Ferrules for GC Column Connection

Use of capillary flow technology (CFT) devices to improve GC and GC/MS system productivity increases the need for robust and reliable fused silica tubing connections. There are tradeoffs when choosing ferrules (Figure 1) to make column connections to the CFT modules.

Composition	Max Temp	Use / Limitation
Vespel	280	Iso thermal only / Shrinks and leaks after thermal cycle
Graphite	450	Not for MS or CFT / Flakes, contaminates
Vespel/Graphite	350	MSD interface / Less shrinking, but retighten after thermal cycle
Original Metal	450	MSD and CFT / columns break or fitting damage

Metal ferrules have been recommended for CFT devices do to the limitations of the other materials. Once a seal is made, metal ferrules retain shape during thermal cycles, eliminating the need to retighten to maintain a leak free seal. However, since metal ferrules do not compress easily during the sealing process, excessive force is frequently used to ensure a good seal.



Additional force on metal ferrules results in either:

- breaking the column or worse,
- permanent damage to the hardware fitting.

Shown in the above figure, original metal ferrules have a non-linear response to force. Contraction of the ferrule ID bottoms out with no further sealing benefit. Due to its bulk, these metal ferrules need a large compression force to assure the solid to solid seal. Excessive force will cause damage to the fitting which can require costly replacement of parts. Silver plating on metal ferrules can cause erosion of the polyimide coating on the column, resulting in column breakage during use.

The Design Challenge

Ferrule Design Requirements

To provide a robust, reliable GC column connection to CFT devices, a ferrule project was launched with the following objectives:

- Leak free seal
- No need to retighten after thermal cycle
- Prevent column breakage at installation
- No degradation of column polyimide coating at high temperatures resulting in column breakage
- Consistent sealing process to obtain a robust and reliable seal
- Eliminate confusion over ferrule size or mixed inventory

The Design Process: column compatibility

To identify the range of compression needed for consistent reliable seals, measurements of standard GC column fused silica tubing ODs were also taken to define requirements for ferrule IDs (Figures 2 and 3)





Benefits of New Flexible Design Metal Ferrule

Flexible design prevents fitting damage or column breakage

A novel design metal ferrule with unique features was developed to provide advantages of both soft and metal ferrules. (Figure 4)



The ID of the ferrules are specified and tightly controlled to ensure compatibility with column tubing. Tight tolerances of the ID allows a consistent process for sealing, reducing the urge to over tighten. Designed to be flexible, these stainless steel ferrules compress easily and gently around the column during installation reducing breakage.

If over tightened, the ferrule squashes rather than being forced into the threads of the fitting. Figure 5 compares a new ferrule (left) to one swaged at 90 degrees (middle) to another over tightened with 180 degrees of torque. Notice thickening of the neck of the ferrule as it is compressed from the force.



Made from stainless steel the flexible metal ferrules hold shape in the fitting during use and temperature cycles, eliminating the need for retightening. The polyimide coating on the column tubing remains intact even after long exposure to high temperatures, preventing spontaneous column breakage during use.

Inertness with UltiMetal Plus

In environmental or toxicology labs where samples are in heavy matrix, capillary flow technology (CFT) devices are widely used to back-flush high boiling material after analytes of interest have eluted. Such samples often contain active or labile samples, prone to adsorption or degradation in the GC flow path.

A small surface of the metal ferrule is exposed to the sample in the fittings connecting the columns to the CFTs (Figure 6). Active sites on the surface of the metal ferrule negatively impact the results of sensitive analytes.



Figure 7 below compares chromatograms of sensitive organophosphorous pesticides when using UltiMetal Plus Flexible Metal ferrules and untreated Flexible Metal ferrules. UltiMetal Plus deactivation treatment improved recoveries of these sensitive analytes.

UltiMetal Plus deactivation is a standard feature of the new Flexible Metal ferrules to provide inert column connections. The rainbow hue seen on the ferrules shown in Figures 5 and 9 is normal variation in color after treatment.



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Additional Benefits

Features that provide Ease of Use

The wide opening designed into the back of the new flexible metal ferrule allows column to be threaded through ferrule easily. Ferrules can be installed onto the GC column tubing while the ferrules are still in the package (Figure 8). The Touchless installation accomplished with the new dial pack design not only prevents contamination of the ferrules but reduces dropping ferrules.



A family of ferrules was designed to accommodate the range of column tubing sizes, including UltiMetal columns, plus a no hole or plug ferrule, to address all application needs. To eliminate confusion or inventory mix-ups, a physically unique design was created for each part number within the family as shown in Figure 9. Note also the rainbow hue on the ferrules as a result of the UltiMetal Plus surface deactivation.



Conclusions

Robust, Reliable and Inert

The new flexible metal ferrule meets the design objectives of making and keeping reliable, robust GC column connections, without damage to the column or fitting.

- Stainless steel maintains shape and seal after thermal cycles but does not flake or contaminate
- ID of each size is tightly controlled to accommodate the range of GC column tubing ODs
- Tight tolerances on the ID reduce the range of torque required to secure the seal, preventing column breakage
- Compresses during installation to prevent damage to the fitting if over tightened
- Inert surface provided by UltiMetal Plus passivation
- Larger opening on the back of the ferrule allows Touchless installation

Ease of use at installation and removal, preventing column breakage, or damage to the fitting, and high reliability of seal during use, these novel design flexible metal ferrules are suitable for use in GC or GC/MS applications. UltiMetal Plus Flexible Metal ferrules are components of a total inert flow path.

For more information: www.agilent.com/chem/flexiferrule

