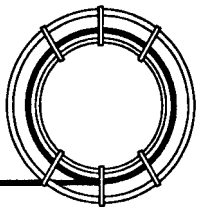
Hints for the Capillary Chromatographer



Selecting the Proper Ferrule for Capillary Columns

Proper ferrule selection is critical for capillary column installation. Characteristics such as thermal stability, ruggedness, and compressibility are determined by the different materials used to make ferrules. It is important to choose the right ferrule type and size to ensure proper column installation. The wrong ferrule type could cause damage to sensitive detectors such as ECDs, ELCDs, and MSDs. The wrong ferrule size or type can cause system leaks that result in decreased sensitivity and deterioration.

Ferrule Materials

Since metal ferrules would damage fused silica tubing, softer materials are used for capillary column ferrules. The two most common materials for capillary column ferrules are graphite and Vespel@. These materials can also be combined to form hybrid ferrules with the benefits of each material. Other ferrule materials, such as Teflon" and silicone, are commonly used with packed columns, but because of their limited thermal stability they are not typically used with capillary columns. Table I lists the maximum operating temperatures and the characteristics of common capillary ferrule materials.

Table I - Common Characteristics of Capillary Ferrules

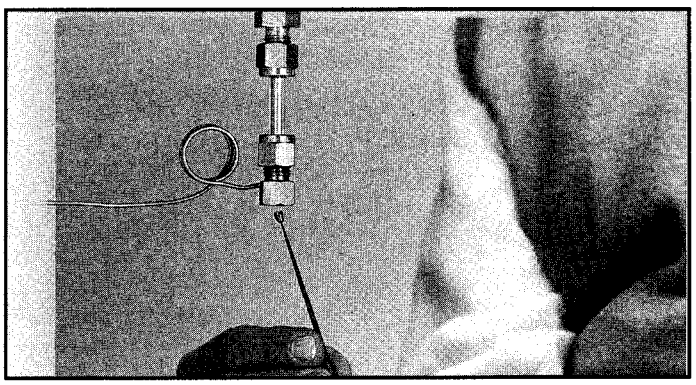
Material	*Max Temp.*	*Characteristics*
Graphite	450°C	Soft, easily conforms to all column sizes. Excellent for high temperature applications. Can flake or deposit particles in inlet & detector fittings. Easily deforms, resulting in limited reusability. Not recommended for vacuum interfaces.
Vespel®/Graphite	400°C	Hard, must be sized to exact column OD. Contracts when cooled causing leakage if not retightened after several thermal cycles. Excellent reusability.

Properties of Graphite Ferrules

Many chromatographers prefer graphite ferrules because they are soft and easily conform to any fitting dimension. Most graphite ferrules are made by tightly winding graphite ribbon around a pin and compressing it into a mold. The graphite ribbon increases ferrule pliability and allows it to deform easily. Increased pliability makes it possible to seal a 0.4mm OD (0.25mm ID) fused silica column with a 0.8mm ID ferrule. In addition, the ferrule can accomodate larger columns if the graphite bore is cored out. These features allow chromatographers to always have the right size ferrule on hand

Graphite ferrules should be tightened using minimal force. Usually 1/4-turn past finger-tight is sufficient to form a leak-tight seal. If a graphite ferrule is over-tightened, it will extrude out of the bottom of the nut, deform into the fitting cavity, and create ferrule fragments. These particles can be driven further into the inlet or make-up gas fitting, causing adsorption or peak tailing when a column is reinstalled. Graphite ferrules can also flake or abrade and emit particles that can .clog small orifices. Because graphite is porous, graphite ferrules leak under vacuum. Therefore, graphite ferrules are not recommended for detectors operated under vacuum, such as MSDs.

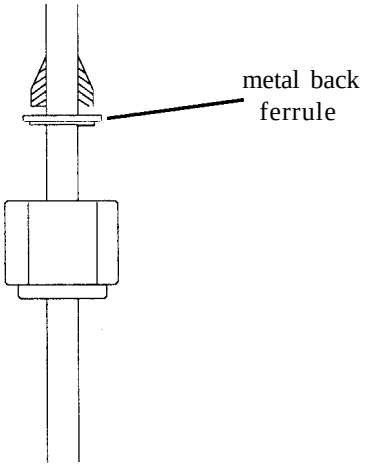
Graphite ferrules must be carefully removed, otherwise fragments and flakes remaining in the fitting can contaminate the GC system. Ferrules are easily dislodged by inserting a tapered needle file into the bore and moving it side-to-side. If the graphite ferrule does not come out in one piece, the inlet or detector fitting should be completely disassembled to ensure that no ferrule fragments remain.



