Installation and Maintenance Instructions for 0.75mm Wide Bore Capillary Columns

These instructions cover instrument preparation, ferrule, and column installation, leak checking, gas flow setting procedures, and maintenance requirements for 0.75mm wide bore capillary columns in packed column (equipped with capillary column conversion hardware) and capillary column GC systems. This information and your instrument's instruction manual will enable you to properly install and maintain wide bore columns in your GC.

Key Words:
• wide bore capillary column • injector • detector • ferrule

Instrument Preparation

Before installing your column, make certain the injector and detector liners (if present) are clean and free of sample residue or septum and capillary fragments. To prevent adsorption problems, the injector and detector liners should be silanized. Cleaning and silanizing procedures can be found under the Maintenance section of this bulletin.

Once the system is clean, set the injector and detector temperatures according to the parameters on the test chromatogram. **Never** set these temperatures above the maximum limit of the stationary phase.

Oxygen and water, normally present in gas cylinders, must be removed from the carrier gas or column life will be shortened. This purified carrier gas is especially important for polar phases, such as SUPELCOWAX™ 10 and SP™-2330. A full line of carrier gas purifiers is available from Supelco. Any carrier gas pressure regulator located downstream of the carrier gas purifier should contain a stainless steel diaphragm to prevent the diffusion of oxygen into the carrier gas (Grob, K., HRC & CC, 173, 1978). Request Supelco Bulletin 848 for carrier gas purification information.

Placing the Column and Cage in the Oven

The fused silica ends on the wide bore column allow you to place the cage in almost any horizontally or vertically oriented oven. Special column hangers are not necessary. Hang the cage or stand it on the oven floor with the extended rod ends down.

Column Installation for Packed or Capillary Column Instruments

1. Squarely cut both sealed ends of the fused silica tubing approximately 1" from each column end (Figure A). Use a Capillary Cleaving™ Tool (Cat. No. 23814 or 23740-U) or other scoring device to cut the fused silica tubing's coating. To break the scored end, bend the tubing slightly while pulling away. **Always point the column end toward the floor when breaking the tubing so particles fall away from the column (Figure B).**

Figure A. Cutting Fused Silica Tubing

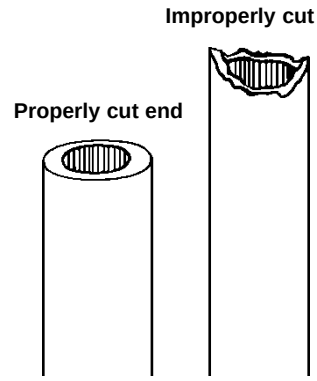

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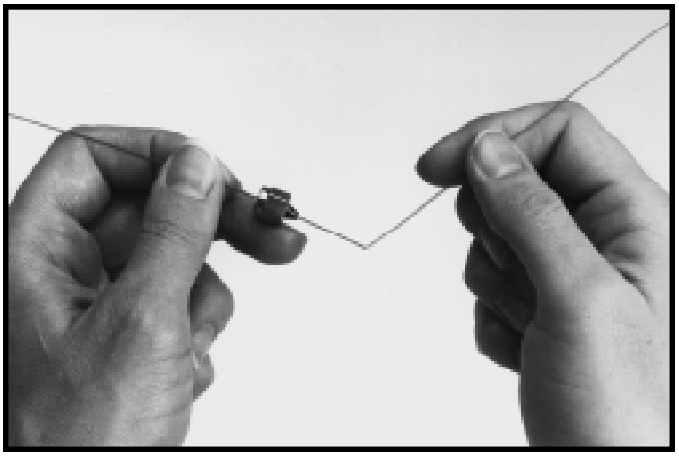
Figure B. Break Toward Floor



