



## Restek Capillary Column Installation Guide

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### Why Read This Guide?

This guide helps chromatographers prevent costly mistakes that could potentially damage a Restek column. Section I describes a brief capillary column installation procedure. Section II provides in-depth installation information necessary to ensure the best performance from your column. The third section provides operational hints that will help extend column lifetime and the last section outlines warranty and return procedures.

### Have Questions or Need Help Troubleshooting?

Whether your problem is straightforward or complex, contact our [technical service](#) chemists online, or call 800-356-1688, ext. 4, Monday through Friday, 8am-6pm EST.

Satisfaction Guaranteed! Our quality is the best and we back our claim! Please contact [Customer Service](#) or call 800-356-1688 (or one of our [distributors](#)) if any Restek product does not meet your expectations or is not better than or equivalent to a competitive product.



## Restek Capillary Column Installation Guide

### Section I: Brief Installation Procedure

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The following procedure is a quick, easy, trouble-free sequence for installing a capillary column. For more in-depth installation information, refer to [Section II](#).

1. Cool all heated zones.
2. Replace spent oxygen and moisture traps.
3. Clean and/or deactivate injector and detector sleeves if necessary.
4. Replace critical injector and detector seals.
5. Replace septum.
6. Set make-up and detector gas flow rates.
7. Carefully inspect the column for damage or breakage.
8. Cut 10 centimeters from each end of the column.
  - o use a sapphire scribe (cat.# 20115) or ceramic scoring wafer (cat.# 20116) to cut fused silica capillary columns.
  - o use the serrated edge of a ceramic scoring wafer (cat.# 20116) or the edge of a sharp file to cut metal capillary columns.
9. Install a nut and ferrule on each end of the column.
10. Cut an additional 10 centimeters from each end of the column to remove ferrule fragments.
11. Mount the capillary column in the oven using a bracket that protects the column from becoming scratched or abraded.
12. Insert column the appropriate distance into the inlet as indicated in the instrument manual.
13. Set the approximate column flow rate by adjusting the head pressure to the value listed on the test chromatogram included with the column.
14. Set split vent, septa purge, and any other applicable inlet gases according to the instrument specifications.
15. Confirm the flow by immersing the column outlet in a vial of solvent (acetone or isopropyl alcohol).
16. Insert column the appropriate distance into the detector as indicated in the instrument manual.
17. Check for inlet and outlet leaks using a thermal conductivity leak detector (cat.#'s 21607, 20130, or 21609). Do not use soaps or liquid-based leak detectors or the column may be damaged.
18. Set injector and detector temperatures. Turn the detector on when the temperatures have equilibrated. Caution - do not exceed the phase's maximum operating temperature!
19. To set the proper dead time (linear velocity), inject methane (cat.# 20197) or a non-retained substance compatible with the detector being used.
20. Verify system integrity by checking the dead volume peak. It should not tail.
21. Condition the column at its maximum operating temperature to stabilize the baseline. (See the test chromatogram included with the column for the maximum

temperature.)

22. Set oven to appropriate temperature and inject methane (cat.# 20197) or an appropriate unretained substance, again to set the proper linear velocity.
23. Inject a duplicate of the original test mixture or your specific test mixture to confirm proper installation, system, and column performance.
24. Calibrate the instrument and inject samples.

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## Restek Capillary Column Installation Guide

### Section II: In-depth Installation Information

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The following section provides in-depth information on instrument preparation procedures for installing and operating fused silica and stainless steel capillary columns.

#### I. **Instrument preparation**

- A. Gas purification
- B. Carrier gas selection
- C. Using hydrogen as a carrier gas
- D. Flow regulated/back pressure pneumatic systems
- E. Head pressure regulated pneumatic systems
- F. Injector maintenance
- G. Protection against dirty samples
- H. Replacing critical seals
- I. Changing septa
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#### II. **Column Mounting and Installation**

- A. Installation preparation
- B. Inlet installation
- C. Establishing flow
- D. Good operating practice
- E. Outlet installation
- F. Leak-checking techniques

#### III. **Setting Optimum Flow Rates**

#### IV. **Confirming Installation Integrity**

- A. Dead volume peak shape test
- B. Solvent peak shape test

#### V. **Conditioning**

#### VI. **Test Mixtures**

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### I. Instrument Preparation

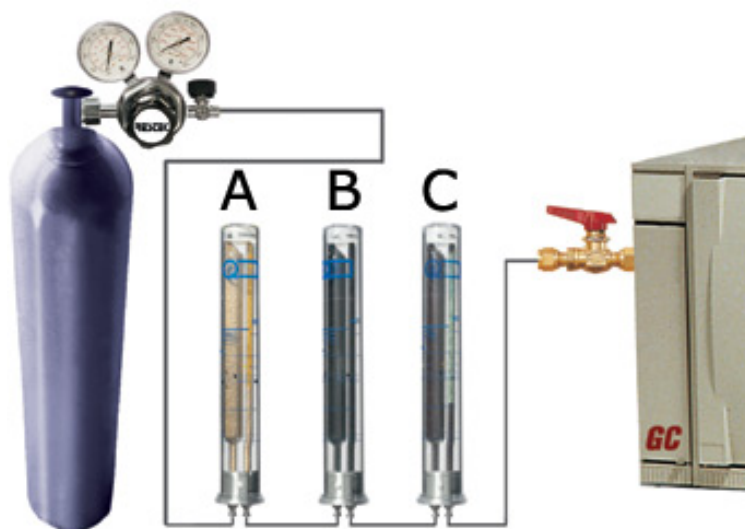
#### Gas Purification

Make sure the carrier gas is clean. The carrier gas **MUST** contain less than 1ppm of oxygen, moisture, or any other trace contaminants to prevent column degradation, decreased column lifetime, and increased stationary phase bleed. Contaminants such as trace hydrocarbons cause ghost peaks to appear during temperature programming and compromise the validity of analytical data. Make-up gas should also be contaminant-free or baseline fluctuations and excessive detector noise can occur. Detector gases, such as hydrogen and compressed air, should be free of water and hydrocarbons or excessive baseline noise can result.

We highly recommend installing a High Capacity Indicating Oxygen Trap on both the carrier and make-up gas lines. Because moisture reacts with most oxygen traps, we suggest installing a Moisture Trap in front of the oxygen trap. *(Install purifiers as closely as possible to the GC's bulkhead fitting, not system-wide. If purifiers are installed system-wide, a leaky fitting downstream of the trap could allow oxygen and moisture to*

enter the gas stream and degrade column performance.) The Moisture Trap can also be used on the FID air line or the ECD make-up gas line to eliminate noisy, rolling baselines when operating at high detector sensitivities. If hydrocarbon contamination is suspected, install a Hydrocarbon Trap between the moisture and oxygen traps. To prevent spontaneous breakage, coil the line leading to and from the purifiers to relieve strain and isolate instrument vibrations.

*A note on equipment:* Because oxygen, moisture, and elastomeric contaminants can migrate through rubber or elastomeric diaphragms and enter the carrier gas, all regulators should be equipped with stainless steel diaphragms.



#### Traps shown:

A. Moisture Trap:

Super-Clean™ Ultra-High Capacity Moisture Filter ([cat.# 22028](#))

B. Hydrocarbon Trap:

Super-Clean™ Ultra-High Capacity Hydrocarbon Filter ([cat.# 22030](#))

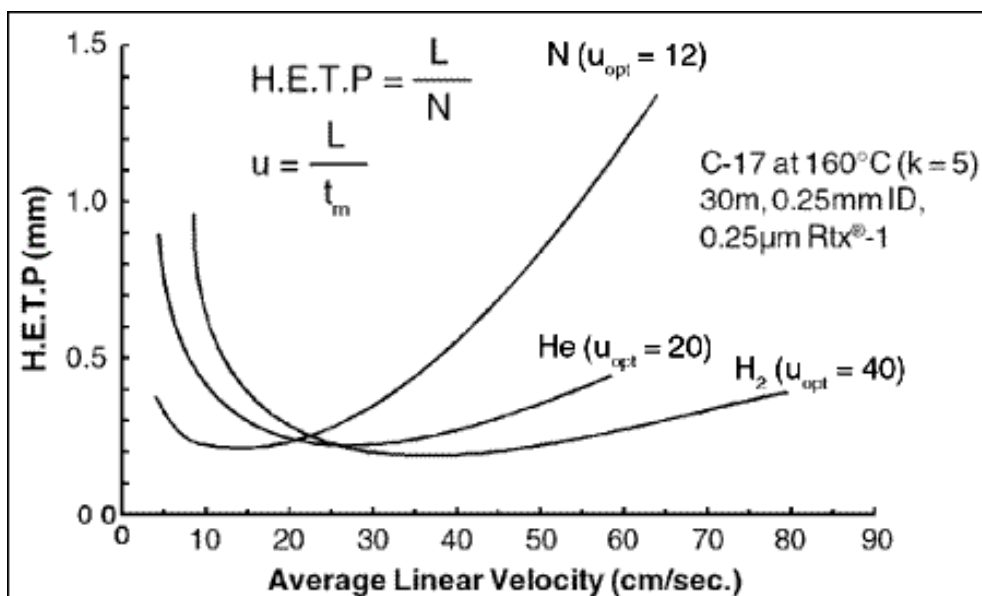
C. High Capacity Indicating Oxygen Trap:

Super-Clean™ Ultra-High Capacity Oxygen Filter ([cat.# 22029](#))

#### Carrier Gas Selection

A fast carrier gas that exhibits a flat van Deemter profile is essential in obtaining optimum capillary column performance. Because capillary columns average over 30 meters in length (compared to 2 meters for packed columns), a carrier gas that minimizes the effect of dead time is important. In addition, because capillary columns are head pressure controlled, not flow-controlled like most packed columns, the carrier gas flow decreases by 40 percent when programming from ambient to 300°C. Therefore, a carrier gas that retains high efficiency over a wide range of flow rates is essential for obtaining good resolution throughout a temperature-programmed analysis.

