Troubleshooting LC Fittings, Part II

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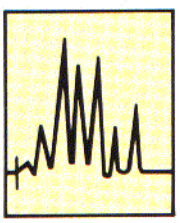

Last month's "LC Troubleshooting" (1) covered the design and assembly of stainless steel fittings used for connecting 1/16-in. o.d. tubing in LC systems. We discussed how fittings from various manufacturers are visually similar and in many cases can be interchanged. This article covers some of the problems that you might encounter if you use fittings improperly. We also discuss the use of finger-tightened fittings as an alternative to stainless steel compression fittings.

FITTING INTERCHANGEABILITY

If you reuse assembled fittings, fitting interchangeability among manufacturers becomes important. When the ferrule is first swaged onto the tube end, it is fixed at a distance dictated by the depth of the inside of the fitting body (the port depth). Thus, for different fittings, different lengths of tubing extend past the swaged ferrule, as illustrated in Figure 1. The extensions shown in Figure 1 result when fittings from different manufacturers are assembled, then disassembled and measured. Two situations that can result from these differences are illustrated in Figures 2a and 2b. When a tube end with a long extension is assembled with a fitting body that has a shorter mating port, the fitting leaks, no matter how tightly the nut is turned (see Figure 2a). Conversely, a tube end with a short extension mated to a fitting body with a long port depth will seal properly, but will leave a small mixing chamber, or dead volume, at the tip of the tube (see Figure 2b). Such dead volumes cause extracolumn band broadening and result in broader peaks, and thus in poorer resolution. Here again, caution is the key word. Make sure that you use only tube ends that are assembled properly for the fitting body. Sticking to one fitting manufacturer is the easiest way to avoid problems.

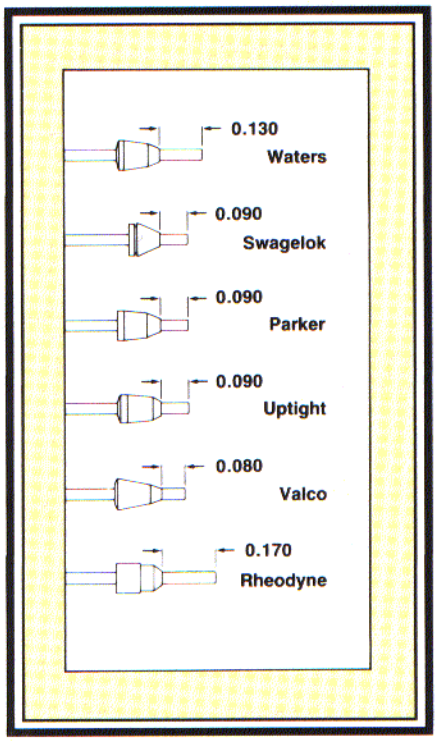

FIGURE 1: Examples of different lengths of tubing that extend past the ferrule after different manufacturers' fittings are made up. Tubing extensions, shown in inches, are experimental values, not necessarily manufacturers' specifications.

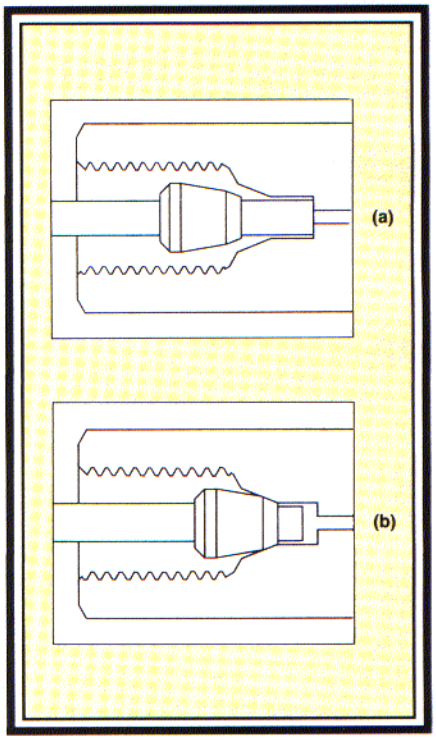

FIGURE 2: Tube ends that are not properly matched with the fitting body. (a) Long tube end with shallower fitting body will result in leakage. (b) Short tube end with deeper fitting body will seal properly, but will add to system dead volume.

