DR-3 Design Report-3, Why Use Analytical Fittings Instead of Industrial Ones*

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Why use analytical fittings instead of industrial ones and why use improved analytical fittings instead of standard analytical fittings in high performance systems ?

The well known double ferrule assembly has a ''swaging" action, i.e. it compresses the tube in some points and increases tube outside diameter beyond those points, as shown in Figure 1. This design is ideal for industrial application like in high pressure system and/or when there is a high level of vibration. The oversize section of the tube beyond the front ferrule makes it very difficult to come out of the fitting even if the nut loosens over time. This creates a safe assembly. This type of fitting is virtually used in all process plants today.

The general acceptance of this design and its ease of availability have led analytical system designers to use it also in analytical instruments and sampling systems. These designs have worked well for many analytical systems by early standard and the available instrument limit of detection.

However by todays standards such fitting design appears to be problematic for instrument manufacturers, system integrators and sampling system builders.

Here are some of the drawbacks of the swaging type fitting that generate some frustrations over the years.

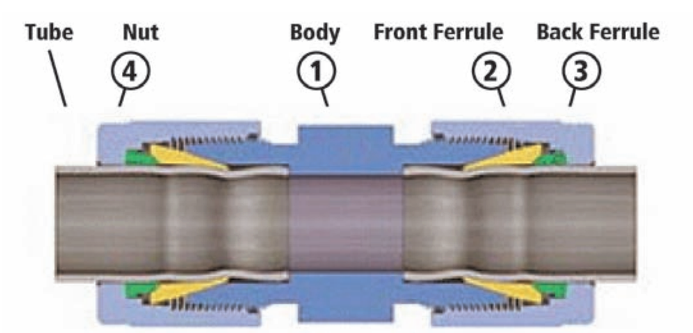

Figure 1: Typical double-ferrule fitting

Scratches, Particles Generation and Eventually Leak

When working with packed columns in gas chromatographic instrument, often these columns must be changed for several reasons. One of the most common ones is simply the need for new impurities type measurement in a new sample background. Such columns have an outside diameter of 1/16"OD, 1/8"OD and, less frequently today but still found, 1/4"OD.

Since the swaging action generates a larger tubing diameter beyond the ferrules, frequent assembly/de-assembly requires more torque to conserve the sealing level. Doing so, the tubing is forced deeper into the fitting detail, but the tube can't move forward, and its outside diameter could not become larger, since the tube is surrounded by the fitting body. When comes the time to de-assemble the system, very often it is difficult to pull out the tube from the fitting body. It is also much more difficult to re-insert the tube back in the fitting body. Doing so generates scratches inside the fitting body which leads to particles generation and to unavoidable leaks at this particular point.

In the attempt to solve this issue, some instrument manufacturers, for example Agilent (formerly Hewlett Packard), have recommended in their user's manual to cut the tubing just after the front ferrule or withdrawing the tubing a little before tightening the nut, as shown in Figure 2. See reference [1].

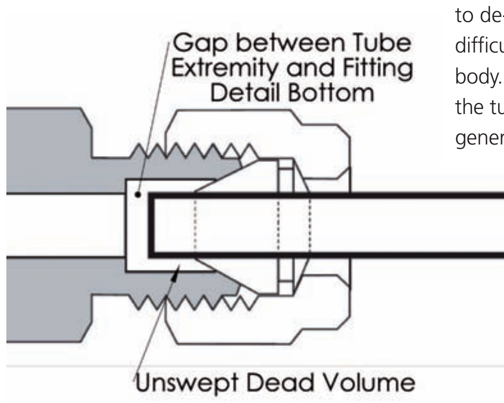

Figure 2: Double-Ferrule Connection as shown in Agilent User Manuals

*: US Pat.#7,503,203 other patents pending

Doing so eliminates the increase in tubing diameter created by the swaging action. This solution eliminates the above cited problems. However it also eliminates the main feature of the double ferrules concept that has made it so successful: safe assembly, vibration/pressure resistance. But much worse, it creates another problem that becomes a nightmare for many chromatographers: unswept dead volume.

Dead Volume

In the attempt to resolve problems caused by the swaging action of the double ferrules design, users have generated an even worse one, i.e. unswept dead volume. Indeed by cutting the tubing this way, a much larger volume is now created since the space or volume previously occupied by the tubing is now creating a relatively large void. Don't forget that this new dead volume is now present on both sides of columns since there is a fitting on each column end.

Problems caused by scratches and generated particles are easy to understand. However, problems caused by dead volumes are much more subtle, sometime such situations are confused with leaks. In fact, dead volumes may be thought as virtual leaks.

