L C T R O U B L E S H O O T I N G

Troubleshooting LC Fittings, Part I

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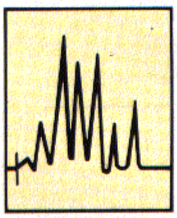

Nearly five years have passed since "LC Troubleshooting" has included an in-depth discussion of LC fittings and fitting problems (1). We have added information on new fittings and have expanded the original discussion to include more details on fitting design, use, and problems. This article, the first of two parts, is primarily descriptive and covers stainless steel fittings used for connecting $^1/_{16}$-in. o.d. tubing in LC systems. We describe the function, proper assembly, and comparison of fittings. The article is based, to a large extent, on the material found in reference 2. Next month we will discuss the major problems that occur with LC fittings and look at some alternatives to the use of stainless steel fittings for high-pressure connections.

FITTING TYPES

The two basic types of high-pressure fittings used in LC are illustrated in Figures 1 and 2. In the early days of LC, the external column endfitting was most popular. This fitting uses a male fitting body (external threads) with a female nut (internal threads), as seen in Figure 1a. Experience has shown that these fittings are not very durable. In most users' hands, the repeated attachment and removal of the $^1/_{16}$-in. tubing distorts the conical sealing surface, as illustrated in Figures 1b and 1c. This problem is minimized by the use of internal fittings, which use female threads in the fitting body and a male nut, as seen in Figure 2a. This design is the standard today and is much less susceptible to distortion than is the external fitting. Problems associated with this design are discussed later.

UNIONS

Along with the column endfittings, the most common LC fittings are unions, fittings that connect two pieces of tubing. The internal-thread zero-dead-volume (ZDV) union (Figure 3) is most widely used. (Analogous external-thread unions are available, but these suffer from the same problems as the column endfittings discussed above.)

Notice that the union of Figure 3 is not drilled through completely but has a small web of metal left between the two pieces of tubing. When GC fittings were modified for LC use, it was common to drill the union all

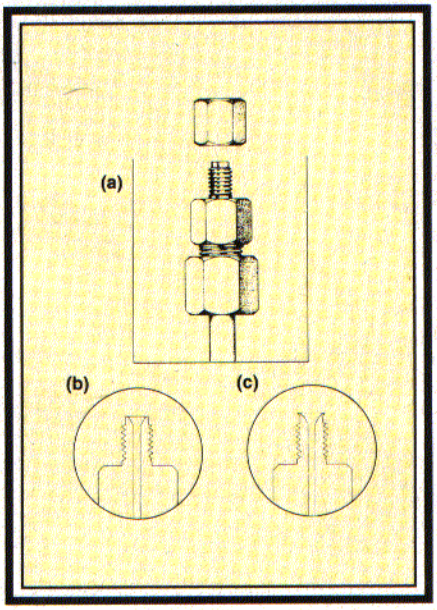

FIGURE 1: External column endfitting. (a) Endfitting with external (male) threads and internal nut. (b) Cross section showing new seat. (c) Cross section showing distorted cone resulting from overtightening the fitting.

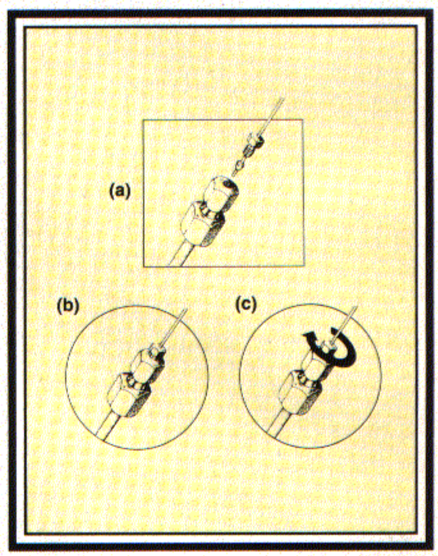

FIGURE 2: Internal column endfitting. (a) Exploded diagram, showing nut, ferrule, and tube end about to be inserted into fitting body. (b) First step in fitting assembly: Finger-tighten nut. (c) Final step in fitting assembly: Turn nut a three-quarter turn past finger-tight to swage ferrule onto the tube end.