FITTING DISTORTION

Another common fitting problem is fitting damage that is due to user abuse. Last month (Figure 1, reference 1) we discussed how external-thread fittings can become distorted when they are used repeatedly. The thicker wall of internal-thread fittings reduces the probability of distortion problems. Unfortunately, distortion problems that do occur with internal-thread fittings are not easy to see because they are inside the fitting body. A ferrule seat in a new fitting should look like that shown in Figure 3a. If the nut is overtightened, the ferrule and mating cone can become distorted (Figure 3b). You may not notice this immediately because the fitting seals well. You can remake this fitting using the same pieces and get a successful seal. When you try to use another nominally interchangeable tube end, however, the ferrule will not seat properly (Figure 3c), and the fitting will leak. The natural tendency is to tighten the nut a bit more, but this further distorts the cone and makes the situation worse.

You can solve problems that result from distorted fittings by replacing the entire fitting. In some cases, such as with check valves and mixers, replacing the LC part that contains the fitting body can be expensive. If you take it easy when tightening the fitting nuts, you should rarely have problems with distorted fittings.

are assembled in the same manner as all-stainless steel fittings — by sliding the nut and ferrule (or nut-ferrule assembly) onto the tube end, pressing the tube end to the bottom of the fitting body, and tightening the nut with your fingers. When finger-tightened, these devices typically hold 2000–5000 psi of back pressure, which is within the practical working limits of most LC systems. If higher pressures are used routinely, a wrench-tightenable metal-and-polymer fitting should be used. The Upchurch fitting (Figure 4a) is assembled just like the other finger-tightened fittings, except it can be tightened further with a wrench in order to provide sealing pressures up to the typical LC system limits of >6000 psi. A similar fitting can be assembled from standard stainless steel parts and nylon or PTFE ferrules. While this configuration may seem less expensive, the ferrules are seldom reusable because they become distorted or stick in the fitting body. If you are concerned that the other finger-tightened fittings do not provide enough security against leakage, the Keystone fitting (Figure 4c) is a good alternative to all-stainless steel fittings. With this fitting, the initial seal is made with a polymeric ferrule, just as with the other models. Added pressure security is given by tightening the second nut against the stainless steel ferrule. This gives a slip-free seal and pulls the tube end into the fitting body so that you are sure that the end bottoms properly in the body.

When you discover a leak with a finger-tightened fitting, it is best to turn off the pump, loosen the fitting, and make sure that the tube end is pressed firmly into the fitting body before retightening the nut. If you were to try to tighten the fitting with the pump on, the tube end might slip out (creating dead volume) because it is lubricated by the leaking mobile phase.

The ferrules used for finger-tightened fittings are made of various polymers that exhibit low distortion and high chemical resistance (for example, Vespel, Kel-F, or PEEK). The fittings that use plastic nuts can break off in the fitting body if you use too much tightening force (for example, if you use pliers). If this happens, you can remove the broken piece from the body by first pulling the tube end free, then pressing the heated tip of a small screwdriver into the stub to melt a slot in the stub. Finally, unscrew the broken piece and replace it with a new nut.

WHERE TO USE FINGER-TIGHTENED FITTINGS

Although finger-tightened fittings can be used on any tubing in the LC system, they generally are used for connections that need to be disconnected regularly, such as the tubing connections between the sample injector, column, and detector. Column connections with finger-tightened fittings are especially convenient; you don't need any tools, and the fitting is easily adjusted so that different manufacturers' columns can be used without concern for fitting assembly problems. Finger-tightened fittings also work well with polymeric tubing such as the waste lines from the injector or detector. For other high-pressure connections (for example, between the pump and the injector), most users prefer conventional stainless steel fittings because they can be made up once and forgotten. Tubing connections for low-pressure applications, as on the inlet side of the pump, generally use specially designed low-pressure fittings (the subject of a future installment of "LC Troubleshooting").

SUMMARY

Compression fittings for connecting 1/16-in. o.d. stainless steel tubing are easy to use and relatively trouble-free when used properly. In order to minimize problems with your fittings, assemble the fittings properly, don't mix fittings among manufacturers (at least not after assembly), and don't overtighten the fittings. Finger-tightened fittings can be used in place of stainless steel fittings, but they generally are reserved for those instances in which the fitting must be assembled and disassembled frequently, such as with column connections.

