

# Ferrules

can be made of graphite, Vespel® (polyimide), Vespel®-graphite mixtures, and Teflon® (polytetrafluoroethylene, PTFE) Even though graphite is not a polymer, its characteristics are more similar to the synthetic polymers than metal ferrules. Attributes of these ferrule materials are summarized in Table 1.

Graphite and polymeric ferrules have several advantages for use in gas chromatography over metal (especially hard metal) ferrules:

- They can seal against imperfect surfaces with little force
- They can be used with virtually any type of tubing or column including glass

and fused silica

- They can also be hand drilled with a pin vise to get the right size for any given tube or column

However, because they are organic and porous, graphite and polymeric ferrules

do have some general weaknesses that constrain their uses as well:

- Polymers have a limited temperature range (compared to metal ferrules)
- They are more permeable to air infiltration (a function of polymer density)
- They sometimes come out of their commercial packaging, or the lab

drawer, contaminated. They are then a source of ghost peaks and baseline disturbances.

Contamination comes from poor manufacturing processes as well as poor choices in packaging (the contamination comes from the packaging).

**A common practice is to leave ferrules in GC oven prior to use for conditioning**

- They can also interact with sample components or solvent causing tailing or losses, especially at trace levels

## Graphite

has been a favorable ferrule material for capillary column use from the beginning of gas chromatography. It is very forgiving because it is so soft and can deform and seal in almost any space. Graphite is easy to identify because it can be deformed, or scratched.

Incorrect size ferrules can sometimes be used easily either to expand the hole to accommodate a larger column or to compress a larger ferrule just by putting it on the column and tightening the fitting a little more than usual.

The graphite is easily reformed in both cases to create a near perfect seal.

However, this malleability can lead to one of the biggest problems with graphite ferrules as well.

This ability of graphite to reform can cause it to extrude through openings in fixtures into adjacent spaces.

Graphite pieces can end up contaminating areas like the bottom of the inlet or the detector jet.

These graphite pieces can interact with sample causing losses, tailing, and can become a constant source of contamination as graphite is an absorbent.

A second major problem with 100% graphite ferrules is that they are very permeable to air due to their semi-laminated structure.

So, when using air sensitive columns (e.g., carbowax) or detectors (

e.g., mass spectrometers or ECDs) Graphite/Polyimide are preferred - much more dense & impermeable.

Some inlet and detector designs use graphite ferrules wherein the graphite is contained within a secondary metal tube.

This provides the benefits of graphite ferrule material, while greatly addressing the issues of deformation, extrusion, and air diffusion.

Graphite Ferrules are resilient enough for glass and ceramic tube use.

Chromalytic have a unique market extending sizes to fit tube size from 1/16"x0.3 up to a Maximum od 2.0inches (50mm) ID



## Polyimide

is a polymer with high temperature stability ( 310-35-degC max ) and low outgassing.

Because of its relatively high temperature stability, polyimide is the coating of choice for the outside of fused silica columns. Polyimide ferrules are easy to identify because they are brown (see Figure 1).

Polyimide is very hard but is softened with admixtures of Graphite and under pressure in-situ can be molded into shapes that match fitting profiles, and devices typically designed for metal ferrules.

Even though it has high temperature stability, polyimide softens when heated, problematic in high-heat zones. It tends to bind onto the tube and can fix inside the fitting.

For this reason, 100% polyimide ferrules find most use outside the GC oven.

These are preferred over metal ferrules for room temperature or low temperature connections

since they are much more forgiving than metal ferrules when sealing against flawed tubing,

they can also be removed without having to be cut off the tubing.

They also have low air permeability.





## Graphite/Polyimide (15% is common) greatly reduces binding .

The mixture retains the benefits of:

- Being able to be molded into desired shapes
- High temperature stability
- Ferrule does not extrude when tightening
- Can seal against imperfect surfaces
- Has low air permeability
- Soft enough for use with fused silica

Because of these benefits, graphite/polyimide ferrules are the dominant ferrule type when making fused silica column connections in the GC oven.

