Designation: E 1510 – 95 (Reappproved 2000)

Standard Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs[1]

This standard is issued under the fixed designation E 1510; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is intended to serve as a general guide for the installation and maintenance of fused silica capillary columns in gas chromatographs which are already retrofitted for their use. This practice excludes information on:

1.1.1 Injection techniques.

1.1.2 Column selection.

1.1.3 Data acquisition.

1.1.4 System troubleshooting and maintenance.

1.2 For additional information on gas chromatography, please refer to Practice E 260. For specific precautions, see Notes 1-4.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific safety information see Section 6 and Notes 2-4. [2]

2. Referenced Documents

2.1 *ASTM Standards:*

E 260 Practice for Packed Column Gas Chromatography[3]

E 355 Practice for Gas Chromatography Terms and Relationships[3]

E 516 Practice for Testing Thermal Conductivity Detectors Used in Gas Chromatography[3]

E 594 Practice for Testing Flame Ionization Detectors Used in Gas Chromatography[3]

E 697 Practice for Use of Electron–Capture Detectors Used in Gas Chromatography[3]

2.2 *CGA Publications:*

CGA P-1 Safe Handling of Compressed Gases in Containers[4]

CGA G-5.4 Standard for Hydrogen Piping Systems at Consumer Locations[4]

CGA P-9 The Inert Gases: Argon, Nitrogen and Helium[4]

CGA V-7 Standard Method of Determining Cylinder Valve Outlet Connections for Industrial Gas Mixtures[4]

CGA P-12 Safe Handling of Cryogenic Liquids[4]

HB-3 Handbook of Compressed Gases[4]

TABLE 1 Typical Splitter Vent Flow Rates (50 to 1 split ratio) (at optimum linear velocity)

Carrier gas	0.25-mm ID, cm^3/min	0.32-mm ID, cm^3/min	0.53-mm ID, cm^3/min
helium	35	80	125
hydrogen	70	160	250

3. Terminology

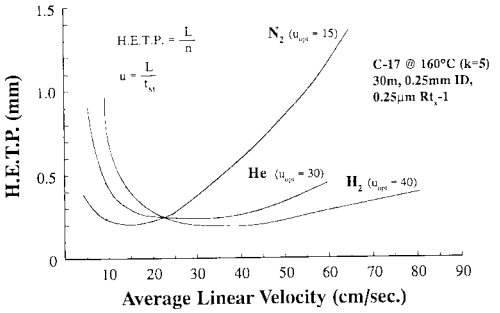
3.1 Terms and relations are defined in Practice E 355.

3.2 Nomenclature for open tubular or capillary columns with a bore of 0.75 mm or less:

3.3 *porous layer open tubular (PLOT)*—refers to columns with particles attached on the inside wall consisting of copolymers such as styrene/divinylbenzene, molecular sieves, or adsorbents such as Al_2O_2 in film thicknesses of 5 to 50 µm.

3.4 *support coated open tubular (SCOT)*—refers to fine particles (silica or fine diatomite) coated with liquid stationary phase which is then deposited on the inside column wall to improve stationary phase stability and sample capacity.

3.5 *wall coated open tubular (WCOT)*—refers to columns coated on the inside wall with a liquid stationary phase in film



NOTE 1—The curves were generated by plotting the height equivalent to a theoretical plate (length of column divided by the total number of theoretical plates, H.E.T.P.) against the column's average linear velocity. The lowest point on the curve indicates the carrier gas velocity in which the highest column efficiency is reached.

FIG. 1 Van Deemter Profile for Hydrogen, Helium, and Nitrogen Carrier Gases[4]

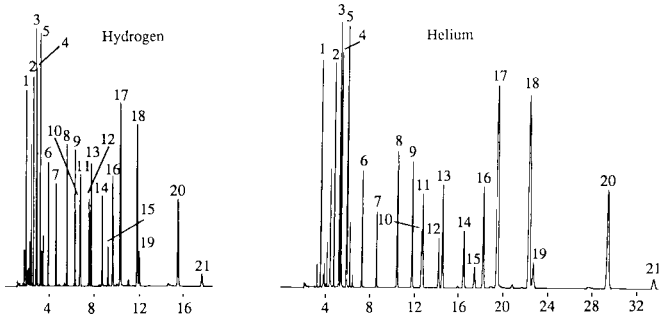
[1] This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.19 on Chromatography.

Current edition approved May 15, 1995. Published July 1995. Originally published as E 1510 – 93. Last previous edition E 1510 – 93.

[2] Reprinted by permission of Restek Corp., 110 Benner Circle, Bellefonte, PA 16823-8812.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from Compressed Gas Association, Inc., 1725 Jefferson Davis Highway, Arlington, VA 22202-4100.



Carrier gas: Hydrogen
Linear velocity: 40 cm/s

Carrier gas: Helium
Linear velocity: 20 cm/s

NOTE 1—Fig. 2 shows that the resolution is similar but the analysis time is reduced by 50 % when comparing hydrogen to helium in an isothermal analysis using optimum flow velocities.

NOTE 2—Hydrogen provides similar resolution in one-half the analysis time of helium for an isothermal analysis.

NOTE 3—

1. Tetrachloro-m-xylene
2. α-BHC
3. β-BHC
4. γ-BHC
5. δ-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. γ-chlordane
10. Endosulfan I
11. α-chlordane
12. Dieldrin
13. DDE
14. Endrin
15. Endosulfan II
16. DDD
17. Endrin aldehyde
18. Endosulfan sulfate
19. DDT
20. Endrin ketone
21. Methyoxychlor

NOTE 4—30 m, 0.25-mm ID, 0.25 µm 5 % diphenyl − 95 % dimethyl polysiloxane 0.1-µL split injection of chlorinated pesticides.

Oven temperature: 210°C isothermal
Injector and detector temperature: 250°C/300°C
ECD sensitivity: 512×10^{-11}
Split vent: 100 cm^3/min

FIG. 2 Hydrogen Versus Helium (Isothermal Analysis)

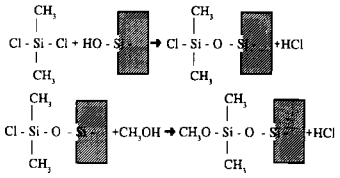


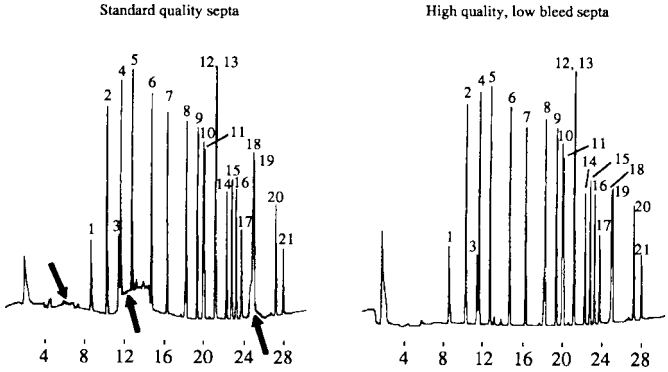
FIG. 3 Capping Silanol Groups with Dimethyl Dichlorosilane (DMDCS)

thicknesses of 0.1 to 10.0 µm. Also referred to as FSOT or fused silica open tubular.

4. Summary of Practice

4.1 The packed gas chromatography system is described in Practice E 260 and is essentially the same as a capillary gas chromatography system except for modifications to the injector and detector to accommodate the low flow rates and sample capacity associated with capillary columns. Refer to the gas chromatography (GC) instrument manual for specific details on injector or detector pneumatics for capillary columns.

4.2 Prior to performing a capillary GC analysis, the capillary column configuration must be determined. The stationary phase type, stationary phase film thickness, column inside diameter, and column length must be selected. It is beyond the scope of this practice to provide these details. Consult a column or instrument supplier for details on selecting the appropriate capillary column configuration.



