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

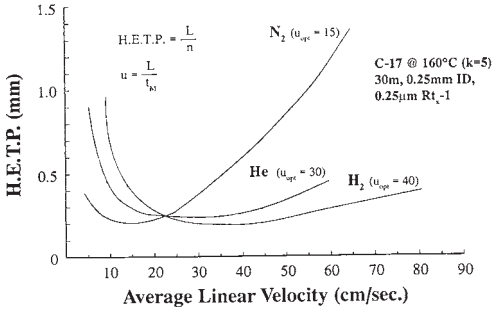
3. Terminology

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_3 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



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]

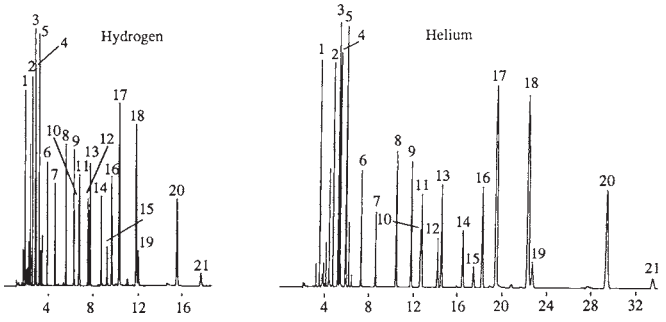
[1] This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.19 on Chromatography.

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

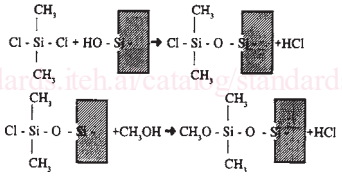


FIG. 3 Capping Silanol Groups with Dimethyl Dichlorosilane (DMDCS)

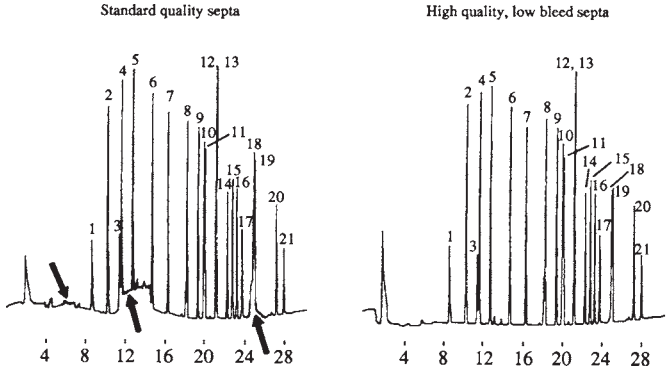
phase which is then deposited on the inside column wall to improve stationary phase stability and sample capacity.

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



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

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

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.

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.

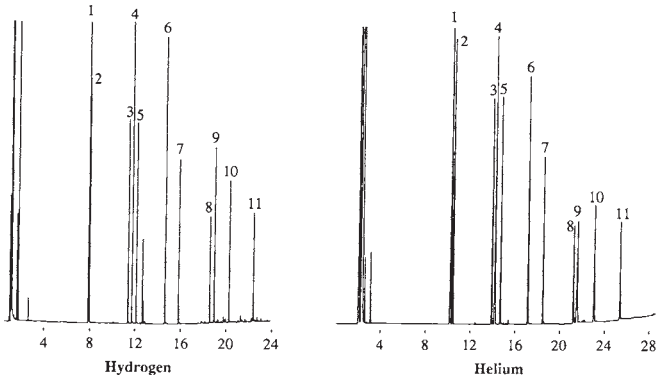
7.2.1.2 Only high–purity gases should be used for capillary chromatography. All regulators should be equipped with stainless steel diaphragms. Regulators equipped with rubber or elastomeric diaphragms should not be used because oxygen, moisture, and elastomeric contaminants migrate through the diaphragm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



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)

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.

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.

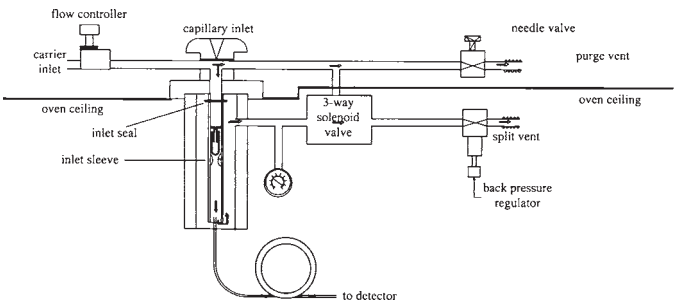


FIG. 6 Flow-Regulated Back Pressure System

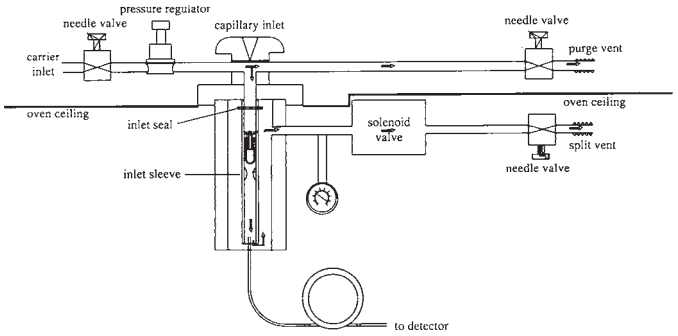


FIG. 7 Head Pressure Regulated System

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 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 re-adsorption 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 non-volatile 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.

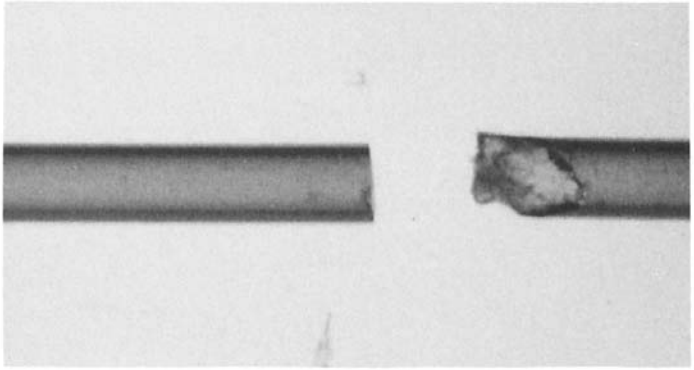
7.2.13.1 Graphite ferrules are the easiest to use, because they are leak-free, universal for most systems, and the choice for most beginning capillary chromatographers. Vespel®/graphite ferrules are preferred for use in mass spectrometers since they do not flake and contaminate the ion source with particles. 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.



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.