Graphite/polyimide ferrules do tend to shrink with temperature cycling, so they need to be retightened several times (after cool down) within the first 10 temperature programmed runs or so.

Extended use above 300degC also tend to "dry out" the polymer and may crack on re-tightening

**PTFE ferrules** and other similar fluoro polymers ( PFA and ETFE ) are very inert and have minimal interaction with samples and solvents.

They are very easy to seal, often requiring only hand tightening.

When disconnecting fittings, PTFE fittings slide right off the tubing and can be re-used multiple times.

However, PTFE ( TEFLON ) they have the most restricted upper temperature limit (250degC)

and are somewhat permeable to air, so use inside the GC oven is quite limited.

PTFE ferrules are used primarily for low temperature applications requiring the most inert connections, and for tubing connections outside the oven - for more reliable, easily adjustable and removable connections.

PTFE ferrules are lower cost and are often used for compressed air supply lines for valve actuators.

**ETFE** (Tefzel) is a melt polymer compare to PTFE (which is sintered) and thus harder and less air permeable than PTFE and often preferred for solvent lines in LC/HPLC applications

**PBI Ferrules** ( experimental by Chromalytic ) . . . offer potential higher temperature usage than Polyimide versions fused silica and GC/MS are targeted uses

Customer feedback on PBI Ferrules s welcome

Ferrule Type	Temp Limit	Typical Uses	Advantages	Limitations
Graphite (100%)	450 °C	General purpose for capillary column connections to inlets and detectors	Easy to use, can be tightened to hold column in place without siezing, column can be readjusted	Allows air diffusion. Not for MS or oxygen-sensitive detectors
		Recommended for high temperature and column-column applications	Can be removed easily	Overtightening can extrude graphite into inlet or detector. Pieces flake off and stay behind, causing peak tailing and sample losses
			High temperature limit	Soft, easily deformed or destroyed
			Most forgiving of receiver imperfections	Interacts with solvent, causing tailing
			Can be re-used	
Polyimide/ Graphite (85%/15%)	350 °C	General purpose for capillary and packed glass columns	Easy reliable connections	Design specific to fitting
		Recommended for MS or oxygen-sensitive detectors	Reusable and remakesable	Shrinks with temperature cycling. Must re-tighten frequently
		Reliable, leak-free connection	Maintains shape, does not flake off particles	Siezes on column, cannot be re-adjusted, must be cut off
Polyimide (100%)	280 °C	Isothermal operation	Fairly forgiving of receiver imperfections	
		Reliable seals even with flawed receiver surfaces, forms to sealing surface	Easy reliable connections	Can shrink at elevated temperatures, must re-tighten frequently
		Excellent sealing material when making metal or glass connections	Reusable and remakesable	
		Excellent for external	Can be removed easily if used at room temperature	Can glue connections together if exceeding recommended temperature limit and destroy fitting
PTFE	<260 °C	External connections not involving carrier gas	Low air permeability	Allows air diffusion. Not for MS or oxygen-sensitive detectors
		Valve actuator gas lines	Easy connections	Limited use inside oven
		Glass packed columns used at low temperatures	Can be re-used	
			Very forgiving of receiver imperfections	
			Can seal with very little pressure.	



**Gas chromatography** relies on tubing, plumbing for gas flows and ferrules for sealing connecting fitting and would not be a practical routine technique without ferrules  
– these essential consumables that provide a pressure seal between the atmosphere and the gas flow path. Ferrules are used in junctions that can be disconnected and remade. Proper selection and use of ferrules ensures a reliable installation. Incorrect selection and/or implementation can cause problems and can affect the quality of analytical results.

## Chromatography Plumbing

The need for a leak-free and reliable flow path is essential in gas chromatography.

In particular the requirements of GC fittings are quite demanding compare to normal plumbing They must be able to withstand high temperatures for long periods of time and wide variations as experienced in GC ovens for example. Connections outside the oven and at near ambient conditions are far less demanding or where constant temperatures are used such as a valve oven, inlet or detector.

