gas cylinders under high pressure:

- **Never move a cylinder with a regulator installed. Make sure safety caps are in place over the gas valve when transporting the cylinder.**
- **Always chain or strap cylinders to stationary objects in the laboratory and while in storage.**
- **Always use cylinder condition labels to show whether tank is FULL, IN USE, or EMPTY.**
- **Always leave at least 200psi residual gas in a depleted cylinder. Store the empty tank in the storage area with the tank valve closed. Mark and date the empty cylinder.**
- **Do not expose cylinders to temperatures above 125°F.**

Gas Generators

As an alternative to gas cylinders, many labs use gas generators. Generators reduce the costs and safety hazards involved with high pressure gas cylinders. Hydrogen generators supply hydrogen from the electrolysis of water. These units are convenient, safe to use, and produce very pure hydrogen. Air compressors can be used for air supply. However, most compressed

air contains hydrocarbons from oil based lubricants. Compressed air that contains hydrocarbons or sulfur gases is not recommended for operating an FID, FPD, TSD, or ELCD. It is advisable to use filters and purifiers to remove hydrocarbon contamination from the compressed air source.

Regulators

The purpose of a pressure regulator is to maintain constant gas pressure to the GC. Regulators may be classified as two types: cylinder regulators and line regulators. Cylinder regulators attach directly to the cylinder valve. The cylinder regulator reduces the gas pressure from the cylinder pressure (usually 2500psi for a new cylinder) down to a more usable pressure (around 100psi for gas chromatography). Cylinder regulators have two pressure gauges: an inlet, or high pressure gauge which reads the cylinder pressure; and a delivery, or outlet pressure gauge. This final delivery pressure is user adjustable by turning the large knob on the front of the regulator.

Cylinder regulators may be either single-stage or two-stage regulators. Two-stage regulators actually employ two regulators back-to-back in one housing. The first stage reduces the cylinder pressure to 200-600psi, while the second stage performs the final pressure reduction. Two-stage regulators are less prone to "creep" (a slow increase in delivery pressure as the tank empties) but have a lower flow capacity than single-stage regulators. Although more expensive, two-stage regulators should be used when a very constant delivery pressure is required, such as when controlling gas flows for a gas chromatograph.

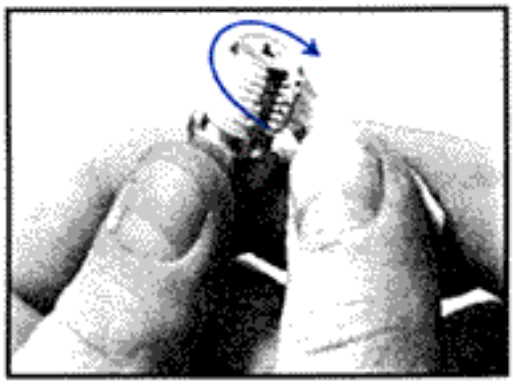
Line regulators have a lower allowable inlet pressure (typically 300psi) and must never be attached directly to a gas cylinder. Line regulators are used to further reduce the pressure of a gas from the supply line to that required at the point of use. Line regulators are always single-stage regulators, and may be equipped with a single pressure gauge to indicate outlet pressure.

Oxygen, moisture, and elastomeric contaminants can migrate through rubber or elastomeric diaphragms and enter the carrier gas. Therefore, all regulators should incorporate metal (PTFE coated stainless steel) diaphragms to assure that contaminants will not enter the gas line. Consult **Table II** to determine the proper type and size of cylinder valves as described by the Compressed Gas Association (CGA) numbers for each regulator.

Table II: High Purity Two-Stage Regulators
Proper type and size of cylinder valves

CGA 580	N_2, He, & Ar	cat. # 20606
CGA 350	H_2	cat. # 20607
CGA 590	Air	cat. # 20608

Figure 5



Wind Teflon® tape clockwise to ensure a good seal.

When installing any regulator, be sure to use high quality PTFE tape on all machine thread connections (**Figure 5**). DO NOT use PTFE tape on Swagelok®-type compression fittings.

Flexible Pigtails

Pigtails (cat.# 20615) are commonly installed between the two-stage regulator and the gas lines. They allow the necessary flexibility in removing regulators from the cylinder. Pigtails are constructed of braided stainless steel with an inert PTFE core.*
*Some O_2 and moisture will diffuse through the PTFE core stainless steel braided pigtails. Always use oxygen and moisture traps downstream of flexible pigtails.