Here are some real situations that happened to many of us. To explain it, refer to Figure 3, which shows the simplest gas chromatographic configuration. Lets select a very common GC application, where the carrier is helium, column is based on a 1/8"OD molecular sieve and the detector is a helium ionisation type. Such configuration is used for permanent gases measurements. On both sides of a column end, there is a double ferrule type column end fitting.

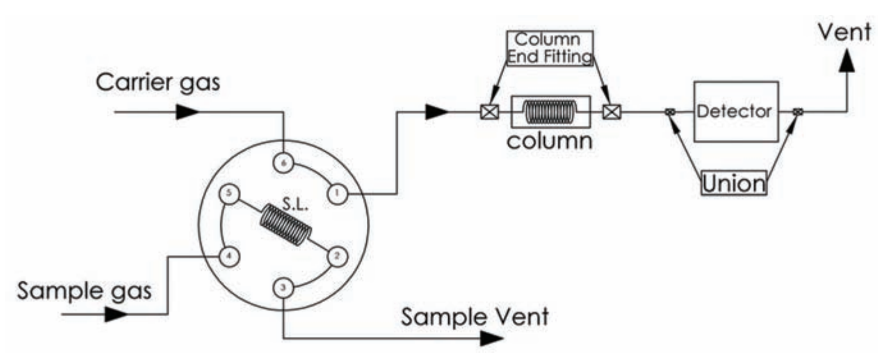

Figure 3: Simple GC configuration

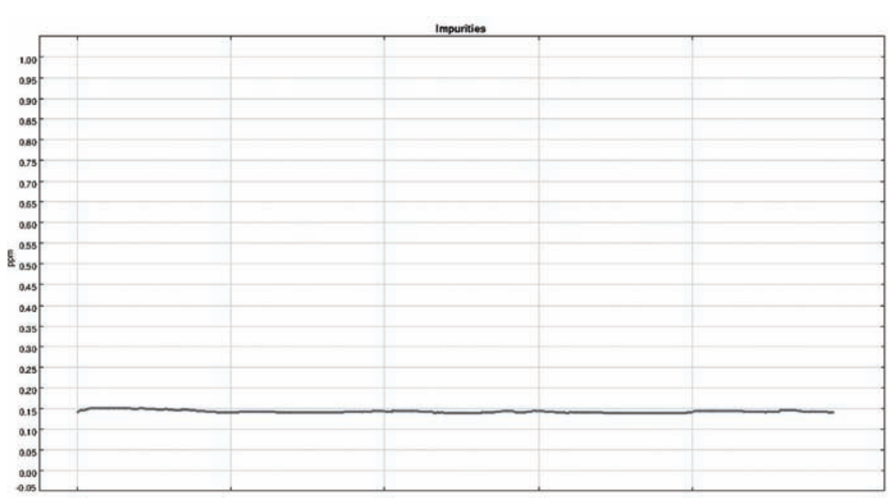

Figure 4: Stabilized detector signal after start-up

After system starts up, helium is flown and the column is regenerated to purge away any contaminants. Figure 4 shows the detector signal after system stabilisation.

Figure 5 shows the same signal when carrier flow is decreased and then restored. When carrier flow is decreased, the signal increases due to dead volume accumulated gas, diffusing back into the carrier. This increases the impurity level into the detector, then increasing the signal.

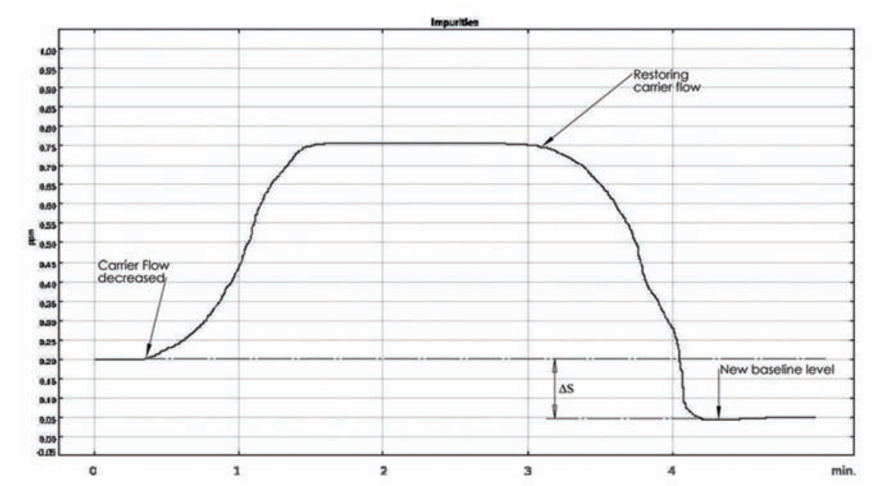

Figure 5: Effect of flow variation over signal

Restoring the flow dilutes the impurity level into the carrier gas, so signal is going down. As it can be observed on Figure 5, the signal is now lower compared to the beginning of the trend. This is due to the fact that there is less contaminant entrapped into the dead volume. Varying system flow or pressure is an excellent method for finding leaks into gas chromatographic system.