ACKNOWLEDGMENTS

Figure 4b was supplied by Alltech Associates (Deerfield, Illinois), and Figure 4c by Keystone Scientific (State College, Pennsylvania). The remaining figures are courtesy of Upchurch Scientific (Oak Harbor, Washington).

REFERENCES

(1) J.W. Dolan and P. Upchurch, *LC•GC* **6**(9), 788 (1988).

"LC Troubleshooting" editor John W. Dolan is president of LC Resources Inc. of Lafayette, California, USA, and is a member of the Editorial Advisory Board of LC•GC.

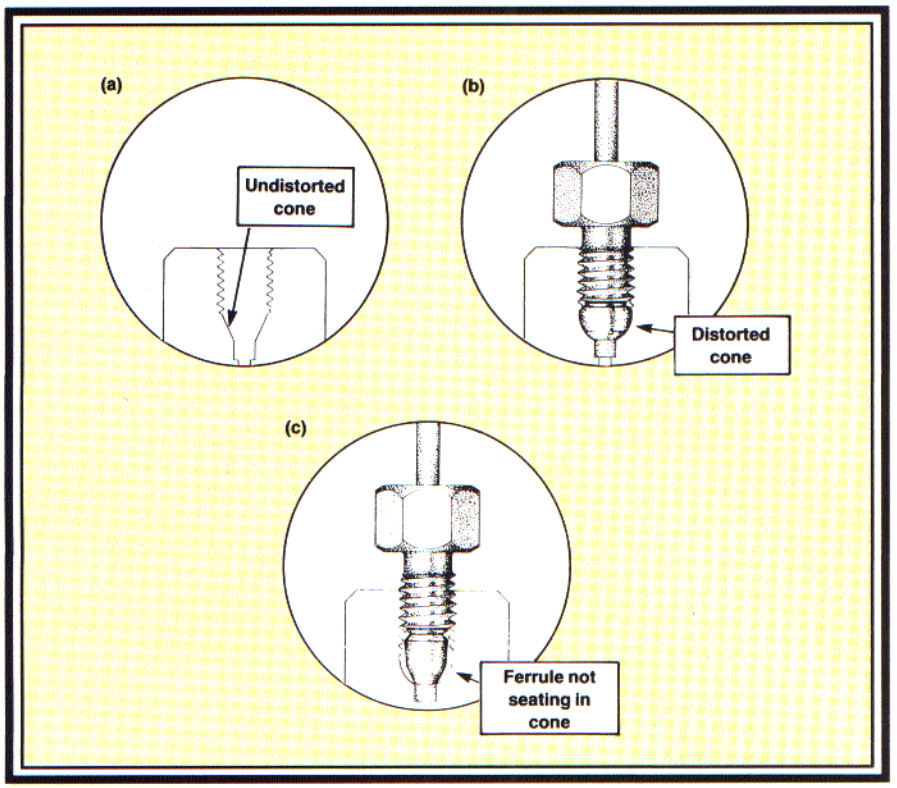

FIGURE 3: Problems resulting from an overtightened fitting. (a) New fitting body, showing undistorted ferrule seat. (b) Distorted ferrule and seat resulting from overtightening the nut. (c) Leaky fitting caused by assembly of a new ferrule in a distorted fitting body.