NOTE 1—Septum bleed can obscure or co-elute with compounds of interest, thus decreasing the analytical accuracy.

NOTE 2—

1. 2,4,5,6-tetrachloro-m-xylene (IS)
2. α-BHC
3. β-BHC
4. γ-BHC
5. δ-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. γ-chlordane
10. Endosulfan I
11. α-chlordane
12. Dieldrin
13. p,p-DDE
14. Endrin
15. Endosulfan II
16. p,p-DDD
17. Endrin aldehyde
18. Endosulfan sulfate
19. p,p-DDT
20. Endrin ketone
21. Methyoxychlor
22. Decachlorobiphenyl (IS)

NOTE 3—30 m, 0.53-mm ID, 0.50 µm 5 % diphenyl − 95 % dimethyl polysiloxane 0.1 µL direct injection of 50 pg pesticide standard.

Oven temperature: 150 to 275°C at 4°C/min, hold 15 min
Injector temperature: 250°C
Detector temperature: 300°C
Carrier gas: Helium
Linear velocity: 40 cm/s (Flow rate: 10 cm^3/min)
ECD sensitivity: 8×10^{-11} AFS

FIG. 4 ECD Septum Bleed

4.3 Apply caution during handling or installation to avoid scratching or abrading the protective outer coating of the column. Scratches or abrasions cause the fused silica capillary column to spontaneously break or fail during usage.

5. Significance and Use

5.1 This practice is intended to be used by all analysts using fused silica capillary chromatography. It contains the recommended steps for installation, preparation, proper installation, and continued column maintenance.

6. Hazards

6.1 *Gas Handling Safety*—The safe handling of compressed gases and cryogenic liquids for use in chromatography is the responsibility of every laboratory. The Compressed Gas Association, a member group of specialty and bulk gas suppliers, publishes the following guidelines to assist the laboratory chemist to establish a safe work environment:

7. Installation Procedure for Fused Silica Capillary Columns

7.1 A brief outline of the steps necessary for installing fused silica capillary columns in capillary dedicated gas chromatographs is as follows:

7.1.1 Cool all heated zones and replace spent oxygen and moisture scrubbers,

7.1.2 Clean or deactivate, or both, injector and detector sleeves (if necessary),

7.1.3 Replace critical injector and detector seals,

7.1.4 Replace septum,

7.1.5 Set make-up and detector gas flow rates,

7.1.6 Carefully inspect the column for damage or breakage,

7.1.7 Cut approximately 10 cm from each end of the column using a ceramic scoring wafer or sapphire scribe,

7.1.8 Install nut and appropriately sized ferrule on both column ends,

7.1.9 Cut an additional 10 cm from each end of the column to remove ferrule shards,

7.1.10 Mount the capillary column in the oven using a bracket to protect the column from becoming scratched or abraded and to prevent it from touching the oven wall,

7.1.11 Connect the column to the inlet at the appropriate distance as indicated in the instrument manual,

7.1.12 Set the approximate column flow rate by adjusting the head pressure (see column manufacturer's literature),

7.1.13 Set split vent, septa purge, and any other applicable inlet gases according to the instrument specifications,

7.1.14 Confirm flow by immersing column outlet in a vial of acetone or methylene chloride,

7.1.15 Connect the column to the detector at the appropriate distance as indicated in the instrument manual,

7.1.16 Check for leaks at the inlet or outlet using a thermal conductivity leak detector (do not use soaps or liquid-based leak detectors),

7.1.17 Set injector and detector temperatures and turn on detector when temperatures have equilibrated (**Caution**—Do not exceed the phase's maximum operating temperature),

7.1.18 Inject a non-retained substance (usually methane) to set the proper dead time (linear velocity),

7.1.19 Check system integrity by making sure dead volume peak does not tail,

7.1.20 Condition the column at the maximum operating temperature for 2 h (consult column manufacturer's literature) to stabilize the baseline,

7.1.21 Reinject a non-retained substance (usually methane) to set the proper linear velocity,

7.1.22 Run test mixtures to confirm proper installation and column performance, and

7.1.23 Calibrate instrument and inject samples.

7.2 The following section provides in-depth information on instrument preparation procedures for installing and operating fused silica capillary columns in capillary dedicated gas chromatographs:

7.2.1 *Gas Purification*—The carrier gas must contain less than 1 ppm of oxygen, moisture, or any other trace contaminants. Otherwise, oxygen and moisture degrade column performance, decrease column lifetime, and increase background stationary phase bleed. Contaminants such as trace hydrocarbons cause ghost peaks to appear during temperature programming and degrade the validity of the analytical data. Make-up gas should also be contaminant-free or baseline fluctuations and excessive detector noise may occur. Detector gases such as hydrogen and compressed air should be free of water and hydrocarbon or excessive baseline noise may occur.

7.2.1.1 Install purifiers as closely as possible to the GC's bulkhead fitting, rather than system-wide. If purifiers are installed system-wide, a leaky fitting downstream of the purifier could allow oxygen and moisture to enter the gas stream and degrade column performance.

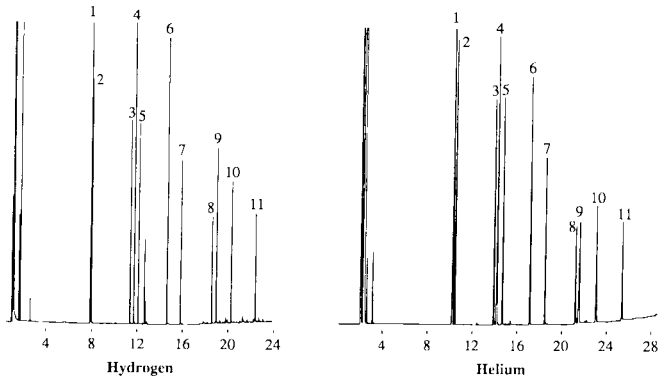
7.2.1.2 Only high–purity gases should be used for capillary chromatography. All regulators should be equipped with stainless steel diaphragms. Regulators equipped with rubber or elastomeric diaphragms should not be used because oxygen, moisture, and elastomeric contaminants migrate through the diaphragm and enter the flow.

7.2.1.3 Both indicating and non-indicating traps are available from most capillary column suppliers. Indicating purifiers are recommended since they allow analysts to visually assess whether the purifier has exceeded its useful life. Also, a moisture trap should be installed prior to the oxygen trap. If hydrocarbon contamination is suspected, a hydrocarbon trap should be installed between the moisture and oxygen trap. Since most indicating traps are made from glass, care should be taken not to apply lateral torque on the fittings, or they will snap. To prevent spontaneous breakage of the trap, the line leading to and from the purifier should be coiled to relieve strain and isolate instrument vibrations.

7.2.2 *Carrier Gas Selection*—A fast carrier gas which exhibits a flat van Deemter profile is essential to obtain optimum capillary column performance. Because capillary columns average 30 m in length (compared to 2 m for packed columns), a carrier gas that minimizes the effect of dead time is important. In addition, capillary columns are usually head pressure controlled (not flow controlled like most packed columns), which cause the carrier gas flow rate to decrease by 40 % when the column is programmed from ambient to 300°C. Therefore, a carrier gas which retains high efficiency over a wide range of flow rates is essential towards obtaining good resolution throughout a temperature–programmed chromatographic analysis.

7.2.2.1 The optimum average linear gas velocity for hydrogen (u_{opt}: 40 cm/s) is greater than all the others, and hydrogen exhibits the flattest van Deemter profile. Helium is the next best choice (u_{opt}: 20 cm/s). Note that head pressures at optimum flow rates are similar for hydrogen and helium because hydrogen has half the viscosity but double the linear velocity as helium. Because of the low optimum linear velocity (u_{opt}: 10 cm/s) and steep van Deemter profile, nitrogen gives inferior performance with capillary columns and is usually not recommended.