Tubing

The next step in plumbing GC gases to the instrument is choosing the appropriate tubing size. Most GCs are plumbed with 1/8" bulkhead fittings, therefore 1/8" tubing is commonly used. The location of the gas cylinders also determines if a larger diameter tubing is required between the tanks and the GC. If only one GC is being plumbed from tanks located in the same room, 1/8" tubing is sufficient. However, if the tanks are located outside the room or if several GCs are being plumbed from the same source, 1/4" tubing is recommended to reduce pressure drop in the line and supply adequate gas for several instruments. When using 1/4" tubing, plumb the GC with a 1/4" line from the tanks to back of the GC. Then use a 1/4" to 1/8" female reducer (cat.# 21825) to allow attachment to the first purifier. Connect both purifiers to the carrier gas line with 1/8" tubing loosely coiled. When measuring the 1/8" tubing, provide extra length to coil the tubing into shock loops. Shock loops will prevent instrument vibrations from being transferred to the supply lines and loosening fittings or breaking gas purification traps. Additionally, shock loops allow an instrument to be moved on the lab bench. Complete the installation by plumbing 1/8" tubing from the outlet of the last purifier to the GC bulkhead fitting.

It is essential to use clean chromatographic grade tubing prior to installing a GC. Tubing can contain residual hydrocarbon contamination from the drawing process. These contaminants can migrate into the gas stream causing elevated background noise and increase instrument down time. Tubing can be solvent rinsed with methanol or other various solvents that do not provide a response on the detector being used. (Caution: do not use methylene chloride when using ECDs.) Restek offers a full line of pre-cleaned, heat treated tubing to plumb GCs without the need for solvent rinsing. GC manufacturers recommend copper or stainless steel tubing for

plumbing gas lines between the gas source and the instrument. Plastic tubing material such as PTFE, polyvinyl chloride, or Tygon® should not be used when plumbing GCs since these materials will allow air and water to diffuse into the gas lines. In addition, plastic tubing can give off organic impurities which can cause ghost peaks and baseline instability.

Tubing Cutting and Bending

The first step toward leak free plumbing is correctly cutting and bending the tubing. Either a hand-held or a motorized tubing cutter can be used to cut tubing. With a hand-held device, the tubing is scored by guiding a cutting wheel along the outside surface of the tubing. By increasing the pressure, the cutting wheel is forced into the tubing, thereby making a cut. With a motorized cutter, the cutting wheel is driven by a high speed motor and the tubing is hand-fed onto the spinning wheel. The mechanical cutter*, though more expensive, will easily pay for itself when plumbing several instruments since it is faster and makes a clean, open cut.

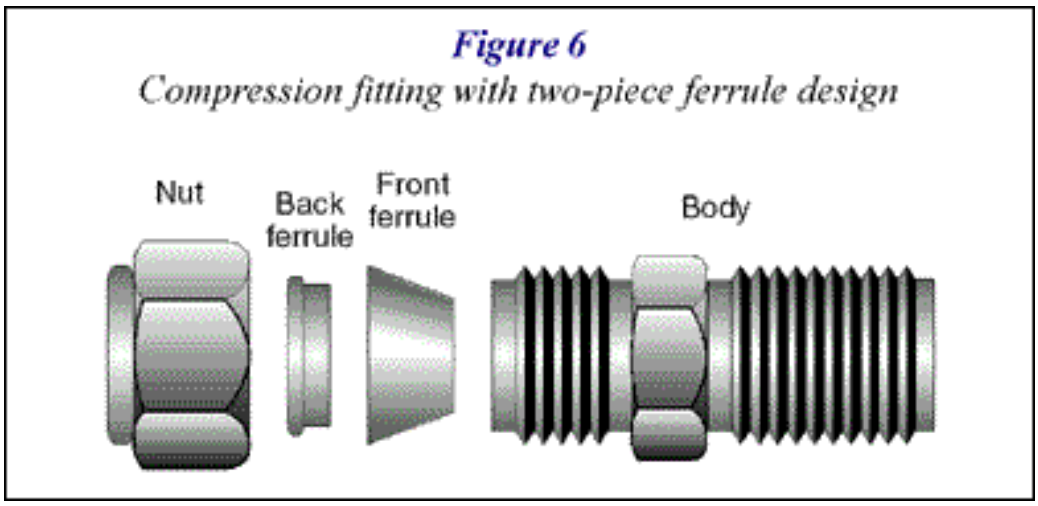
When using a tubing cutter, a burr or ridge will form on the tubing end. This burr must be removed to allow unobstructed gas flow and to obtain a leak-free connection with the compression fittings. Use a file or exterior deburring tool to remove the burr on the outside of the tubing and an interior deburring tool for the inside. Restek also offers a special tool that deburrs the inside and outside of tubing simultaneously (cat.# 20134 for 1/4" and 1/8" tubing, or cat.# 20188 for 1/16" tubing). Always hold the tubing open end down when deburring to prevent fragments from falling into the tubing.
*Catalog number 20186 is recommended for 1/16" and 1/8" tubing only.