**Figure A** shows the van Deemter profile for hydrogen, helium, and nitrogen carrier gases. The curves were generated by plotting the Height Equivalent to a Theoretical Plate (H.E.T.P., the length of the column divided by the total number of theoretical plates) against the column's average linear velocity. The lowest point on the curve indicates the carrier gas velocity at which the highest column efficiency is reached.



Hydrogen is the fastest carrier gas ( $u_{\text{opt}}$ : 40cm/sec.) and exhibits the flattest van Deemter profile. Helium is the next best choice ( $u_{\text{opt}}$ : 20cm/sec.). The head pressures at optimum flow rates are similar for hydrogen and helium because hydrogen has half the viscosity and double the linear velocity of helium. Nitrogen's performance is inferior for capillary columns and is usually not recommended because of the slow optimum linear velocity ( $u_{\text{opt}}$ : 12cm/sec.) and steep van Deemter profile.

**Figure B** compares hydrogen to helium in an isothermal analysis using optimum linear velocities. The resolution is similar, but the analysis time is reduced by 50 percent when using hydrogen. However, helium does improve the separation of very low boiling or early eluting compounds since they allow more interaction with the stationary phase. Temperature programming usually provides similar analysis times between hydrogen and helium since the elution of most compounds strongly depends on the oven temperature. Therefore, the savings in analysis times are not as noticeable as when using isothermal oven conditions.

**Figure C** illustrates that hydrogen is only slightly faster than helium when both carrier gases are operated under the same temperature-programmed conditions. Also, note that helium improves the resolution of the early eluting compounds (peaks 1 & 2).

### Exert Caution when using Hydrogen as a Carrier Gas

Hydrogen is explosive when concentrations exceed 4 percent in air and should only be used by individuals who have received proper training and understand the potential hazards. Proper safety precautions should be taken to prevent an explosion in the oven chamber. Some gas chromatographs are designed with spring loaded doors, perforated or corrugated metal oven chambers, and back pressure/flow controlled pneumatics which minimize the hazards when using hydrogen carrier gas. Additional precautions include:

- Frequently checking for leaks using a thermal conductivity leak detector (cat.# 22451).
- Minimizing the amount of carrier gas that could be expelled in the oven chamber if a leak were to occur by installing a needle valve, restrictor, or flow controller prior to the carrier inlet bulkhead fitting (only necessary for head pressure controlled systems).
- Purging an inert gas (N<sub>2</sub>) into the oven chamber to displace oxygen and prevent an explosive atmosphere from forming.

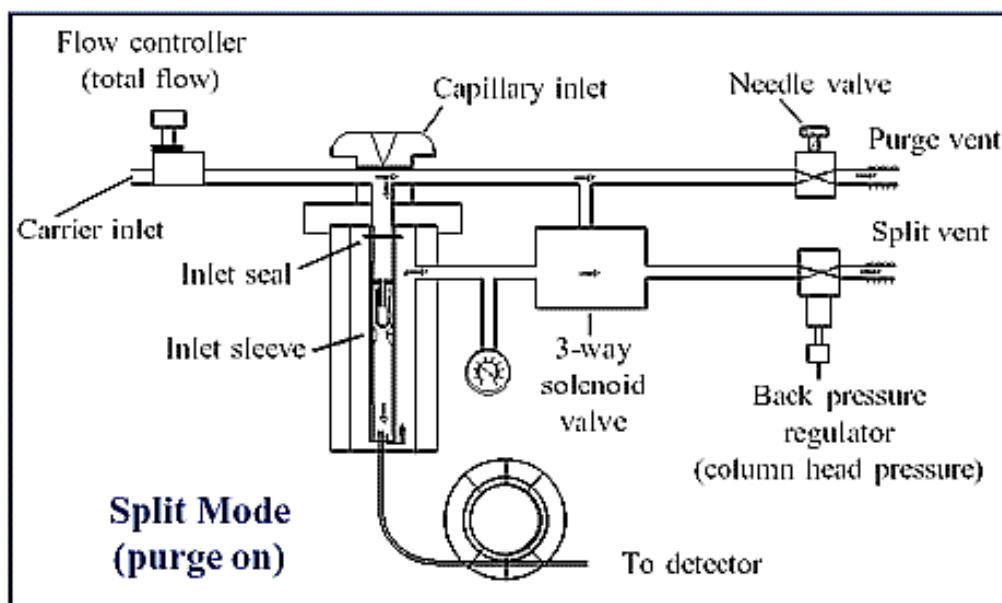
Hydrogen is expelled from both the split vent and septum purge when it is used as a carrier gas. Because of hydrogen's fast diffusivity, an explosion in a laboratory setting is highly unlikely. However, a spark from static electricity can ignite the hydrogen exiting from a septum purge or split vent which could cause a flame. Precautions to minimize

the problems with hydrogen exiting the split vent or septum purge include:

- Plumbing the exit lines to a hood or venting the escaping gas outside.
- Plumbing the lines to exit into a vial of water.
- Plumbing the exit lines to a position where analysts could not get burned if inadvertent ignition occurred.

### Flow Regulated/Back Pressure Pneumatic Systems

**Figure D** illustrates a flow regulated/back pressure system commonly used for split/splitless inlets (HP-5890, Varian 3300, 3500, & 3600 GCs). A flow controller positioned upstream of the injector controls the total amount of carrier gas that is expelled from the split vent, septum purge, and column (knob labeled "Total Flow" on HP 5890 GCs). The back pressure regulator (knob labeled "Column Head Pressure" on HP 5890 GCs) stops or reduces the flow from exiting the split vent until the desired column head pressure is reached. The flow controller provides the increase of pressure necessary to meet the requirements of the back pressure regulator. It is the back pressure regulator, located downstream of the split point, that actually controls the capillary column flow rate. One of the primary benefits of a flow controlled/back pressure regulated system is that adjustments to the capillary column flow rate (via head pressure changes) do not affect the amount of carrier gas exiting the splitter vent. Once the desired split vent flow rate is achieved, the flow controller setting should not have to be changed when installing different columns.

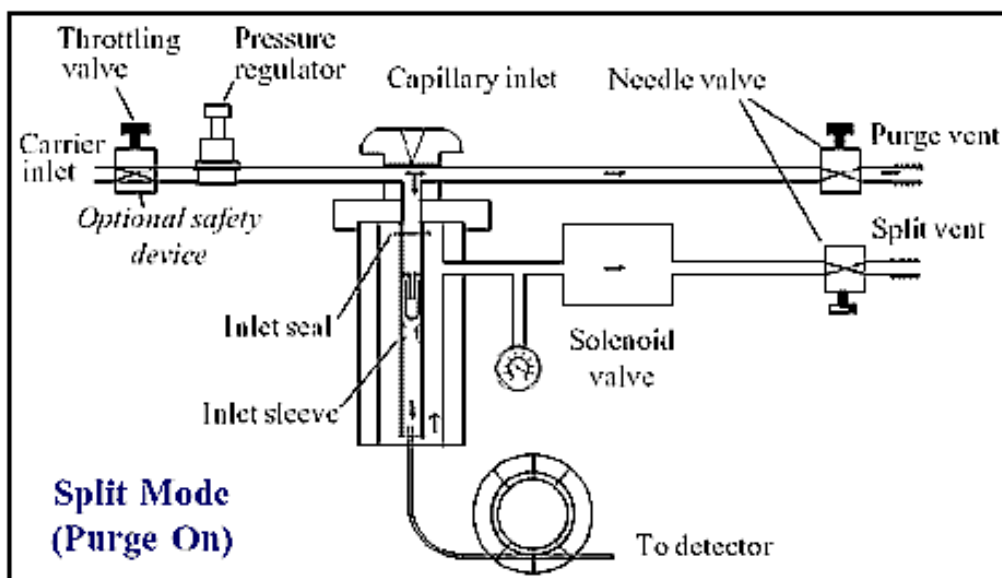


Flow regulated/back pressure systems prevent a drastic carrier gas loss that can happen if an inlet fitting or column leak occurs. Leaks are indicated by a failure to obtain the proper operating pressure with the capillary column. A common mistake is to increase the total system flow by turning the flow controller up when a proper head pressure cannot be obtained rather than checking for inlet leaks.