Now by doing so and looking at the signal trend of Figure 5, this would lead to think that there is leak and air diffusion into the system. The normal reflex would be to retighten fittings until the signal is going down.

By retightening the fittings, the ferrules are pushed forward and tubing OD increases once again, decreasing dead volumes. Doing so, the entrapped contaminant is forced back into the carrier gas and detector.

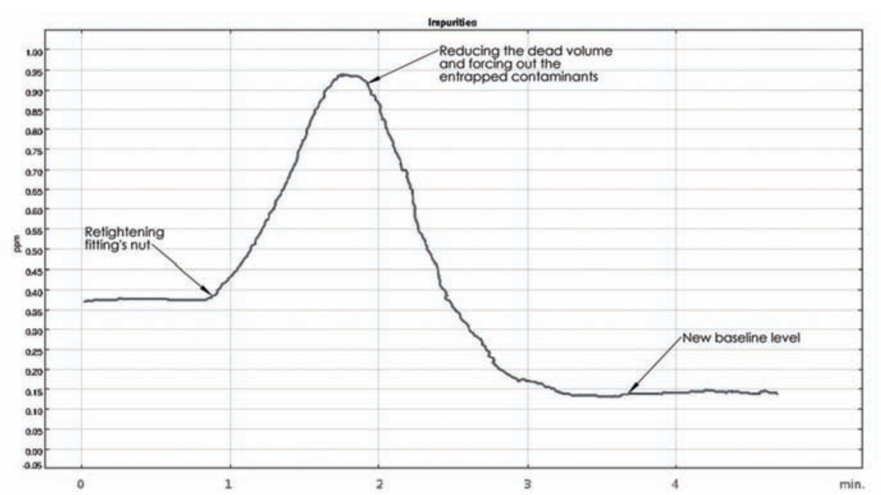

Figure 6: Effect of retightening a double ferrules fitting

Signal shown in Figure 6 is typical of such situation. Varying the flow or pressure to recheck for leaks would again generate a signal similar to Figure 5, but with less amplitude. Again, with the best intention in mind, naturally someone observing this would retighten even more fittings, still thinking that there are leaks. Since there are also unions and other fittings at various points in the system, it makes this problem even worse! At the end, in the attempt to resolve these virtual leaks, fittings will become overtightened, and now real leaks will be generated.

GC System Erratic Working Behaviour Caused by Dead Volume

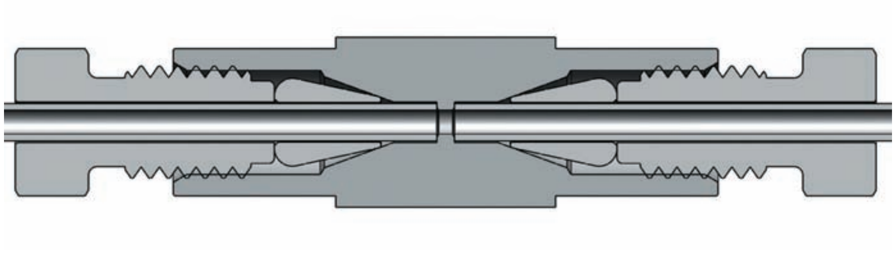

Figure 7: Typical single ferrule based analytical fitting

Another erratic behaviour could appear when injecting relatively large sample volume. Indeed, injecting such large volume suddenly reduces system pressure, generating a "ghost" peak. This is caused by some trapped contaminants in dead volume, diffusing back into the carrier. Larger is the tubing size or higher is the system sensitivity, worse will be the problem.

As a former process GC manufacturer, we have experienced this problem several times before. Other colleagues in the field had reported similar system behaviours.

No Dead Volume Analytical Fitting?

Talking about analytical oriented fitting, here's one of the most popular style in gas chromatography. See Figure 7. Similar designs are used by Waters®, Upchurch Scientific® and VICI®. This is a single ferrule design that doesn't use swaging action to hold the tube in place. When the nut is screwed in fitting body, the ferrule edge will grip on the tube creating a first sealing point. Another sealing area is done between the external surface of the ferrule, close to its end, and the fitting body.

The required torque is small compared with the double ferrule design one. Normally, there is no tubing deformation and the tube stays round and straight between the ferrule end up to the fitting detail where the square end of tubing seats against the bottom of the square fitting detail.