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2. Slide the injection port fitting over the column end. The threads on the fittings in the Supelco™ Direct Injection Conversion Kit should face the column end.
3. Slide a 0.5mm ID ferrule over the fused silica tubing with the column end pointing down. If you are using the Supelco Direct Injection Conversion Kit, slide the tapered end of the ferrule over the column. In some cases, the 0.5mm ID ferrule will not fit over the 0.32mm ID fused silica tubing because the protective coating may be oversized. If this happens, use a Pin Vise Drill Kit (Cat. No. 23820-U) or Needle File (Cat. No. 23783) to enlarge the ferrule ID.
4. Cut approximately 1 to 2" off the column end (pointing it down and cutting squarely) to remove ferrule fragments that may have fallen inside the capillary bore. These fragments can cause peak tailing and adsorption of reactive sample components.
5. Adjust the ferrule and fitting to the proper height and insert the column end into the GC injection port fitting. If you are using the Supelco Direct Injection Conversion Kit, adjust the ferrule so that 1/16" of fused silica tubing protrudes from the back of the ferrule (Figure C). For capillary inlet systems, efer to your instruction manual for the correct insertion distance. A convenient way to ensure correct insertion distance is to place a mark **behind** the capillary fitting with either typewriter correction fluid or a felt tipped marker (Figure D). If the tubing moves upon insertion, it can be repositioned using the reference mark.
6. Tighten the fitting (approximately 1/4 to 1/2 turn past fingertight) until the fused silica tubing is held firmly by the ferrule. If the tubing moves, reposition to the correct insertion distance.

Figure C. Cross Section of Direct Injection Conversion Kit and Column After Installation

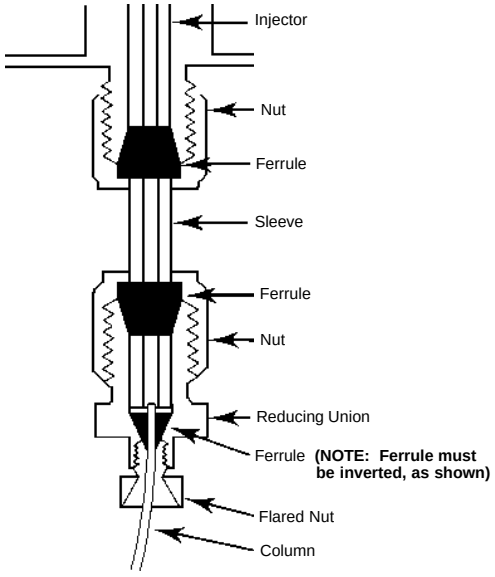

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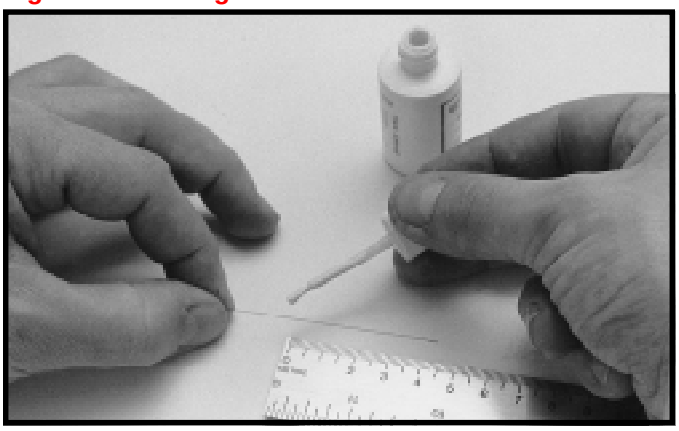
Figure D. Marking the Insertion Distance