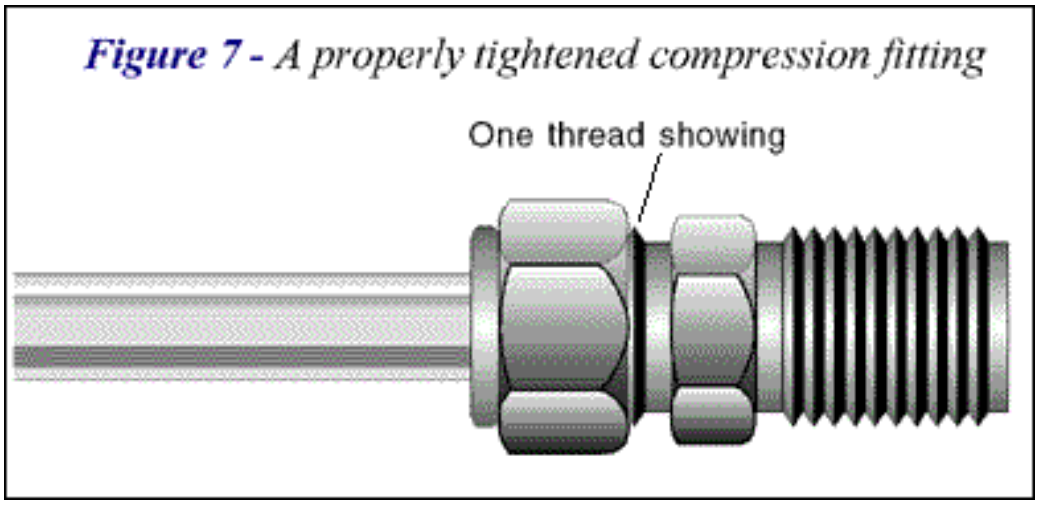
Tubing Benders

Tubing often requires bending during installation. Copper and thin walled 1/8" stainless steel tubing can easily be bent by hand. However, heavy walled 1/8" and 1/4" stainless steel tubing will require a tubing bender. A tubing bender incorporates the use of lever arms that reduce the force required to bend the tubing. Bends should be made with a uniform radius and should not kink or deform the tubing in a manner that obstructs flow. Try the bending procedure on some spare tubing first to help avoid costly mistakes on expensive tubing.

Fittings

Compression fittings provide gas-tight, leak-free connections without the use of PTFE tape or adhesives. A Swagelok®-type compression fitting consists of a nut, a back ferrule, a front ferrule, and the fitting (union, elbow, tee, etc.) as shown in **Figure 6**. Slide the nut and ferrules onto clean, deburred tubing and insert the tubing into the fitting as far as possible. Hand tighten the nut, then use a wrench to tighten further. For 1/8" tubing, tighten the nut 3/4-turn past finger-tight. For 1/4" tubing, tighten the nut 11/4-turns past finger-tight. When tightened, the back ferrule forces itself into the front ferrule causing it to compress and grip the tubing forming a leak-free seal. Be careful not to over-tighten the nut, or the tubing and ferrules can become deformed and not seal. A properly tightened compression fitting usually shows one thread from the back of the nut (**Figure 7**). Overtightened fittings show no thread and are prone to leakage.