### Head Pressure Regulated Pneumatic Systems

**Figure E** illustrates a head pressure regulated inlet system used in some split/splitless inlet systems (PE Autosystem, 8500, and Shimadzu 9A and 14A GCs). A single-stage pressure regulator is used to control the flow rate in the capillary column by increasing or decreasing the upstream inlet pressure. The split vent and septum purge flow rates are controlled by a needle valve or variable restrictor located downstream of the pressure regulator. Head pressure systems require adjustment of the needle valve controlling the septum purge or split vent every time a change is made in the column's head pressure.





We recommend placing a throttling valve (needle valve or restrictor) on the carrier gas inlet bulkhead fitting of pressure-regulated systems to prevent a catastrophic carrier gas loss should an inlet leak occur. If several GCs are attached to a common carrier gas source, a leak in one GC could drain the carrier gas from all other GCs, causing a loss of flow and subsequent damage to all capillary columns in the entire system. To prevent this from happening, limit the flow of carrier gas to each gas chromatograph (via a throttling valve) until it matches the flow requirements of the inlet system. This can be detected when the column's head pressure starts to decrease if the throttling valve is closed any further.

### Injector Maintenance

Perform injector maintenance prior to installing a capillary column. Periodic maintenance is required after installation, depending on the number of injections and the cleanliness of the samples. Maintenance includes cleaning and deactivating inlet sleeves, replacing critical inlet seals, and replacing the septum. Review the instrument manual inlet diagram prior to disassembly.

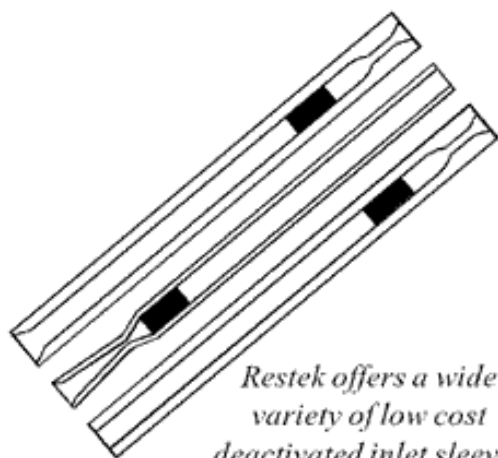
### Cleaning and Deactivating Injector Sleeves

Don't install a new Restek column with a dirty injector sleeve! For optimum column performance, the inlet sleeve needs to be free of septum particles, sample residue, and ferrule fragments. Use deactivated inlet sleeves when analyzing samples with active functional groups or compounds prone to decomposition or adsorption onto untreated glass surfaces.

If the sleeve is deactivated and not excessively dirty, cleaning with organic solvents will not affect the integrity of the deactivation layer. First, use non-swelling organic solvents such as methanol or isopropyl alcohol to remove septum particles that adhere to the sleeve wall. Next, use solvents such as pentane, methylene chloride, or toluene to remove sample residue. Nylon tube brushes and pipe cleaners (cat.# 20108) are ideal for cleaning sleeves. Do not use laboratory detergents, acids, or bases to clean sleeves because they will remove the deactivation layer and require sleeve resilanization.

Sleeves that are very dirty or contain pyrolyzed residue can be difficult to clean.

Heating borosilicate or quartz sleeves overnight in a muffle furnace at 550°C will remove most contaminants. Etching with a 1:1:1 mixture of hydrofluoric acid, sulfuric acid, and deionized water for ten seconds is also very effective. However, heating to



*Restek offers a wide variety of low cost deactivated inlet sleeves to fit most instruments.*



550°C or using the acid-etch procedure will remove the deactivation layer and require sleeve resilanization. Caution - Exert extreme caution when using hydrofluoric acid. Hydrofluoric acid can cause severe burns and nerve damage if it is ingested, inhaled, or brought into contact with the skin. Only properly trained professionals equipped with the appropriate safety devices should attempt to handle strong acids.

Most new sleeves received from instrument manufacturers are not deactivated. Before optimum column performance can be achieved, sleeves must be deactivated. Restek has developed a unique, high temperature silanization procedure for sleeve deactivation that ensures a highly inert surface. These sleeves can be purchased from Restek at a cost below many instrument manufacturer's cost.

Refer to Restek's Chromatography Products Catalog to obtain the part number for your specific instrument. For more information on inlet sleeves, request a copy of Restek's bulletin Operating Hints for Split/Splitless Injectors or Guide to Direct/On-column Flash Vaporization Injection.

### Protection Against Dirty Samples

Sleeve packing materials such as fused silica wool (cat.# 20790), glass wool (cat.# 20789), or fused silica beads (cat.# 20791) act as filters when analyzing samples containing high molecular weight residue or particulates. However, both wool and beads greatly increase the surface area that the sample contacts and can be a source of adsorption or breakdown. It is critical that the wool or beads be properly deactivated. If you plan to use wool, be careful inserting it into the sleeve because active sites can be created as the fibers break. We do not recommend using packings coated with stationary phases. (For more information on protection against dirty samples, request a copy of Restek's bulletin A Guide When Injecting Dirty Samples.) Alternative sleeve designs that minimize sample interaction with non-volatile residue are also available.

### Replacing Critical Seals

Replace the critical seal prior to installing an inlet sleeve (see instrument manual for seal location). Most capillary injection ports use a rubber o-ring or graphite ferrule to seal the sleeve inside the injection port body. The seal must fit tightly around the sleeve to prevent the carrier gas from leaking around the outside of the sleeve. If your GC uses a ferrule as the inlet seal, always pre-swage the ferrule to fit the sleeve before tightening it in the inlet (especially Varian inlets).



### Changing Septa

Always use a high quality, low bleed septa. We recommend replacing the septum frequently to prevent leaks and fragmentation. Otherwise, multiple injections and continuous exposure to a hot injection port will decompose the septum, causing particles to fall into the sleeve. Septum particles are a potential source of ghost peaks, loss of inertness, and carrier gas flow occlusion as shown in **Figure F**. It is best to install a new septum at the end of an analytical sequence so that it can condition in the injector and reduce the incidence of ghost peaks. Always use clean forceps when handling septa to avoid contamination.

### Setting Detector and Make-up Gas Flow Rates

Confirm that the make-up gas, detector fuel, and oxidant flow rates are set according to the instrument's specifications (**Table I**, below). Make-up gas flow rates set too low will cause tailing solvent peaks, baseline disturbances, decreased sensitivity, and detector noise. Some instruments do not have leak-tight detector cavities and require flow rate verification before the column is installed into the detector. However, for GCs with leak-tight detector cavities, it is usually easier to check detector and make-up gas flow rates after the column is installed. We highly recommend using the Restek Flowmeter 6000 (cat.# 21622).

**Table I Typical FID Flow Rates**

Instrument	H <sub>2</sub> (ml/min.)	Make-up (ml/min.)	Air (ml/min.)
Hewlett-Packard	30	20	400
Varian	30	20	300
Shimadzu	30-60	40	500
PerkinElmer	45	--	450

## II. Column Mounting and Installation



**Do not remove the high-temperature string holding the fused silica capillary column inside the Restek cage.** The high-temperature string is capable of withstanding more than 400°C and does not decompose during continuous operation at oven temperatures used in capillary chromatography.

When hanging the column on the oven support rod, be careful that fused silica tubing does not contact any metal parts. Stainless steel

columns can be placed directly on the oven support rod. If there is not an oven support rod, one can be made by inserting a temperature resistant pegboard hook into the corrugated oven wall or by hanging a 1/16-inch "S" hook from the oven ceiling. Be careful not to damage the oven thermocouple or interfere with the fan operation when installing homemade brackets.

Position the column so that it is midway between the injector and detector. This reduces thermal gradients and enhances retention time reproducibility. Uncoil one or two loops of tubing. When using fused silica columns, be careful not to scratch the column surface against the metal cross bars when removing loops. This abrasion of the polyimide coating could lead to spontaneous breakage.

**Caution** - When removing loops from 0.53mm ID columns, pull the tubing from the cage at the point with the widest gap between the metal crossbars. Avoid sharp bends that will break the tubing.