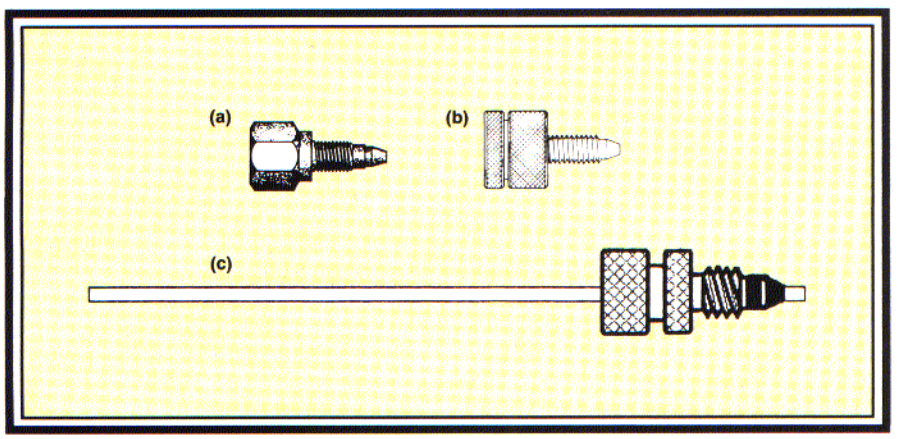

FIGURE 4: Typical finger-tightenable fittings made up of a stainless steel nut and polymeric ferrule. (a) Wrench-tightenable high-pressure Fingertight from Upchurch. Tightening with a wrench will provide a seal up to 10,000 psi. (b) Universal column adapter from Alltech Associates. The nut on this fitting is internally threaded so that it can be reversed to connect with either male or female threads. (c) Slip-Free from Keystone. This fitting has a second ferrule of stainless steel that provides added security from tubing slippage.

SPECIAL PRECAUTIONS FOR MICROBORE

When you use microbore LC columns (<2 mm i.d.), extracolumn dead volume is especially critical. It is therefore essential that the dead-volume contribution from the fittings be kept to an absolute minimum. Because of manufacturing tolerances, slight differences in the port depth exist between nominally identical fittings from the same manufacturer. For this reason, in microbore LC it is best always to use exactly the same tube end and fitting as a pair, rather than interchanging fittings during use. The slight differences from one fitting to the next can add enough dead volume to cause problems in certain microbore applications. Clearly mark the fitting sets with adhesive labels or by scribing an identifier on the fitting body and nut.

FINGER-TIGHTENED FITTINGS FOR HIGH-PRESSURE APPLICATIONS

In recent years, it has become increasingly popular to use fittings that can be finger-tightened to securely hold stainless steel tubing under normal operating pressures (2000–6000 psi). These are now available from a variety of manufacturers (see Figures 4 and 5). In all cases, these fittings rely on a polymeric ferrule to make the seal between the tubing and the fitting body in the same manner as for standard stainless steel fittings (for example, Figure 3 of reference 1). There are three main advantages to a polymeric ferrule: First, it does not become permanently swaged onto the tubing, so the tube end can be detached and reassembled with another brand of fitting body and still seal properly. That is, the ferrule can be slid in or out on the tube end to match the extension dictated by the fitting body (see Figure 5). The second advantage of the polymeric ferrule is that it can be somewhat distorted, allowing it to make a good seal in fittings with ferrule seats of different tapers. It will even provide a good seal with a distorted fitting seat, such as that of Figure 3. Finally, because the polymeric ferrule is softer than the stainless steel body, it will deform before damage is caused to the ferrule seat. Thus, if a fitting is overtightened, a relatively inexpensive ferrule is destroyed, not an expensive fitting body.

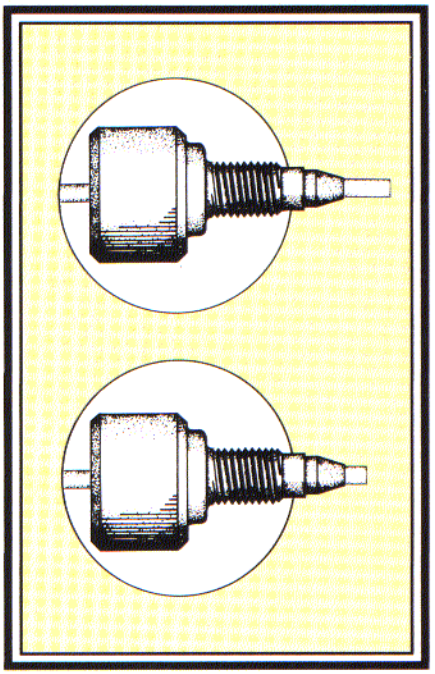

FIGURE 5: Adjustability of finger-tightened fittings. Polymeric ferrule grips the tube end to provide a leak-free seal, yet will slide freely on the tube end when disassembled so that the extension can be adjusted for fitting bodies of different depths.

The finger-tightened fittings of Figures 4 and 5 provide the same sealing function, although they differ in design. The fittings of Figure 4 have a stainless steel nut and a polymeric ferrule. The all-plastic fittings (Figure 5) are available in a single- or two-piece design. Both kinds of finger-tightened fittings