Installation and Maintenance Instructions for 0.53mm ID Fused Silica Capillary Columns

These instructions cover instrument preparation, column installation, leak checking, gas flow setting procedures, and maintenance requirements for 0.53mm ID 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 these columns in your GC.

Key Words:
● fused silica capillary column ● injector ● detector ● ferrule

CAUTION: Always wear safety glasses when handling 0.53mm ID fused silica columns, especially when cutting the tubing. Although the tubing is flexible, small particles often fly off when the tubing breaks. Mishandling can cause injury.

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.

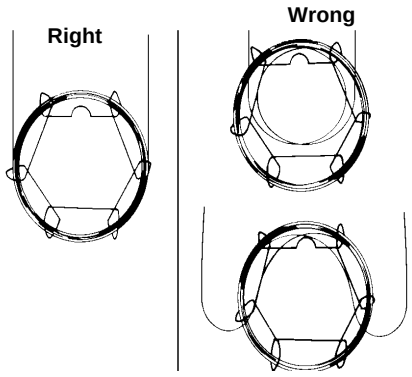
Once the system is clean, set the injector and detector temperatures according to the specifications provided with the test chromatogram. **Never** set these temperatures above the maximum limit of the stationary phase.

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 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 diffusion of oxygen into the carrier gas (Grob, K., HRC & CC, *3*, 173, 1978.) Request Supelco Bulletin 848 for carrier gas purification information.

Column Hanging Procedure

Your capillary column must be suspended properly in the oven — supported by its metal cage, not by the fused silica tubing. Sharp bends in the tubing can weaken and eventually break the column. Avoid them by making sure column ends cross at the *bottom* of the cage, not at the top, when connecting the column to the injector and detector (Figure A).

Figure A. Proper Column Installation Prevents Stress on the Tubing



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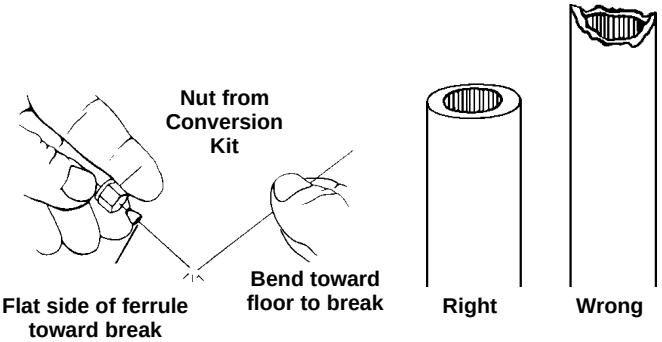
Column Installation for Packed or Capillary Column Instruments

1. Cut both sealed ends of the fused silica tubing approximately 1" from each column end. Use a Capillary Cleaving Tool (Cat. No. 23814 or 23740-U) to cut the fused silica tubing's protective coating. To break the scored end, bend the tubing slightly while pulling away. **Always point the column end down when breaking the tubing (Figure B). Otherwise, shards might fall into the large bore of the tubing.**

Figure B. Breaking Fused Silica Tubing



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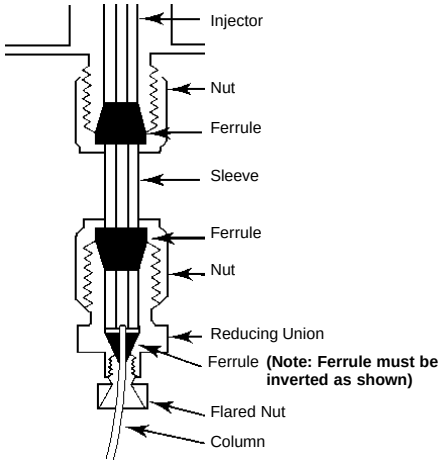
2. Slide the injection port fitting over the column end. The threads on the fittings in the Supelco™ Direct Injection Conversion Kit should face the column end.
3. Slide a 0.8mm ID ferrule over the fused silica tubing with the column end pointing down. If you are using the Supelco Direct Injection Conversion Kit, slide the ferrule tapered end first over the column end.
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, refer to your GC's 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. If the tubing moves upon insertion, it can be repositioned using the reference mark.