7.2.2.2 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 great as when isothermal oven conditions are utilized. In addition, slower carrier gases, such as helium, can improve the separation of very low boiling or early eluting compounds since they allow more interaction with the stationary phase. Fig. 5 illustrates that hydrogen is only slightly faster than helium when both carrier gases are operated under the same temperature to programmed conditions. Also, note that helium improves the resolution of the early eluting compounds (Peaks 1 and 2) as compared to hydrogen for a temperature programmed analysis.



Carrier gas: Hydrogen
Linear velocity: 40 cm/s

Carrier gas: Helium
Linear velocity: 20cm/s

NOTE 1—Hydrogen is only slightly faster than helium when both carrier gases are operated under the same temperature-programmed oven conditions.

NOTE 2—

1. Phenol
2. 2-chlorophenol
3. 2-nitrophenol
4. 2,4-dimethyl phenol
5. 2,4,dichlorophenol
6. 4-chloro-3-methyl phenol
7. 2,4,6-trichlorophenol
8. 2,4-dinitrophenol
9. 4-nitrophenol
10. 2-methyl-4,6-dinitrophenol
11. Pentachlorophenol

NOTE 3—30 m, 0.25-mm ID, 0.25 µm 5 % diphenyl – 95 % dimethyl polysiloxane 0.1-µL split injection of phenols.

Oven temperature: 50°C (hold 4 min) to 250°C at 8°C/min (hold 5 min)
Injector and detector temperature: 280°C
FID sensitivity: 32×10^{-11}
Split vent: 40 cm^3/min

FIG. 5 Hydrogen Versus Helium (Temperature-Programmed Mode)

NOTE 1—**Warning:** Exert caution when using hydrogen as a carrier gas. Hydrogen is explosive when concentrations exceed 4 % in air and should only be used by individuals who have received proper training and understand the potential hazards. Proper safety precautions should be utilized 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 used by analysts include:

(*a*) Frequently check for carrier gas leaks using a sensitive electronic leak detector,

(*b*) Use electronic sensors that shut down the carrier gas flow should an explosive atmosphere be detected,

(*c*) Purge an inert gas (N_2) into the oven chamber to displace oxygen and prevent an explosive atmosphere from forming, and

(*d*) Minimize 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 (head pressure regulated systems only).

NOTE 2—**Warning:** Analysts should also be aware that hydrogen will be expelled from both the split vent and septum purge when it is used as a carrier gas. Because of the fast diffusivity of hydrogen, an explosion in a laboratory setting is highly unlikely. However, a spark from static electricity (particularly the case if a lab is carpeted) can ignite the hydrogen exiting from septum purge or split vent, which could cause a burn or a fire. Since hydrogen flames are colorless, an analyst would not know that the split vent was ignited unless he inadvertently touched it. Precautions to minimize the problems with hydrogen exiting the split vent or septum purge include:

(*a*) Plumbing the exit lines to a hood or venting the escaping gas outside,

(*b*) Plumbing the lines to exit into a vial of water, and

(*c*) Plumbing the exit lines to a position where analysts could not get burned or a fire could not be started if inadvertent ignition occurred.

7.2.3 *Flow-Regulated Pneumatics*—Fig. 6 illustrates a flow-regulated back pressure system commonly used today for split splitless inlets. A flow controller positioned upstream of the injector serves to control the total amount of carrier gas that is expelled from the split vent, septum purge, and column. The back pressure regulator stops or reduces flow from exiting the split vent until the desired column head pressure is reached. The flow controller, sensing that no flow is exiting the split vent, provides the increase of pressure necessary to meet the requirements of the back pressure regulator. Thus, it is the back pressure regulator which is 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 (by means of head pressure changes) do not affect the amount of carrier gas exiting the splitter vent. Thus, once the desired split vent flow rate is achieved, analysts should not have to change the flow controller setting when installing different columns.

7.2.3.1 Flow-regulated back pressure systems prevent a drastic carrier gas loss that could occur if an inlet fitting or column leak were to occur. Leaks are indicated by a failure to obtain the proper column operating pressure by adjusting the back pressure regulators. A common mistake made by analysts not familiar with flow-regulated back pressure systems is to increase the total system flow by turning up the flow controller (split vent adjustment knob) when a proper head pressure can't be obtained rather than checking for inlet leaks.

7.2.4 *Head Pressure-Regulated Pneumatic Systems*—Fig. 7 illustrates a head pressure regulated inlet system used in some split/splitless inlet systems. 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, downstream of the pressure regulator. Head pressure systems require readjustment of the needle valve controlling the septum purge or split vent every time a change is made in the column's head pressure.

7.2.4.1 It is recommended that a throttling valve (needle valve or restrictor) be placed 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

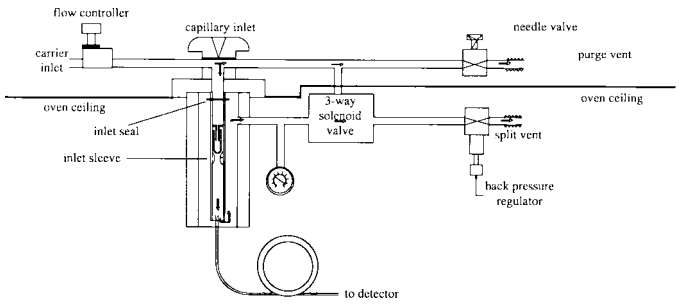


FIG. 6 Flow-Regulated Back Pressure System

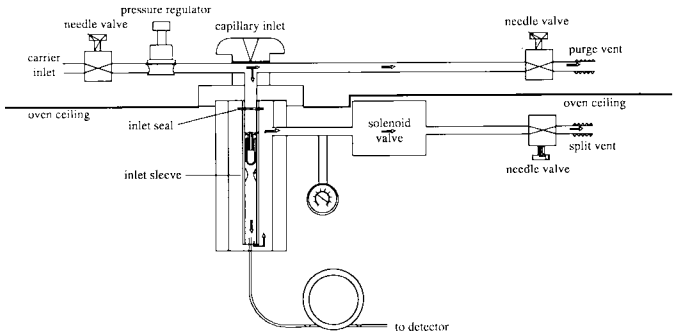


FIG. 7 Head Pressure Regulated System

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 (by means of a throttling valve) until it matches the flow requirements of your inlet system. This throttle point can be detected when the column's head pressure starts to decrease if the throttling valve is closed any further.

7.2.5 *Injector Maintenance*—Maintenance should be performed on the injector prior to installing a capillary column and periodically, depending on the number of injections made and the cleanliness of the samples. Maintenance should include cleaning and deactivating inlet sleeves, replacing critical inlet seals, and replacing the septum. Review the instrument manual's inlet diagram prior to disassembly.

7.2.6 *Cleaning and Deactivating Injector Sleeves*—The inlet sleeve should be free from septum particles, sample residue, and ferrule fragments to obtain optimum column performance. The inlet sleeve must be deactivated when analyzing samples with active functional groups such as alcohols, acids, aldehydes, phenols, bases, or other compounds prone to decomposition or adsorption on untreated glass surfaces. If the sleeve is deactivated and not excessively dirty, it may be cleaned with organic solvents without affecting the integrity of the deactivated layer. First, use non-swelling organic solvents such as methanol or isopropyl alcohol to remove septa particles which adhere to the sleeve wall. Then use solvents such as pentane, methylene chloride, toluene, or any other solvent that will solubilize and remove sample residue. Nylon tube brushes and pipe cleaners are ideal for cleaning sleeves. Do not use laboratory detergents, acids, or bases to clean sleeves. This removes the deactivation layer and requires re-silanization of the sleeve.

