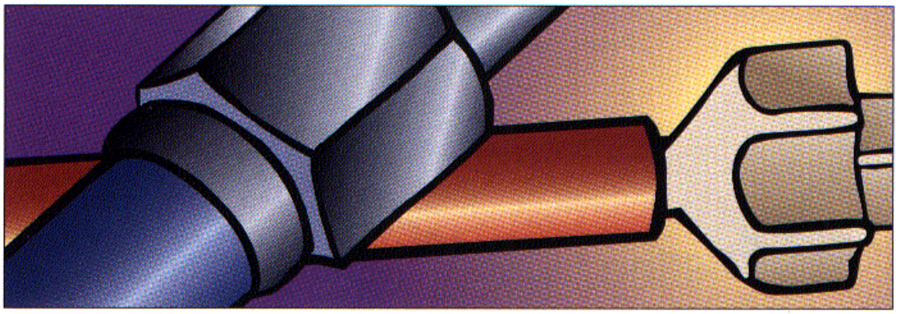
LC Troubleshooting



Pump Preventive Maintenance

John W. Dolan

Routine maintenance will extend the life of your pump, a vital module in any liquid chromatography system.

Proper pump operation is essential for obtaining optimum performance from liquid chromatography (LC) systems. The pump delivers mobile phase at a precise flow rate, which in turn determines the precision of retention times and the confidence in peak identification. In many systems, the pump also blends the mobile-phase components to the exact mixture required for the LC method. This month's "LC Troubleshooting" covers the basics of pump operation and the key elements of preventive maintenance. With regular preventive maintenance, your LC pump will provide you with many years of satisfactory service.

CENTRAL DESIGN

All commercial LC pumps are based on some form of the reciprocating piston pump, which is diagrammed in Figure 1. The essential parts are a motor, a mechanism to translate the rotary motion of the motor into a reciprocating action to drive the piston, a pair of check valves, and a pump seal. Manufacturers sell many variations on this basic design, but all have these key features in one form or another. The most popular variations are dual-piston pumps in which two parallel pistons work alternatingly. Another style uses two pistons, with one piston feeding the other in a tandem flow path. A third common style has a metal diaphragm and pumps oil against the diaphragm rather than having the piston make direct contact with the mobile phase.

In the generic pump depicted in Figure 1, the piston slides back and forth in the pump head as the motor shaft rotates. The pump seal keeps liquid from leaking around the piston and enables the pump to generate substantial pressures inside the pump head. When the piston retracts, it creates a low-pressure region inside the pump head. This low-pressure region allows the outlet check valve to close; a slight solvent pressure on the inlet check valve causes it to open so that solvent flows into the pump head. When the piston reverses and slides into the pump head, this solvent is pressurized. As the pressure increases, the inlet check valve settles on its seat and the pressure continues to grow until it exceeds the system pressure. At this point, the outlet check valve opens and solvent flows to the rest of the system.

Check valves — the weak link: The check valves are the most failure-prone pump parts. The check valves typically comprise a ruby ball and sapphire seat. The seal is much like a tennis ball sitting on a bagel — the actual seal is a very narrow ring where contact is made. Any bit of dust or contamination that gets in this sealing area will cause the check valve to leak. This susceptibility to valve contamination and leakage is why chromatographers should keep mobile phases free of particulates. Filtering the mobile phase through a 0.5-µm or finer membrane filter is necessary if you want long-term check valve reliability.

The seal — not really: The pump seal is another delicate pump part. Figure 2 shows a cross-sectional diagram of a pump head and seal. The sealing lip of the seal touches the piston only in a narrow ring. This seal will hold at pressures as high as 10,000 psi in some cases, but a little damage will make it leak. In fact, it always leaks to a certain extent, which enables the mobile phase to lubricate the piston. This lubrication allows the seal to slide smoothly on the piston for longer life. The piston typically is sapphire, and the seal is a polymeric material such as Kel-F, polyethylene, or another chemically inert polymer.

