LC Troubleshooting

Pump Seal Problems

John W. Dolan

A reader complains of leaks and short pump seal life.

Recently, I received a letter about pump seal problems:

Question: I am having a problem with extremely short pump seal life in my liquid chromatography (LC) system. The reversed-phase method uses a simple mobile phase consisting of 35% acetonitrile and 65% 50 mM phosphate buffer (pH 3.0) pumped at 1 mL/min. We typically spend the day preparing samples, then load the autosampler and let the method run overnight. When the sample set is finished — usually in 8–10 h — the system automatically shuts off. When we restart the system the next day, invariably the pump seals leak. What are we doing wrong, and how can we correct this problem?

John W. Dolan: A pump seal certainly should last longer than 8–10 h. The shortest seal lifetime I ever encountered in my work was 4–5 days, and that was using 100 mM buffer and operating 24 h/day. Your conditions do not appear to be excessively demanding. Let's look at the possible causes.

POSSIBLE CAUSES

Commonly, rapid pump seal wear results from buffers or salts crystallizing on the pump piston when the pump is out of service. When the pump is turned on again, the buffer crystals abrade the sealing surface for a few seconds until they redissolve. This failure is most common with mobile-phase salt concentrations greater than 100 mM. To remedy this problem, flush the salts from the system before turning off the pump, as described below.

Another source of problems is installing the seal backward. The open side of the seal must face the liquid end of the pump (see Figure 1). Many pumps use a seal with a flange on the back edge, so you can install the seal only in one direction. Some pumps use a flangeless seal (as the pump in Figure 1), and users can accidentally install these seals backward. I have seen cases in which new, backward-installed seals worked properly at low pressures (for example, less than 1000 psi) but leaked after a few hours of use at higher pressures. Figure 1 shows that when the open end of the seal faces the liquid, the increasing liquid pressure will force the seal onto the piston, effectively sealing it. On the other hand, a reversed seal would be pushed off the piston as the pressure increased.

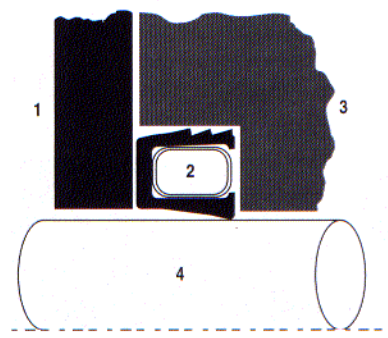

FIGURE 1: Cross section of a pump seal mounted in a pump head. 1 = backup washer, 2 = pump seal, 3 = pump head, 4 = piston. (Reprinted with permission from reference 1.)

A third cause is using solvents that are incompatible with the pump seal material. Most pumps come equipped with a seal made of a polymeric material filled with graphite or glass fiber. These universal seals can be used with most, if not all, LC solvents. However, some companies manufacture seals that are incompatible with certain LC solvents. I once used a system with a seal that exhibited extended life when used with water–acetonitrile and water–methanol mobile phases but was unstable in tetrahydrofuran. After an extended troubleshooting session, we found the source of system contamination — the seal was dissolving in the mobile phase. So it is wise to double-check the solvent compatibility of your pump seals and match them to your application.

A damaged piston also could shorten pump seal life. If the piston surface is scratched or otherwise damaged, it can quickly abrade the seal. A scratched piston can allow mobile phase to leak under the lip of the seal, resulting in a premature buildup of salt crystals behind the seal. These salt crystals abrade the seal as described above. You should inspect the piston each time you change the seal. A simple way to do this is to hold a small flashlight to the end of the piston. The glow of the light in the piston will allow you to identify any imperfections on the piston surface. Pistons typically are made of sapphire, which should be visibly smooth. Scratches appear as longitudinal lines on the piston. A scratched piston should be replaced. If you do not have a spare piston and you need to put the pump back into service, replace the seal and continue to use the piston but order a new one for the next maintenance cycle. A scratched piston will result in premature seal wear but seldom hinders pump operation.

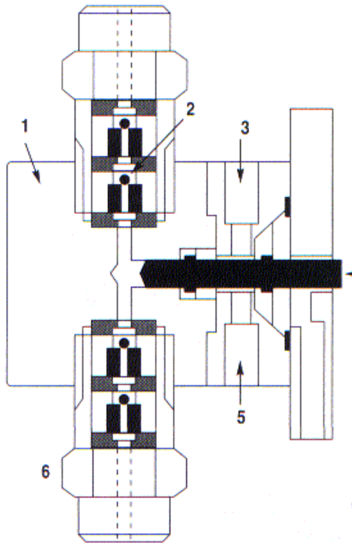

FIGURE 2: Cross section of a pump that enables users to flush behind the pump seal. 1 = pump head, 2 = outlet check valve, 3 = inlet to flushing channel, 4 = piston, 5 = drain from flushing channel, 6 = inlet check valve. (Reprinted with permission from reference 1.)

REPLACING SEALS

One key to maximizing pump seal life is to install the seals meticulously. First, review the manufacturer's suggested pump seal replacement procedure outlined in the pump operation and service manual. For most pumps, you first must remove all tubing connections from the pump head. You may need to remove the check valves. If there is any possibility of confusion about the direction of flow through the pump head, mark an arrow on the pump head indicating the flow direction so you won't reinstall the pump head upside down.