7.2.6.1 Sleeves that are very dirty or contain pyrolyzed residue can be difficult to clean and may not justify the cost to do so. Heating sleeves (borosilicate or quartz glass) in a muffle furnace at 550°C overnight will remove most contaminants. Etching with a 1 to 1 to 1 mixture of hydrofluoric acid, sulfuric acid, and deionized water for 10 s is also very effective.

7.2.6.2 High–quality deactivated sleeves are available from some capillary and instrument suppliers. If deactivated sleeves are not available, they can be deactivated by using a three–step procedure: acid cleaning, dehydration, and silanization.

7.2.7 *Acid Cleaning*—The first step involves etching the sleeves in an acid solution (such as 1 to 1 to 1 sulfuric/hydrofluoric/deionized water) for a 10 s duration. Then rinse the sleeves thoroughly with deionized water and blow dry (do not use methanol or acetone to help the drying process).

NOTE 3—**Warning:** Exert extreme caution when using hydrofluoric acid. Only professionals properly trained and equipped with the appropriate safety devices should attempt to handle strong acids. Hydrofluoric acid could cause severe burns and nerve damage if it comes in contact with skin, is ingested, or inhaled.

7.2.7.1 *Dehydration*—The second step involves the removal of surface water. Heat the sleeves in an oven at 250°C for 1 h. Program slowly (approximately 4°C per min) from ambient to 250°C to prevent water staining or spotting.

7.2.7.2 *Silanization*—The third step involves a reaction of the glass surface silanol groups with a chlorosilane to prevent them from adsorbing or degrading sample compounds. Silanization should be performed within 1 h after the sleeves have cooled from the dehydration process to prevent readsorption of atmospheric moisture. Soak the sleeves for 5 min in a 5 % volume to volume mixture of dimethyldichlorosilane in toluene. Rinse thoroughly with toluene to remove the excess chlorosilane reagent. Finally, any unreacted chlorine groups should be capped by soaking for 5 min in methanol. Blow dry after removing from the methanol. The sleeves are now ready to be used.

NOTE 4—**Warning:** Exert caution when handling chlorosilanes. Chlorosilanes give off HCl vapors when reacted with silanol groups, methanol, or if it comes in contact with atmospheric moisture. Only professionals properly trained and equipped with the appropriate safety devices should attempt to handle chlorosilanes.

7.2.8 *Protection Against Dirty Samples*— Precautions such as packing sleeves with a small plug of fused silica wool should be employed when analyzing samples containing high molecular weight residue or particulates. Use fused silica or glass wool cautiously because, if not deactivated properly, it could degrade the system's inertness to sensitive compounds prone to breaking down in hot inlets. Alternative sleeve designs, which minimize interaction of the sample with nonvolatile residue, are available from some capillary manufacturers. See 8.3 for more information about analyzing dirty samples.

7.2.9 *Replacing Critical Seals*—Review the GC manual and replace the critical seal prior to reinstalling the inlet sleeve. Most capillary injection ports use a rubber O-ring or graphite ferrule to seal the sleeve inside the injection port body. It is critical that the seal fits tightly around the sleeve and prevents the carrier gas from leaking around the outside of the sleeve.

7.2.10 *Changing Septa*—Replace the septum frequently (usually before 100 injections) 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 inlet sleeve. These particles are a potential source of ghost peaks, loss of inertness, and carrier gas flow occlusion. It is best to install a new septum at the end of an analytical sequence so it can condition in the injector and reduce the incidence of ghost peaks. Always use a high-quality, low-bleed septum to prevent the ghost peaks from interfering with the compounds of interest. Never handle septa with bare hands. Always use forceps to avoid contamination.

7.2.11 *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 manual's specifications. 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.

7.2.12 *Mounting the Column in the Oven*— Most instrument manufacturers provide universal hanging brackets that hold the column in the center of the oven and prevent the fused silica tubing from abrading or rubbing against the oven wall. The brackets also properly position the column in the oven chamber to reduce thermal gradients which enhance retention time reproducibility. If there is not a column support bracket, one can be made by inserting a temperature-resistant peg board rod into the corrugated oven wall or by having an "S" hook with 1⁄16 -in. tube hanging from the oven ceiling. Be careful not to damage the oven thermocouple or interfere with the fan operation when making homemade brackets, and do not allow fused silica to contact metal parts. Once the column is mounted, uncoil two loops of fused silica tubing from the cage to provide adequate length for installation with the appropriate ferrules and fittings.

7.2.13 *Choosing Ferrules*—Usually graphite or Vespel®/[5] graphite ferrules are used to seal the column to the injector and detector in capillary gas chromatography. Both ferrule types have advantages and disadvantages. Because graphite ferrules are soft, they easily conform to all column–outside diameters and different types of instrument–fitting configurations. However, they can flake or fragment upon removal, causing particles to lodge in the injector or detector sleeves. Vespel®/graphite ferrules are hard and must match the column and fitting dimensions closely to seal properly. In addition, Vespel®/graphite ferrules can deform and shrink upon initial heating and subsequently should be re-tightened or leakage will occur. Vespel®/graphite ferrules do not shed fragments and can be reused many times. Always check the upper temperature limit of the ferrule for your application.

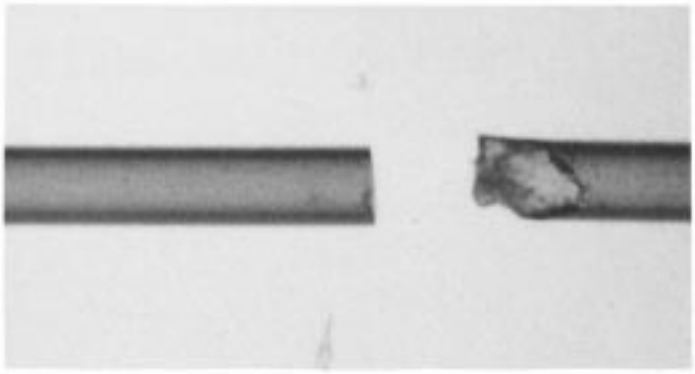
7.2.13.1 Graphite ferrules are the easiest to use, because they are leak-free, universal for most systems, and the choice for most beginning capillary chromatographers. Vespel®/graphite ferrules are preferred for use in mass spectrometers since they do not flake and contaminate the ion source with particles. In all cases, it is best to choose a ferrule that will fit snugly on or be slightly larger than the outer diameter of the capillary tubing used. This minimizes the need for excessive torque in order to properly seal the ferrule to the column.

7.2.14 *Cutting Column Ends*—Fused silica tubing breaks easily when the outside polyimide layer is scratched or scored. An improper cut allows polyimide or glass fragments to interact with the sample stream and degrade the inertness of the system. Scoring devices which utilize a blade are preferred over pointed ones because a better, squarer cut is made. Silica scoring wafers, sapphire blades, or tungsten carbide scoring blades usually produce the best cuts and are available from most common suppliers.

7.2.14.1 To obtain a square cut, place the column end against the forefinger and score the polyimide layer lightly and rapidly. Score only one side of the column. Point the column end down (to prevent glass shards from falling inside) and quickly flick the column just above the score. Examine the quality of the cut with a small 10× pocket magnifier and make sure the cut is clean and square. See Fig. 8 for a good versus poor column cut. It may require several cuts to obtain one that is square and desirable. Use an old column to practice and develop the skill needed to consistently make square cuts.

7.2.15 *Installing the Connecting Nut and Ferrule*—Capillary columns are usually shipped from the manufacturer with the ends flame-sealed or capped with septa. Cut approximately 10 cm off of each end as described in 7.2.14. Install the inlet–connecting nut followed by the appropriately sized ferrule in the manner described in the instrument manual. Be sure to point the column end down when installing the ferrule to prevent shards from falling into the capillary bore. Slide the connecting nut and ferrule approximately 20 cm down the length of the column to make installation easier. Cut an additional 10 cm off of 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.