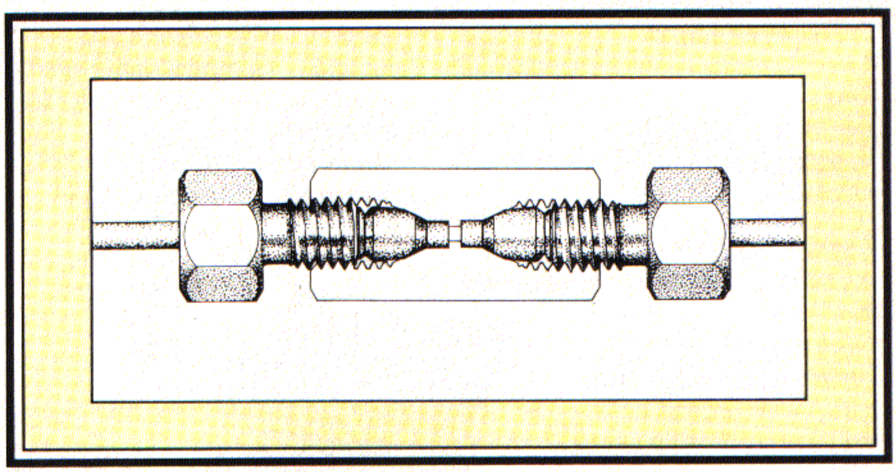

FIGURE 3: Zero-dead-volume union. Note narrow passageway between the two tube ends through the fitting body.

the way through so that the two joined pieces of tubing would butt against each other with no volume between them (Figure 4a). Because no extra volume was added to the system by the connector, the term "zero-dead-volume" was used. This fitting design usually worked well when it was first used or when the original pieces were reassembled. However, when poor assembly techniques or mismatched parts were used, numerous problems occurred, as illustrated in Figures 4b and 4c, in which extra volume was unintentionally added to the system or fitting leakage occurred. The answer to these problems was to leave a small web of metal between the two halves of the fitting, with a small hole drilled through as shown in Figure 3. Because the length of the web is short (typically 0.020 in.) and the diameter of the through-hole is small (typically 0.020 or 0.010 in.), there is no practical contribution to system dead volume. The advantage of this precisely machined fitting is that the tube ends will be interchangeable once the ferrule is set on the tubing (see discussion below).

MAKING UP FITTINGS

When fittings are made up the first time, they need to be assembled so that the ferrule is properly swaged onto the tube end. There are four parts to the unassembled fitting: the fitting body, the tubing, the ferrule, and the nut, as shown in Figure 2. To assemble the fitting, slide the nut and ferrule onto the tube end as shown in Figure 2a. Next, push the tubing into the fitting body until the tube end bottoms out. Finger-tighten the nut (Figure 2b), then tighten it a three-quarter turn more with a wrench (Figure 2c). Now the ferrule is permanently attached to the tubing. It is important that you hold the tubing securely in the fitting while tightening the nut, or improper assembly can result (these problems will be discussed next month). To replace the ferrule, you must cut the tubing, discard the old ferrule, and attach a new ferrule.

Once the fitting has been assembled initially, you should be careful not to overtighten it during reassembly. The procedure is simple. Just insert the preformed tube end into the fitting body and finger-tighten the nut. Then, using a wrench, tighten the nut a one-half turn (not a three-quarter turn as above). Turn on the system and start the mobile phase flow. If the fitting leaks, tighten it a little at a time until it stops leaking. Do this each time the fitting is reassembled, and your fittings will last longer. Make sure you avoid overtightening, which can cause leaks and destroy the fitting.

AVOIDING FITTING PROBLEMS

Be aware of design differences among manufacturers. Nuts, ferrules, and fitting bodies may vary in design. Some of these differences are fairly insignificant in terms of practical fitting usage, but other differences can result in problems when fittings from different manufacturers are interchanged. We'll examine the nuts first.