Valves

To expedite troubleshooting, the entire GC network should incorporate valving. 1/8" brass plug valves (cat.# 21889) are recommended to isolate the system to check for leaks or to allow the GC to be taken off-line for repairs. Plug valves use a rotating cylinder to control gas flow in one direction only. Ball valves use a ball encased in PTFE packing and allow flow in either direction. One drawback of ball valves is the potential for PTFE to flow form and cause the valve to leak when used under fluctuating temperature conditions. Valves should be placed before gas purification traps to allow simple trap replacement without shutting down other GCs on the same line. For easy identification and troubleshooting, label or color code each valve throughout the system to help identify each gas type. After the system is pressurized, leak check valves in all possible positions using a thermal conductivity leak detector (cat.# 21607).

Caution: Two different gas types should never be connected together by a tee or a valve to allow easy change-over of carrier gases. Mixing will inevitably occur making troubleshooting very difficult.

Back:Plumbing Gases to the GC Next:Leak Checking

GC Gas Purification

Indicating vs Non-Indicating | Proper Order of Trap Installation | Purifiers for Other Gases? | Important! Split Vent Traps

Clean carrier gas is the key to longer column lifetime and less detector noise. Oxygen and moisture can enter downstream of the carrier gas cylinder through fitting leaks or connectors that utilize rubber o-rings. Also, contamination of the tubing with solvents or lubricating oils can increase background noise and cause ghost peaks with GC systems. Therefore, traps should always be used (even with ultra high purity gases) to prevent impurities from entering the GC system. Individual traps are designed to remove moisture, oxygen, hydrocarbons and other contaminants from the gas supply. Traps are available with either 1/4" or 1/8" compression fittings and are typically constructed with metal or glass bodies. Plastic bodied traps should never be used since oxygen and moisture permeation will occur. Several common carrier, make-up, and detector gas purifiers are discussed in the following section.

The most common contaminants in carrier gas are oxygen, water, and hydrocarbons. Both oxygen and moisture degrade the stationary phase and shorten column lifetime. Hydrocarbons cause ghost peaks or increase detector noise. Oxygen contamination in carrier gas can produce excessive column bleed at high temperatures (**Figure 8**). Although some stationary phases are more resistant to oxidation (methyl and phenyl/methyl polysiloxanes), all stationary phases will eventually degrade when exposed to oxygen in the carrier gas at high temperatures.

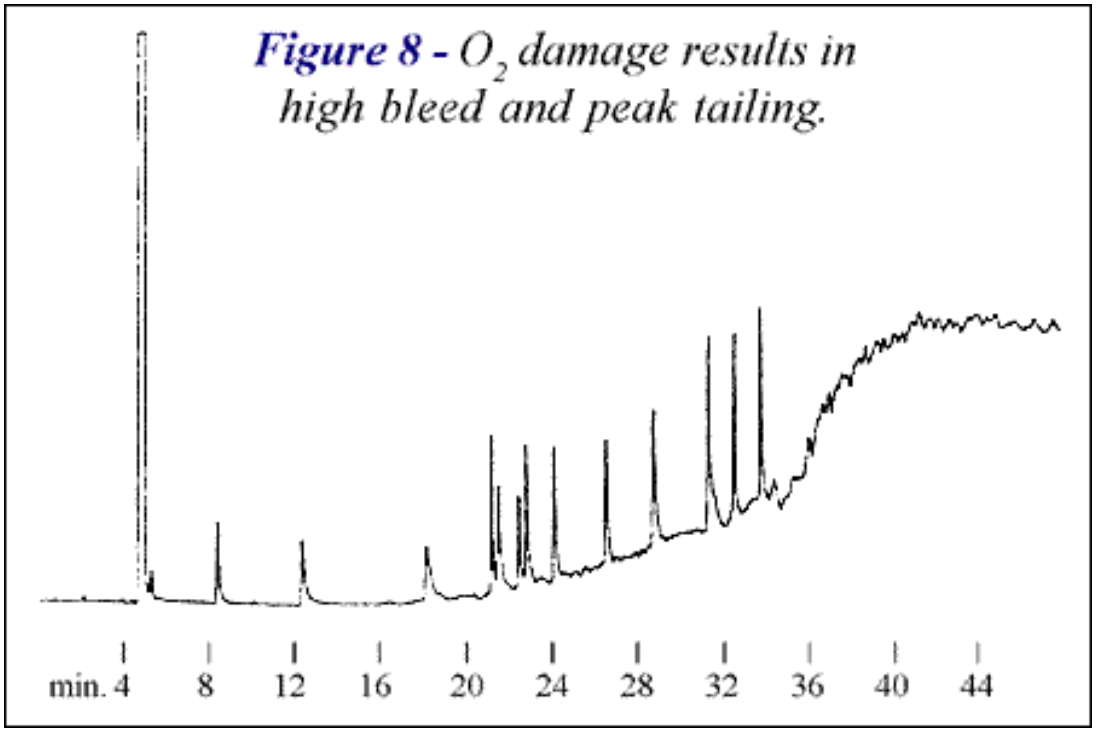


Figure 8 - O_2 damage results in high bleed and peak tailing.