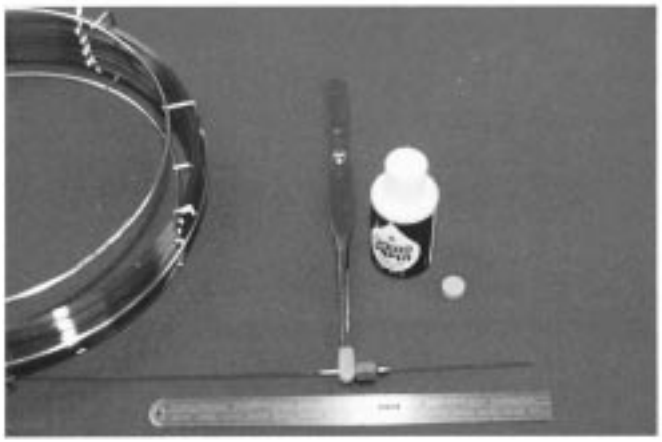
7.2.16 *Connecting the Column to the Inlet*— It is important to install the column at the exact distance recommended in the instrument manual, or poor peak symmetry and quantitation could occur. Determine what this distance is before proceeding. Lay the column end beside a ruler and position the nut and ferrule to the exact distance required for installation. Install the column, being careful to hold the nut and ferrule at the correct distance. This can be accomplished in several ways. Some analysts use white typewriter correction fluid or a black marker to indicate the correct insertion distance. Make sure the ferrule does not slide over the correction fluid, or it will carry fragments into the GC sample stream. Another way to maintain the correct distance is to use rubber-tipped slide-lock tweezers. Clamp the slide-lock tweezers at the appropriate distance to hold the column and nut firmly during installation (see Fig. 9). Other analysts have used a septum to hold the nut and ferrule during installation. Push the column end through the septum center prior to installing the nut and ferrule (see 7.2.16). Position the septum on the column to the correct installation distance. This prevents the nut and ferrule from sliding during



NOTE 1—This photo shows a good and bad column cut. The good cut leaves the end of the column square and free of fragments or fractures.

FIG. 8 Good Versus Bad Column Cut

[5] Vespel is a registered trademark of the *DuPont* Company.



NOTE 1—Rubber-tipped slide-lock tweezers hold the column firmly at the correct distance during installation.

FIG. 9 Different Measuring Techniques

installation. The septum can be slid away from the nut after the fitting is tightened and can remain in the oven cavity while performing an analysis.

7.2.16.1 Gently insert the column end into the inlet fitting, making sure the end is not crushed or scraped against the metal injection port fittings. While maintaining the correct distance, tighten the ferrule 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 when gentle pressure is applied.

7.2.16.2 Make sure the fused silica tubing is not sharply bent when installing the column. The tubing should gently bend from the cage to the fitting in angles less than 90° or a diameter less than 15 cm. Sharp bends weaken the fused silica and eventually cause spontaneous breakage while being used in the oven (see Fig. 10). If the tubing cannot be positioned to avoid sharp bends, repeat the installation process (7.2.13) and uncoil the appropriate amount of tubing from the cage.

7.2.17 *Establishing Flow*—Turn the carrier gas on and set the column head pressure, septum purge flow, and split vent flow rate to the values indicated.

7.2.18 *Setting Column Flow Rates*—The column head pressures listed in Table 2 provide flow rates close to the optimum value. The exact optimum pressures and flow rates for a particular column will be set at a later time. (See 7.2.22.3.)

7.2.19 *Septum Purge Flow*—Set the flow rate exiting the septum purge between 2 and 5 cm^3/min.

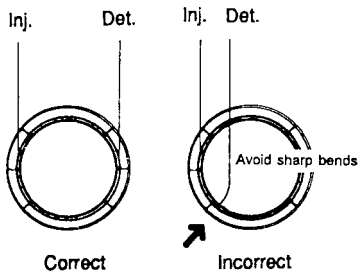


FIG. 10 Avoid Sharp Bends When Installing Fused Silica Capillary Column

7.2.20 *Split Ratio*—The split ratio is the amount of carrier gas exiting the split vent versus 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, split ratios of 50 to 1 are used by most analysts. Use the following equation to calculate the split ratio:

$$\text{Split ratio} = \frac{\text{Split vent flow } (cm^3/min)}{\text{Column flow rate } (cm^3/min)} \quad (1)$$

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.

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

where $\pi = 3.1459$, column radius and length are in centimetres, and time is in minutes. For example, a 30-m × 0.53-mm inside diameter column operated at 20-cm/s linear velocity with helium has a flow rate of 2.65 cm^3/min.

$$\text{Flow} = \frac{(3.14159)(0.0265\ cm)^2\ (3000\ cm)}{2.50\ min} = 2.65\ cm^3/min \quad (3)$$

7.2.21 *Connecting the Column to the Detector*—It is equally important to install the column to the exact distance in the detector as recommended in the instrument manual. Determine what this distance is before proceeding. Lay the column end beside a ruler and position the nut and ferrule to the exact distance required for installation. Install the column, being careful to hold the nut and ferrule at the correct distance. Use the same positioning techniques as described in 7.2.17.

7.2.21.1 Gently insert the column end into the outlet fitting, making sure it is not crushed or scraped against the metal detector parts. While maintaining the correct distance, tighten the ferrule 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 when gentle pressure is applied.

7.2.21.2 Regardless of the GC manufacturer, a higher degree of inertness and better peak symmetry results if the column end can be terminated a short distance (1 to 3 mm) from the detector jet orifice (FIDs, NPDs, FPDs, etc.). Be careful not to push the column beyond the jet orifice or the column end burns 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 2 mm away from the narrowed point to prevent flow occlusion or loss of inertness.

7.2.22 *Leak Checking Techniques*—The best way to leak check a capillary column system is to use a highly sensitive thermal conductivity leak detector (TCD). These portable devices detect minute traces of helium or hydrogen carrier gas without contaminating the system, and are available from most column and instrument manufacturers. Leaks in mass spectrometers can easily be determined by monitoring for mass 28 (N_2) or 32 (O_2).

7.2.22.1 Liquid leak detectors that contain soaps or surfactants should never be used in capillary chromatography. Leaks draw these materials inside the system and contaminate the

TABLE 2 Column Head Pressure (Helium or Hydrogen Carrier Gas)

NOTE 1—If you are having difficulty establishing the column head pressure for back pressure regulated systems, then suspect septum or inlet ferrule leaks.

Column Length, m	Column Inside Diameter					
	0.10-mm ID, psig	0.20-mm ID, psig	0.25-mm ID, psig	0.32-mm ID, psig	0.53-mm ID, psig	0.75-mm ID, psig
10	15	12	6	4	2	2
15	15	12	6	4	2	2
25	30	30	12	12	8	5
30	30	30	12	12	8	5
50		60	30	15	12	10
60		60	30	15	12	10
100		90	40	30	20	15
150			60	50		

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.

7.2.22.2 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. **Caution**—Do not exceed the maximum operating temperature of the column. Then light or turn on the detector.

7.2.22.3 Alternatively, butane from a disposable lighter can be squirted on a fitting and the detector signal monitored for a response. Leaks at the inlet will take several minutes to respond, since the butane must travel through the entire column before it reaches the detector. The butane leak–checking technique is not nearly as sensitive as a TCD leak detector. Spraying argon gas and monitoring mass 39 has also been found to be very effective for mass spectrometers.