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7. Turn on the carrier gas to purge the column before connecting the column to the detector. Head pressure should be about 4psig for a 30 meter and 7psig for a 60 meter wide bore column. Later you will fine tune the pressure when op-timizing the column flow rate.
8. Prepare the other column end as described in steps 1-4.
9. Insert the column end into the detector fitting at the proper height and tighten the fitting as described in steps 5 and 6. If you are using a Supelco Make-Up Gas Adapter, insert the column end so it protrudes 3/16" or more beyond the fer-rule. For most capillary detector fittings (including Supelco's make-up adapter kit), the column end can be inserted very close to the detector orifice. This will prevent column eff-luent from contacting adsorptive surfaces inside the detector fitting. When connecting the column to an FID, make sure the flame is out. Otherwise the column end could be charred if accidentally pushed through the jet orifice. The column end must exit below the jet orifice (or the radioactive foil in an ECD) or chromatographic performance will be impaired.
10. Position the column away from the oven door or other cold spots in the oven. Cooler air blowing across the fused silica tubing causes irregularly shaped peaks with jagged leading and tailing edges.

Checking for Leaks

Once the column is connected to the instrument, turn on the carrier and make-up gases and check the fittings for leaks. **Do not use liquid leak detectors.** These liquids can be drawn into the column or column fittings and contaminate the system. The best way to leak-check a capillary system is with GOW-MAC® Gas Leak Detectors (Deluxe Model, Cat. No. 22409; Mini Model, Cat. No. 22807 or 22808). These detectors operate on the same principle as a thermal conductivity detector. They are highly sensitive to low concentrations of He, H_2, and N_2 and cannot contaminate the instrument or column.

If GOW-MAC Gas Leak Detectors are unavailable and you are using Supeltex™ M-2A or Supeltex M-4 ferrules, minimize the risk of leaks by tightening the ferrules until the tubing no longer moves in the fittings. (Be sure to readjust insertion distances.) One-fourth turn past fingertight is usually sufficient. But, be careful: oxygen entering a leaking connection could shorten the life of your column.

Gas Flow Setting Procedure

Packed Column Instrument with Supelco Direct Injection Conversion Kit

1. Using carrier gas flow controller, adjust the flow of helium▼ through the column to about 5cc/min. Measure the flow rate at the detector. Be sure all other gases are turned off or erroneous flow readings will be obtained. Allow 15 minutes between the adjustments for the flow controller to stabilize the flow.
2. Set the make-up gas flow. This flow plus the 5cc/min flow through the capillary column should equal the manufacturer's recommended flow rate for your detector (30-60cc/min).

▼Helium is superior to nitrogen as the carrier gas. It offers faster analysis times and provides more resolving power at flow rates higher than optimum. (See discussion in R.R. Freeman's *High Resolution Gas Chromatography*, p. 18, Figure 1.5. This book is available from Supelco as Cat. No. 23512.)

Capillary Column Instrument

There are three flows to adjust in a capillary system: (1) make-up gas, (2) splitter vent, and (3) column flow. These flows should be set in the above order at ambient oven temperature, until otherwise specified in these instructions.

1. The first and easiest flow to set is the make-up gas. Once set, it should not require altering. This flow, plus the 5cc/min flow through the capillary column, should equal the manufacturer's flow rate for your detector. Refer to your instrument manual for instructions and specifications for setting this flow — typically 30-60cc/min.
2. The second flow to set, the splitter vent flow, determines the split ratio. Unlike the make-up gas flow, the splitter vent flow must be readjusted each time the starting oven temperature or carrier gas head pressure is altered. We recommend setting the splitter vent flow at 240-250cc/min to provide an approximate split ratio of 50:1. Other samples may require lower split ratios to improve detection, or higher ratios to prevent column overload.
3. Turn on your detector and inject 25-50µL of a 1% methane in N_2 gas blend (Cat. No. 23443) onto the column to see if the linear velocity (i.e., column flow) is correct. See Table 1 for the appropriate methane retention times for the most common carrier gases.
4. Adjust column head pressure until the CH_4 peak elutes at the appropriate time indicated below. In volume, this will be about 5cc/min. If tailing is evident, there could be dead volume in the system. If there is no peak at all, suspect a hook-up or detector problem. Before proceeding with the conditioning procedure, make the appropriate corrections.

