

Don't Touch That Column Read Me First

GC Capillary Column Installation Manual



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Chromatography Associates capillary columns are manufactured using the highest quality material and the most advanced techniques. They are all individually tested to insure compliance with our high Quality Standards. They arrive in your lab in perfect condition. The rest is up to you.

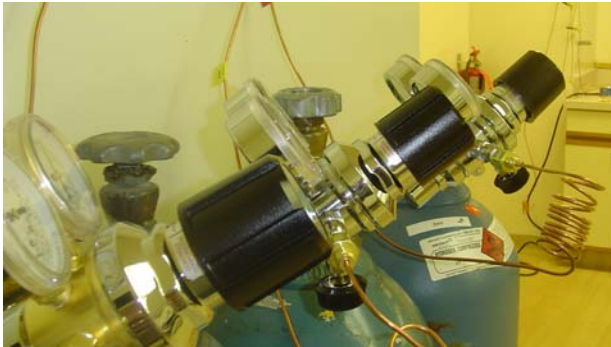
Before you even remove the column from the box there are steps that should be taken to insure the best performance and the longest lifetime from your column. Oxygen and water present in the carrier gas is the easiest way to damage a capillary column. So here are the important steps to avoid this problem.

Examine the **Carrier Gas Pathway** from the gas source all the way to the column.

Carrier Gas

Carrier gas quality should be 99.99% or better. We still recommend the use of moisture, hydrocarbon and oxygen carrier gas purifying traps.

Regulators



Tank regulators should be Dual Stage to minimize fluctuations of the outlet pressure as the tank pressure decreases. Any regulator in a carrier gas line must have a Stainless Steel diaphragm. This prevents the migration of water and oxygen into the carrier gas, which would occur with polymeric



diaphragms. Don't forget to use Teflon tape on the pipe threads of the regulator.

Carrier gas lines



All tubing used for carrier gas lines must be clean. Pre-cleaned tubing is available from most vendors. Stainless steel is recommended although copper may be used. Just remember copper tubing is more subject to cracking or breaking.

Connections



Connection should be made with compression fitting (Swagelok , Parker etc.). All connection must be leak free. Due to Venturi effects any leak will not only vent carrier gas but also draw in ambient air and hence water and oxygen.

Purifiers



Purifiers are a must for optimum performance. They should be located as close to the GC as possible. This minimizes the number of fittings after the purifiers and possible leaks. The purifiers should be in series in the following order: Moisture, Hydrocarbon, Oxygen and optionally an additional Indicating Oxygen trap.

Septa



Replace the septa at the beginning of any new analysis and when installing a new column. Degraded septa are a source of background interferences and another means of water and oxygen getting to the column.

Liners

Liners should be inspected and cleaned or replaced. Keep in mind that liners should be deactivated whenever active components are present in your samples. Some cleaning techniques such as acidic or basic washes will remove the deactivation. Replacement is often the least complicated solution.



O-rings



Inspect the o-rings or ferrules used to seal the liners in place. They are another source of leaks. If the specific injector system has other critical seals or sealing disks,

these should also be inspected for contamination and damage.

Now you can take the column out of the box.

Heated Zones



Cool the oven, injector and detector to below 100°C. For obvious reasons this minimizes burnt fingers. Remember that once the capillary column ends have been cut air will begin to diffuse into the column ends. If these air filled ends are installed in a heated injector or detector then several inches of each end will be exposed to high heat in the presence of oxygen. Essentially you will have burnt the column ends before you even begin analyzing samples.

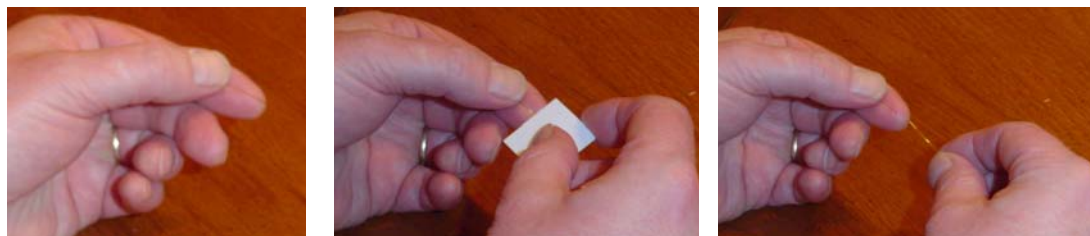
Ferrules and their behavior



Capillary ferrules are commonly made from Vespel, Vespel/graphite mixtures or graphite. Ferrules using Vespel will shrink somewhat after they are subjected to high temperatures. Therefore after installing a new capillary and running the column up for a brief conditioning, you should cool the oven and retighten

the capillary connections.