### Choosing Ferrules



Graphite or Vespel®/graphite ferrules are used to seal the column to the injector and detector in capillary gas chromatography. Both ferrule types have advantages and disadvantages. Graphite ferrules are the easiest to use, and they are leak-free, universal

for most systems, and preferred by most beginning capillary chromatographers. Because graphite ferrules are soft, they easily conform to column outside diameters and different types of instrument fittings. However, they can flake or fragment upon removal, causing particles to lodge in the injector or detector sleeves, and they will not hold a seal under vacuum. Vespel®/graphite ferrules are hard and they must match the column and fitting dimensions closely to seal properly. In addition, because Vespel®/graphite ferrules can deform during initial heating, they need to be re-tightened or leakage will occur. Vespel®/graphite ferrules do not fragment, can be reused many times, and are preferred by mass spectroscopists since they do not contaminate the ion source with particles and maintain their seal under vacuum. In all cases, it is best to choose a ferrule that fits snugly or is slightly larger than the capillary tubing OD (see table below). This minimizes the need for excessive torque to properly seal the ferrule to the column.

Nominal Tubing ID	Nominal Tubing OD			
	MXT		Fused Silica	
0.05mm	---	---	0.363mm	+/-0.012mm
0.10mm	0.23mm	+/- 0.0254mm	0.363mm	+/-0.012mm
0.15mm	0.41mm	+/- 0.0254mm	0.363mm	+/-0.012mm
0.18mm	0.36mm	+/- 0.0254mm	0.34mm	+/-0.01mm
0.25mm	0.41mm	+/- 0.0254mm	0.37mm	+/-0.04mm
0.28mm	0.56mm	+/- 0.0254mm	---	---
0.32mm	0.41mm	+/- 0.0254mm	0.45mm	+/-0.04mm
0.53mm	0.74mm	+/- 0.0254mm	0.69mm	+/-0.05mm
0.75mm	0.93mm	+/- 0.0254mm	---	---

Installation Preparation

Cut each column end squarely, approximately 10 centimeters from the end seals. To obtain a square cut with fused silica columns, place the column end against the forefinger and score the polyimide layer lightly and rapidly with a sapphire scribe (cat.# 20115) or a ceramic scoring wafer (cat.# 20116). Score only one side of the column. Point the column end down to prevent polyimide or fused silica shards from falling inside, and quickly flick the column just above the score.



Proper and improper fused silica cuts.

Cut metal capillary tubing by scoring the tubing wall (without cutting completely through) with the edge of a sharp file or ceramic scoring wafer. Wipe any filings off the tubing and bend it away from the score. Once the score opens, bend the tubing in the opposite direction (toward the score) until it snaps into two pieces. If the hole is not round or there is a burr on the tubing, try the procedure again. The flat side of a ceramic scoring wafer can be used to polish or round the column end into a smooth conical shape. We do not recommend using high speed wheels or grinders to cut the metal tubing since they may introduce metal filings into the tubing or ruin the polymer near the cut from the high temperatures created.



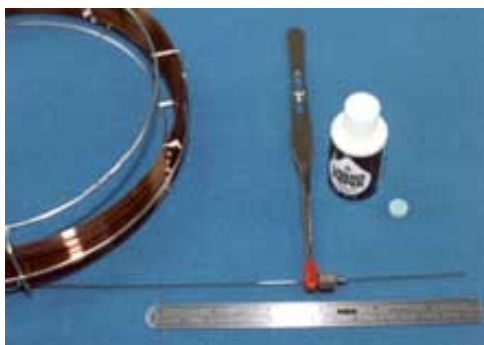
Proper and improper MXT® column cuts.

Next, install the nut and ferrule to the inlet in the manner described in the instrument manual. Use a pin vise and drill (cat.# 20103) to enlarge the ferrule ID if it does not slide easily onto the column. Prevent shards from falling into the column bore by pointing the column end down when installing the ferrule. Slide the connecting nut and ferrule approximately 20cm down the length of the column to make installation easier. Cut an additional 10cm from the column end after the nut and ferrule have been installed to remove any ferrule fragments that might have been forced into the column bore. Examine the quality of the cut with a small 10x pocket magnifier (cat.# 20124) and make sure that the cut is square. Jagged silica edges or exposed polyimide cause adsorption and tailing peaks, so it is very important that the column ends are cut uniformly. It may take several times, but once a square cut has been obtained, proceed with the installation. (Use an old column to practice making consistently square cuts.)

### Inlet Installation

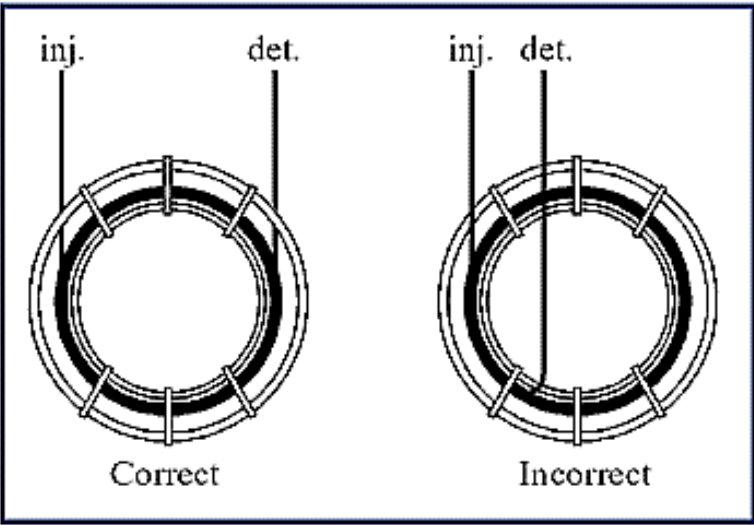
Consult the instrument manual to determine the correct insertion distance for the injector. It is important to install the column at the exact distance recommended by the injector manufacturer or poor peak symmetry and quantitation could occur. Lay the column end beside a ruler and position the nut and ferrule to the exact distance required for installation. Next, mark the insertion distance. This can be done one of three ways: with a pair of Slide-Lok Tweezers (cat.# 20101), with typewriter correction fluid, or by positioning a septum on the column to hold the nut and ferrule. If using a septum, slide it away from the nut after the fitting is tightened and leave it in the oven cavity while performing the analysis.

Gently insert the column end into the inlet fitting, making sure that the end is not crushed or scraped against the metal injection port fittings. While maintaining the correct distance, use a capillary wrench to tighten the nut approximately one-half turn past finger-tight until the column is held firmly. The ferrule is tight if the column cannot be pulled from the fitting while applying gentle pressure.



*Various ways to mark the insertion distance*

Make sure the fused silica tubing is not sharply bent when installing the column (**Figure G**). The tubing should gently bend from the cage to the fitting in angles greater than 90° or in diameters greater than 15cm. Sharp bends weaken the fused silica and eventually cause spontaneous breakage during use. If the tubing cannot be positioned to avoid sharp bends, then repeat the installation process and uncoil the appropriate amount of tubing from the cage.



Establishing Flow

Turn the carrier gas on and set the column head pressure to the values indicated in **Table II** \*. These values represent approximate head pressures and flow rates. The exact optimum pressures and flow rates for a particular column will be set at a later time.

Table II Approximate Column Head Pressure (He or H<sub>2</sub> carrier gas)

length (m)	0.18mm ID	0.25mm ID	0.32mm ID	0.53mm ID
15	—	6psig	3psig	2psig
20	14psig	—	—	—
30	—	12psig	8psig	4psig
40	30psig	—	—	—
60	—	24psig	16psig	8psig
105	—	40psig	30psig	14psig
Septum Purge Flow: between 2 and 5 cc/min.				

\* If you are having difficulty establishing the appropriate column head pressure for back pressure regulated systems, then suspect septum or inlet ferrule leaks.

The split ratio is the amount of carrier gas exiting the split vent vs. the amount of carrier gas entering the capillary column. The split ratio should be adjusted so the sample amount reaching the column does not exceed the column's capacity. Typically, a split ratio of 50 to 1 is used. **Table III** lists common split vent flow rates found using hydrogen or helium carrier gases. Use the equation below to calculate the split ratio.

Split ratio =  $\frac{\text{Split vent flow \& column flow}}{\text{Column flow rate (cm}^3\text{/min.)}}$

While the flow rate exiting the split vent is easy to measure with conventional bubble meters, the low flow rate exiting a capillary column can be difficult to measure. The following equation can be used to approximate the column flow rate.

Flow =  $\frac{(\pi) (\text{column radius cm})^2 (\text{column length cm})}{\text{dead volume time (min.)}}$

where pi = 3.1459, column radius and length are in centimeters, and time is in minutes.

For example, a 30 meter x 0.53mm ID column operated at 20cm/sec. linear velocity with helium has a flow rate of 2.65cm<sup>3</sup>/min.



$$\text{Flow} = \frac{(3.14159) (.0265\text{cm})^2 (3000\text{cm})}{2.50\text{min.}} = 2.65\text{cm}^3/\text{min.}$$

Table III Typical Split Vent Flow Rates (50 to 1 split ratio)

Carrier gas	0.18mm ID	0.25mm ID	0.32mm ID	0.53mm ID
helium	15cc/min.	35cc/min.	80cc/min.	125cc/min.
hydrogen	30cc/min.	70cc/min.	160cc/min.	250cc/min.