7.2.23 *Setting the Optimum Flow Rate*— The most accurate and reproducible way to set the capillary column flow is by injecting a non-retained substance to determine the column's linear velocity (or 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 since this practice does not account for column-to-column variations. Relying on head pressure readings is not recommended due to both 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.

7.2.23.1 Because capillary columns are usually operated in a pressure- (not flow-) controlled mode, the temperature at which the linear velocity is set is critical. To obtain optimum performance, linear velocity should always be set at the operating temperature for an isothermal analysis. For temperature programmed analyses, the column's linear velocity should be optimized at an oven temperature where a critical or 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 record the temperature at which the linear velocity was set and the non-retained compound used for the analysis so the practice can be easily reproduced.

7.2.23.2 To set a dead time, inject 2.0 µL of a non-retained substance (Table 3) that is compatible with the detector by means of a standard 10-µL syringe with a TFE-fluorocarbon-tipped plunger. Draw 2 µL from the head space over the dead volume compounds in a vial used on selective detectors such as NPDs, ECDs, or ELCDs. Accurately mark the injection starting time and peak elution time with an electronic integrator.

TABLE 3 Recommended Dead Volume Compounds

NOTE 1—These compounds may be slightly retained on thick film phases (1.0 to 7.0 µm) giving slightly erroneous dead volume times. However, they will be reproducible for similar column types.

Detector Type	Dead Volume Compound
FID	methane, propane, butane
TCD	air, methane, butane
NPD	acetonitrile headspace vapors
ECD	methylene chloride headspace vapors
ELCD (halogen mode)	dichlorodifluoromethane headspace vapors
MS	air, methane, propane, butane, argon
ELCD (sulfur mode)	hydrogen sulfide
ELCD (nitrogen mode)	air (without scrubber)

7.2.23.3 Adjust the column head pressure until the correct dead time (Table 4) is obtained for the appropriate column length and carrier gas. Once the dead volume time has been finalized, recheck the split vent and septa purge flow to make sure they did not significantly change. (Head pressure-regulated capillary systems require readjustment if the column head pressure changes significantly. However, back pressure-regulated capillary systems should not require readjustment.)

TABLE 4 Dead Volume Time for Commonly Used Capillary Columns

NOTE 1—The values in the table were obtained using the formula for average linear velocity (u). The optimum u is 40 cm/s for hydrogen, 20 cm/s for helium, and 10 cm/s for nitrogen.[A] To obtain the required dead volume time for column lengths not listed, insert the appropriate values in the following equation.

$$\text{Dead volume time} = \frac{\text{column length (cm)}}{u \text{ of carrier gas (cm/s)}} \quad (4)$$

Column Length, m	Hydrogen (40 cm/s)	Helium (20 cm/s)
10	0 min 25 s	0 min 50 s
15	0 min 38 s	1 min 15 s
25	1 min 03 s	2 min 05 s
30	1 min 15 s	2 min 30 s
50	2 min 05 s	4 min 10 s
60	2 min 30 s	5 min 0 s
100	4 min 10 s	8 min 20 s
105	4 min 23 s	8 min 45 s
150	6 min 15 s	12 min 30 s

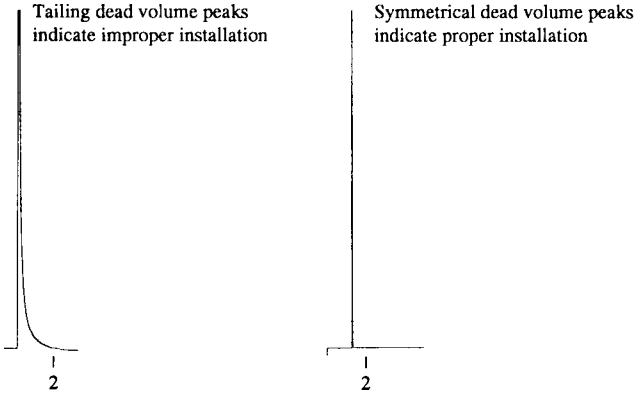
[A] Nitrogen is not recommended as a carrier gas for most capillary columns because inadequate resolution and longer analysis times result.

7.2.24 *Confirming Installation Integrity*— There are two recommended tests to confirm installation integrity: the dead volume peak shape test and the solvent peak shape test.

7.2.25 *Dead Volume Peak Shape Test*—Examine the shape of the peak obtained while setting the dead volume time (column flow). A sharp, narrow dead volume peak that shows no sign of tailing indicates an unobstructed pathway for the sample and a correct installation. Tailing dead volume 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 column which has outlived its useful life (see Fig. 11). The cause of a tailing non-retained peak must be corrected before using the column analytically.

7.2.26 *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 (for example, methane), they are not extremely sensitive indicators of system or installation problems. A 1-µL injection of a liquid solvent such as methylene chloride can expand to 500 µL 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.

7.2.26.1 To perform the test, inject 1 µL of a solvent compatible with the detector (for example, methylene chloride for an FID or pentane for an ECD, etc.) in the split mode at 40°C isothermal and examine the peak shape. 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. See examples in Fig. 12.

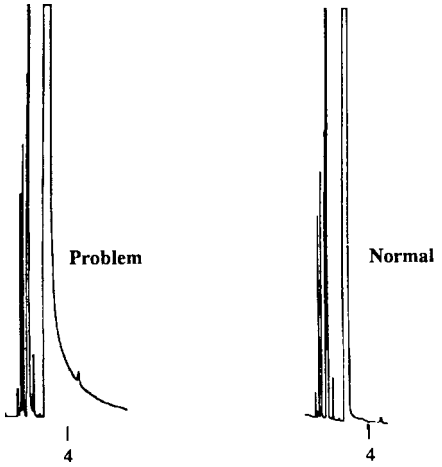


NOTE 1—Non-retained compounds such as methane that show tailing indicate a system or installation problem that must be corrected before using the column analytically.

NOTE 2—30 m, 0.32-mm ID, 0.25 µm 5 % diphenyl − 95 % dimethyl polysiloxane 2.0-µL injection of methane.

Oven temperature: 40°C isothermal
Injector and detector temperature: 325°C
Carrier gas: Hydrogen
Linear velocity: 40 cm/s
FID sensitivity: 4×10^{-11} AFS
Split ratio: 35:1 (vent flow: 80 cm^3/min)

FIG. 11 Methane Peak Symmetry Test for FID, NPD, and TCD



NOTE 1—The shape of the solvent peak is a good indicator of system integrity.

NOTE 2—30 m, 0.32-mm ID, 0.25 µm 5 % diphenyl − 95 % dimethyl polysiloxane 1.0-µL injection of methylene chloride.

Oven temperature: 40°C isothermal
Injector and detector temperature: 325°C
Carrier gas: Hydrogen
Linear velocity: 40 cm/s
FID sensitivity: 4×10^{-11} AFS
Split ratio: 35:1 (vent flow: 80 cm^3/min)

FIG. 12 Solvent Peak Shape Test

7.2.26.2 Some injection modes and types of detectors cause solvent peaks to tail more than others. For instance, a 0.53-mm inside diameter column installed in the direct injection mode will cause slightly more solvent tailing than split systems. To make sure that the column is installed properly and that no system–related problems are present, consult the instrument manual or the capillary column manufacturer's literature to determine the appearance of a normal solvent peak.

7.2.27 *Column Conditioning and Stabilization*—Even though most capillary columns are pre-conditioned by the manufacturer, additional conditioning is necessary by the end-user to stabilize the system. Before conditioning a column at an elevated temperature, make sure that flow is present (that is dead volume test), that no leaks are present, and that 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 to destroy its utility at high-operating temperatures.