Here the idea is to reduce or eliminate any tube deformation in order to minimize the formation of unswept dead volume. To help achieving this goal, tube diameter must be just enough smaller to slip into the fitting detail. Furthermore, tube end must be square and have a good finish, in order to create a face seal with the square bottom of the fitting detail.

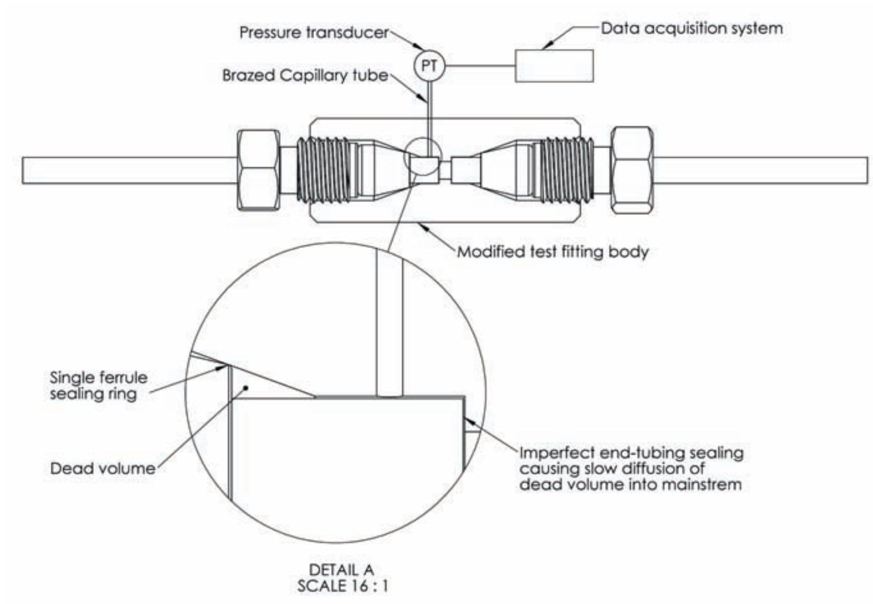

Figure 8: Monitoring apparatus of dead volume pressure variation

This concept works well with tubing size smaller than 1/8"OD. This concept has been called "Zero Dead Volume" fitting. Obviously, there is still a dead volume, as small as it is, it's still there. In some applications, where system sensitivity is high, like mass spectrometer and plasma emission detector, the effect of this small volume could be observed. There's still a dead volume since tube must be inserted in the fitting, so the clearance between the outside diameter of the tube and the internal fitting hole will be eventually filled with fluid. Don't forget that helium molecule diameter is about .25nm. There is also a larger void that will be filled between ferrule contact point with fitting body and where the tube enters into the pilot zone. When temperature or pressure suddenly changes, these various volumes will be filled with some fluid. We have demonstrated this by the setup of Figure 8. We will show later how we have decreased further this volume and its adverse effect.

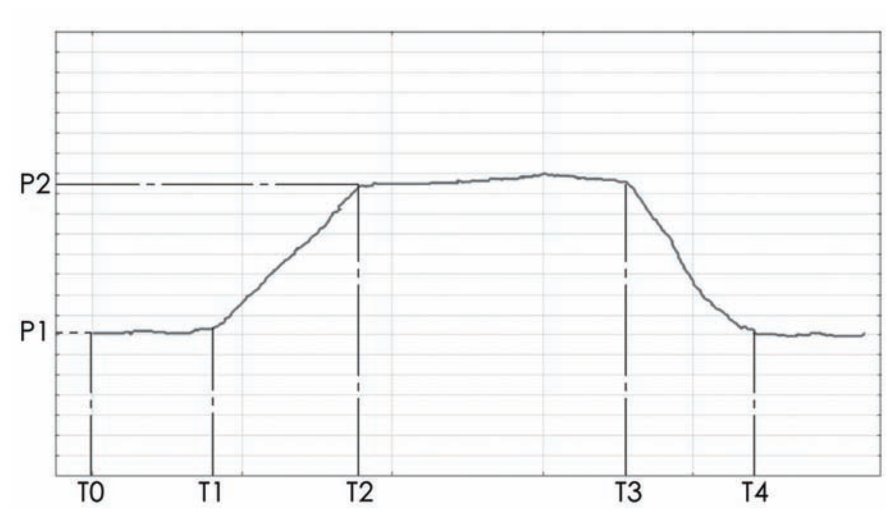

Figure 9: Rise and fall of dead volume pressure

A so called Zero Dead Volume has been modified to monitor the pressure variation on the internal volume. A capillary hole has been done and an external capillary tube has been brazed on the fitting body. On the other end of the capillary tube, a pressure transducer has been connected. Pressure signal was monitored and trended.