Safety Tip: Always use a split vent trap when injecting hazardous or carcinogenic chemicals into a split/splitless inlet system.

Good Operating Practice

Operating a column without carrier gas flow causes irreparable damage to the stationary phase. Confirm flow by inserting the column outlet into a vial of solvent such as acetone or isopropyl alcohol prior to installing it into the detector. The appearance of bubbles at the column outlet confirms carrier gas flow. Allow the column to purge with carrier gas for fifteen minutes before installing the column outlet into the detector to remove any room air that may have diffused inside the column.

Outlet Installation

Install the nut and ferrule to the detector in the manner described in the instrument manual. Gently insert the column end into the outlet fitting making sure that it is not crushed or scraped against the metal detector parts. Regardless of the GC manufacturer, a higher degree of inertness and better peak symmetry results if the column end can be installed 1 to 3mm from the detector jet orifice. Be careful not to push the column beyond the jet orifice or the column end will burn in the flame. Some jets are too narrow to insert the column close to the jet orifice. If this is the case, pull the column end approximately 2mm away from the narrowed point to prevent flow occlusion or loss of inertness. While maintaining the correct insertion distance, use a capillary wrench to tighten the nut approximately one-half turn past finger-tight until the column is held firmly. The ferrule is tight when the column cannot be pulled from the fitting while applying gentle pressure.

Note — Be cautious when using stainless steel or aluminum-clad columns in gas chromatographs or GC/MS systems with electrically energized detector jets or orifices. These columns will conduct electricity and cause a short if the end of the column is installed too far into the energized detector. Always turn off the electrometer with Varian, PerkinElmer, and Shimadzu FIDs (since the detector is not grounded) when installing stainless steel or aluminum clad columns.

Leak-Checking Techniques

The best way to leak-check a capillary column system is to use a thermal conductivity leak detector (cat.#22451)\*. These portable devices detect minute traces of helium or hydrogen carrier gas without contaminating the system. Leaks in mass spectrometers can easily be determined by monitoring for Mass 28 (N2) or 32 (O2).\*

Never use liquid leak detectors that contain soaps or surfactants in capillary chromatography. Leaks draw these materials inside the system and contaminate the column, making high sensitivity operation difficult. In addition, liquid leak detectors can cause permanent damage to the capillary column by depolymerizing the silicone stationary phase.

Once the system is leak-free, set the injector and detector temperatures approximately 20°C above the final operating temperature of the analysis or at the column's maximum operating temperature. Then light or turn on the detector. Caution: Do NOT exceed the maximum operating temperature of the



column.

\* Spraying argon gas and monitoring for Mass 39 is also effective for mass spectrometers.

### III. Setting Optimum Flow Rates

The most accurate and reproducible way to set the capillary column flow is by injecting a non-retained substance (see table IV) to determine the linear velocity (dead volume time) and adjusting the head pressure until the linear velocity is at its optimum value. Measuring the flow rate at the column outlet is not recommended because it does not account for column-to-column variations. Relying on head-pressure readings is not recommended due to instrument and column variations. Exact flow rate values for a particular column can only be determined after the linear velocity is set at its optimum value.

Because most capillary columns are operated in a pressure (not flow) controlled mode, the temperature at which the linear velocity is set is critical. To obtain the optimum performance, linear velocity should always be set at the operating temperature for an isothermal analysis. For a temperature-programmed analysis, the column's linear velocity should be optimized at an oven temperature where a hard to separate peak pair elutes. If there are no critical peak pairs, raise the oven temperature to the temperature reached midway through the programmed run. Always document which non-retained compound was used and the temperature at which the linear velocity was set in order to easily reproduce the analysis.

To set dead time, inject 2.0µl of a non-retained substance that is compatible with the detector (**Table IV**). Accurately mark the injection starting time and peak elution time with an electronic integrator.

**Table IV**

Detector Type	Recommended Dead Volume Compound
FID/TCD	CH <sub>4</sub>
NPD	acetonitrile vapors
ECD	methylene chloride vapors or air
ELCD	dichlorodifluoromethane vapors
MS	O <sub>2</sub> or N <sub>2</sub> (air)
PID	ethylene or acetylene

The compounds listed above may be slightly retained on thick film phases (1.0 to 7.0µm) giving erroneous dead volume times. However, they are reproducible for similar column types on subsequent analyses.

Adjust the column head pressure until the correct dead time is obtained for the appropriate column length and carrier gas (**Table V**). Once the dead volume time has been finalized, check the split vent and septa purge flow to make sure they did not change significantly. (Head pressure regulated capillary systems require adjustment of the split vent flow if the pressure changed significantly. Back pressure regulated capillary systems should not require adjustment.)

The values in **Table V** were obtained using the formula for average linear velocity (u). The optimum u is 40cm/sec. for hydrogen, 20cm/sec. for helium, and 12cm/sec. for nitrogen\*. Insert the appropriate values in the equation below to obtain the required dead volume time for column lengths not listed.

$$\text{Dead volume time} = \frac{\text{Column length (cm)}}{u \text{ of carrier gas (cm/sec.)}}$$

**Table V Dead Volume Times (for commonly used capillary columns)**



length (m)	hydrogen	helium
15	0.63 min./TCD	1.25 min.
30	1.25 min.	2.5 min.
60	2.5 min.	5.0 min.
105	4.38 min.	8.75 min.

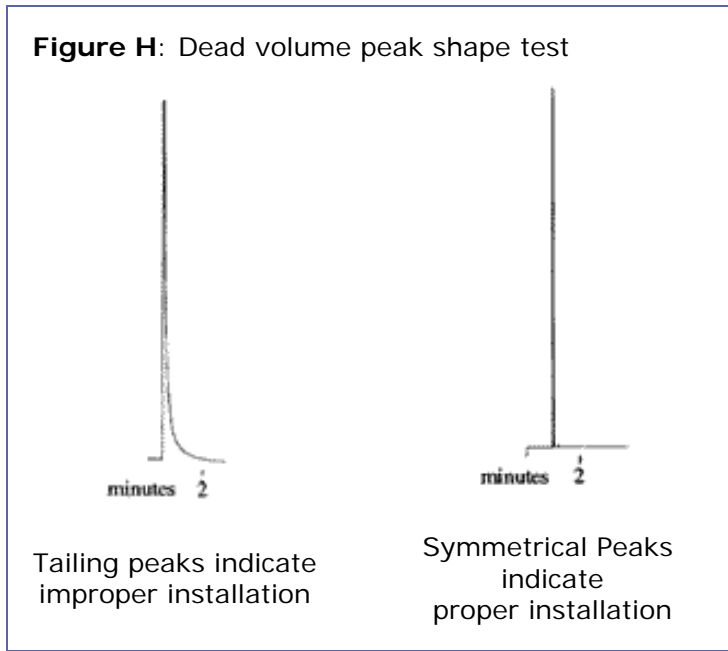
\* Nitrogen is not recommended as a carrier gas for most capillary columns because inadequate resolution and longer analysis times result.

#### IV. Confirming Installation Integrity

We highly recommend using the dead volume peak shape test and the solvent peak shape test to confirm installation integrity.

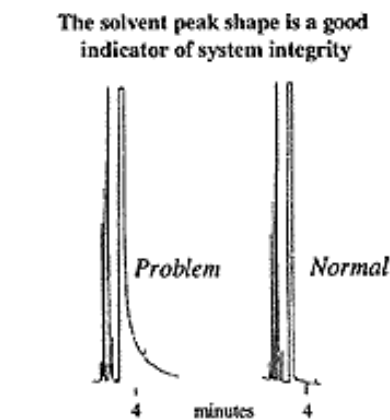
##### Dead Volume Peak Shape Test

Examine the dead volume peak. A sharp, narrow peak that shows no sign of tailing indicates an unobstructed sample pathway and correct installation (**Figure H**). Tailing peaks indicate improper column installation, gross contamination of the splitter sleeve, a cracked splitter sleeve, improper sweeping of the column end by make-up gas, a crushed column end, or a column that has degraded. The cause of a tailing non-retained peak must be corrected before using the column analytically.



##### Solvent Peak Shape Test

The solvent peak shape test is an additional indicator of proper column installation in the inlet and outlet. Since compounds used to set the dead volume are usually gases at room temperature (methane), they are not extremely sensitive indicators of system or installation problems. A 1 $\mu$ l injection of a liquid solvent, such as methylene chloride, expands to over 500 $\mu$ l of gas volume, making any potential installation or system problem readily apparent. A tailing solvent peak is a sensitive indicator of broken, undeactivated, or contaminated inlet sleeves. Tailing solvents also indicate problems with inadequate make-up gas or improper column insertion into the detector.