Note: If the carrier gas flow rate must be altered by more than a few cc/min, readjust the make-up gas.

Table 1. Recommended Methane Retention Times◆

Column Length (m)	Carrier Gas (min:sec) H_2	He	N_2
30	1:15	2:30	5:00
60	2:30	5:00	10:00

◆There are different optimum average linear velocities for different carrier gases: i.e., H_2 — 40cm/sec, He — 20cm/sec, N_2 — 10cm/sec.

Conditioning the Column

To prevent phase oxidation, purge the column with carrier gas for 30-60 minutes before heating the oven. The column is then ready to program up in temperature (see conditioning instructions with your chart). For most applications, further column conditioning is unnecessary. To minimize bleed or baseline rise during a temperature programmed analysis at high detector sensitivities, we recommend program conditioning the column. Set the instrument to repetitively cycle the oven temperature up and down overnight, following the same temperature program to be used for the analysis. Programmed conditioning stabilizes the baseline much faster than conditioning at a high isothermal temperature. Remember to heat and cool capillary columns **slowly**. Use temperature programming rates of less than 25°C/min and allow the oven cooling mechanism to operate automatically (see conditioning instructions with your column). Thermal shocks could damage a capillary column by causing the phase to puddle.

Injecting the Test Mix

To determine if the system is operating properly, you must make a test mix injection. A vial of test mix is included with all columns except those that are specially tested. Refer to the appropriate procedure below. If you cannot obtain efficiencies and peak height ratios similar to those on the test chromatogram, you may have installation or instrument problems. If so, troubleshoot the system using these instructions, or contact our Technical Service Department. When you can duplicate the test results, your column will be performing at optimum.

Packed Column Instrument

For direct injections, dilute the mix 1:10 in carbon disulfide and inject 0.2µL onto the column. (If carbon disulfide is not available, methylene chloride may be used, but the resulting solvent peak will be wider). This injection will provide 10ng of each component on column. To check the system, set all parameters as indicated on the test chromatogram. Poorly shaped peaks indicate excess dead volume or some other installation problem. To correct this, refer to the Direct Injection Conversion Kit and/or test mix instruction sheets.

Note: This wide bore column was tested in a capillary system, using split injections. In your packed column system, with direct injection conditions, the solvent peak will be wider than that on the chromatogram accompanying the column. You should, however, obtain similar efficiencies and peak height ratios.

Capillary Column Instrument

Use the test mix undiluted. For conditions, see the QA test chromatogram. To correct problems, refer to the test mix instruction sheet.

Optimizing Resolution and Analysis Time for Your Sample

After testing the column with the test mix, slowly adjust the temperature to the desired operating level for your sample. Inject a trial aliquot of your sample and optimize resolution and analysis time. Determine the fastest oven temperature program rate or the highest isothermal operating temperature that will continue to resolve your components. Increase your column flow rate to the fastest analysis time that will still allow sample resolution. You may operate the column at flow rates up to 20cc/min. However, resolution will be lost at flows above the optimum.

Although a sample size up to 5µL can be injected, use the smallest possible injection to minimize the width of the solvent peak. Smaller sample size will also increase the resolution of closely eluting peaks.

Precautions

Prevent thermal shock — Nonbonded phases are particularly sensitive to thermal shocks that can permanently damage a capillary column. Never heat or cool any capillary column at more than 25°C/minute. Cool the column by allowing the oven door mechanism to operate automatically. **Do not** force the column to cool faster by opening the door wide or using cryogenic cooling.

Change the fused silica tubing when necessary — The deactivated fused silica tubing on the inlet end of your wide bore column acts as a guard column. When you perform on-column injections, the guard column helps prevent nonvolatile and insoluble sample components from contaminating and ultimately damaging the analytical column. Replace this tubing when it becomes contaminated (see Replacing the Fused Silica Column Ends).