Between T0 and T1, system was at atmospheric pressure. At T1, system is pressurized at P2 value. Slowly, but surely, the pressure transducer signal ramped up until P2 is reached. At T3, system pressure is turned down at P1. For some time, starting at T3, pressure transducer signal decreased until it reached P1. So between T3 and T4, the fluid entrapped in this small volume is diffusing or depressurizing back in the main stream. This is where there is a contamination risk. Don't forget in some analytical applications, we are counting molecules and there are a lot of them in this volume. Reducing this volume as much as possible could only be beneficial.

Torque Related Problem

Tube size of 1/16"OD or 1/32"OD requires less pushing force from the nut to get proper sealing, so less torque from the user. However, a problem arises with this concept when tubing size is 1/8"OD and higher. Remember, most of packed columns are made with 1/8"OD 304 stainless steel tubing, file cut. Indeed, when the nut becomes in contact with the ferrule, it becomes very hard to rotate. Higher rotating torque is required to move forward the nut in order to get the ferrule gripping on the tube. It's difficult to do so while holding the fitting body with your hand. It requires longer tools and very often a vise is used to hold the fitting body. So imagine yourself replacing such fittings on a column inside a GC oven or on a critical and fragile component of your system.

Often this rotational torque transfer makes the ferrule to rotate or twist. Since the area in contact between the nut and the ferrule is much larger than the area in contact between the tip of the ferrule and the fitting body, this large rotational traction force can't be counterbalance by the ferrule. So until the ferrule becomes really compressed on the tube and forced against the fitting body, it will rotate and/or twist. Such ferrule rotation causes scratches and leaks.

This is a common problem for 1/8"OD tube size and, believe it, even worse for ¼"OD size. This phenomenon is shown in Figures 10.

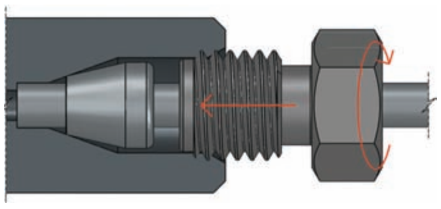

Figure 10a: Motion of ferrule and nut before the contact of the ferrule with the fitting taper

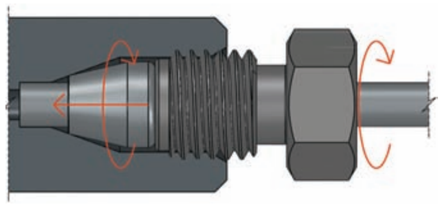

Figure 10b: Motion of ferrule and nut at the beginning of the contact of the ferrule with the fitting taper

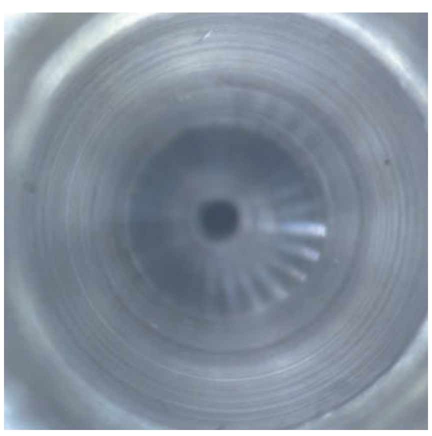

Figure 10c: Initial surface finish of fitting taper

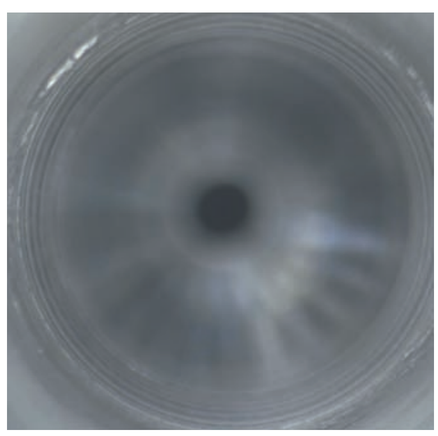

Figure 10d: Surface damages caused by ferrule rotation

Need For Improvement

Reducing further the dead volume of analytical fitting,eliminating the ferrule rotation and improving the sealing between the ferrule and the fitting body will be very beneficial for any analytical high sensitivity instrumentation.

Here's how we did it.