To perform the test, inject 1 $\mu$ l of a solvent in the split mode at 40°C isothermal and examine the peak shape (**Figure I**). The solvent peak should be symmetrical and show minimal tailing. If tailing appears, suspect an installation or system problem. The cause of a tailing

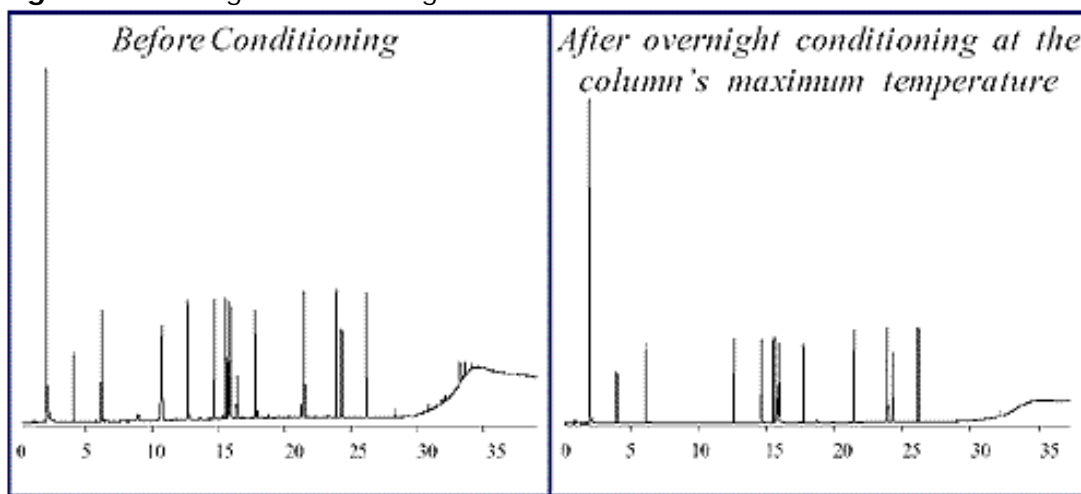
solvent peak must be corrected before using the column analytically.

## V. Conditioning

Before conditioning a column at an elevated temperature, make sure there is proper flow, there are no leaks present, and there is an ample supply of oxygen-free carrier gas for the conditioning period. Conditioning at elevated temperatures without flow permanently damages or destroys the performance of the capillary column. Conditioning with an oxygen leak present causes the column to exhibit permanent high bleed and destroys its utility at high operating temperatures.

To condition the column, set the GC oven at 40°C, hold fifteen minutes, then program at 10°C/min. to the maximum operating temperature (see the test chromatogram included with the column). Alternatively, the column can be conditioned 25°C below the maximum operating temperature if it is going to be used at relatively low temperatures. Hold the column at this temperature for two hours or until the baseline stabilizes. Overnight conditioning is not necessary with Restek capillary columns operated at moderate detector sensitivities (approx.  $8 \times 10^{-11}$  AFS). Overnight conditioning is necessary when the column is going to be operated at high detector sensitivities ( $< 4 \times 10^{-11}$  AFS) and at oven temperatures close to the maximum operating temperature (see **Figure J**). Extra conditioning may be required if operating the column at high sensitivity ( $< 1 \times 10^{-11}$  AFS) or using thick films ( $> 1\mu\text{m}$ ). The column should not be installed in very sensitive or hard-to-clean detectors such as ECDs, NPDs, FPDs, PIDs, ELCDs, or Mass Spectrometers during the initial conditioning period. This practice is particularly important with very thick film columns ( $> 3\mu\text{m}$ ) which produce more stationary phase bleed. (Before conditioning thick film columns, cap the detector.) The Crossbond® procedure used by Restek produces columns with very low bleed levels. If your column is experiencing higher bleed than shown on the test chromatogram, contact us immediately at 800-356-1688 (ext. 4).

**Figure J:** Overnight conditioning reduces column bleed



## VI. Test Mixtures

Restek tests every column with a stringent test mix to determine that the column and GC systems are performing correctly. It is good analytical practice to run the test mixture before analyzing samples to assess system problems or chemical incompatibilities that may be present. It is also good practice to inject the test mix weekly to monitor column performance and to indicate when maintenance procedures are needed. Refer to the "[Column Maintenance and Rinsing](#)" section in this guide.

Inject a column test mixture according to the test chromatogram conditions. Review the test chromatogram to determine peak identities for your specific column. Carefully compare Restek's test chromatogram and your analytical run, noting changes in peak shapes. In general, tailing hydrocarbon and Fatty Acid Methyl Ester (FAME) peaks indicate dead volume or contamination in the inlet or detector. Check the inlet and outlet sleeves for ferrule or septa fragments and reinstall the column. Excessively tailing solvent peaks and tailing or adsorbed peaks such as 2,3-butanediol, octanol, 2-

ethylhexanoic acid, or dicyclohexylamine indicate the need for cleaning and reactivating the split/splitless sleeve or that there is a problem with the make-up gas system. **Figure K** shows the Grob mix run on a relatively non-polar stationary phase.



## Restek Capillary Column Installation Guide

### Section III: Operational Information

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- A. Standby operation
  - B. Column removal and storage
  - C. Protection against dirty samples
  - D. Connecting fused silica tubing
  - E. Connecting stainless steel tubing
  - F. Column maintenance and rinsing
  - G. Injecting water and other solvents
- 

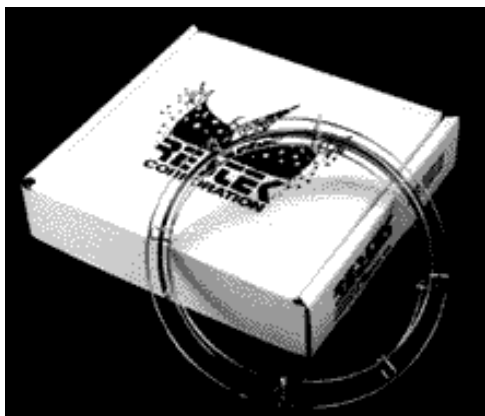
#### A. Standby Operation

If the column is not going to be used for several days but the instrument must remain on standby, reduce the splitter vent flow rate to 10cc/min. (to conserve carrier gas) and maintain an oven temperature between 100°C and 150°C. Keeping the oven slightly hot minimizes septa bleed from accumulating at the head of the column and reduces the need for excessive conditioning when the column is used. Make sure there is a sufficient carrier gas supply when leaving the column in standby operation. If the column will be unused for three or more days, it is best to shut down all heated zones and leave the column in the GC with a low flow of carrier gas. Never leave the column in the GC without carrier gas flowing.

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#### B. Column Removal and Storage

Remove the column from the GC. Make sure that there are no ferrule fragments in the injector or detector fittings. If a graphite ferrule sticks in the fitting, remove it by using a tapered needle file (cat.# 20106). Insert the file and spin it slightly until it is latched inside the ferrule. Then move the file slightly from side-to-side, while applying force, until the ferrule slips out of the fitting. If the ferrule does not come out in one piece, disassemble the fitting and inspect all parts to make sure that all fragments are removed.



Seal the column ends with a high temperature (2000°C) Microflame™ torch (cat.# 20125) immediately after removing the column from the GC. If a Microflame™ torch is not available, or when using stainless steel columns, insert the column ends into a septum to prevent phase oxidation during storage. Always store the column in the original box, away from sunlight or fluorescent lights, to avoid polymer damage. In particular, Stabilwax®, Rtx®-1301, and Rtx®-1701 columns (polyethylene glycol and cyanosilicone polymers) are especially susceptible to uv induced degradation and should be shielded from light.

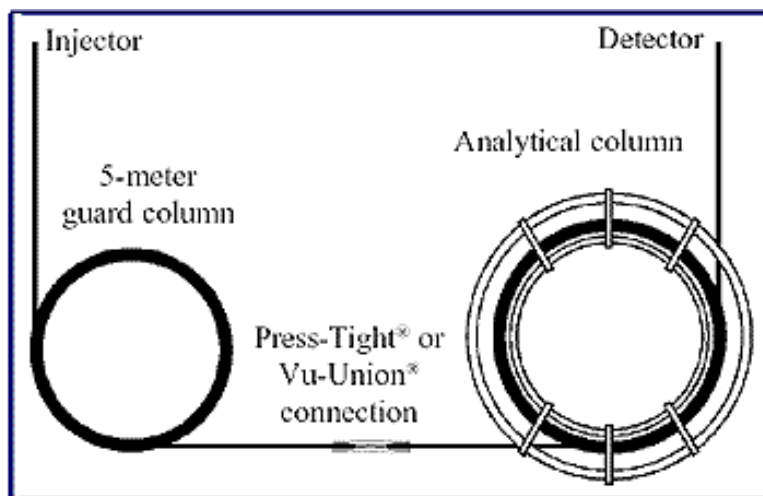
### C. Protection Against Dirty Samples

Samples that contain non-volatile or high molecular weight residue can contaminate the stationary phase, decrease quantitative accuracy, and cause a loss of peak resolution after a limited number of injections. Packing the inlet sleeve and using a guard column are two specific precautions recommended to help increase the number of analyses performed before column and inlet sleeve maintenance is required.