Oxygen can be removed using getters, or materials that adsorb or chemically react with oxygen. Some getters must be heated to effectively remove oxygen, while others can be used at room temperature. Getters also differ in their capacity and mechanism to remove oxygen. Heated getters may release hydrogen or other impurities into the carrier gas stream, whereas most room temperature getters simply bind or react with oxygen. Some room temperature getters are extremely reactive when broken, therefore care must be taken not to break the trap or expose the trap material. Getters can also remove trace moisture but this diminishes their capacity to remove oxygen. Removing moisture with molecular sieve traps is more effective and will extend the lifetime for most getters. Molecular sieve traps exhibit excellent capacity for removing trace levels of moisture from carrier gas. Indicating molecular sieve traps are available, however, the indicating media is only sensitive to high levels of water and are not usually recommended. The 1/8" “S” type molecular sieve traps (cat.# 20686) are usually the best choice for chromatographers. They are packed, activated at oven temperatures of 300°C, sealed, and are ready to use. Because of their small size, they can be reconditioned in a GC oven when contaminated.

Hydrocarbon impurities in the carrier gas lines will result in detector instability, ghost peaks, and in extreme cases will result in column contamination. High levels of hydrocarbon impurities are not usually present in commercially available carrier gas sources, therefore most chromatographers do not find hydrocarbon traps necessary.

Hydrocarbon and solvent contamination is frequently removed using activated coconut charcoal. Since indicating hydrocarbon traps are not available for carrier gas lines, the analyst must note the date of installation and change the trap after approximately six months of use. Indicating traps, which are available from compressed air lines (1/8": cat.# 20637), should be used when oil lubricated air compressors are used as the FID air source.

What are the differences between indicating and non-indicating traps?

Some traps can indicate oxygen, moisture, or hydrocarbon removal by changing color. Indicating traps are made with glass housings to allow visual inspection of the color change. Although glass housings are fragile, they prevent oxygen from diffusing into the carrier gas and allow visual indication of the purifier activity level. Plastic materials are permeable to oxygen and are not recommended for any trap installed on a carrier gas line. Non-indicating traps are generally contained in a metal housing for strength and ruggedness. It is important that indicating oxygen traps are made with either glass or metal housings.

Indicating traps have an advantage over non-indicating traps since you can visually determine when to install a new trap. With non-indicating traps, it is impossible to accurately determine when the trap needs to be replaced. Non-indicating, high capacity traps should be installed prior to an indicating trap. When the indicating trap shows a color change, the non-indicating trap has been depleted and should be changed.

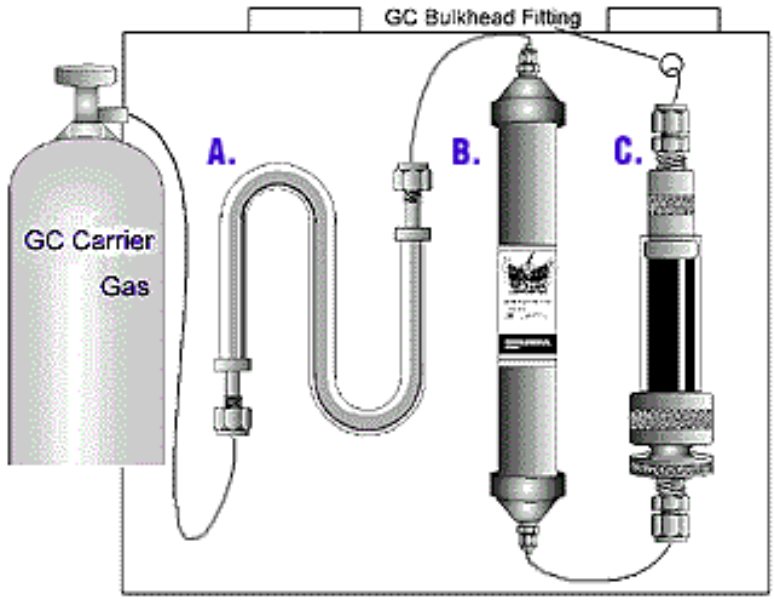
In what order should the traps be installed?