Maintenance

Accurate qualitative and quantitative capillary chromatography requires a strict program of maintenance, described in detail below.

Removing the Column from the GC

Loosen the nut holding the column and let it slide down the column. Place thumb and forefinger immediately below the connection and gently wiggle the column while pulling downward. Both column and ferrule should come free. If the column is removed but the ferrule remains in the connection, carefully insert the tip of a small needle file into the ferrule bore. Twist file clockwise to tighten, then wiggle the file to remove the ferrule. If an M-4 ferrule was used, the fitting and glass sleeve may have to be disassembled to remove graphite fragments.

Injector/Detector Liners

Injector and detector liners are in direct contact with the sample during the analysis. They can, if dirty, adsorb sample components. In many analyses only 1-5ng of a sample component pass through the column. Therefore, clean, inert liners are important. Most available liners are not deactivated, although they should be. Deactivated liners for most GCs can be found in the Supelco catalog.

Since injector sleeves can become contaminated with septum fragments and sample residue, examine the sleeves each time the septum is changed. If dirty, clean them by rinsing with pentane, methylene chloride or acetone. These solvents do not affect the deactivated surface. If a harsher chemical clean-up is necessary, or if a water-soap solution is used, the surface may have to be redeactivated with Sylon™ CT (Cat. No. 33065-U). If a sleeve cannot be cleaned with organic solvents, we recommend discarding it and using a new, deactivated sleeve.

Routine Column Maintenance

To ensure that your chromatographic system is performing optimally, we recommend you make a weekly injection of the appropriate isothermal test mix. If you cannot duplicate the original test chromatogram from week to week, a problem exists and must be corrected. Interpretation of the test results are covered in the test mix instruction sheet.

Depending on use, capillary columns may eventually show tailing, broadening peaks, or retention changes. If your column shows tailing peaks and dirty liners are not the cause, the problem could be septum fragments or sample residue contaminating the inlet end. You can quickly cure this by replacing the tubing (see Replacing the Fused Silica Column Ends). If necessary, a bonded phase column can be rinsed with a solvent to remove contaminants (see Rinsing a Bonded Phase).

If a gradual loss of column efficiency or decrease in retention times is observed, there are two possible causes: (1) the guard column is dirty (simply replace the fused silica tubing) or (2) phase has gradually bled from the inlet end of the column and recondensed farther down. This phase gradient results from having a continual one-directional flow. Avoid this by periodically (about every two weeks) reversing the inlet and detector ends of the column, and thus the direction of flow. This procedure should be used as a preventative, not a cure.

Replacing the Fused Silica Column Ends

If the fused silica tubing at the inlet end of the column becomes contaminated, replace it to maintain column performance. Use the following procedure:

1. Carefully remove the nut on the end of the butt connector using two 1/4" box-end wrenches. To avoid breaking the column end, do not allow the butt connector body to rotate.
2. Remove the ferrule from the housing by carefully pushing the glass column end toward the butt connector body.

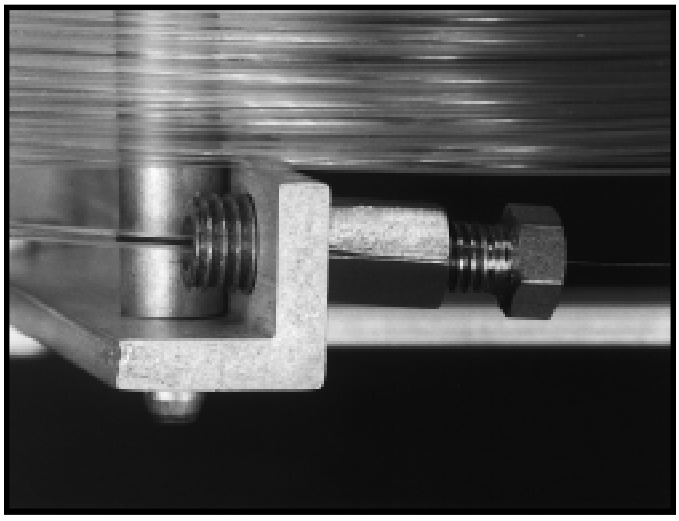
2a. If the ferrule is stuck in the housing, cut the glass column as close to the butt connector as possible. Use a needle file or paper clip to push the ferrule out of the connector. Obtain a new ferrule (Cat. No. 22456) and proceed to step 4 below. Follow previous instructions for cutting fused silica tubing or glass capillary tubing.