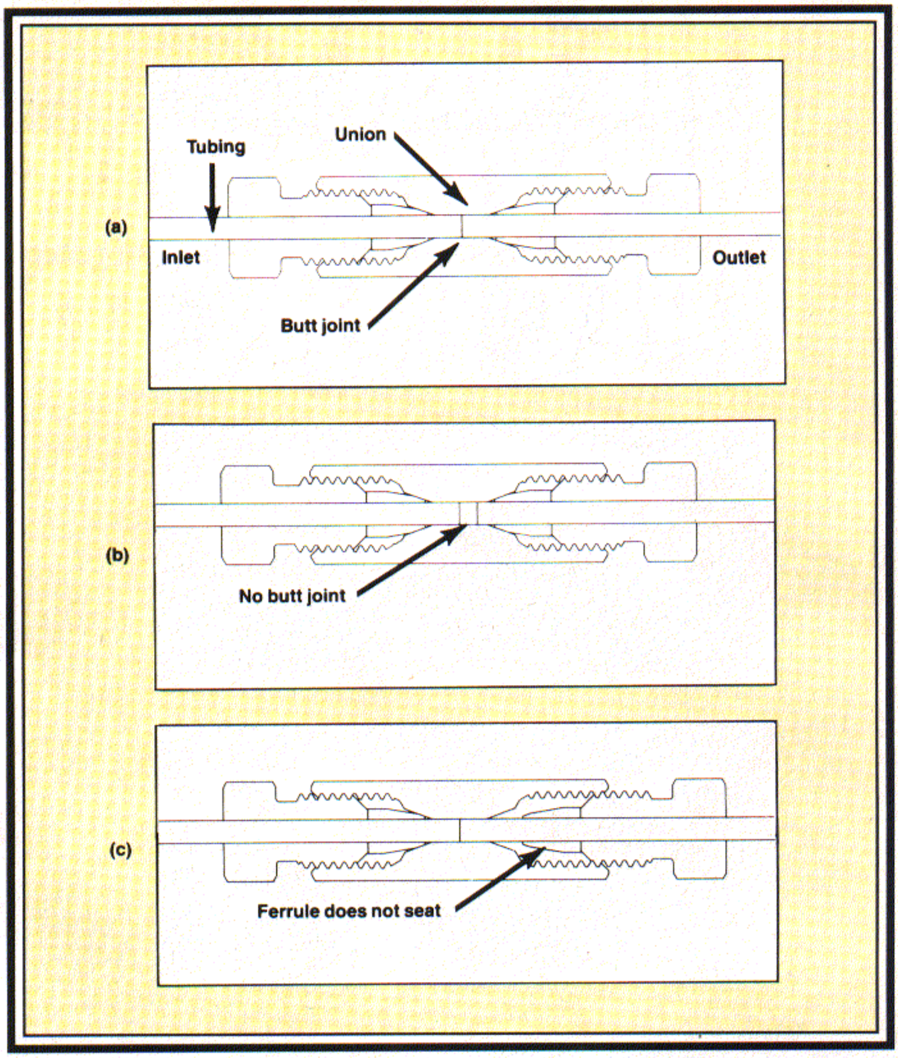

FIGURE 4: Early versions of zero-dead-volume union. (a) Properly assembled union, with tube ends properly butted. (b) Connection resulting when tube ends are too short: Space between tube ends adds dead volume, resulting in band broadening. (c) Connection resulting when tube ends are too long: Tube ends butt, but ferrule does not seat, so fitting leaks.