Cutting fused silica tubing



Capillary tubing is composed of the actual fused silica tubing surrounded by a coating of polyimide. This polyimide coating protects the fused silica from abrasion and from water. When you cut capillary tubing you are actually cutting through the polyimide and scoring the fused silica. If you actually tried to cut the tubing you would end up crushing it and scattering broken pieces into the end. Pieces of broken tubing in the sample pathway will cause tailing of even non-active sample components. The simplest way to cut the columns is to place the column end along the end of your index finger, gently cut through the polyimide

and score the column with a ceramic scribe. If the column falls apart immediately you have probably scored too hard and crushed the column. Try again. When properly done you can gently flick the cut end downward and the cut is complete. Always inspect the column end to see how good you've done. A magnifier is helpful for eyes over 40. It's a good idea to practice on an old column. Soon it will be like riding a bicycle.

Inlet liners

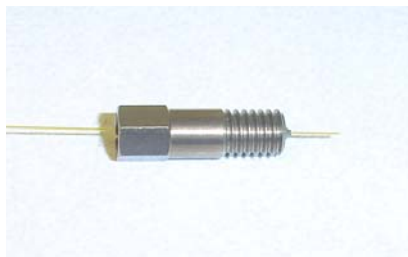
In the GC the first thing your sample interacts with is the inlet sleeve or liner. It's a good idea to minimize the interaction, otherwise analyte adsorption can lead to poor chromatography and misleading results. There are two basic types of analytes: non-active and active. Non-active analytes would be hydrocarbons, and active analytes would be other compounds containing groups that can chemically interact with the sleeve or contaminants. Tailing of hydrocarbon materials does not mean a chemical interaction. It means a disruption of the laminar flow of your analyte stream. In other-words, there is an obstruction or particle in the carrier gas pathway. The most likely source of this would be an improperly cut column end. Reinstallation of the column would be advised. Temporary or transient interaction of an active analyte with the sleeve or a contamination present in the sleeve is clearly evident by tailing of these peaks. This usually results from active sites in the inlet liner due to contamination or improper deactivation. Replace, deactivate or change the liner.

Glass wool



Glass wool inserted into a straight sleeve is a great inexpensive alternative to costly split sleeves. Make sure the wool you purchase is deactivated and you don't contaminate it when you handle it.

Install the column in the injector



Pay close attention to the GC manufacturers recommended insertion distance. Remember always to cut 5-10 mm from the column end whenever a ferrule is placed on the column. There is always the possibility of scraping off a piece of the ferrule and getting it inside the column.

Establish carrier gas flow



It is always a good idea to confirm that there is flow. One easy way is to dip the column end into a vial of solvent. A stream of bubbles indicates good flow. Purge the column with carrier gas for a minimum of 15 minutes.

Heat the Injector and Detector

Once the column has been purged heat the injector and detector. Remember not to exceed the upper isothermal temperature of the column.

If there is a need to exceed the upper isothermal temperature limit of the column with the injector and/or the detector, use 1 meter guard columns on the front and back of the column.

Methane test



The **methane test** should be performed to establish the proper linear velocity of the carrier. Simply inject a methane standard at either your isothermal test temperature or the GC temperature when your most difficult separation occurs. Determine the linear velocity in cm/sec by dividing the column length in cm by the retention time of the methane in seconds. Optimum values are 20-22cm/sec for helium and 38-40cm/sec for hydrogen. Nitrogen is not recommended as a carrier gas due to long dead times, longer isothermal analyses and poor performance during programmed runs. Note that if

the peak symmetry for the methane is poor (tailing) the column should be removed and reinstalled. Tailing methane peaks usually indicate a broken column end or a fragment of column (or ferrule) in the carrier gas pathway.



Call or email for Help
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Chromatography Associates
Capillary Columns are
manufactured in
State College, PA USA