The order in which the traps are placed in the carrier gas flow path and their proximity to the GC is very important. **Figure 9** shows the recommended order for installing carrier gas traps. The molecular sieve trap should be placed first in line from the carrier gas tank. This trap will remove moisture, and prevent condensation in the carrier gas line. The hydrocarbon trap should be placed next, to prevent hydrocarbons from contaminating the oxygen trap. The oxygen trap should be placed closest to the GC bulkhead fitting. In general, traps should be installed on each GC as close to the bulkhead fitting as possible. Traps installed near the gas cylinder will not remove oxygen that may enter the carrier gas from leaky fittings downstream.

Traps should be installed vertically to avoid channeling. Channeling results from the packing material

settling which, when a trap is positioned horizontally, may allow carrier gas to pass through without sufficient interaction with the packing.

Figure 9 - *Trap recommendations, order, and installation tips.*



Traps Shown: (with cat. #s)

A. Molecular Sieve “S” Trap 20686

B. Hydrocarbon Trap 20627

C. High Capacity Indicating Oxygen Trap 20624

Hints for Using Traps:

- Install close to GC bulkhead fitting.
- Install vertically.
- If using a high capacity, non-indicating O_2 trap, use a low capacity indicating trap after it.

Trap Recommendations:

Carrier gas: oxygen, moisture, and hydrocarbon (optional)
Make-up gas: none required*
Air (FID, etc.): hydrocarbon (when trace oils are suspected)
H_2 (FID, etc.): none required
ELCD reaction gas: hydrocarbon

* Use oxygen and moisture traps on ECD make-up gas.

Should purifiers be used for other gases?

In addition to carrier gas, traps can also be used for other gases such as make-up and detector gases. Make-up gas for Flame Ionization Detectors (FID) does not require purification unless the FID is operated at high sensitivities. However, oxygen and moisture traps are highly recommended for make-up gas when operating sensitive detectors such as Electron Capture Detectors (ECD). The hydrogen reaction gas used for sensitive Electrolytic Conductivity Detectors (ELCD) also requires a hydrocarbon trap to remove trace impurities. These impurities can cause baseline instability and decrease the lifetime of the nickel reaction tube.

Many analysts use “house” compressed air from oil based compressors which can emit hydrocarbon vapors. Trace oil vapors can increase the background noise and contaminate FID detectors. A new compressed air trap (cat.# 20637) is available which reduces oil vapors to levels less than 5ppm. This trap changes color as oil vapors are detected to indicate when the trap is depleted. Because of its plastic body, the indicating compressed air trap is not recommended for carrier gas lines.

After running the gas supply lines to the lab bench, it is time to plumb the gas supply lines to the GC. Special care should be taken to connect the correct gas type to the appropriate fitting on the GC. The gas inlets for the GC will be located in different places depending on the make and model of your instrument. Refer to your installation manual for the location of the gas inlets.

Hints for ECDs: Always use high purity moisture traps like our 1/8" “S” trap (cat.# 20686) and 1/8" high purity oxygen trap (cat.# 20624) on ECD carrier and make-up gas lines.

Traps are Necessary for Split Vents to Prevent Breathing Air Contamination

Potentially hazardous or carcinogenic chemicals can enter the lab atmosphere through the split vent in a capillary GC. As much as 99% of the sample injected vents to the air where chemists working nearby breathe these pollutants. This problem is further magnified when multiple GCs are used in the same lab. Split vent traps, packed with charcoal, reduce the uncontrolled release of hazardous materials into the lab.

The best trapping material is activated coconut charcoal due to its tenacious trapping ability. Narrow 1/4" trap bodies cause increased back pressure on the inlet system and severely increase retention times. In addition, the excessive back pressure on the split vent outlet can cause the back pressure regulator to perform erratically when the solvent expansion pulse occurs. Therefore, a large trap body design maximizes the quantity of charcoal that comes in contact with the sample vapor stream without causing unreasonable back pressure. Trap bodies made from solvent resistant plastics either crack or leak with continuous solvent exposure. A glass trap body provides the best resistance and longevity from repeated solvent injections.