1. First, we did change the pitch and shape of the thread on the nut and inside the fitting body. Ultra fine pitch threads are used instead of the ones normally used. Doing so, the force transfer to the ferrule is done in a much smoother way.
2. An anti-friction and anti-galling coating has been applied to the front and the threaded portions of nut. This reduces the friction at least by an ordered of 10, eliminating the rotation of the ferrule when the nut is screwed in. So, now the fitting can be easily assembled with small tool and this without the need of a vise.
3. In order to decrease further the required torque to make a proper fitting connection and still getting excellent sealing performance, a relatively thick layer of gold has been deposit on the front portion of the ferrule, on the area where the ferrule seals on the fitting body. Figure 11 shows our new design.
4. We added a fine lip at the bottom of the detail. So, when the nut is screwed in, firstly, the ferrule grips the tubing. Then, as the ferrule is pushed forward by the turning nut, the tubing is compressed against this new fine lip. When proper tubing is used, this creates an effective metal to metal seal. Normally, tube material softer than union material will be preferred. For example tubing made of annealed SS304 and fitting body made of hardened SS316L have resulted in excellent performance. Having a fine lip, or sealing ring, distributes the mechanical force on a much smaller area increasing the effective seating force; the sealing lip penetrates the tube end. In fact, this concept creates a first metal to metal seal section, with no dead volume. The second section is the one done by the coated ferrule against union body. Optimally, an additional soft layer (ideally gold) would be deposit on the sealing lip, further improving the sealing, and reducing total force required for the desired sealing level. It also improves sealing with tubes that have some imperfection on their end.
5. We have made the front portion of the ferrule longer, in order to occupy more of the empty volume. Doing so reduces the dead volume, as shown in Figure 11.

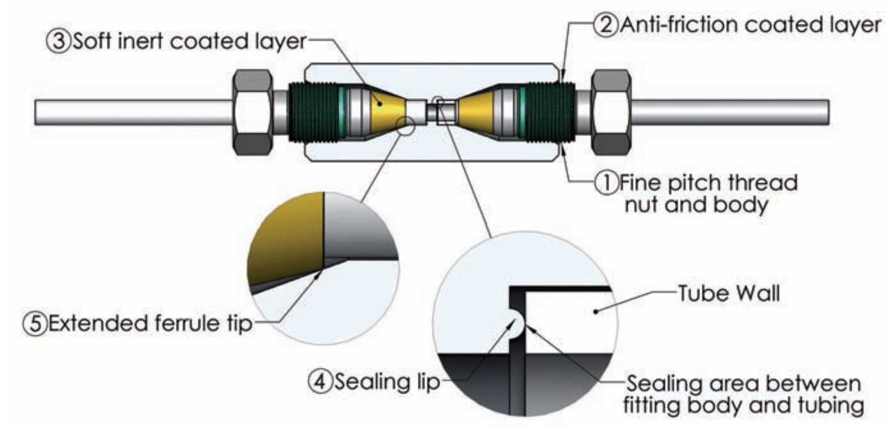

*Figure 11: Improved fitting design** **Patent pending*

Material Considerations

Many GC column manufacturers ship their packed columns with industrial type fittings, often made of brass. It is common knowledge within industrial fitting manufacturers that the fitting material should always be harder than the tubing material. In fact, protection of the fitting assembly against loosening coming from pressure pulsation and mechanical vibration comes from the swaging effect of the ferrule on the tubing. See reference [2], page 2-1.

However, in the analytical world, things are different. Pressure and vibration are less critical; sealing is a top priority. A ferrule material softer than the tubing one is important for the good performance of such fitting assembly. It makes sure that the tubing remains undeformed (not swaged) while the ferrule creates a sealing ring on the surface of the tubing.

There comes the choice of gold for the coating of our ferrule. The gold layer improves a lot the sealing; it is softer and easier to mate the stainless steel surface of the fitting body. Gold inertness is also well renowned.

Ferrule Modifications Results

With these modifications, there is less torque required to get the same and even better sealing level of previous design. Less torque reduces further the risk to get ferrule rotation when making connection.

Figure 12 shows the difference between rates of rise and fall of the dead volume pressure. Test is done with similar set-up of Figure 7; the only difference is the use of AFPTM extended tip ferrule.

As all components, except the ferrule, are the same in both tests, leak rate at the end of tubing is assumed the same. The higher rate of rise/fall of pressure while using this new ferrule shows that the dead volume is pressurized (or depressurized) faster, indicating that the dead volume is smaller. Note: This test is done with a union body that doesn't have a sealing lip. Exact same tubing outside diameter was used in both tests.