Ferrules are used to provide a seal between a tube and a fitting; union, a valve, a transfer line, or device requiring attachment of a tube or chromatography column. For permanent connection, then the tubes can be soldering, braising, cementing, etc. can be used.

A ferrule is used to make a reusable connection. The simplest ferrule seal uses some easily deformable material such as a rubber gasket or O-ring common in many industrial and domestic situations.

Unfortunately, such a connection cannot satisfy the special requirements of GC and liquid chromatography is more demanding requiring sealing material with unique characteristics.

- Use of gases with very high diffusivities – these gases (He and H<sub>2</sub>) can be prone to leakage
- Air can also back diffuse through leaks, even against a pressure gradient
- Many of the detectors used in GC can detect air leading to raised detector baseline and increases drift
- Many of the stationary phases used in GC are sensitive to air through oxidation which will lower column and increase column activity to polar compounds, as well as increasing detector noise due to bleed of liquid phase decomposition products
- Larger leaks of course will waste gas, which can be quite expensive (e.g., when using High Purity grades required for some sensitive detectors; GC-MS, Helium Ionisation and even TCD)
- Leaks in the sample flow path will vent sample (inlet side) or effluent (detector side) causing errors in the result in loss of sample and cause errors and inconsistencies in results
- Where mass flow control is required, leaks can cause serious errors in retention time (packed column inlets but more so with low flow rate capillary columns), with variations in split ratios.

Exacerbated by variations in temperature, vibration, and time. Some of these effects can be offset by using chemical standards where such leaks are constant,

## Metal Ferrules

come in two basic designs: one- and two-piece.

**Swagelok** is the premier manufacturer of two-piece ferrule designs and the de facto standard over many years.

The two-ferrule design has some advantages (at a price premium) over single ferrule designs.

Swagelok fittings are ubiquitous in the GC laboratory.

Single-ferrule formats serve the same purpose as two-ferrule designs. Parker and VICI-Valco are examples

It is MOST important to match both the ferrule and fitting with those from the same manufacturer and NOT to mix them..

**Hard metal ferrules** (#316SS) are only used with similar hard tubing material.

The seal is actually made over a relatively narrow region near the tip of the ferrule.

The cone design of the ferrule directs the tightening force from the nut to the back of the ferrule to its cone tip which in turn compresses onto the underlying tube.

***If the tube is a glass or fused-silica column, it would break.***

Soft metal (#304SS) or polymeric tubes, in contrast, will deform, allowing the ferrule to “bite” into them permanently.

• Metal ferrules can be used at high temperatures and can withstand wide temperature cycling, and are ideal for fixed installation in heated zones such as valve ovens and heated inlets and detectors.

• However, when used for extended periods at high temperatures, the ferrule can seize into the fitting.

• If overtightened they can also distort both the ferrule and the fitting so that on reuse orientation may be different requiring even more excessive force to re-swage them into a good seal

- In some situations they just won't reseal and in fact may need to be cut off the tubing and replaced

• Hard metal ferrules usually come in either brass or stainless steel.

Stainless steel is more inert than brass and is required for normal sample paths.

Outside the sample path, use the same ferrule material as the fitting or fixture.

One should never use a stainless ferrule with a brass fitting or fixture because steel is harder and can permanently damage the fitting. A brass ferrule can be used with a stainless fitting if you are prepared to cut off the ferrule if it becomes scored.

**Metal Ferrules** have a number attractive attributes . . .

- Metal has the least permeability to air
- Metal fittings and ferrules can effectively seal over the largest pressure and temperature range
- Metal fittings and ferrules are extremely rugged and can take a lot of abuse
- Metal fittings can be and remade many times (if not over tightened)

Problems arise . . . however, because of there hardness, and failures can occur:

- Improper matching of ferrule and seat designs (avoid using/mixing generic fittings and ferrules)
  - Over tightening can cause irreversible damage to the fitting as well as the ferrule.
  - Improper installation (inserting hoving tubing into fitting too far and/or over tightening)
- can cause the end of the tubing to bulge in the fitting, making it difficult or impossible to remove.
- Seizing of the ferrule to the fitting after long periods at elevated temperature.
  - Inability to seal over fine-lined striations in the metal tubing itself and scoring of the ferrule material