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



6. Tighten the fitting (approximately 1/4 to 1/2 turn past fingertight) until the column is held firmly by the ferrule. If the tubing moves, reposition the column to the correct insertion distance.
7. Turn on the carrier gas to purge the column before connecting the column to the detector. Head pressure should be about 5psig for a 15 meter, 0.53mm ID column, 10psig for a 30 meter column, and 20psig for a 60 meter column. Later you will fine tune the pressure when optimizing 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 ferrule. 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 effluent 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 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 2.5cc/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 2.5cc/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 sent in the above order at ambient oven temperature, unless otherwise specified in these instructions.

1. The first and easiest flow to set is the make-up gas. This flow, plus the 2.5cc/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. The splitter vent flow in a pressure regulated carrier gas system must be readjusted each time the starting oven temperature or carrier gas head pressure is altered. We recommend setting the splitter vent flow at 240-270cc/min to provide an approximate split ratio of 100:1. 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.

If you are using an electrochemical detector, use an inert halogenated gas, such as Freon® 22, to set the liner velocity.

Table 1 — Recommended Methane Retention Times◆

Column Length (m)	Carrier Gas (min: sec) H_2	He	N_2
15	0:38	1:15	2:30
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.

4. Adjust column head pressure until the CH_4 peak elutes at the appropriate time indicated below. In volume, this will be about 2.5cc/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.

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 column). 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. Thermal shocks could damage a capillary column by causing the phase to puddle.

Injecting the Test Mix

To ensure that your chromatographic system performs at optimum, make a weekly injection of the appropriate isothermal test mix and compare the results to the original test chromatogram. (Test mixes are available for all Supelco capillary columns.) If you do not obtain similar efficiencies and peak height ratios, you may have instrument or installation problems. If so, troubleshoot the system using these instructions and correct the problem. When you can duplicate the test results, your column will be performing at optimum. Interpretation of the test results are covered in the test mix instruction sheet.

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 0.53 ID 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. Be sure to set the methane retention time (Table 1) at the temperature on the instructions 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.

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.

Minimize contamination — When you perform on-column injections, nonvolatile and insoluble sample components can contaminate and ultimately damage the column. Sample cleanliness prolongs column life. (See Rinsing a Bonded Phase.)

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, the problem 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. 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 column inlet is dirty or (2) phase has gradually bled from the inlet end of the column and recondensed farther down. A 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.

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%.

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, methyl chloride or acetone. Other solvents are unpredictable and therefore not recommended. We suggest solvent volumes of 3-5mL.

In some cases, contaminants polymerize in the column inlet and rinsing with solvents will not restore the column to acceptable performance. In such cases, cut off two loops (1/2 meter) from the column inlet, using our Capillary Cleaving™ Tool. If samples are excessively dirty, attach a guard column to the column inlet, with a capillary butt connector to prevent contamination from reaching the analytical column. Refer to the Supelco catalog for more information regarding columns.

Storing the Column

To minimize phase exposure to atmospheric oxygen, seal the ends of the column before storing it. For short term storage, you can place septum over each end. For prolonged storage, we recommend flame sealing the ends with a microtorch. This method provides an essentially nonpermeable seal. Before sealing the column by either method, fill it with an inert gas: nitrogen or helium.

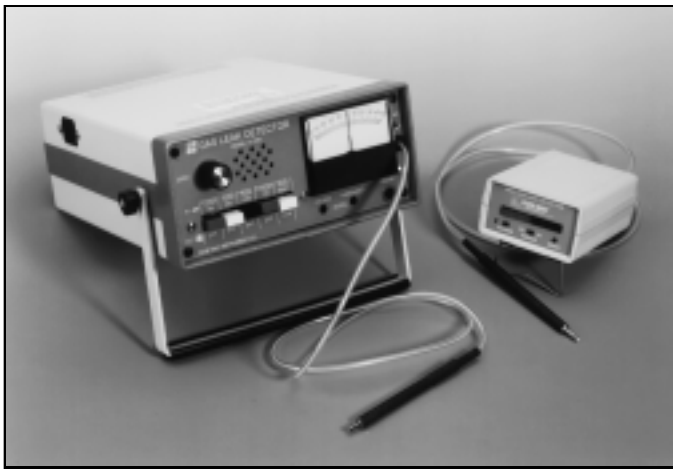
Note: When flame sealing columns, use dark welder's safety glasses to avoid possible eye damage from radiation.

