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

Troubleshooting Pump Problems

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It has been several years since "LC Troubleshooting" has focused on pumps (1,2). Although the basic principles of pump operation, maintenance, and troubleshooting have changed very little, new pump models have been introduced in the interim, and the pump remains one of the most error-prone components of the LC system. Therefore, it is useful to review this topic again.

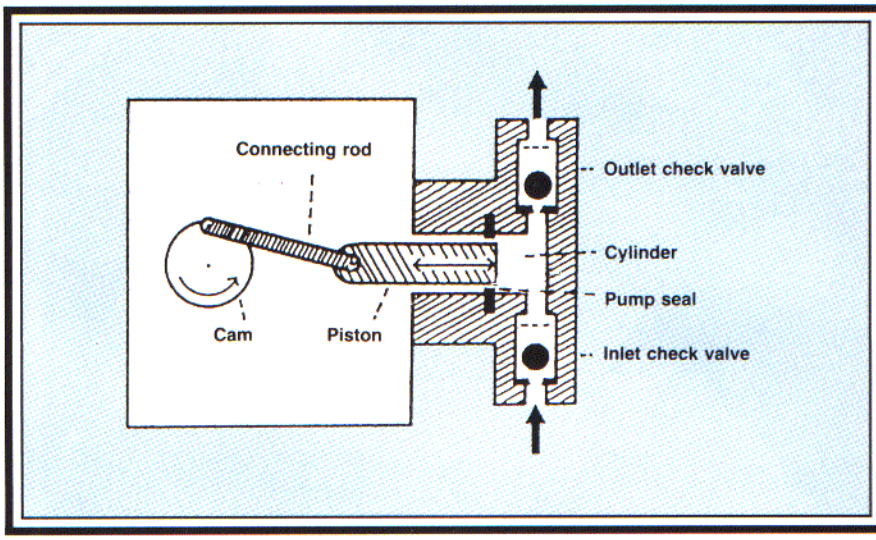

FIGURE 1: Schematic showing the common pump parts of reciprocating piston pumps. (Reprinted from reference 3 with permission.)

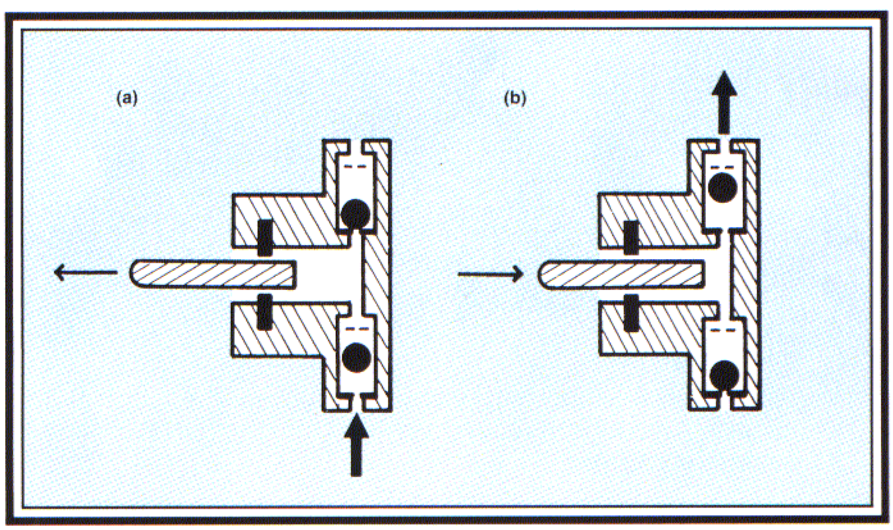

FIGURE 2: Check valve action. (a) Intake or fill stroke. Piston withdraws, opening inlet check valve. (b) Delivery stroke. Piston pressurizes mobile phase, forcing inlet check valve closed and outlet check valve open. (Reprinted from reference 3 with permission.)