Whilst not ideal sandpaper can be used with care to improve imperfections in tubing

Rotating the tube is far bett than length wise

**Soft Metal Ferrule** material (e.g., **Siltite-SGE and Aluma-Seal** Restek) is now available

which can be used for fused-silica column connections,

- They can deform slightly so that they seal with less force than is necessary with hard metal ferrules otherwise impossible with glass or fused silica.
- They are soft enough to be used with fused-silica capillary columns.
- They are a little more forgiving of imperfect surfaces than hard metal ferrules.
- They have very low (or no) air permeability and can be used over the full range of GC temperatures.
- Unlike polymeric ferrules, soft metal ferrules do not shrink and therefore do not need periodic retightening.
- They are specially coat to reduce the risk of seizing in fittings.

Soft metal ferrules seize onto the column upon first use, so connections must be done with some care.

These **soft metal ferrules** are particularly suited for MS transfer lines or capillary column connections where one does not typically need to trim the swaged column end regularly.

Even though they deform slightly soft metal ferrules should still be used with fittings that have the proper corresponding taper design. To benefit from using this style ferrule, spare replacement parts such as a new baseplate for the inlet or a special reverse nut for transfer line connections.

**Glass and Fused Silica capillary** connections - -

- Most ferrules require close tolerance matching of Tubing OD and ferrule ID
  - Hard ferrules have no give and tend to "crunch" the tubing
- Polymeric Ferrules are softer and mor forgiving where applicable
- Graphite probably priveds the best all-round ferrule materiial

**For glass and ceramic tubing** . . . reducing Graphite Ferrules overcome many adaptions of metric tube size and Imperial size fittings.

Care is needed to select tubing as round as possible is important in obtaining a "re-doable" and effective seal. With care Graphite Ferrules can re reused atthough extrusion into the thread of the Nut does occur making them difficult to remove.

For Reducing Ferrule application end-pressure on the tubing is a limitation with Graphite Ferrules

- the tube can be forced out the back of the nut

. . . graphite after all is a lubricant and glass is smooth . . . of course

Extreme application with large OD tubing may benefit by centreless grinding of the glass tube / ceramic and/or grooving or roughening of the glass surface

Glass tubes to 2" diameter and ceramic tubes to 1000degC are possible - although th graphite does tend to oxidise and burn away in contact with air Caution PRESSURE LIMITATIONS Low Pressur High Flow applications are OKthrough . . . **with CARE !**

. Enclosed in an inert atmosphere temp to >1500degC are possible ( ceramic tubes )

**- often there are Just NO Alternatives to Graphite ferrules**



# Inertness - SS Fittings and Ferrules

**Silcosteel** is a patented process where a film of fused silica is vacuum deposited onto SS surfaces dramatically improving the inertness to many aggressive fluids and reactive compounds in sampling and in chromatography use.

Coated tubing and fittings even extend to anti-corrosion, anti-coking and Ultra High vacuum applications. Silco-treated Swagelok and Parker fittings are available from Restek and a custom coating service of customer supplied parts is available from SilcoTek (enquiries to **Chromalytic Tech.**)

**\* IMPORTANT - SS Ferrules must NOT be "Silco-treated"**

Ordinary uncoated SS ferrules are perfect when used in a "silco-treated" fitting but "silco-on silco" will NOT seal

