TROUBLESHOOTING

Potpourri

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My goal as the writer of this column is to provide practical troubleshooting information that is of use to many readers. This can be a difficult task, however, because my view of high performance liquid chromatography (HPLC) and HPLC problems is, of course, biased by my particular laboratory experience. Many readers use HPLC with different constraints and thus see problems that I rarely encounter. This month, along with the usual troubleshooting tips, I am including some questions for readers. I hope that some of you will respond to these questions; that way, many of us can learn from the experience of a few. If you have troubleshooting tips on proprietary products, your description can be phrased in such a way that no trade secrets are revealed.

POLYMERIC SUPPORTS

Polymeric column packings have been available for years in the form of gel-permeation or gel-filtration columns. Care is required with these columns to avoid pressure shock that could collapse the column bed. Also, the choice of solvents for the mobile phase is limited by chemical incompatibility between some mobile phases and the packing. Finally, the cost of these columns is quite high; experimenting with conditions during method development is risky, because a column can be destroyed in just a few minutes.

A number of manufacturers now offer columns based on rigid polymeric supports; these columns are reportedly much more robust than earlier products. Of particular interest are the reversed-phase polymeric supports, which are free of the silanol effects common with silica-based reversed-phase columns.

I have not had hands-on experience with this new generation of columns, but many of you have used them, and I would like to solicit your reactions. Are they as stable as the manufacturers claim? Are special techniques such as high- or low-pH flushing useful in prolonging column life? Is column-to-column reproducibility adequate, or is it necessary to mix additives with the mobile phase to mask undesirable effects? Have you encountered any unexpected problems?

LOW-PRESSURE TUBING

One common problem with making up low-pressure fittings is how to get a good grip on the Teflon tubing. This is not a problem when a slip-on ferrule is used, as with the inverted-ferrule fittings from Upchurch (Oak Harbor, Washington). However, flared fittings (for example, Cheminert, from LDC/Milton Roy, Riviera Beach, Florida) and fittings that use inserts (for example, General Valve, Fairfield, New Jersey) require a firm grip to prevent the tubing from slipping during assembly. I follow the advice given in the General Valve literature and carry a piece of 600-grit sandpaper in my lab coat pocket so that it is handy for gripping the tubing when the need arises. Some people use a Kimwipe instead of sandpaper, but I've never had much luck with that. At a recent seminar, someone suggested using a latex glove to grip the tubing; a pair of finger-cots should also work well.

Omnifit (Cambridge, United Kingdom) suggests cutting Teflon tubing diagonally so that it can be threaded easily through the firm's grippers (ferrules). Then cut the tube end squarely before installation. Tapering the end of the tubing is also useful for threading the tubing through stainless steel ferrules or through the access holes in column ovens or detectors. Once the tubing is positioned properly, trim the end squarely with a razor blade.

FINGER-TIGHTENED FITTINGS

The plastic finger-tightened fittings popularized by Upchurch are widely used, especially for column connections. Three problems commonly associated with these fittings can easily be solved.

First, readers complain that the tubing slips under pressure. This usually occurs if the fitting is made up while the mobile phase is flowing; there are fewer problems if the fitting is tightened under no-flow conditions. The second generation of finger-tightened fittings, available from most LC supply houses, consist of a metal body and a polymeric ferrule joined into one piece. The metal body allows the fitting to be more firmly tightened, alleviating the slippage problem.

Second, the all-plastic fittings can strip out if they are tightened too much. Never use pliers to tighten finger-tightened fittings or you will strip the threads. If this happens, be sure to clean any thread shards from the mating portion of the fitting (for example, the column end). Tiny pieces of the threads can block the fitting, tubing, or column frit. (Do any of you know of a good technique for removing the pieces of stripped-off threads?) Of course, the problem can be avoided by using the metal and plastic fittings mentioned above.

Finally, at one time or another, we all overtighten finger-tightened fittings and break them off in the column endfitting. A reader suggests that the broken piece can be removed with a heated screwdriver. Just heat up the end of a pocket screwdriver (be careful not to use an open flame around solvents!), push it into the broken piece, and unscrew it. What a neat idea! I can hardly wait to break a fitting so that I can try this. . . . Once again, using the metal and plastic fitting will prevent this problem.

STATIC ELECTRICITY

Static electricity can sometimes be a problem with microprocessor-based HPLC systems even though commercial systems are supposed to be immune. I've caused system crashes by touching the chromatograph after shuffling across the lab in crepe-soled shoes. Static is particularly troublesome in the Midwest and East during the winter and in the Western states during summer months. You can't do much to solve the instrument problems, but you can minimize the source of static. One good method is to spray an antistatic aerosol on the lab floor in front of the HPLC unit. This is especially effective on the thick rubber fatigue mats often found in labs. Antistatic spray is available from some laboratory suppliers, but the most convenient source is the local grocery store — just get the spray sold for use in clothes driers.

If you still have a static problem after spraying the floor, try grounding yourself before touching the instrument; simply touch a water pipe or electrical outlet at the back of the bench to dissipate the static charge.

FOR COMPUTER USERS

Here are a few tips for those of you who have computer data systems attached to your chromatograph. First, handle floppy diskettes with care. The diskettes are durable and are hard to damage, so many of us get sloppy about storing them properly (I have about a dozen of them collecting dust in a pile next to my computer as I write this article). It takes only one accident to make one realize that

valuable data may well be gone for good once a disk is damaged. For this reason, be sure to make a backup copy of any data diskette that you feel is worth saving.

Beware of stir-bar magnets. A stir-bar can instantly erase the data from your disk. One person I know destroyed a disk of valuable data when he inadvertently held the disk next to his lab coat pocket, which contained a stir-bar. If you are in the habit of keeping a few stir-bars in your lab coat pocket, perhaps now is the time to stop.

If a mouse is part of your computer's hardware, you should be careful not to contaminate the mouse with solvents or other chemicals that may have spilled on the bench. If possible, move the monitor to one side and operate the mouse on the top of the computer's system unit. Not only will this keep the mouse out of puddles, but also the textured surface of the system unit enhances the performance of the ball used in mechanical mice.

If you are concerned about spilling liquid into the computer keyboard or otherwise damaging it, try sliding a plastic bag over the keyboard. Most keyboards will operate properly with a plastic cover, although you may have to experiment with several different thicknesses of bags before you find the best one for your system.

If your system has a hard disk, be sure to park its heads before you shut off the power. Head-parking utilities are available as public-domain programs from many sources and are included with the later versions of DOS. These programs move the heads to the unused inside track of the hard disk before the power is turned off, preventing the heads from crashing into another part of the disk and destroying data that may be stored there. This kind of crash is rare, but it can be disastrous. I once had a hard-disk head-crash that destroyed three megabytes of data. Now I *always* park the heads before switching off the power.

CASE STUDIES

When troubleshooting "case studies" are presented in this column to illustrate specific problems and solutions, the response is always very positive. If you have a case study that would be instructive to other readers, I would like to hear from you. Reading about someone else's HPLC problem not only is informative, but it also provides practice in troubleshooting as the problem is traced from its detection to its solution.

AT YOUR SERVICE

It usually takes four to six months for a question or topic submitted to Troubleshooting to get into print. I realize that readers often need specific problems solved immediately, however, so I respond as soon as possible with either a letter or a phone call to help you get right back to work. If you have troubleshooting problems and/or tips, send me a note (c/o *LC•GC*, P.O. Box 10460, Eugene, OR 97440), or call me at (408) 266-8371.

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