ERROR-PRONE BY DESIGN

In the typical LC system, the pump contains the most moving parts, with the possible exception of certain autosamplers. This mechanical complexity inevitably makes the pump subject to problems. Although each manufacturer has specific design features that distinguish its pump from others, most pumps have the common parts illustrated in Figure 1. In operation, a motor turns a cam, which, by a connecting rod, converts the rotary motion of the motor into reciprocal motion so that the piston moves back and forth in the pump head. An inlet and outlet check valve plus a pump seal make pumping a liquid possible, as illustrated in Figure 2. Figure 2a shows the *fill* stroke of the pump: The piston withdraws from the cylinder, lifting the ball of the inlet check valve from the seat and creating a suction, thereby drawing mobile phase into the pump head. On the *delivery* stroke (Figure 2b), the piston moves into the cylinder, forcing the inlet check valve to seal against its seat and opening the outlet check valve. Mobile phase is then pumped into the system.

Commercial pumps may vary greatly from the stylized pump of Figures 1 and 2. Pistons commonly are driven directly by a shaped cam, which varies the piston speed during different portions of the pumping cycle in order to produce more even flow and fewer pressure pulses. The number and design of check valves vary from manufacturer to manufacturer. Finally, electronic control of the motor helps to improve the uniformity of pump delivery. However, this rather simplified description of pump operation is sufficient for understanding general pump troubleshooting procedures.

ENEMY #1: AIR

Air bubbles in the pump cause most of the problems with LC pumps. Air comes from three primary sources. The most obvious occurs when a mobile phase reservoir is pumped dry or the solvent inlet line is lifted above the level of the mobile phase while the pump is operating. This causes air to be drawn into the pump. A more insidious source of air is from leaks at the fittings that connect the inlet tubing to the pump. And the most common source of air is from bubbles generated when the mobile phase components are mixed or from cavitation of the mobile phase in the inlet line or pump head. Let's look at each of these sources and how to prevent problems that can result from them.

In each case of air in the pump, the symptom is either the pump's inability to pump or fluctuating pressure. Because most workers use pumps with more than one piston or multiple pumps in one LC system, total flow stoppage is rare. More commonly, you will see a dip in system pressure corresponding to the delivery cycle of the pump head that is at fault. You see this pressure dip because the fill stroke causes the bubble to expand, and the delivery stroke compresses it; in both cases, the pressure differential is low enough that the check valves don't operate, or don't operate properly. When the bubble leaves the pump, the pressure returns to normal.

Preventing the air that is in the reservoir from reaching the pump is a simple fix: Just attach an inlet-line frit ("sinker frit") to the end of the inlet tubing to keep it at the bottom of the reservoir. If you work with biological samples that cannot tolerate the presence of stainless steel components, you can use a "biocompatible" inlet-line frit made of plastic. Unfortunately, these frits are not heavy enough to hold the inlet line in the bottom of the reservoir, but you can usually keep the tubing submerged by fastening the tubing at the mouth of the reservoir with a piece of Parafilm or a tight-fitting cap.

Air leaks on the inlet side of the pump can be difficult to track down. Carefully tighten each of the fittings (including the check valve) on the inlet side of the pump, and see if the problem goes away. Be especially careful when tightening the plastic fittings — it is easy to strip or distort them. If the problem does not go away, even when degassed mobile phase is pumped, you will need to disassemble the fittings and examine them for damage. Again, the plastic fittings are usually the problem. Most manufacturers ship their systems with the flared-style (Cheminert) fittings for the low-pressure plastic tubing. Overtightening one of these fittings will cause the flare to distort, which can allow air to leak in or solvent to leak out. Remake any suspect tube end either by cutting off a portion of tubing and reflaring it or by replacing the fitting with another style of low-pressure fitting (these generally are interchangeable among manufacturers). If a compression fitting is suspect, clean and reassemble the fitting or cut the old ferrule off the tubing and remake the end with a new ferrule (be sure to use the same brand as the one you removed).

Air also can leak when a proportioning valve diaphragm becomes perforated or other damage to the solvent proportioning manifold occurs. Because proportioning valve leaks are hard to pinpoint, it is best to bypass the proportioning manifold by connecting the manifold inlet and outlet tubing with an appropriate union. If the bubble problems go away, replace the entire manifold; or if you are adventuresome, try your hand at further isolation of the problem. With complex parts such as these, eliminating the part from the system or replacing it with a known good part is a good way to isolate the problem source.