Needle files easily remove graphite** ferrules **injector and detector fittings or nuts. Gently insert** the **file into the ferrule bore and move it from side-to-side to dislodge the ferrule.

The life of a graphite ferrule is limited because they compress so easily. Some chromatographers obtain new life from a crushed ferrule by installing a reversed Swagelok-type back ferrule between the fitting and the ferrule (Figure 1). The back ferrule raises the graphite ferrule higher in the fitting, allowing it to seal again.

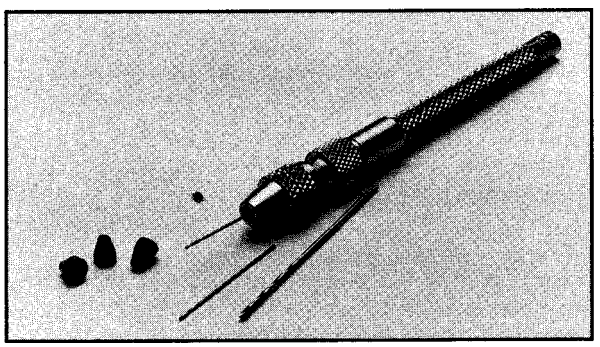
Figure 1 - Give a used graphite ferrule new life by installing a reversed metal back ferrule in the fitting.



Properties of Vespel or VespelGgraphite

Both 100% Vespel and Vespel/raphite ferrules are available. Vespele-type ferrule are often preferred because they do not flake, deposit particles, or fall apart in a fitting. Most chromatographers choose the Vespel/graihite ferrule combination. These ferrules are made by compressing a graphite/polyimide powder under high pressure in a heated mold. They retain their shape and can easily be removed intact. Vespel/graphite has a higher thermal stability than Vespe1° (400°C vs. 350°C) and the graphite impregnation makes the ferrule feel softer and seal with less torque. Vespel/graphite ferrules are currently available in combinations ranging from 85% Vespel/15% graphite to 60% Vespe1/40% graphite. The 60/40 Vespel/ graphite combinations are preferred by most chromatographers because they seal with the least amount of torque.

Unlike graphite, the inside diameter of Vespel-type ferrules must be very close to the column OD in order to seal properly. If the ID of a Vespel-type ferrule is too large for the column OD, it will not compress properly and allow a leak. Usually, the ferrule forms an oval shape, gripping the tubing but not sealing at the ends of the oval. If the ID of a Vespel-type ferrule is too small to fit over the column, the bore must be enlarged with a small drill.



If *the* ***Vespel/graphite ferrule's ID is too small to fit over the column, a pin vise drill can be used to enlarge the bore.***

Vespel/graphite ferrules will deform to the exact fitting dimension when heated Usually this deformation process causes the ferrule to become loose and leak during the cool down cycle of a GC oven. Therefore, they must be subsequently retightened after several thermal cycles or carrier gas leakage will occur. No additional shrinkage or loosening occurs once the ferrule has conformed to the internal dimensions of the fitting cavity.

Vespe1° ferrules can be removed from a fitting using a tapered needle file in the same manner as a graphite ferrule. Vespe1 ferrules sometimes stick to the fitting and column after they have been in use for a prolonged period. Stuck ferrules can be removed by tapping the fitting with a solid object such as a wrench and gently pulling outward on the column. This problem is greatly minimized by using Vespel/graphite combination ferrules.

What are common ferrule sizes?

Most column connections in the GC inlet and detector are made using 1/16" Sagelok-type fittings. The ID or opening of the ferrule depends on the outside diameter of the column. Table II lists common fused silica capillary column IDs, ODs, and recommended ferrule sizes.

Table II - Common Ferrule Sizes for Fused Silica Capillary Columns

Column ID	*Column OD*	*Ferrule Opening*
0.18 to 0.25mm	0.35 to 0.40mm	0.4mm
0.32mm	0.45 to 0.48mm	0.5mm
0.53mm	0.69 to 0.72mm	0.8mm

The choice of ferrule material is often personal preference. If you are installing a capillary column for the first time, we suggest using a graphite ferrule. Graphite easily forms a leak-tight seal and conforms to any column OD. If you frequently install new columns, Vespel/graphite is recommended to eliminate particle evolution and minimize maintenance down-time. However, when connecting columns to MSDs or Mass Spectrometer transfer lines, Vespel/graphite is the only ferrule you should use to ensure a leak-free seal under vacuum. We recommend trying both ferrule types to choose a ferrule that best fits your needs. ■

'This article is a reprint from the The ***Restek Advantage,*** Vol. 4 No. 2, March 1993, p. 12-13.