2b. If the butt connector ferrule is easily removed from the housing, use a gentle twisting motion to remove the column end from the ferrule. Similarly, remove the fused silica tubing from the ferrule.

3. Make sure the glass column and the new piece of fused silica tubing easily fit into the appropriate ends of the butt connector ferrule. Occasionally the outside diameter of the column or fused silica tubing may be larger than the ferrule ID. If this is the case, use the pin vise drill kit to slightly enlarge the ferrule ID until it fits over the column or tubing end. After drilling, be sure to blow out any ferrule fragments with N_2.

4. Insert a new 1/2 meter piece of deactivated 0.32mm ID fused silica tubing in the small hole of the butt connector ferrule, pushing the tubing completely through the ferrule with the column end pointed down. Cut one inch off the tubing to remove ferrule fragments, then position the ferrule so that the fused silica tubing protrudes 1/16" to 1/4" from the larger hole.
5. Using a magnifier, make sure both tubing ends are squarely cut and clean.
6. Push the glass column end through the butt connector housing until the column end protrudes approximately 1/2".
7. Carefully guide the end of the fused silica tubing into the glass column, using a gentle, rotating motion to work the ferrule onto the glass column.
8. Move the butt connector ferrule into the housing.
9. Slide the butt connector nut over the fused silica tubing, threaded end first. Tighten the nut 1/4 to 1/2 turn past fingertight. If the glass column loop rotates upon tightening, slide it back to relieve the strain. The fused silica end should be visible within the glass column (Figure E).
10. Gently pull on both pieces of tubing. If either moves in the ferrule, push the ends back in place and tighten the butt connector fitting further.
11. Leak check the connection with a GOW-MAC gas leak detector or other TC detector (see Checking for Leaks).

Figure E. Fused Silica Tubing and Wide Bore Column, Properly Installed in a Butt Connector♦



♦ Butt Connector US Pat. No. 4,529,230

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Removing and Reinstalling Columns in a Cage

To remove the column from the cage:

1. Make sure the cage top (with rods riveted to it) is down, or the column may fall to the floor when the cage is disassembled.
2. Disconnect the column from the butt connectors (see Replacing the Fused Silica Column Ends).
3. Remove the springs that hold the cage together by pulling the spring end out of the hole in the frame with a pair of needle-nosed pliers. Allow the spring to contract **slowly**.
4. Gently pull the top part of the cage from the bottom part. The column will now slide off the retaining rods.
5. Reinstall the column (or its replacement) by reversing the above order. Put the springs in the inner holes in the cage when mounting a 30 meter column. Use the outer holes when mounting a 60 meter column.
6. The column must be realigned in the cage to reduce strain at the butt connections. Place the cage on its side and pull outward at the centers of the springs, then gently release them. This will allow the coils to fall into a strain-free position.

Rinsing a Bonded Phase

You can rinse a Supelco bonded phase capillary column with certain solvents to remove soluble contaminants that cause adsorption and peak tailing. Rinsing, however, also removes polymer fragments formed by thermal degradation of the phase during analyses. Therefore, each time the column is rinsed and subsequently heated, analysis time decreases by approximately 5%.

We recommend routine replacement of the fused silica tubing attached to either end, rather than column rinsing. This fused silica tubing protects the column from sample nonvolatiles. Thus, prompt replacement when the column performance begins to deteriorate will almost always eliminate the need to rinse the column.