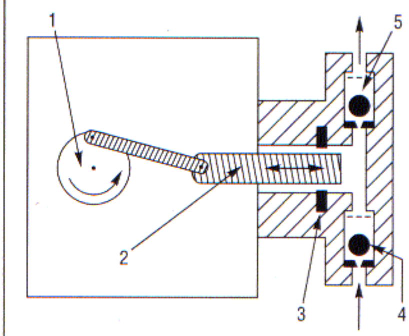


FIGURE 1: Schematic diagram of a generic LC pump. 1 = motor cam, 2 = piston, 3 = pump seal, 4 = inlet check valve, 5 = outlet check valve. (Reprinted courtesy of LC Resources Inc. [Walnut Creek, California])

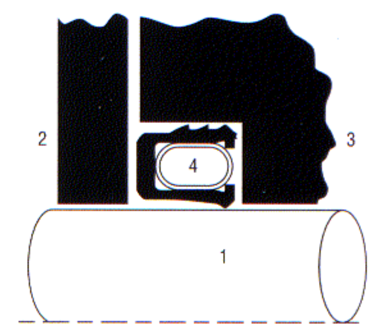


FIGURE 2: Cross section of a pump head. 1 = piston, 2 = pump body, 3 = pump head, 4 = pump seal. (Reprinted courtesy of Bal Seal Engineering Co., Inc. [Santa Ana, California])

Many of today's pumps come equipped with a second seal or diaphragm mounted behind the main seal, such as the one shown in Figure 3. This design allows you to flush buffers or salts from behind the main seal to remove these corrosive substances and reduce maintenance requirements.

THE PROBLEM AREAS

Let's take a look at the key problem areas for our generic pump, how the problems originate, and how to correct or prevent them. Be-

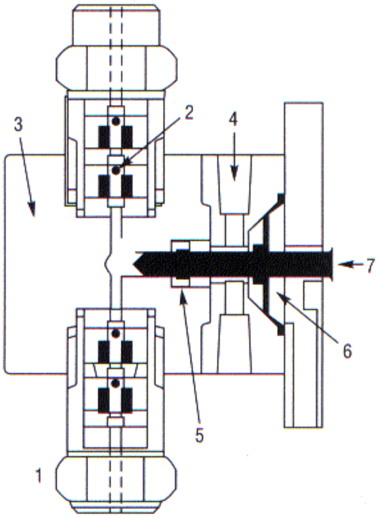


FIGURE 3: Schematic showing a flushing passage behind the main pump seal. 1 = inlet check valve, 2 = ball in outlet check valve, 3 = pump head, 4 = flushing passage, 5 = main seal, 6 = flushing seal, 7 = piston. (Reprinted courtesy of reference 1.)

TWO SIMPLE TESTS

Try these two simple tests to check your LC pump's performance. My laboratory includes the tests in the quarterly standard operating procedure for LC system performance and uses them to verify that check valve and pump seal replacements were successful.

Pressure decay: Set the upper pressure limit to approximately 1000 psi higher than the normal operating pressure. I normally run the pumps at approximately 2000 psi, so I set the limit to 3000 psi. Block the pump outlet with a compression-fitting plug or cap. Turn on the pump and allow it to reach the upper pressure limit and then shut off the flow and watch the pressure drop off.

A properly performing pump should lose less than 15% of its pressure within 10 min after being shut off. For example, if the pump is shut off at 3000 psi, the pressure should be no lower than 2550 psi after 10 min.

Problems? Check for leaks, tighten all the check valves and fittings, and try again. Don't overtighten the fittings because they will break or distort. If tightening doesn't help, remove the fitting, rinse the seat, and reassemble it. Don't forget that a faulty pump seal also can cause excessive pressure bleed.

Flow check: After completing the pressure decay test you'll know if the pump is leak-free and the check valves and pump seals are working properly. The real test of a pump, however, is how accurately it will deliver solvent. Connect a column to the system and time how long it takes to collect 10 mL of effluent in a volumetric flask at a flow rate of 1 mL/min. Although most pumps specify delivery to ±0.1%, we accept ±0.2% or 10 min ± 12 s for a 10-mL collection.

cause each pump model is a little different, you'll want to look in your operator's manual to see how this discussion applies to your pump.