A rare and elusive air-leakage problem related to check valves can occur with certain pumps. Some manufacturers use a hard plastic washer to make the seal between the inlet check valve and the pump head. Overtightening the inlet check valve can crack this washer, causing air to leak in or fluid to leak out. If you suspect this, remove the inlet check valve and examine the seat for cracks (a small mirror can help you to do this without removing the pump head).

The last source of air in the LC pump is from outgassing of the mobile phase. When reversed-phase solvents are mixed (for example, methanol and water), the mixture has a lower capacity for dissolved gas than do the pure starting components. This is why you often see bubbles evolving from a freshly mixed mobile phase. When mixing is done by hand, the excess gas bubbles from solution, but the mobile phase remains air-saturated. Thus, when the pump starts the fill cycle, the pressure is reduced and gas once again evolves from solution, leaving bubbles in the pump head. When low-pressure mixing is used, the solvents are combined just before the pump, so the mobile phase entering the pump is supersaturated with air; it takes very little disturbance to cause this solution to outgas in the pump. With high-pressure mixing, the solvents are not mixed until after the pump, so bubble problems in the pump should not be encountered.

A final source of bubbles from the mobile phase is a phenomenon called "cavitation." This occurs when the pump tries to draw solvent through a line that has too great a flow restriction, creating a partial vacuum in the line. This vacuum causes any dissolved gas to boil out of solution and form bubbles in the inlet line or the pump head. The most common cause of cavitation problems is blockage of the inlet filter in the mobile phase reservoir. If cavitation is suspected, remove the inlet-line filter; if the problem goes away, a restricted filter is likely. Replace the filter with a new one and the problem should be solved. A tightly fitting reservoir cap that is not vented can also cause cavitation problems. To remedy this situation, loosen the cap or drill a small (≤ 1 mm) vent in the cap.

DEGASSING IS THE KEY

The best way to avoid bubble problems is to thoroughly degas the mobile phase. Helium sparging is the most effective and convenient degassing method. Use a commercial degassing setup or make your own from a piece of PTFE tubing and an extra inlet-line frit attached to a helium supply. Immerse the frit in the reservoir and vigorously bubble helium through the mobile phase for a few minutes (generally, 3–4 psi at the helium tank is sufficient). Then reduce the helium flow to a trickle and start using the LC system. In cases in which volatile mobile phase components might be lost, you can use a pressurized reservoir or a presaturation system, as described in an earlier "LC Troubleshooting" installment (4). Vacuum degassing can be used, but it is less effective than helium sparging. For a detailed discussion of solvent degassing, see references 5 and 6.

Degassing is recommended whenever reversed-phase methods are used. Generally, degassing is not required for normal-phase work. It has been assumed that degassing is not necessary when high-pressure mixing is employed, but problems due to poorly degassed mobile phase can still occur under certain conditions (4).

Degassing the mobile phase can prevent bubble problems from occurring, but it can also correct existing bubble problems. Bubbles in the LC system can be removed by pumping degassed mobile phase through it. The degassed mobile phase, having a higher capacity for air than nondegassed mobile phase, acts as a "sponge" as it passes through the system, redissolving bubbles that are lodged in various parts of the LC system.

ENEMY #2: DIRT

Although bubbles in the mobile phase cause the most frequent problems in the LC pumping system, dirt is the source of the most damaging problems. By "dirt," we generally mean particulate matter introduced by the mobile phase, with the sample, or as a result of buffer evaporation. In extreme cases, seal wear can also introduce particulate matter into the pump. Two main pump problems are observed when dirt is allowed in the system. The primary problem is malfunctioning check valves, and the second problem is premature pump seal wear. Let's look at each of these problems.