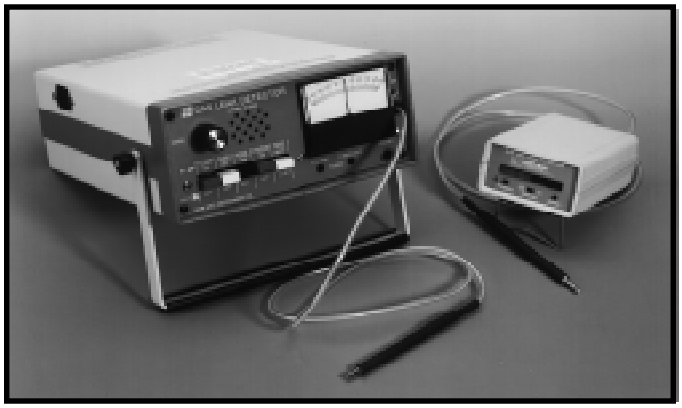
If necessary, however, you can rinse your Supelco bonded phase wide bore column in one of several ways. To flush contaminants from the inlet, solvent should always flow to the inlet from the outlet. You may find it easiest to attach the inlet end of the column to a vacuum source and pull the solvent through the column.

Alternatively, solvent may be pushed through the column from a pressurized reservoir at approximately 30psig. All Supelco bonded phase columns can be rinsed with pentane, methylene chloride or acetone. Other solvents are unpredictable and therefore not recommended. We suggest solvent volumes of 3-5mL.
Rinsing with solvents will not restore the column if contaminants have polymerized in the column inlet. However, the fused silica guard columns on Supelco wide bore capillary columns preclude this problem.

Ordering Information:

Helpful Products Simplify Column Installation

GOW-MAC Gas Leak Detectors for GC Fittings



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- Deluxe model with meter readout and audible leak alarm
- Smallest mini detector available, only one with a rechargeable battery

GOW-MAC Gas Leak Detectors use thermal conductivity to accurately locate gas leaks. The units pinpoint leaks by detecting gases that have a thermal conductivity value different from that of air. Helium leaks of 1 x 10^{-5}cc/sec and refrigerant leaks of 1 x 10^{-4}cc/sec are easily detected. Argon, CO_2, fluorocarbon, and H_2/He (40:60) leaks can also be detected at very low levels. This clean and efficient method of leak detection completely eliminates the risk of system contamination that can result from using soap solution.

Description	Cat. No.
Deluxe Model	**22409**
Mini Model	
115VAC/60Hz	**22807**
230VAC/50Hz	**22808**
Carrying case for mini model	**22809**

Make Your Capillary Columns Last Longer with a High Capacity Carrier Gas Purifier

- Removes oxygen at high concentrations when disposable purifiers cannot
- Can triple the life of a capillary column
- Use with all carrier gas (except hydrogen) with flow rates as high as 1100cc/min

Supelco's high capacity gas purifier prevents carrier gas with high concentrations of oxygen or water from destroying your capillary column. By ensuring that only pure gases enter the column, this purifier can extend the life of your column dramatically.

Power (VAC)	Fitting (inches)	Cat. No.
115	1/8	**23800-U**
115	1/4	**23802**
220	1/8	**23801**
220	1/4	**23803**

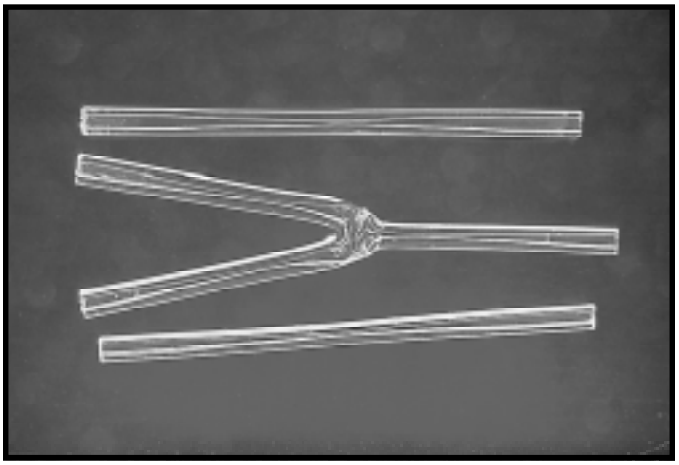
Capillary Cleaving Tool Permits Perfect Butt Connections