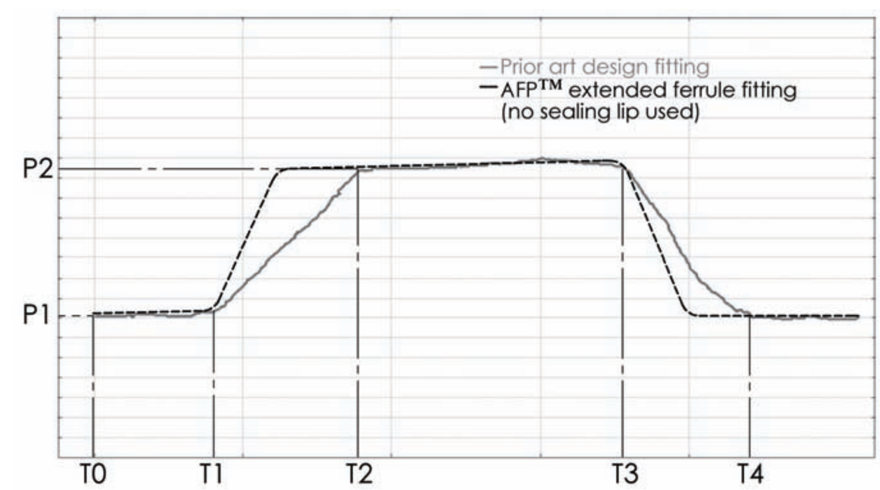

Figure 12: Difference in rates of rise and fall of the pressure in the dead volume

Fittings Performance Comparison

To further validate the benefits brought by our improvements over existing design, we did comparison tests of sealing performance of all the three fittings related here: old design fitting, and new AFP™ fittings with and without the sealing lip. To do so, we needed specifically modified fittings. An old design Zero Dead Volume fitting (Figure 13a) and an AFP™ new fitting without sealing lip (Figure 13b) have been changed to allow the pressurization of the internal volume between the threads and the sealing area between the ferrule and the fitting body. Small holes in these areas have been done and external 1/16"OD tubes have been brazed on the both sides of fitting bodies. One the tube is used to send in pure nitrogen in the tested area.

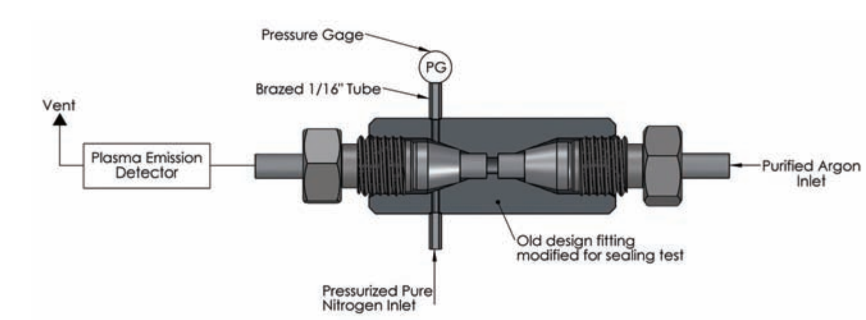

Figure 13a: Test apparatus for sealing of old design fitting

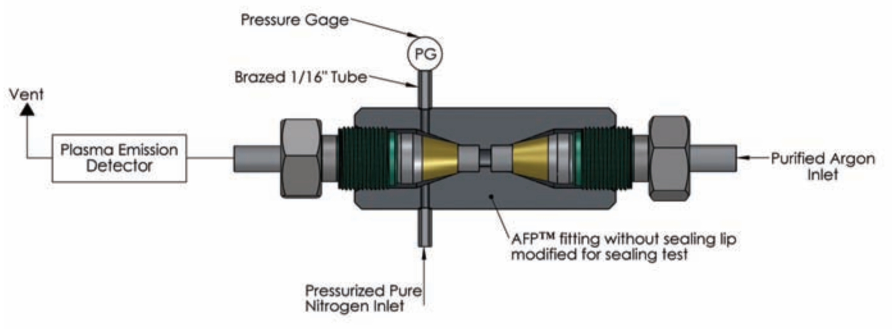

Figure 13b: Test apparatus for sealing of AFP™ new fitting without sealing lips

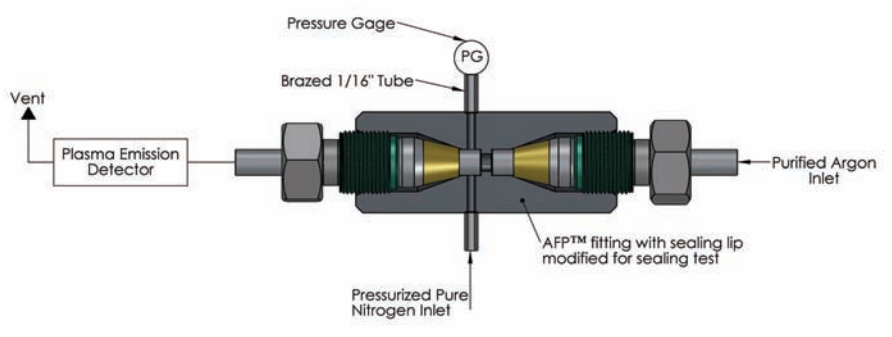

Figure 13c: Test apparatus for sealing of AFP™ new fitting with sealing lips

On the other tube, a pressure gage has been installed. In the case of the AFP ™ new fitting with sealing lip (Figure 13c), the small holes are located between the sealing lip and the sealing area between the ferrule and the fitting body, as this is the area to be tested for this fitting.