When a check valve fails because of particulate contamination, a speck of dirt gets lodged between the ball and the seat, preventing proper sealing. This results in pressure fluctuations and poor pump delivery. When high-pressure mixing is employed, a dirty check valve can create proportioning problems. Dirt in the check valves and pump head also can increase the chances of bubble problems because the particulates create microscopic rough spots that tend to collect bubbles. If you suspect that check valves are the problem and a cursory flushing does not solve the problem, it is best to confirm that you have a check valve problem by substituting known good check valves for the questionable ones. If the problem goes away, you know you had a check valve problem; if not, look elsewhere. It is not always possible to clean dirty check valves, so you may want to leave the good check valves in place and return the questionable ones to the manufacturer to be rebuilt. Often you can clean the check valve by removing it from the pump and flushing it with clean solvent. If this is not successful, try sonicating the check valve in dilute nitric acid (for example, 10% nitric acid in water); then rinse with HPLC-grade water. (Use extra care when working with acid.) If neither of these cleaning procedures is successful, replace the check valve with a new one.

The pump seal, by design, does not provide a 100% seal around the piston. If it did, the piston would not be able to move. The mobile phase wets the piston, lubricating it so that it can slide back and forth through the seal. As a result, the portion of the piston behind the seal is slightly damp when the pump is operating. When the pump is shut off, the mobile phase behind the seal evaporates. This is acceptable when the mobile phase contains only solvents, but when buffered mobile phases are used, they must be washed fect pump operation, samples that contain particulates or are cloudy should be filtered before analysis.

DRIP, DRIP, DRIP

Mobile phase leaks occur occasionally with all pumps, no matter how carefully you handle the fittings. Generally you can tighten the nut on the leaky fitting a little and eliminate the leak. Be careful not to overtighten the fittings, because overtightened fittings can leak. Furthermore, overtightening can permanently damage the part; this can be an expensive mistake if the fitting threads into the check valve or pump head. If tightening does not stop a leak, disassemble the fitting and reassemble it, replacing the nut and ferrule if necessary. When disassembling fittings anywhere in the LC system when buffers are in use, it is a good practice to flush the fitting with nonbuffered solvent before reassembly. This will prevent buffers from drying in the fitting, causing the ferrule to seize or the fitting to leak.

OTHER PREVENTIVE MAINTENANCE

Solvent filtration and degassing are the primary techniques to prevent pump problems. In the past, we have encouraged regular changing of the pump seals — for example, on a quarterly basis. This is an easy and rather inexpensive procedure that is wise in many cases. However, manufacturers are claiming extended seal lifetimes in the new pumping systems. For example, the manufacturer of the unit shown in Figure 3 advertises a five-year guarantee on the pump seals. Discussions with other manufacturers indicate that they are experiencing extended seal lifetimes as well. In reality, you probably will have to figure out how long your pump seals last in your operating environment. In general, it is a good idea to replace the seals when about 80% of their useful life is gone. In any event, replacing the seals at least once a year is probably a good practice. When you replace the seal, inspect the piston for scratches. Hold the piston so that a bright light reflects off the surface. Lines parallel to the piston axis indicate piston abrasion, and you should replace the piston if you see them.

Additional preventive maintenance depends on the specific model of pump that you have; consult the operators' manual for suggestions. Many pumps require occasional lubrication where the piston driver contacts the driving cam. Check for other points to lubricate.

SUMMARY

Common sense is the guide for minimizing problems with LC pumps. Degassing the mobile phase will keep bubbles out of the pump and will remove the occasional bubble that does get in. Filtering the mobile phase so that particulate matter does not enter the system will greatly improve check valve reliability. Flush the pump with nonbuffered mobile phase at the end of each day's work if buffers are used in the mobile phase. If high buffer concentrations are used, flush behind the pump seal daily to remove buffer residues and to extend seal life. The expected lifetime of pump seals is changing; it is a good idea to replace seals in older pumps every three months, but you might experience extended seal lifetimes with newer pumps.

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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.

Erratum: Please note that in the December 1988 installment of "LC Troubleshooting" (*LC•GC* **6**[12], 1052–1056), an error appears on p. 1054. The sentence following equation 4 should read: "Now you can use data such as those shown in Figure 2 along with equation 4 to make a relative resolution map such as that of Figure 3." The sentence as published erroneously refers to Figure 4 rather than to Figure 3.