912-0177

Make scalpel-like cuts in both polyimide and fused silica — no jagged edges to create problems. Industrial sapphire cutting edges remain sharp indefinitely. The spring-loaded retractable blade version reduces the chances of breakage if the tool is dropped.

Description	Cat. No.
Capillary Cleaving Tool with fixed blade	**23740-U**
Capillary Cleaving Tool with retractable blade	**23814**

GlasSeal Capillary Column Connector — One Size Fits All



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GlasSeal connectors immediately connect fused silica tubing of the same or different diameter — no tools, no leaks. Use to connect a guard column or transfer line, repair a broken column, or connect columns having the same or different phases. "Y" connectors split a sample to two columns or a column effluent to two detectors. Silanized for an inert inside surface. Choose borosilicate glass or fused silica. For use with our 0.25mm-0.53mm ID tubing.

Description	Cat. No.
GlasSeal Capillary Column Connectors	
Borosilicate, pk. of 12	**20479**
Fused Silica, pk. of 25	**23628**
"Y" GlasSeal Capillary Column Connectors	
Borosilicate, each	**20480**
Fused Silica, pk. of 3	**23632**

Ferrules for Use with Wide Bore Capillary Columns

- 1/4″ ID ferrules will connect the sleeve in our 1/4″ conversion kits to the instrument and to the special adapter
- 1/8″ ID ferrules will connect the sleeve in our 1/8″ conversion kits to the instrument and to the special adapter
- 0.5mm ID ferrules will connect the fused silica lines on a Supelco 0.75mm ID column to the special adapters in our conversion kits

	Quantity	1/4"	1/8"	0.5mm❑
Supeltex M-2A■ (15% graphite/85% polyimide)				
	10	**22481**	**22483-U**	**22461**
	50	**22471**	**22472**	**—**
Supeltex M-4 (flexible graphite)				
	10	**22492**	**22491**	**22462**
	50	**22478**	**—**	**—**

Double-Tapered Ferrules for Capillary Column Butt Connector (0.5 to 1.2mm ID)

Description	Cat. No.
Supeltex M-2▼ Ferrules, pk. of 2	**22466**
Supeltex M-2B● Ferrules, pk. of 2	**22456**

■ DuPont VESPEL® SP-21 part

▼ DuPont VESPEL SP-1 part (100% polyimide) max. temp. 350°C

● DuPont VESPEL SP-211 part (100% Teflon®, 15%graphite, 75% polyimide) max. temp. 350°C

❑ Use with 1/16" fittings

Butt Connector US Pat. No. 4,529,230.

Trademarks

CARBOWAX — Union Carbide Corp.
Cleaving — Sigma-Aldrich Co.
Freon — E.I. du Pont de Nemours & Co., Inc.
GlasSeal — Sigma-Aldrich Co
GOW-MAC — GOW-MAC Instrument Co.
SP — Sigma-Aldrich Co
Supelco — Sigma-Aldrich Co
SUPELCOWAX — Sigma-Aldrich Co
Supeltex — Sigma-Aldrich Co
Swagelok — Crawford Fitting Co.
Sylon — Sigma-Aldrich Co
Teflon — E.I. du Pont de Nemours & Co., Inc.
VESPEL — E.I. du Pont de Nemours & Co., Inc.
VOCOL — Sigma-Aldrich Co

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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