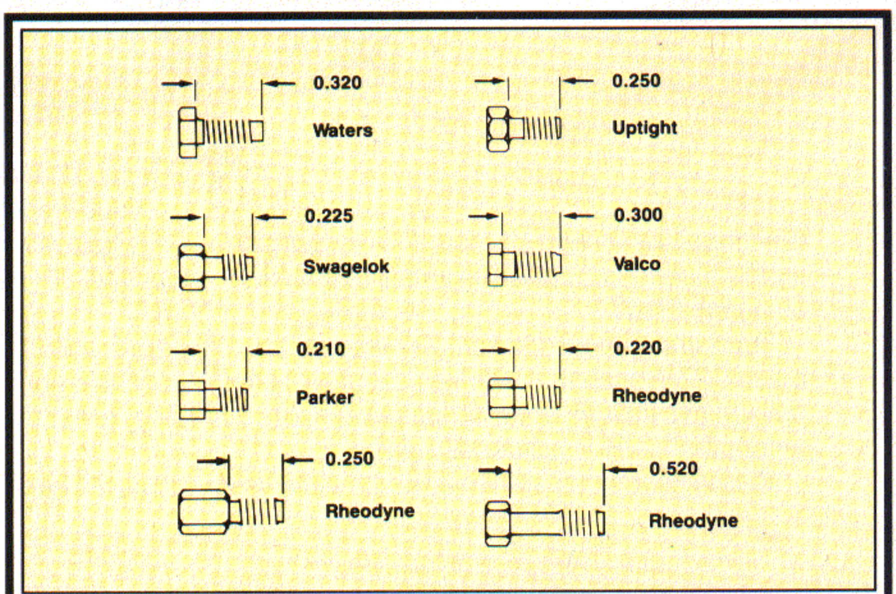

FIGURE 5: Comparison of 10–32 threaded nuts. Measurements shown (in.) are typical, but not necessarily manufacturers' specifications.

At first glance, the male nuts are all the same, but upon closer examination, you'll notice some important differences (see Figure 5). First, the nuts are not the same length. (The measurements in all the figures are typical experimental measurements and may not accurately reflect manufacturers' specifications.) Usually, the mating fitting body will have a complementary thread length (for example, a long nut usually will have a long threaded body). You will also notice that some nuts have threads that extend to the tip of the nut, whereas others have a short unthreaded portion at the tip (compare Waters and Uptight in Figure 5). As mentioned above, properly mated fittings will cause no problems, and in many cases the nuts from one manufacturer will work in the fitting body of another manufacturer. Problems can occur, however, as illustrated in Figure 6. At one extreme (Figure 6a) is the case in which a short nut is used with a fitting body with deep threads. Here, the shoulder on the nut bottoms out before an adequate seal is made; the resulting fitting leaks. The other extreme is illustrated in Figure 6b, in which an inadequate number of threads are in contact between the nut and body; damage to the nut or body will probably result.

The remaining difference between the nuts is the type of threads that are used. Two English threads (1/4–28 and 10–32) and one metric thread (M6) are available. Most American manufacturers use 10–32 threads, meaning there are 32 threads per inch on a no. 10–size shank (Figure 7a). One U.S. manufacturer (Scientific Systems, Inc., State College, Pennsylvania) uses 1/4–28 threads (28 threads per inch on a 1/4-in. shaft, Figure 7b), and European manufacturers often use the M6 thread size (1 thread per mm on a 6-mm shaft, Figure 7c). These nuts are not at all interchangeable. Fortunately, the 1/4–28 nut cannot be misassembled with the other fittings. However, the 10–32 and M6 nuts are close enough in size that it is possible to thread them into the wrong fitting body. This can result in a loose fitting, a leak, or permanently damaged threads. So in order to avoid this problem, it is important to clearly label 10–32 and M6 fittings if they are used in the same laboratory (for example, use a diamond-tipped scribe to mark "US" or "M6" on these nuts).

The main operating practice to glean from this discussion is that you will be much less likely to have assembly problems if you stick to one manufacturer for nuts and fitting bodies. If you do choose to interchange nuts and fittings, do so only with unassembled fittings (see the discussion of assembly problems below for potential problems).

Design differences also exist among the ferrules sold by different manufacturers. Typical examples are shown in Figure 8. Swagelok (Solon, Ohio) uses a two-piece ferrule, whereas the other manufacturers use a one-piece design. The shape of the ferrules, as seen in Figure 8, also differs noticeably among vendors. These differences are of little practical importance, because all of these ferrules are interchangeable when new ferrules are first used with a fitting. This interchangeability is due to the distortion of the ferrule that takes place at its leading edge when it is swaged onto the tubing. Thus, the ferrule deforms to take the shape of the mating fitting at the sealing surface (see the contact between the ferrule and fitting body in Figure 3). In other words, while it is a good idea to use ferrules made by the same manufacturer as the nut and fitting body, it is not essential (precautions on interchangeability will be given in next month's column).