Most pump parts succumb to three types of failure. Every part will fail eventually through normal wear and tear, which is unavoidable, but often can be delayed by good maintenance practices. A second mode of failure is accelerated wear resulting from poor laboratory practices. For the most part, careful users can convert this type of failure to the first type. The final failure mode is catastrophic failure in which a part suddenly fails. This failure may result from normal processes of materials aging, operator error, or unexplained causes. Once again, you can convert much of this type of failure to the first type.

Next, let's look at three key areas of the pump, their failure characteristics, and some maintenance tips.

Driving cam: Most pumps use a cam-and-follower mechanism to transfer the rotary motion of the pump into the reciprocating action of the piston. The cam has a computer-designed shape that helps minimize flow and pressure pulses for the particular pump design. Little can go wrong with the cam as long as the contact surfaces are lubricated.

Many pumps include a small sponge that continuously rubs oil on the cam to keep it lubricated. Some designs use heavy grease on the surfaces, and others immerse the working surfaces in an oil-filled sump. Because this connection is inside the pump cabinet and inconvenient to inspect, users often ignore it until problems occur.

Excessive wear resulting from poor lubrication may require factory service. Once when the cam of a pump in my laboratory went dry, the piston vibrated as it moved back and forth emitting a disconcerting audible chatter and excessive pressure fluctuations. I fixed the problem by placing a few drops of light oil on the lubricating sponge. After consulting the instrument manual, I discovered that the sponge should have been lubricated once a year with a few drops of oil. Now lubrication is part of our standard operating procedure for yearly maintenance on each LC system. Consult your pumps' manuals for specific lubrication recommendations.

Piston and pump seal: As mentioned earlier, the pump seal is less than 100% effective, so a little mobile phase leaks under the seal to lubricate the piston. This leakage is acceptable until the pump is turned off. If the mobile phase comprises HPLC-grade solvents, the solvents evaporate, leaving a dry piston that becomes rewetted when the pump restarts. If the mobile phase contains buffers or salts, they leave behind a crystalline residue. This residue is very abrasive and tears at the pump seal for the first few piston strokes the next time the pump is used. When the piston is rewetted, the residue dissolves, and the pump works normally.

This buffer abrasion is the primary cause of pump seal wear, so avoiding this problem is one of the best ways to extend pump seal life. I recommend flushing the system with nonbuffered mobile phase before shutdown. For example, if the mobile phase was 45:55 (v/v) buffer–methanol, flush the systems with 45:55 (v/v) water–methanol. Allow approximately 10 mL of solvent to pass through the pump. Then if you want to flush the column, switch to a strong solvent such as methanol. I don't recommend switching directly from buffer to 100% strong solvent because of the high probability of precipitating the buffer, especially if you use acetonitrile as the organic solvent. An additional method of ensuring that the piston is rinsed is to increase the flow rate and thus the pressure, so that clean solvent is forced under the seal.

If your pump has a flushing passage behind the main pump seal, you can remove any salt residues by flushing a syringe full of water through the passage. This flushing is especially useful for ion-exchange applications, hydrophobic interaction chromatography, and other methods that use mobile-phase salts and buffers with concentrations of greater than 100 mM. Be sure to use a flushing solvent that is compatible with all exposed pump parts.

Once I was surprised to find that the flushing seal in one of my laboratory's pumps was incompatible with isopropanol; when I flushed the pump with isopropanol the seal deteriorated. The resulting mess was a bother to clean up. Since then my laboratory replaces all these seals with new PTFE ones.

Pump seals wear out, but they will last for many months with regular flushing. Use the pressure test (see the accompanying sidebar) to test the integrity of your pump seals. If you can determine how long your pump seals last, set up a maintenance program to replace the seals at 75–80% of their expected life. When seals fail, they shed small particles of seal material, which can foul check valves and column frits. The trick is to replace the seals before they get to this state. In any event, the pump seals should be replaced once a year, so add this procedure to your standard operating procedure for yearly pump maintenance.

When removing an old pump seal, be careful not to scratch the pump head. Some pump manufacturers provide a tool for seal removal and insertion. A wood screw used as a cork screw is another good alternative for seal removal. If you use a small screwdriver or spatula to pry out the seal, you likely will damage the pump head.