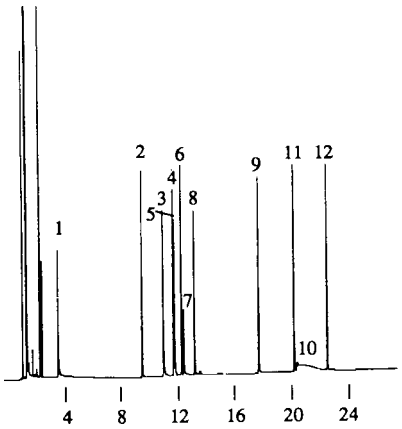
7.2.27.1 To condition the column, set the GC oven to hold 15 min at 40°C, and then program at 10°C/min to the maximum operating temperature as indicated by the column manufacturer. (Alternatively, the column can be conditioned 25°C below the maximum recommended operating temperature if the column is going to be used at relatively low temperatures.) Hold the column at its maximum temperature for two hours or until the baseline stabilizes. Overnight conditioning is necessary when the column is going to be operated at high detector sensitivities (for example, $<4 \times 10^{-11}$ AFS) and at oven temperatures close to the maximum operating temperatures.

7.2.27.2 It is a good practice to condition the column without installing it into the detector in some cases. Very sensitive detectors or hard–to–clean detectors such as ECDs, NPDs, FPDs, PIDs, ELCDs, or mass spectrometers should not be attached to the column during the initial conditioning period. This practice is particularly important with very thick film columns (>3 µm) which produce much more stationary phase bleed. In these cases, remove the column from the detectors, cap off the detector, and proceed with the conditioning procedure.

7.2.28 *Using Test Mixtures to Evaluate Capillary Column Performance*—Most capillary columns are tested by the column manufacturer with a multifunctional test mixture to evaluate the column's separating power and compatibility for various classes of compounds. By running a similar test mixture on your particular GC system, you can determine whether the column is performing optimally or if an instrumental problem should be suspected. In addition, tailing or adsorption of certain test mixture compounds indicate incompatibility with the same class of compounds on that particular column. Therefore, quantitative results for those classes of compounds that exhibit peak tailing and adsorption should be closely monitored for spurious or unreliable results. It is a good analytical practice to run the test mixture prior to analyzing samples to assess system problems or chemical incompatibilities that may be present. It is also good analytical technique to inject the column test mix weekly to monitor the column's performance and to indicate when maintenance procedures such as cleaning inlet sleeves, breaking off contaminated loops, or solvent rinsing are needed.

7.2.28.1 Obtain a test mixture from the column manufacturer and inject it according to the conditions printed on the test chromatogram. Alternatively, the test mixture developed by Grob[6] is very powerful and popular for determining the column's chemical compatibility or system integrity. These Grob mixtures are available from chemical standard manufacturers. Read Grob's articles for a detailed interpretation of the test mixture results. In general, tailing hydrocarbon and FAME (fatty acid methyl ester) 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. Tailing or adsorbed peaks such as 2,3-butanediol, octanol, 2-ethylhexanoic acid, or dicyclohexylamine may indicate the need for cleaning and re–deactivating the split/splitless sleeve. (Some polar columns may adsorb these compounds due to their nature.) An excessively tailing solvent peak also indicates an undeactivated split/splitless sleeve or a problem with the make-up gas system. Figs. 13 and 14 show a column that is performing improperly and properly with the Grob mixture on a relatively non-polar stationary phase. However, analysts should be aware that more polar stationary phases can exhibit a higher degree of tailing for the active probes because of the inherent nature of the stationary phase.

7.2.29 *Running Samples and Calibration Mixtures*—At this point, the column should be properly installed and fully operational to run samples as indicated in the analytical method.



NOTE 1—The Grob mix on this column shows poor performance. Tailing 2,3-butanediol, octanol, and dicyclohexylamine peaks may indicate inadequate split/splitless sleeve deactivation. Asymmetrical hydrocarbon and FAME peaks may indicate that the column has been installed improperly.

NOTE 2—

1. 2,3-butanediol
2. n-decane
3. 1-octanol
4. 2,6-dimethylphenol
5. Nonanal
6. n-dodecane
7. 2-ethylhexanoic acid
8. 2,6-dimethylaniline
9. Methyl decanoate
10. Dicyclohexylamine
11. Methyl undecanoate
12. Methyl dodecanoate

NOTE 3—30 m, 0.32-mm ID, 0.50 µm 100 % dimethyl polysiloxane 0.8-µL injection of the Grob test mixture.

Oven temperature: 40 to 250°C at 6°C/min
Injector and detector temperature: 325°C
Carrier gas: Hydrogen
Linear velocity: 40 cm/s
FID sensitivity: 4×10^{-11} AFS
Split ratio: 35:1 (vent flow: 80 cm^3/min)

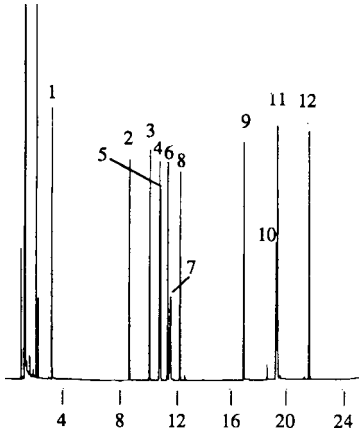
FIG. 13 Grob Mix Showing Poor Chromatographic Performance

8. Operational Information

8.1 *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 10 cc/min (to conserve carrier gas) and maintain an oven temperature between 100 and 150°C. Keeping the oven slightly hot minimizes septum bleed from accumulating at the head of the column and reduces the need for excessive conditioning when the column is subsequently 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.

8.2 *Column Removal and Storage*—Remove the column from the GC, being careful to remove all ferrule fragments from the injector and detector fittings. If a graphite ferrule sticks in the fitting, remove it by using a tapered needle file. 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 is not removed in one piece, disassemble the fitting and inspect all parts to make sure all fragments are removed.

[6] *Journal of Chromatography*, Vol 156, 1978, p. 1 and Vol 219, 1981, p. 13.



NOTE 1—The Grob mix shows that this column is performing adequately. Symmetrical hydrocarbon and FAME peaks indicate that the column has been installed properly (that is, no dead volume exists in column connection). Active probes such as 2,3-butanedial, octanol, nonanal, and dicyclohexylamine are symmetrical which indicates adequate deactivation of split/splitless sleeves.

NOTE 2—

1. 2,3-butanediol
2. n-decane
3. 1-octanol
4. 2,6-dimethylphenol
5. Nonanal
6. n-dodecane
7. 2-ethylhexanoic acid
8. 2,6-dimethylaniline
9. Methyl decanoate
10. Dicyclohexylamine
11. Methyl undecanoate
12. Methyl dodecanoate

NOTE 3—30 m, 0.32-mm ID, 0.50 µm 100 % dimethyl polysiloxane 0.8-µL injection of the Grob test mixture.

Oven temperature: 40°C to 250°C at 6°C/min
Injector and detector temperature: 325°C
Carrier gas: Hydrogen
Linear velocity: 40 cm/s
FID sensitivity: 4×10^{-11} AFS
Split ratio: 35:1 (vent flow: 80 cm^3/min)

FIG. 14 Grob Mix Showing Proper Column Installation

8.2.1 The column ends should be sealed immediately upon removal from the GC, using a high-temperature (2000°C) torch available from most column and instrument manufacturers. If a high-temperature torch is not available, insert the column ends into a septum to prevent oxidation of the phase during storage. Always store the column in the original box, away from sunlight or fluorescent lights, to avoid polymer damage. In particular, polyethylene glycol and cyanosilicone polymers are especially susceptible to uv–induced degradation and should be shielded from light.

8.3 *Protection Against Dirty Samples*— Samples that contain nonvolatile or high-molecular weight residues can decrease quantitative accuracy and cause a loss of peak resolution after a limited number of injections. Two specific precautions are recommended to help increase the number of analyses performed before column and inlet sleeve maintenance is required: packing the inlet sleeve and using a guard column.