The trap capacity determines the number of injections that can be performed before solvent breakthrough occurs. High capacity traps provide protection for thirteen hundred injections or fifty days if one analysis is performed per hour.

Leak Checking

Many GC problems can be avoided by leak checking the system during the plumbing process. Loss of GC gases, reduced trap lifetime, damage to capillary columns, and increased detector maintenance will result if a leak is present. Leak checking the instrument before column installation and conditioning prevents column degradation indicated by high bleed and short lifetime. Irreversible damage can occur if a column is exposed to oxygen at high temperatures. Additionally, some detectors (for example, an ECD) are very sensitive to oxygen and can easily be damaged by oxygen exposure. Leak checking should be performed from the tanks to the GC, including all the fittings inside the GC. The GC fan should be turned off during leak checking. Next, check the external fittings along the carrier gas line for leaks. Leak detectors such as the Restek Leak Detective™ (110 volts: cat.# 21607, 220 volts: cat.# 21609), Gow-Mac Leak Detector (cat.# 20130), or Compact Leak Detector (cat.# 21605), detect minute traces of helium or hydrogen without contaminating the system. Never use liquid leak detectors that contain soap or surfactants. Liquids can be drawn inside the fitting at the site of a leak by the Venturi effect and contaminate the system.

If a thermal conductivity leak detector is not available, a pressure decay test can also be used to find major gas leaks. To perform a pressure decay test, first cap off all possible gas outlets including the injection port and the detector fittings. Next, shut off the gas supply at the cylinder. In a leak free system, the line pressure observed at the two-stage regulator will hold constant for 15 minutes or longer. A rapid loss of pressure indicates that leaks are present. If this is the case, isolate smaller sections of the plumbing by capping off the line closer to the cylinder and recheck the pressure drop after closing off the gas supply. Repeat this process until the leak is found, then retest the entire system to ensure pressure is maintained.

Back:Leak Checking

Instrument Logbook

Another step in setting up a GC is creating an instrument logbook. Detailed documentation is crucial to the operation of any testing lab, therefore, record all of the steps involved in installing the new GC in your laboratory. Additionally, develop a GC maintenance schedule and document all maintenance performed (**Figure 10**). A routine GC maintenance schedule will minimize system troubleshooting, increase sample throughput, and improve analytical accuracy. A good GC maintenance program focuses on the inlet, capillary column, detector(s), oven calibration, traps and purifiers, and leak checking. GC documentation should also include analytical information (number of analytical sequences run, type and number of samples analyzed, appearance of sample) and any troubleshooting or repair work performed on the instrument. Documentation and routine maintenance will make future troubleshooting efforts less time consuming.

Figure 10:

GC & Column Documentation	GC1	GC2	GC3	GC4	GC5	GC6	GC7	GC8
Column Information								
Column installed								
Column catalog #								
Column serial #								
Date of installation								
Analysts initials								
GC #								
Ferrules used with column								
Carrier gas used								
Linear velocity								
Dead time								
Date column removed from GC								
Analyst's initials								
Columns sealed with								

Analytical Information								
# of standards run								
Type of standards run								
# of samples run								
Type of samples run								
Solvent sample standards were diluted with								
Sample concentration								
Injection size								
Septa used								
Inlet sleeve								
Packing materials?								
Injector temperature								
Injection mode								
Split vent flow								
Septum purge								
Splitless hold time								
Routine GC Maintenance	**GC1**	**GC2**	**GC3**	**GC4**	**GC5**	**GC6**	**GC7**	**GC8**
Injector								
Replace septa								
Replace sleeve								
Replace column ferrule								
Replace injector fitting								
Replace inlet seal								
Replace o-ring								
Clean needle guide								
Clean needle disk								
Clean inlet seal								
Clean injection port								
Clean septum nut								
Leak check								
Detector								
Replace detector sleeve								
Replace detector fitting								
Replace detector ferrules								
Clean detector								
Clean detector port								
Clean collector								
Leak check								
Instrument								
Replace GC traps								
Replace chemical filters								
Leak check all fittings								
Check gas flow rates								
Check make-up gas flows								
Miscellaneous								
Sweep oven								
Special problems								