If you prevent buffers from drying behind the pump seals, pump pistons should last indefinitely. When replacing the pump seal, wipe the piston with a damp wiper to remove any buffer residue. If the residue does not wipe off easily, use a little toothpaste to remove the deposit — toothpaste is abrasive enough to remove deposits but will not scratch the piston.

Abrasive buffer residues can scratch the piston surface, causing longitudinal lines that you can see by holding a flashlight up to the end of an exposed piston. The piston will glow in the light, and you can see any scratches in the normally smooth surface. A scratched piston should be replaced because it will allow more mobile phase to leak under the seal than normal. Pistons most commonly are broken during pump head assembly. You can minimize this problem by adjusting the pump so that the piston is fully retracted before assembly and wetting the piston and seal with a few drops of methanol.

Piston breakage during normal operation is extremely rare because most pumps use a self-aligning mechanism to ensure that the piston is centered properly. I recently encountered a broken piston under normal circumstances, but it was the first case that I can remember in my 25 years of LC experience. The symptom was obvious in hindsight but perplexing at the time. The dual-piston pump delivered roughly half the flow rate and pressure, and it was unresponsive to normal purging or check valve replacement. When I used a syringe to pressure prime the pump, I saw that only one pump head was accepting solvent. Next, I discovered a broken piston.

Check valves: The check valves are the most problematic portion of the pump. Because all the mobile phase must pass through the check valves, any particulate matter in the mobile phase is likely to cause check valve problems. The key to check valve longevity is keeping them clean. Use filtered mobile phases and rinse the buffers from the system at the end of the day. Be sure to leave the system in sufficient organic solvent to prevent the growth of microorganisms when it is not in use. Leaving a system in an aqueous buffer is just asking for microbial growth and buffer precipitation.

Some workers like to remove check valves and sonicate them periodically in methanol. Although this practice is unlikely to cause any harm, I don't have any good evidence that it is an effective preventive maintenance technique. My philosophy about check valves is to leave them alone if they are working — you are just as likely to create problems as to prevent them.

Check valve failure shows different symptoms depending on the check valve and the system design. Pressure pulses that correspond to the cycle of one piston represent the most common symptom. Purge the pump well to ensure that the pressure problem is not the result of a trapped bubble. If this purging fails to fix the problem, the most expedient diagnostic tool is to systematically replace the check valves one at a time until you locate the problem valve. Don't forget to reinstall the good valves when you finish.

Some manufacturers and aftermarket parts suppliers make cartridge-type check valves that allow you to change the ball-and-seat unit while reusing the threaded portion of the check valve. These parts save a bit of money, and some designs allow you to reduce inventory by using the same cartridge for inlet and outlet valves and use the same cartridge for different models or brands of pumps. Because substitution is the most convenient way to isolate check valve problems, be sure to keep at least one set of inlet and outlet check valves on hand for each model of pump in your laboratory.

DEGASSING — THE BEST MEDICINE

The most common pump problem is unrelated to hardware failure; it is air bubbles trapped in the pump. Fortunately, this problem also is the easiest problem to prevent. Although some pumps will work without degassing the mobile phase, every system will work more reliably if you degas the mobile phase. Helium sparging is the most effective way to remove excess dissolved air, but other techniques such as vacuum, membrane, and sonication degassing may be satisfactory for some systems. The key to degassing is removing enough gas that the system works reliably, and some systems are more tolerant of dissolved gas than others.

IN CONCLUSION

The user-serviceable parts of the LC pump are quite simple. You can find specific directions in the instruction manuals that come with your pump. Follow the guidelines discussed here — modifying and adding steps as necessary for your specific LC pump — and you will obtain many years of satisfactory performance from your pump. Although a 6–7 year-old pump generally is considered geriatric for an LC system, many laboratories still rely on LC pumps that have been in continual use for 20 years or more. Most changes in LC pumps during that time have been in electronics and control — not in the physical pumping mechanism.

REFERENCE

(1) J.W. Dolan and L.R. Snyder, *Troubleshooting LC Systems* (Humana Press, Clifton, New Jersey, 1989), p. 188.

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