8.4 *Packed Inlet Sleeves*—The first precaution involves the use of an inlet sleeve packed with silanized fused silica or glass wool or highly inert deactivated fused silica or glass beads to catch sample residue and prevent it from entering the capillary column. Too much wool or beads can detrimentally affect the performance of the analytical column. Pack the inlet sleeve according to the specifications indicated in the GC instrument manual.

8.4.1 The use of packings coated with a stationary phase is not recommended since the packings tend to be adsorptive and can bleed stationary phase onto the analytical column. This problem is particularly damaging if a methyl silicone inlet packing is used with a polyethylene glycol (PEG) type capillary column.

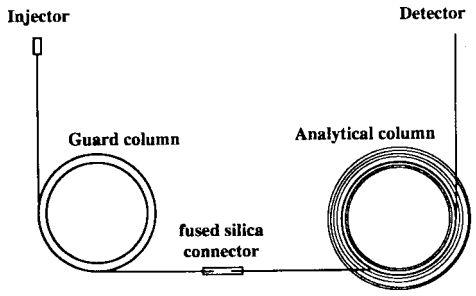
8.5 *Guard Columns*—The second precaution involves the use of a 5-m deactivated, uncoated guard column at the column inlet (see Fig. 15). The guard column serves to trap nonvolatile contaminants before they enter the coated analytical capillary column, thus prolonging the life of the analytical column. A 5-m guard column also allows several 1-m segments to be removed as the front becomes contaminated without having to reconnect the column each time.

8.6 *Connecting Fused Silica Tubing*— Connecting fused silica tubing together is necessary for attaching a guard column, attaching a restrictor to the column outlet, or for repairing a broken analytical column. Either metal connectors, which incorporate a single ferrule to encompass the column connection point, or tapered fused silica connectors (sometimes called presstight or pressfit connectors), which compress the column end and make a seal with the polyimide coating, can be used. Metal connectors offer a mechanically stable seal. However, tapered fused silica connectors provide better inertness and do not cause solvent or other analyte peaks to tail.

8.6.1 It is especially important to cut the column end squarely using a sapphire blade or scoring wafer to obtain a seal with fused silica compression connectors. (See 7.2.15 for cutting guidelines.) Tapered fused silica connectors will not form a tight seal if pointed-tipped diamond or tungsten carbide scribes are used. It is also equally important to clean and lubricate the column end prior to inserting it into the tapered fused silica connector. Moisten a towel with DI H_2 O, methanol, toluene, or another solvent and wipe the last 2 cm of the column end in a motion towards the column outlet.

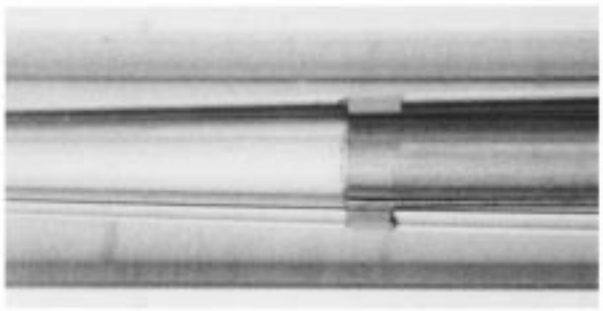
8.6.2 Firmly press the column end tightly into the connector, being careful not to apply so much force that the column end crushes. Visually examine the compression sealing ring to make sure it is continuous around the end of the column (see Fig. 16) and leak check the connection as described in 7.2.23.

8.6.3 Polyimide glues can be used to strengthen the connection. The polyimide glue should be placed sparingly halfway around the end of the connector and should not encompass the entire column end. Otherwise, polyimide glue will only dry on the outer skin and force solvents and glue deep inside the



NOTE 1—A guard column protects the analytical column from damage by nonvolatile residue, particulates, or derivatization.

FIG. 15 Fused Silica Guard Column



NOTE 1—A proper seal is made when a continuous ring forms at the column end and connector mating point.

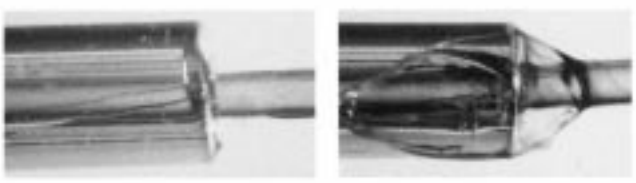
FIG. 16 Proper Column Connection to Tapered Fused Silica Connector

connector and column bore rendering the connection useless. Dry the glue slowly by programming the oven from 40 to 150°C at 4°C/min and hold for 30 min. Continue the temperature program from 150 to 250°C at 1°C/min to strengthen the bond between the column end and the tapered fused silica connector. (See Fig. 17.)

8.7 *Column Maintenance*—Column 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 1 m) 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.

8.8 *Removing Loops from the Column Inlet*—Two loops from the inlet end and one loop from the outlet end should be cut and removed from the column to remove pyrolyzed contaminants that are not solvent soluble. Closely examine the inlet end of the column to make sure all contaminated tubing has been removed. Occasionally, it becomes necessary to remove more than two loops to restore the column to acceptable performance if it is heavily contaminated.

8.9 *Solvent Rinsing Bonded Phase Capillary Columns*—Solvent rinsing removes only soluble contaminants and may not always restore a bonded phase capillary column to its original performance. Choose a series of polar and non-polar volatile solvents that will solubilize the suspected contamination. Verify its compatibility with the bonded phase by consulting the manufacturer's literature.



NOTE 1—These photos show proper and improper amounts of polyimide glue.

FIG. 17 Applying Polyimide Glue

8.9.1 Some analysts have found that a mixture of 60 % methanol, 20 % deionized water, and 20 % methylene chloride is ideal for rinsing and cleaning most bonded stationary phases. The methylene chloride tends to swell the polymer and the methanol DI H_2 O penetrates deeply 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. Do not use this mixture unless the compatibility with the specific bonded phase is verified.

8.9.2 Always rinse the column from the detector end to the inlet end (that is, backflushing) to prevent contaminants lodged on the inlet from being carried deeper inside the column bore. Columns can be rinsed by either forcing solvent through the column under pressure or by pulling solvent through under vacuum. Most capillary column suppliers offer devices that connect the column to a pressurized glass reservoir.

8.9.3 The amount of solvent used to rinse a column should be approximately equal to three to five times the column's internal volume. Table 5 lists typical solvent volumes and pressures used to rinse each column inside diameter.

8.9.4 Sometimes very thick films (>3 µm) can swell shut and occlude the column flow while solvent rinsing. Do not try to remove the blockage by increasing the rinsing pressure. Attach both column ends to a vacuum system and evacuate for 24 h. The vacuum evaporates the solvent and opens the column bore. Use non-swelling solvents such as methanol or acetone to minimize swelling while solvent rinsing thick film columns.

8.9.5 Allow the column to purge with a dry carrier gas for 4 to 6 h after rinsing. A dry carrier gas purge is necessary to decrease polymer swelling after exposure to solvents. If the column is installed in a GC and heated rapidly before the solvent swelling has diminished, extensive damage to the polymer occurs. After the column has purged, install it in the GC and hold it for 30 min at 40°C. Program slowly (4°C/ min) to the column's maximum operating temperature and condition for 2 h prior to use.

9. Keywords

9.1 capillary columns; capillary gas chromatography; gas chromatographs; installing capillary columns

TABLE 5 Typical Solvent Volumes and Pressures Used to Rinse Bonded Phase Capillary Columns

Column ID, mm	Solvent Volume, mL	Reservoir Pressure, psig
0.10	5	60
0.18	10	50
0.20	10	50
0.25	15	40
0.32	25	30
0.53	50	15
0.75	100	10