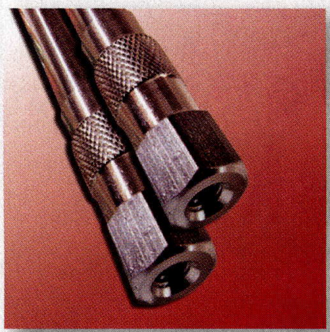


LC TROUBLESHOOTING

Drip, Drip, Drip . . .

Some things never change

John W. Dolan
LC Troubleshooting Editor

This "LC Troubleshooting" installment marks the beginning of the 26th year that I have been writing this column. Over that time, changes in instrument design, and especially column technology, have occurred that have made liquid chromatography (LC) an easy-to-use and reliable process. Some of the major problems with routine operation now are minimal. For example, bubbles resulting from poorly degassed mobile phase no longer hold first place on the top 10 LC problems list — automatic in-line degassers are largely responsible for this change. Some problems, however, have not gone away and probably never will. Leaks fall in this category. Leaks are so ubiquitous that it is easy to assume that everyone knows how to handle them, so we don't even train new workers on how to deal with them. This is highlighted by a quick search through my database of past "LC Troubleshooting" columns — "leak" or "leaks" only showed up as a keyword in 18 of approximately 280 columns. So, I guess it is time to go back to the basics and spend a little time on leaks for this month's topic.

How Do I Know There's a Leak?

Leaks rarely occur without giving some other warning at the same time. Most commonly, leaks are accompanied by a low or fluctuating pressure readout. Many LC systems are equipped with leak detectors. These are simply a pair of electrical contacts located in a low spot in the column oven, autosampler, pump, or other system component. When a leak occurs, the mobile phase runs to the low spot and completes the circuit between the contacts and triggers an alarm. With the help of a leak detector, you usually will know which module has the problem, but you might need to look further to identify the specific source of the leak.

If the leak causes a steady drip, drip, drip of mobile phase, it is fairly easy to track to its source. Smaller leaks can be a bit more of a challenge. Some leaks are so slow that the mobile phase evaporates before a visible drop is formed — these can leave buffer residues behind. One thing to look for is a white deposit or crystalline fuzz of buffer salts at one of the connections — this is a sign of a past or present leak. Some very small leaks can be frustrating to identify — probing them with a paper towel or laboratory wiper does not seem to show any moisture present. One of my favorite leak-detection tools for such cases is a small piece of thermal printer paper. Thermal paper is becoming a rare commodity in the laboratory, but you might find it in a balance printer, a fax machine, or in a drawer where you keep printer supplies. Another source is a charge-card receipt, most of which come printed on thermal paper. Cut a thin pointer from the paper, for example, 1 cm wide at one end by 5–10 cm long, tapering to a point. Thermal paper is very sensitive to small amounts of organic solvent (you can test this by placing a drop of alcohol or acetonitrile on the paper), so you can use this probe to poke at the fittings or other location where you suspect a leak. If a leak is present, the tip of the paper will turn black. (Note to self: do not throw away that last "useless" roll of thermal fax paper.)

Where Is It?

Because mobile phase is the lifeblood that flows through the LC system, leaks can occur nearly anywhere in the system. Most of the time, a leak is discovered initially by a pressure problem, a leak detector alarm, or a drop of fluid you happen to notice during some other operation. I recommend following a procedure that will help to identify leaks and some other problems before they reach the data-threatening stage. Each time the LC system is started, during the equilibration period, simply trace the flow path of solvent through the LC system and look for obvious problems, among them a bead of liquid indicating the presence of a leak. Start at the mobile-phase reservoir: Is there sufficient mobile phase, is junk floating in the buffer? Follow the flow path to the pump: Any leaks at low-pressure fittings? Crimps in tubing? Is the degasser working? At the pump, look at all the connections for signs of leaks. Does everything sound normal? Open the door to the autosampler: Are there white spots indicating leaks or other visible signs of trouble? Do the connections on the injection valve look OK? Is there enough wash solvent in the reservoir or wash vial? Open the column oven and look around for any signs of leakage: Does everything look OK at the detector? Is there room in the waste reservoir for the mobile phase from the next batch of samples? This simple five-minute survey of the LC system often will identify problems before they become serious. They also will make you very familiar with the normal performance of the system so that you will be more likely to spot a problem when one occurs.

Specific Places to Look

Anywhere a high- or low-pressure connection is made is a good candidate for a leak. Connections that are made and broken regularly, such as during installation or removal of a column, are usually the first place to look. Otherwise, check all the low-pressure fittings on the upstream side of the pump. Because air can leak into a gap too small to allow liquid out, sometimes tightening each low-pressure fitting with your fingers is an effective search technique to find a loose fitting. At the pump, look around the check valves, especially if you remove them regularly for service. The drain hole below the pump heads between the inlet check valve and the pump body can drip, indicating a failed pump seal. The seal between the sample injection needle and the injection valve is a common wear point that eventually will leak and need adjustment or replacement. Because the detector is operated at a lower pressure than the rest of the system, detector leaks are less common. But if you use a narrow-bore waste tube to generate backpressure so as to avoid bubbles in the flow cell, high flow rates will generate higher-than-normal pressures, which can cause the detector flow cell or connections to leak.

Most of the time, a leak can be stopped by tightening a fitting or component. Low-pressure fitting nuts often come with flat surfaces that invite the use of a wrench. Don't! I recommend against tightening any low-pressure fitting with anything other than your fingers.