You should remove the pump seal using a wood screw of sufficient diameter to thread into the seal. Use the screw like a corkscrew to grip and remove the seal. You can pry out seals with spatulas or screwdrivers, but this practice risks scratching the inside of the pump head. Discard the old seal promptly so you don't confuse it with a new one during reassembly. Rinse the pump head with water, solvents, or both to remove any buffer residue. If you use sonication to clean the pump head, remove the check valves first (be sure to mark the inlet and outlet check valves to avoid confusion).

The piston should be cleaned to remove any residue of buffer salts or seal debris. Generally you can use a squirt bottle to rinse the piston with water, alcohol, or acetone; you should wipe the piston with a lint-free tissue. Sometimes the seal will leave a residue that cannot be rinsed off easily; you can remove it with toothpaste. Squeeze a small amount of toothpaste on the problem area and rub with a soft cloth or tissue. The toothpaste contains enough abrasive to remove stubborn deposits but not enough to scratch the piston. Rinse the piston thoroughly with water and inspect it as described earlier.

Place a new pump seal in the pump head and make sure it faces the correct direction (open edge toward the liquid end of the pump). Some pumps require a special tool for seal installation. Before reassembly, squirt a few drops of alcohol on the seal and piston to lubricate it. Next, turn the pump on and stop it when the piston is fully retracted into the pump head. I have found that I am less likely to break the piston during assembly if it is fully retracted.

After the head is in place, tighten it to the manufacturer's specifications and reinstall the inlet tubing. Run the pump for a few cycles to purge any air from the head before connecting the outlet tubing.

MINIMIZING PROBLEMS

You can take at least three steps to minimize pump seal problems and extend pump seal life. First, avoid conditions that promote pump seal wear such as high buffer concentrations — most methods will work satisfactorily with 25–50 mM buffer. I try to avoid buffer concentrations in excess of 100 mM unless I have evidence that these levels are necessary. Salts in the mobile phase can cause similar problems. High buffer concentrations are more susceptible to precipitation, especially when acetonitrile is used. On-line mixing can aggravate buffer solubility problems because buffers that are readily soluble in intermediate concentrations of acetonitrile can precipitate if they encounter 100% acetonitrile at the point of mixing. High buffer concentrations also will cause more rapid accumulation of deposits behind the pump seals when the volatile mobile-phase components evaporate.

A second step to maximize pump seal life is to remove buffers from the system when it is not in use. First, replace the buffered mobile phase with nonbuffered mobile phase — 65% acetonitrile–35% water in your case — and wash the system with approximately 20 mL of this buffer-free mobile phase. As with any flushing or equilibration step, it is the volume, not the time, that is important, so you can increase the flow rate if the pressure is reasonable. After the initial wash, you can flush the column with strong solvent to remove any strongly retained materials. As an added measure, I like to increase the flow rate so the pressure is higher than normal, which ensures that clean mobile phase leaks under the seal to remove or dilute any buffer residues behind the seal. You could program a buffer-free mobile-phase flush at the end of your series of runs before shutting off the pump.

Some pumps allow users to flush behind the pump seal to remove buffer residues. Figure 2 illustrates a pump with a passage that directs the flush solvent to the back side of the piston seal. (Note the two pump seals in Figure 2: the normal seal on the left side of the flushing channel and the backup seal on the right side that prevents liquid from entering the pump mechanism.) Generally, flushing approximately 10 mL of water through the flushing passage will remove any buffer residue. It is a good practice to follow this flush with a 10-mL alcohol flush to remove the water. This technique is especially useful with ion-exchange methods or applications using salt or buffer concentrations in excess of 100 mM.

A final technique to reduce pump seal wear is to keep the pump running at a low flow rate. As long as the piston cycles fast enough to keep volatile solvent components from evaporating and leaving salt crystals behind the pump seal, wear should be reduced. Some laboratories leave their LC systems running at 0.1 mL/min when they are not in use. With isocratic methods, the detector outlet can be directed back into the reservoir to eliminate solvent consumption.

AND FINALLY . . .

The last step in changing pump seals (or performing any instrument maintenance, for that matter) is to order replacement parts. Be sure to order the proper parts for your pump and your application. As stated earlier, some manufacturers provide pump seals with optimal performance in certain mobile-phase solvents, so match the seals to your application. As long as you are ordering parts, be sure you have a spare set of check valves. Generally, seals and check valves are the only pump parts that fail on a regular basis. Keeping a spare piston on hand usually is unnecessary unless downtime is critical and piston breakage is likely; a scratched piston usually will provide satisfactory service until the next seal replacement cycle.

If you institute the various cleaning and preventive maintenance measures described above and still have seal problems, you may have a problem with piston–seal alignment. Most of today's pumps have a floating piston that self-aligns when the head is assembled. In the early days of LC, the pistons were glued to the cam follower in the pump, forming a rigid assembly. This design was highly susceptible to misalignment and premature wear. If your pump has the floating piston design, be sure that the piston has some lateral movement — it could be frozen into place with buffer deposits. If your piston has fixed alignment, you may need to request a service call or a factory rebuild to correct the problem.

REFERENCE

(1) J.W. Dolan and L.R. Snyder, *Troubleshooting LC Systems* (Humana, Clifton, New Jersey, 1989).

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