Afterward, the evaluation was done with the test bench shown in Figure 13. Purified argon flows at atmospheric pressure through axial tube of the fitting, while the brazed tubes of the fitting are pressurized with pure N_2. The fitting is connected to a Plasma Emission Detector tuned to measure the intensity of the N_2 emission line at 337.1 nm. Other types of N_2 sensitive detectors could also be used. Such systems have found leaks that were undetectable with a helium based mass spectrometer leak detector. This set up is also very sensitive to measure inboard leakage. Indeed the fitting is surrounded or "immersed" in a "sea" of air, which has about 79% N_2. Compared to the 5 ppm of helium normally found in atmospheric air, which the helium mass spectrometer based leak detector relies on to find such a leak. The N_2 as a tracer is more sensitive and the system is much less complex.

This set up is used by AFP™ for the leak rate certification of all our valves. The gas is passed through a 0.5 micron particle filter before being introduced to the fitting under test. This is to make sure that fitting performance test will not be affected by particles being introduced into the fitting. This system has 4 to 5 times the sensitivity for leak detection than a standard helium mass spectrometer leak detector, see ref. [3].

Once the test bench properly installed, we pressurized the test area with a specific pressure of N_2; the signal increasing as the detector sees the increasing amount of N_2 coming from the badly sealed area. So, we tightened the nut to eliminate the leak and get back system baseline.

Once done, we recorded both pressure and torque, and repeated these steps for many pressure and for all three fittings.

Ones can see on Figure 14 the torque requirement reduction given by the AFPTM new improvements, compare to old existing design. Furthermore, our experience has demonstrated that ¼-past tube gripping is not enough to achieve proper sealing while using those old design unions. So, torque requirement is much higher getting of the old design union the same sealing level.

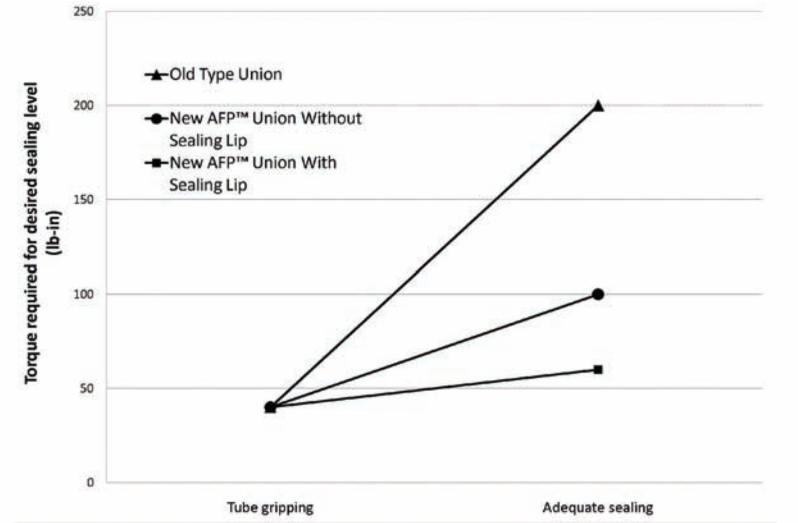

Figure 14: Torque required achieving adequate sealing

Conclusion

We have demonstrated why the characteristics that make an excellent industrial fitting are giving some serious problems in high performance analytical applications. While the existing single ferrule based analytical fitting avoids problems generated by swaging action, its required rotating torque for dimension of 1/8"OD and higher generates serious problems.

Modification of ferrule, nut designs and fitting detail, as described previously, has resulted in tremendous benefits, like eliminating torque related problems (ferrule rotation and twisting), reducing dead volume, better sealing and increasing the possible number of remakes.

However, ones should not forget that an important part of the fitting is the quality of the tubing.

Reader comments are welcome. The author may be contacted at: ygamache@afproducts.ca

Reference

[1]: Agilent, 6890 User's Manual and, Site Preparation and Installation Manual. Manual.

[2]: Swagelok, Tube Fitter's Manual by F.J. Callahan.

[3]: Varian Mass Spectrometer Manual, Part Number

0981-6999-09-070, September 1995, P.P. 3-10, 3-11

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