**Basically the mating surfaces are like sand-paper on a micron scale**

## some Applications

Ferrule Type	Temp Limit	Typical Uses	Advantages	Limitations
Metal (1-piece or 2-piece)	> 450 °C	External connections	High pressure limit	No at all forgiving of flaws in fitting/fixture
		Gas lines to GC	High temperature limit	Ferrule and fitting designs must match, no mixing of designs
		Gas sampling valves and associated tubing	Very low (no) diffusion of air through ferrule	Can seize after long use at high temperature
			Extremely rugged	Tightening technique is critical (not too loose, not too tight) for proper seal and ability to re-use
Soft metal	> 450 °C	Capillary column connections to inlets and detectors	Low (or no) air permeability	Permanently attaches to tubing upon first use. Must be cut off to be replaced.
		Capillary column connection to MSD transfer line	Much more forgiving of fitting imperfections than hard metal	Can be easily overtightened sealing the first time but significantly decreasing probability of resealing a second time
		Column connections to Capillary Flow Technology and other styles of gas phase microfluidic devices	Connections can be disconnected and reconnected multiple times	Ferrule and fitting designs must match for proper seal and re-seal
		External connections of 1/16" gas lines	High temperature use	

Swagelok 2p  
Ferrules

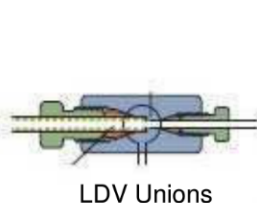


VICI LDV  
1-P Ferrules



## High Performance Chromatography

Require absolute minimum of dead-volume effects and maintaining "stream-line" flow often requiring perfect matching of ferrules and fittings - to ensure high column efficiency. VICI-Valco has many options for **LDV connections** including **fused silica adaptors** and profile ferrules.



LDV Unions



Polyimide/Valcon  
"profiled" ferrules



SILCOSTEEL  
MXT Unions

# Graphite Ferrules

the "PERFECT" Connection for GC and Glass Tubes

also Graphite/Polyimide for GC/MS ( Vacuum Application )

**500degC** in air

"unlimited" in inert gas

Soft and Malleable

No phase transitions

like plastics

## Graphite Ferrules - ALL sizes from 2mmOD to 2.0 in ID

- have been proven in over 25 years production by **Chromalytic Technology** with "millions" being sold "world-wide" mainly through some of the leading chromatography distributors and OEMs.

Full range of sizes from 0.3mm ID for capillary columns right up to 2.0-in for preparative columns and industrial applications;

Industrial Applications : **glass/ceramic tube** reactor/catalyst tubes, pump glands. **there is a size to fit ALL applications !**

**If we haven't got it already we will guarantee to make it for you !**

- Maximum temperature use to 500 deg-C or much higher in a "capture fitting" or if protected in an inert atmosphere.
- They are soft and malleable and extrude into shape to fill minor imperfections in tubing and "distorted fittings".
- They have an ideal "feel" for use with glass and fused silica capillary tube to help avoid crushing on over-tightening.
- They will not stick or bind to tubing and the nut/ferrule can simply be slid off the tubing for re-use.

**Specifically for GC . . . ALL types of GCs** are catered for including OEM specific ferrules; for example HP/Agilent inlet seals, injector liner O-rings; 8mm, 1/4, 5mmOD for Carlo Erba / Fisons /Thermo / Shimadzu; capture type cup GC column seals. etc

**OEM Enquiries Welcome**

### 3 basic Profiles are available . . .

- SF** - Single (Piece) Ferrule for **Swagelok** and similar fittings eg **Parker**.
- GF** - Front ferrule design for use with a "backing ferrule" or in Chromalytic "butt-type" connection fittings, injection systems.
- Short** profile - "short" specially designed for HP/Agilent GC capillary GC column inlet seals.

Also . . .

**Graphite O-rings** (6.35mm and 6,5mm ID) for HP/Agilent GC injector liners.

**Cup Graphite Seals** for CE/Fisons/ThermoQuest , Shimadzu

and the NEW **CT** graphite capture fittings - **Enquire !**

**Capture Ring** graphite seal for 8mm OD Fisons/CE/TQ liners.

## Graphite/Polyimide Ferrules - 1/8"ID max

**: material tends to be cost prohibitive - Other Sizes to special Order**

These are precision machined ( and NOT moulded ) to give a perfect fit in all Swagelok and Parker 1/16-in fittings.

**Temperature use** is limited to about 320 deg-C.( phase transition )

They are made from **15% graphite/polyimide** selected by . . .