The first precaution involves the use of an inlet sleeve packed with silanized fused silica or glass wool (cat.# 20790, 20789) or highly inert silica beads (cat.# 20791) to trap sample residue and prevent it from entering the capillary column. Insert a loosely packed 2cm wool plug approximately 1cm below the maximum penetration distance of the syringe needle. Too much packing material can detrimentally affect the performance of the column. Refer to the instrument manual for specific inlet packing instructions.

Stationary phase coated packings are not recommended since they tend to be adsorptive and can bleed stationary phase onto the column. This problem is particularly damaging if a methyl silicone inlet packing is used with a Carbowax® PEG-type capillary column.

The second precaution involves using a 5-meter deactivated, uncoated guard column at the analytical column inlet (**Figure L**). The guard column prolongs column life by trapping non-volatile contaminants before they enter the analytical column. Sample components travel at the same rate as the carrier gas on the uncoated guard column inlet, but slow down considerably when they reach the analytical column's stationary phase. Because the

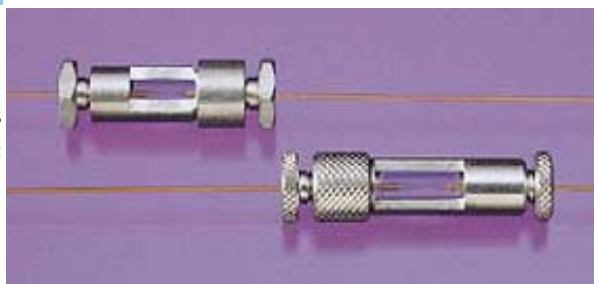


sample's rate of travel is faster through a guard column, the sample has a limited interaction time with residue and, therefore, is not as affected by its presence. Without a guard column, sample residue affects chromatographic performance more rapidly due to increased interaction times. A 5-meter guard column also allows contaminated segments to be removed without having to reconnect the column each time. Restek's Integra-Guard™ is a built-in guard column configuration that eliminates connectors. For more information on protection against dirty samples, request Restek's bulletin A Guide When Injecting Dirty Samples.

### D. Connecting Fused Silica Tubing

Connectors are necessary to attach guard columns and restrictors, or to repair broken analytical columns. Both Press-Tight® connectors and Vu-Union® connectors are effective at making low dead volume, inert connections. Press-Tight® connectors are best for standard applications at temperatures below 325°C. Polyimide resin (cat.# 20445) can be used to strengthen and create permanent press-tight connections, preventing the possibility of premature separation. Vu-Union® connectors are appropriate for high temperature or high pressure applications, or when attaching an analytical column to a mass spectrometer transfer line. Call Restek's Technical Service at (800) 356-1688 (ext. 4) for more information on the use of these two connectors.



**Press-Tight® connectors****Vu-Union® connectors**  
(vacuum/high pressure & capillary/microbore)**Connecting Columns with Press-Tight® Connectors**

1. Cut the column ends squarely.
2. Wipe the column ends with methanol to lubricate and clean.
3. Carefully insert the ends into the Press-Tight® Connector.
4. Visually inspect (see photo).
5. Leak-check using a thermal conductivity leak detector.

**E. Connecting Stainless Steel Tubing**

In order to connect MXT® columns, special connectors must be used. The MXT® Low Dead Volume connector (cat.# 20397, 20394) and MXT® Low Dead Volume "Y" connector (cat.# 20396, 20395) are Silcosteel®-treated and deactivated to make them inert to active compounds. They will not cause peak tailing or affect system inertness and can be used up to 400°C without degrading the deactivation layer. Stainless steel replacement ferrules are available to fit 0.28mm ID MXT columns (cat. # 20398) and 0.53mm ID MXT columns (cat. # 20399).

MXT® tubing can be connected to fused silica tubing by using a capillary Vu-Union® connector (cat.# 20418) provided the end of the MXT® column is properly burnished into a conical shape. The flat side of a ceramic scoring wafer can be used to polish or round the column end into a conical taper. An MXT® connector with a special ferrule (cat. # 20259) can also be used with a fused silica column. A fused silica Press-Tight® connector cannot be used with MXT® columns because without polyimide coating on the outside of the tubing, a leak-free connection cannot be obtained.

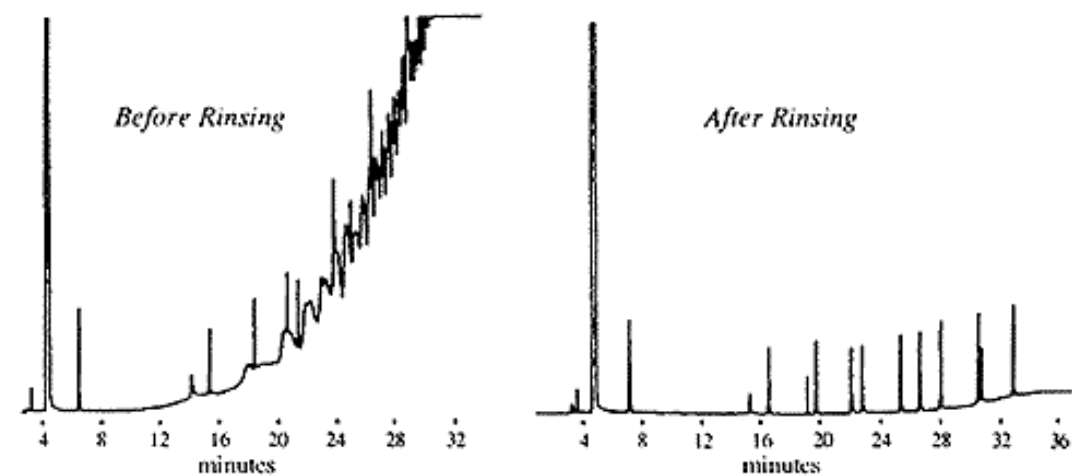
**F. Column Maintenance and Rinsing**

Maintenance is necessary if a column experiences high bleed (that cannot be decreased by overnight conditioning), adsorption of active components, peak tailing, or changes in relative retention times. Cutting two loops (approximately one meter) from the column inlet is often sufficient to restore a contaminated column. However, high molecular weight residue carried deep into the column bore can only be removed by solvent rinsing. To remove pyrolyzed contaminants that are not solvent-soluble, cut two loops from the column's inlet and one loop from the outlet. Closely examine the inlet end of the column to make sure that all contaminated tubing has been removed. Occasionally, it becomes necessary to remove more than two loops from a heavily contaminated column to restore it to acceptable performance. Solvent rinsing removes only soluble contaminants and may not always restore a bonded phase capillary column to its original performance. **Figure M** illustrates the performance of a column that has been successfully solvent-rinsed. Choose a series of polar and non-polar volatile solvents that will solubilize the suspected contamination. Only use solvents shown in **Table VII** to rinse Restek columns. If you are



using a solvent that is not listed, call Restek BEFORE rinsing to prevent permanent damage to your capillary column.

**Figure M:** Solvent rinsing removes soluble contaminants



**Table V Dead Volume Times (for commonly used capillary columns)**

Phase	Magic Mix	Water	Methanol	CH <sub>2</sub> Cl <sub>2</sub>	CHCl <sub>3</sub>	Acetone	Toluene	Pentane
MXT/Rtx-1	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx/XTI-5 & Rtx-5MS	OK	OK	OK	OK	OK	OK	OK	OK
Rtx-5 Amine	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-Volatiles	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-502.2	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-20	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-35	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-50,-56, & 65TG	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-1301 & 624	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-1701	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-200	OK	OK	OK	OK	OK	OK	OK	OK
Rtx-225	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-BAC1 & BAC2	OK	OK	OK	OK	OK	OK	OK	OK
MXT/Rtx-WAX & Stabilwax	Avoid	Avoid	OK	OK	OK	OK	OK	OK
Stabilwax DB	Avoid	Avoid	Avoid	OK	OK	OK	OK	OK
Stabilwax DA	Avoid	Avoid	OK	OK	OK	OK	OK	OK

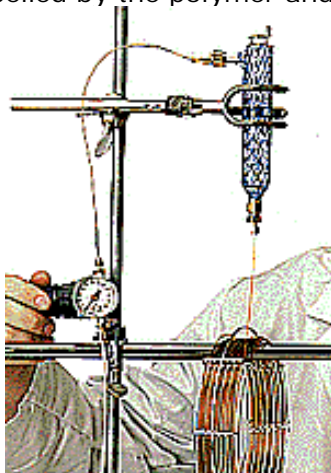
Magic Mix = 20% DI H<sub>2</sub>O, 60% CH<sub>3</sub>OH, 20% CH<sub>2</sub>Cl<sub>2</sub>

**Do not rinse the following columns** because they are either partially or not bonded: Rtx-2330, Rt-2330, Rt-2340, Rt-BDEX, Rt-γDEX, Rt-TCEP & Micropacked

Restek's chemists have found that a mixture of 60% methanol, 20% DI H<sub>2</sub>O, and 20% methylene chloride (i.e. magic mix) is ideal for rinsing and cleaning most bonded stationary phases. (Do not use this mixture unless these solvents are recommended for rinsing.) The methylene chloride tends to swell the polymer which allows the methanol/DI H<sub>2</sub>O to deeply penetrate into the swollen polymer lattice to remove polar contamination.