Fixing Leaks

Most of the time, a leak can be stopped by tightening a fitting or component. Low-pressure fitting nuts often come with flat surfaces that invite the use of a wrench. Don't! I recommend against tightening any low-pressure fitting with anything other than your fingers. It is too easy to over-tighten these plastic fittings. Over-tightening can distort the fitting and cause more serious leaks, or in extreme cases can strip the threads. And we all know that Murphy's law reminds us that if a $5 Delrin nut is threaded into a $1000 proportioning manifold, the threads in the manifold will be damaged from over-tightening, not those of the nut!

Leaks at stainless steel fittings usually can be stopped with a one-fourth-turn tightening of the nut. If this does not correct the problem, disassemble the fitting, rinse it out with a little water or solvent, and try again. If it still leaks, replace the ferrule with a new one. Leaky PEEK (polyetherether ketone) fittings require a little more care. First turn off the pump, then loosen the PEEK nut, push the tubing to the bottom of the fitting body, and retighten the nut. If PEEK fittings are tightened with the pump flow on, it is possible for the tubing to slip out of the fitting because the PEEK ferrule does not grip the tubing as tightly as the stainless steel ones and is designed to move in such situations. The tube might not slip all the way out of the fitting, but rather slip a millimeter or two, leaving a small gap in the fitting that will add to extra-column volume and cause peak broadening or poor peak shape.

A loose check valve that shows external leakage can be treated as the stainless steel fittings earlier. Just tighten it a bit to see if the leak will stop. If it does not, remove the check valve and examine the bottom of the fitting port where the check valve seals against the pump head. The seal usually is made with a hard plastic piece that sometimes can crack if tightened too tightly. If this is the case, replace the seal and reinstall the check valve. When tightening check valves, be sure to support the pump or pump head so that it does not twist.

A leak at the drain hole behind the check valves indicates that the pump seal is worn out. Follow the pump seal replacement procedure in the pump operator's manual. Usually this involves removing the pump-head retaining nuts and gently pulling the pump head from the pump. The old seal is removed with a pump seal removal tool, if supplied. If no tool is supplied, use a plastic probe to pry out the seal. Another good seal-removal tool is a brass wood screw threaded into the seal to pull it out, much like a corkscrew operates. Be especially careful during this process not to scratch the pump head, so prying out the seal with a pocket screwdriver is asking for trouble. Rinse all the parts generously with alcohol and wipe off the piston. If any buffer residue remains on the piston, rub the piston with a little toothpaste to remove it, and rinse again (avoid fluoride-containing toothpaste if you are doing ion chromatography). Examine the piston for any chips or scratches. This can be facilitated by holding a laser pointer against the end of the sapphire piston, causing it to light-pipe, highlighting any imperfections in the surface. Replace the piston if any damage is observed. Install the new seal and lubricate the seal and piston with a squirt of alcohol before sliding the parts back together. Be careful to tighten the head-retaining screws evenly and do not over-tighten them.

Drips at the injection valve that are not corrected by tightening or replacing the fittings might be due to a worn out injection rotor seal. Rotor seals are designed to last for 100,000 cycles or more, so failure can be a rare event, but it will occur eventually. Service the injector according to the manufacturer's instructions. Usually this involves disassembling the injector, rinsing it thoroughly, installing a new rotor, and reassembling the valve.

Worn pump seals and injector rotors often result in the shedding of particulate matter, which can cause frit blockage downstream, such as at the head of the column. For this reason, it is a good idea to purge the pump and autosampler thoroughly following seal replacement before reconnecting the column. For example, pump 25–50 mL of solvent through the system at a high flow rate to remove any residual particles. Then as a safety measure, install a 0.5-μm porosity in-line filter just downstream from the autosampler. This will catch particles that might otherwise block the frit at the inlet of the column.

Leaks at the fittings at the ends of the column usually can be fixed by tightening them as described for the other high-pressure fittings mentioned earlier. If the column hardware itself is leaking, you can try tightening it up, but in my experience, if the column leaks, it is time to replace it.

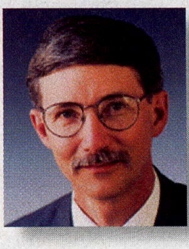
It might or might not be possible for you to fix a leaky detector cell. UV detector cells most commonly comprise a block of stainless steel with a hole drilled through it. Quartz windows are attached to each end and sealed with a polymeric seal. Sometimes the design is such that user servicing is possible; in other cases, factory service or replacement with a new cell might be required. Consult the detector manual for instructions.

Summary

Fixing leaks in the LC system is one of the less exciting, mundane tasks that seems to be part of every chromatographer's life. As might be expected, leaks are more common when the system parts are removed and replaced regularly, such as in a system that is used for many different methods, each of which requires a column change. Systems dedicated to one method often will be more trouble-free. There's nothing magic about identifying where the leak occurs — use your eyes, the system alarms, and the thermal-paper probe to help you locate the problem. Most of the time a leak can be stopped by a little more tightening of a fitting and you should be back in business.

For more information on this topic, please visit www.chromatographyonline.com/dolan

John W. Dolan



"LC Troubleshooting" Editor John W. Dolan is Vice-President of LC Resources, Walnut Creek, California; and a member of LCGC's editorial advisory board.

Direct correspondence about this column to "LC Troubleshooting," LCGC, Woodbridge Corporate Plaza, 485 Route 1 South, Building F, First Floor, Iselin, NJ 08830, e-mail John.Dolan@LCResources.com.

For an ongoing discussion of LC troubleshooting with John Dolan and other chromatographers, visit the Chromatography Forum discussion group at http://www.chromforum.com.