Supelco 0.53mm ID capillary columns are of the highest quality commercially available. Every column is backed by our full warranty (see our catalog for details). With proper installation and maintenance, performance should be excellent.

Ordering Information:

Helpful Products Simplify Column Installation

GOW-MAC Gas Leak Detectors for GC Fittings



995-0110

- 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

994-0140

- 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

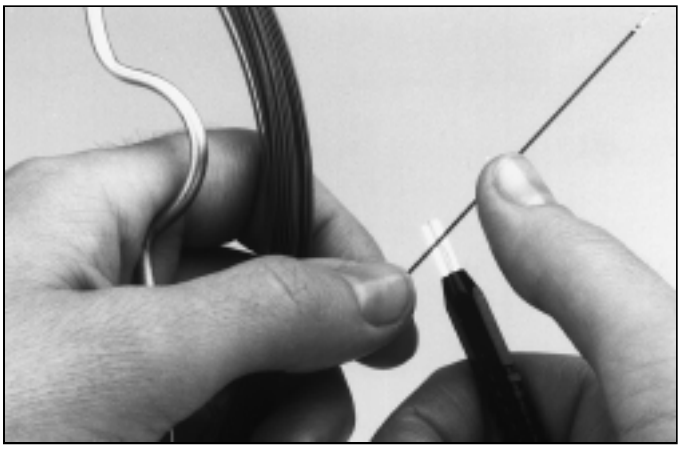
Supelco's high capacity gas purifier prevents carrier gas with high concentrations of oxygen or water from destroying your capillary column. By ensuring that only pure gases enter the column, this purifier can extend the column's life dramatically.

This high capacity unit can remove 23 grams of oxygen at concentrations up to 2000ppm. When purifying gas containing 10ppm oxygen and water, a single converter tube will usually last about nine months. When necessary, you can order a new tube, or refill the spent one. Either way offers better long-term economy than disposable purifiers.

The stainless steel converter tube is 12" x 1/2" OD and has Swagelok® fittings. The split-sided furnace is 10" long. Mounting brackets enable you to bolt the unit to a bench top or wall.

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

Capillary Cleaving™ Tool Permits Perfect Butt Connections



910-0103

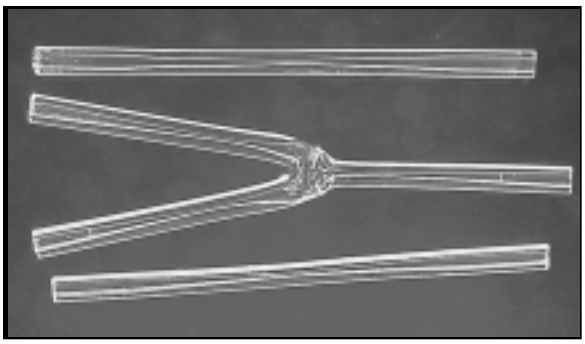
Make scalpel-like cuts in both polyimide and fused silica — no jagged edges to create problems. Industrial sapphire cutting edges remain sharp indefinitely. The spring-loaded retractable blade version reduces the chances of breakage if the tool is dropped.

Description	Cat. No.
Capillary Cleaving Tool with fixed blade	**23740-U**
Capillary Cleaving Tool with retractable blade	**23814**
Replacement Blades	**23815**

Trademarks

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.
VESPEL — E.I. du Pont de Nemours & Co., Inc.

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 0.53mm ID 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
- 0.8mm ID ferrules will connect the 0.53mm ID column to the special adapters in our conversion kits

	Quantity	1/4"	1/8"	0.8mm●
Supeltex M-2A▼ (15% graphite/85% polyimide)				
	10	**22481**	**22483-U**	**22489**
	50	**22471**	**2-2472**	**22473**
Supeltex M-4▼ (flexible graphite)				
	10	**22492**	**22491**	**20628**
	50	**22478**	**—**	**22479**

▼DuPont VESPEL® SP-21 part.

●Use with 1/16" fittings.

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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