Without the addition of methylene chloride, polar solvents such as water or methanol are repelled by the polymer and do not clean deeply inside the polymer lattice.



Always rinse the column from the back (detector side) to the front (inlet side) to prevent contaminants lodged on the inlet side from being carried deeper inside the column bore (backflushing). Columns can be rinsed by either forcing solvent through the column under pressure or by pulling solvent through under vacuum. Restek offers a convenient Capillary Column Rinsing Kit (cat.# 20612, shown at left) to rinse columns. This kit contains a glass reservoir with a fritted disk to prevent particulates from damaging the capillary column. The amount of solvent used to rinse a column should equal approximately three to five times the column's internal volume. **Table VII** lists typical solvent volumes and pressures used to rinse each column ID.

**Table VII Typical Solvent Volumes and Pressure Used for Rinsing**

Column ID	Solvent Volume	Reservoir Pressure
0.18mm	10ml/TCD	50psig
0.25mm	15ml	40psig
0.32mm	25ml	30psig
0.53mm	50ml	15psig

Use non-swelling solvents such as methanol or acetone to minimize swelling while solvent-rinsing thick film columns. Sometimes very thick films ( $>3\mu\text{m}$ ) can swell shut and occlude the column flow while being solvent-rinsed. Do not try to remove the blockage by increasing the rinsing pressure. Attach both column ends to a vacuum system for 24 hours. The vacuum evaporates the solvent and opens the column bore.

After rinsing, allow the column to purge with a dry carrier gas for 4-6 hours to decrease polymer swelling after solvent exposure. If the column is installed in a GC and heated rapidly before the solvent swelling has diminished, extensive damage to the polymer occurs. After purging, install the column in the GC at  $40^{\circ}\text{C}$  and hold for 30 minutes. Then, program slowly ( $4^{\circ}\text{C}/\text{min.}$ ) to the column's maximum operating temperature and condition for two hours before using.

## G. Injecting Water and Other Solvents

In general, injecting water and other solvents will not damage bonded or non-bonded phase capillary columns provided that the solvent is vaporized before it reaches the front of the column (i.e. when split, splitless, or direct injections are performed). However, if operating the capillary column in cold or heated on-column modes, some damage may occur. To reduce the risk of damage, avoid water and methanol with Stabilwax®, Stabilwax®-DB, Stabilwax®-DA, Rtx®-2330, and Rtx®-225 phases when using cold or heated on-column injection techniques. Alternatively, avoid damage from water and methanol by attaching a 5-meter Hydroguard™ guard column to the capillary column inlet. This allows the solvent to vaporize before reaching the capillary column. Injecting water, methanol, or other solvents will not damage the other phases Restek offers, regardless of the injection mode.



## Restek Capillary Column Installation Guide

### Section IV: Column Troubleshooting & Return Procedure

- A. Pinpointing Performance Problems
- B. Contacting Restek for Technical Service
- C. Return Authorization Procedure

Many problems associated with capillary chromatography are due to sample contamination or instrument variables. Our technical service chemists may suggest some troubleshooting procedures to isolate these variables. Preliminary troubleshooting efforts by you help us identify the cause of the problem faster and can save you valuable down time.

#### A. Please follow these steps to pinpoint performance problems:

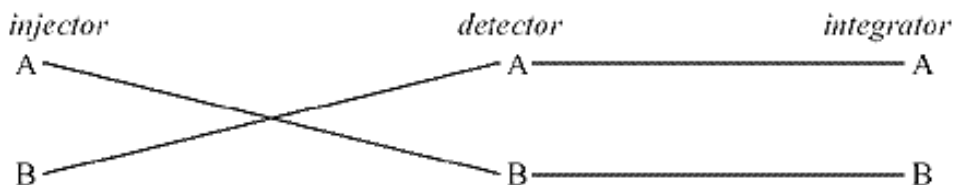
##### Isolate the Column From Instrument Variables:

The first step is to isolate the column from instrument variables. To do this, simply install a new column into the same GC without changing any GC related variables. We highly recommend keeping a spare column on hand at all times, but if you do not have a spare column, then install the questionable column in another GC that is working properly. If the problem does not seem to follow the column, (i.e. the new column does not work in the same GC, or the old column works fine in another GC), then we suggest performing routine maintenance on the injection port and detector as described in Section II of this installation guide. If the problem seems to follow the column, then it is time to perform routine maintenance on the column as described in Section III of this installation guide.

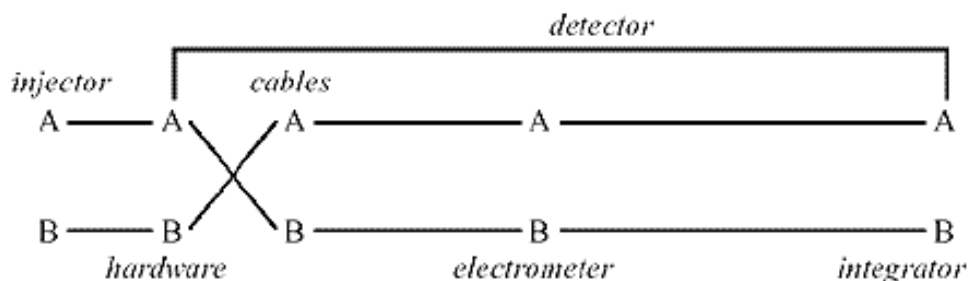
##### Begin Diagramming and Documenting

Document the procedures in chronological order listing times, dates, and important instrument parameters. Label all troubleshooting chromatograms. These steps help to inform anyone else that may be working on the system of the troubleshooting procedures that have been completed.

Start with a simple instrument diagram and try switching column A to detector B and vice versa. If the problem moves to detector B, then the problem is most likely occurring in the injector.



If the problem stays on Detector A when the column outlet is switched, then suspect a detector problem. Begin isolating detector problems by switching hardware, cables, electrometers, integrators, or any suspect part in the pathway. If the problem goes away from the A side, then that detector is most likely the cause and should be replaced.



## B. If you are unable to pinpoint the problem:

Contact our [technical service group](#) at 800-356-1688 (ext. 4). Save all chromatograms and notes on any operating parameters.

### Provide Restek's Technical Service Chemists with This Information:

1. The column's catalog and serial numbers.
2. Where and when the column was purchased and the invoice or purchase order number.
3. Instrument parameters:
  - a. instrument manufacturer and model number
  - b. injection mode
  - c. attenuation and range setting
  - d. oven temperature profile
  - e. injector and detector temperatures
  - f. detector type
4. Sample parameters:
  - a. sample size, list of sample components, and concentrations
  - b. description of the sample components that work and those that are problematic
  - c. derivatization agents, inorganics, or high molecular weight residue which may be present
  - d. number of injections made with the sample on this particular column and the length of time the column has been in service
5. Concise description of the problem:
  - a. observed symptoms
  - b. variables affecting the symptoms
  - c. steps taken to solve the problem
  - d. if the problem has been observed before

Before you call, obtain chromatograms generated when the column was working properly and chromatograms that illustrate the problem you are having.

## C. Return Authorization Procedure:

### Procedure for Returning Capillary GC Columns

If a phone call does not resolve the problem satisfactorily, we will evaluate the column in our lab to identify the problem source. Before returning the column, obtain an [RA#](#) (return authorization number) from a technical service chemist.

**Important Note:** If you purchased the column through a distributor, you must obtain return authorization through that distributor.

To send the column back, place it in its original box and tape. Send it prepaid to this address:

Restek Corporation  
 110 Benner Circle  
 Bellefonte, PA 16823-8812  
 Attn: RA# \_\_\_\_\_

[ *Note: information provided here is specific to Capillary GC Columns. Refer to the current Chromatography Products Guide for return authorization information on other types of products, or call Technical Service at 800-356-1688 (ext. 4).* ]

After receiving the column, it is retested in our QA lab and rinsed if necessary. In most cases, the column evaluation is completed within three working days. A technical service chemist will call and discuss the results and give suggestions. If we determine the column is working satisfactorily or was contaminated and simply required solvent rinsing, it will be returned to your lab with our recommendations to help you avoid this problem in the future. If the column is defective, it will be replaced immediately with a new column. There is no charge for retesting or troubleshooting column problems.

When column problems are suspected and you need the product immediately, we can send you a new column, but we must invoice you for it. Credit is issued to your account if the returned column can be restocked or is found defective. If the problem was system-related or was caused by improper handling, the column will be returned to you and the replacement item invoice is due. We cannot restock damaged or used items.