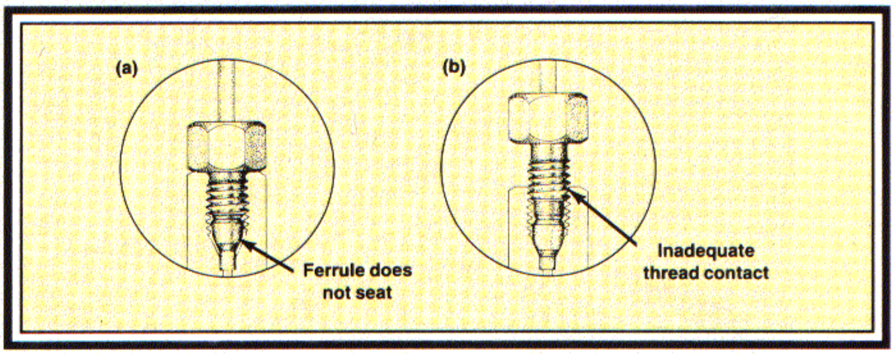

FIGURE 6: Fitting problems resulting from mismatch of fitting body and nut. (a) Short nut with deeply threaded fitting: Ferrule does not seat and shoulder of nut hits fitting body. (b) Nut with unthreaded tip in short fitting body: Stripped threads are likely because of insufficient thread contact.

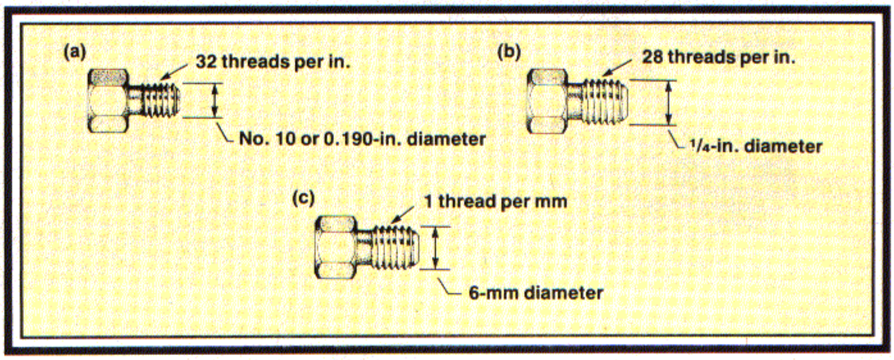

FIGURE 7: Thread types. (a) 10–32 threads, standard for most fittings used in the United States. (b) 1/4–28 threads, used only by Scientific Systems, Inc. (c) M6 threads, common in Europe.

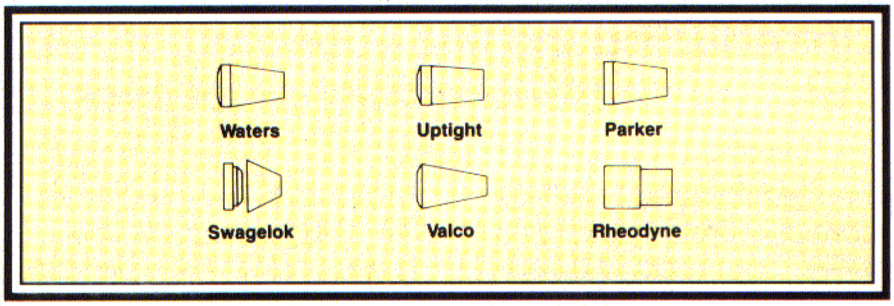

FIGURE 8: Ferrule designs. Although they look different, these ferrules are interchangeable when new.

SUMMARY

We have seen that most high-pressure fittings used in LC systems are the internal-thread (female) type. These have proved to be more durable than the external-thread fittings used in the early days of LC. The fittings produced by different manufacturers are visually similar, and in many cases may be interchanged successfully, especially when the parts are new. However, fewer problems are likely to occur when you use matched parts from a single manufacturer. Next month we will discuss the most common fitting problems and how to avoid them.

ACKNOWLEDGMENTS

The figures provided with this article are courtesy of Upchurch Scientific.

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"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.