**Chromalytic Technology** as being the best compromise in "softness", non-stickability and crack resistance. They are preferred over **Graphite Ferrules** for vacuum operation or use in GC/MS for reduced air diffusion.

For long term use - they may tend to "dry out" and may crack on re-tightening after use at high temperature.



### Single Piece

#### Ferrule Size / Cat.#

1/16" to :	
0.3mm	SF100/0.3G
0.4mm	SF100/0.4G
0.5mm	SF100/0.5G
0.8mm	SF100/0.8G
1.0mm	SF100/1.0G
1.2mm	SF100/1.2G
1/16"	SF100G

1/8" to :	
0.4mm	SF200/0.4G
0.5mm	SF200/0.5G
0.8mm	SF200/0.8G
1.0mm	SF200/1.0G
1.2mm	SF200/1.2G
1/16"	SF200/100G
1/8"	SF200G
3/16"	SF300G

1/4" to :	
0.5mm	SF400/0.5G
0.8mm	SF400/0.8G
1/16"	SF400/100G
1/8"	SF400/200G
6mm	SF400/M6G
1/4"	SF400G
3/8"	SF600G
1/2"	SF800G
5/8"	SF1000G
3/4"	SF1200G
7/8"	SF1400G
1.0-in	SF1600G
1-1/4-in	SF2000G
1-1/2-in	SF2400G
1-3/4-in	SF2800G
2.0-in	SF3200G

Metric	
5mm	SFM5G
10mm	SFM10G
12mm	SFM12G
14mm	SFM14G
16mm	SFM16G
18mm	SFM18G

### Front Ferrule Type

#### Ferrule Size / Cat.#

1/16" to :	
0.4mm	GF100/0.4
0.5mm	GF100/0.5
0.8mm	GF100/0.8
1.0mm	GF100/1.0
1/16"	GF100

1/8"	GF200
1/4" to 3/16"	GF400/300
1/4 to 6mm	GF400/M6
1/4"	GF400

#### "Specials"

Agilent O-rings	GFO-6.35HP
6.35mm	GFO-6.5HP
6.5mm	
Inlet Seals (Short)	GF0.3HP
0.3mm	GF0.4HP
0.4mm	GF0.5HP
0.5mm	GF0.8HP
0.8mm	GF1.0HP
1.0mm	GF1.2HP
1.2mm	

(standard)	
0.3mm	GF100/0.3HP
0.4mm	GF100/0.4HP
0.5mm	GF100/0.5HP
0.8mm	GF100/0.8HP
1.0mm	GF100/1.0HP
1.2mm	GF100/1.2HP

Fisons/CE/	
0.3mm	CF0.3Fis
0.4mm	CF0.4Fis
0.5mm	CF0.5Fis
0.8mm	CF0.8Fis
1.0mm	CF1.0Fis
8mm	CF8.0Fis

### Graphite/Polyimide 1/16"OD . . .Tube Fitting

0.3mm	SF100/0.3VG1
0.4mm	SF100/0.4VG1
0.5mm	SF100/0.5VG1
0.7mm	SF100/0.7VG1
0.8mm	SF100/0.8VG1
1.0mm	SF100/1.0VG1
1.2mm	SF100/1.2VG1
1/16"	SF100VG1
1/8"	SF200VG1
3/16"	SF300VG1
1/4"	SF400VG1

**ANY size**  
to your  
specifications  
**Generous Discounts**  
apply to OEMs  
and for  
**"bulk"orders**

**Other Specials** - for small "runs" a tooling cost may apply

### Double-Hole Ferrules

### No-Hole Ferrules (Blanks)

1/16" Double-hole	
SF100/2x0.5G	
GF2x0.5HP, GF100/2x0.5G	
1/8" Double-hole	
SF200/2xM0.5G	
SF200/2xM0.8G	

1/16"	SF100/00G
1/8"	SF200/00G
3/16"	SF300/00G
1/4"	SF400/00G
3/8"	SF400/00G
1/2"	SF800/00G

Note : **Teflon** and **PEEK** ferrules are also available for HPLC / LC / FIA applications . . . **Enquire !**