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

Reciprocating Piston Pumps

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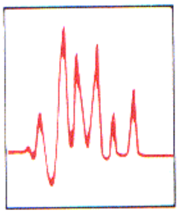

Pumps for liquid chromatography include reciprocating piston pumps, syringe pumps, diaphragm pumps, gas-pressure pumps, and a few others. Reciprocating piston pumps are by far the most common in use today. Troubleshooting for reciprocating pumps, whether they have one, two, or three pistons, is the subject of this month's column. Because there is some overlap in troubleshooting for the various classes of pumps, this column will be of interest to chromatographers who use nonreciprocating pumps as well. Troubleshooting other pump types will be discussed in future columns.

CLASSIFY THE PROBLEM

As was discussed in an earlier article (1) problems with pumps are usually detected by inspecting the chromatogram or the pressure monitor on the instrument. Problems fall into three categories: low flow or pressure, high flow or pressure, and erratic flow or pressure. Detection of pump problems by examination of the chromatogram requires retention-time measurements. If retention times are longer than normal, the pump may not be delivering enough mobile phase. Conversely, shorter than normal retention times may be the result of too much mobile phase being delivered by the pump. Finally, poor reproducibility of retention times may be the result of erratic mobile phase delivery. Problem diagnosis using a pressure monitor is straightforward. An abnormally high pressure corresponds to the delivery of too much mobile phase, an abnormally low pressure corresponds to insufficient mobile phase, and erratic pressure results from erratic mobile phase delivery.

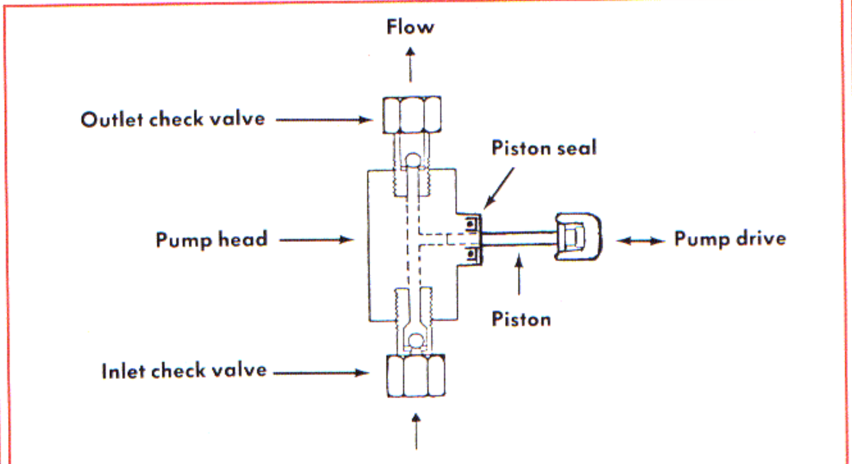

FIGURE 1: Liquid end of reciprocating piston pumps.

CHECK EXTERNAL CAUSES

The very first step to take once a problem with the pump is suspected is to check for the obvious reasons for failure. Check for sufficient mobile phase in the reservoir, use of the proper mobile phase, and check the flow rate and pressure-limit settings. If changes have recently been made in the system, then tubing, column, and other component replacements may be affecting the system's back pressure. Clearly, the reference chromatograms and daily records that you record in your logbook will help to isolate the source of the problem. We will now assume that you have eliminated any problem sources external to the pump itself.

CHECK FOR AIR ENTRAPMENT

By far the most common problem that occurs with reciprocating piston pumping systems is air entrapment in the liquid end of the pump. The liquid end is the portion of the pump that contacts the mobile phase and usually includes an inlet check valve, outlet check valve, piston, seal, and pump head. A typical example is shown in Figure 1. Small bubbles of air get caught on the machined edges of the head and check valves, which then act as nucleation sites. Air-bubble formation usually results from the creation of a slight vacuum within the pump head on the suction stroke of the pump. The conditions of low pressure and nucleation sites are ideal for bubble formation, especially if the mobile phase is at equilibrium with atmospheric air. The other cause of air bubbles is seen in LC systems that use prepump, low-pressure mixing of the mobile phase. In this case, the solubility of air in the mixture may be less than in the pure solvent components of the mobile phase; therefore, bubbles form upon mixing. Once a small bubble forms, it acts as a pulse dampener inside the pump head, dampening the effect of the suction and delivery strokes. The result is a low and often erratic flow from the pump head.

If bubble formation in the pump head is a regularly occurring problem with your pump, you should take measures to eliminate the source of the air. Some manufacturers have designed pumps that do not require degassed solvent, but most pumps operate in a more trouble-free manner if the solvent is degassed prior to use. Common methods of degassing include helium sparging, vacuum degassing, boiling, and ultrafiltration, either alone or in combination. Whichever method you choose, be sure to protect the solvent from reabsorbing atmospheric air. A loosely fitting cap or foil cover on the reservoir may be sufficient for most systems. Constant sparging may be necessary for demanding applications. Avoid tightly fitting bottle caps to prevent a vacuum from forming as solvent is pumped from the reservoir. Other sources of air leaking into the system are permeable tubing (thin-wall Teflon inlet lines, for example) and loose compression fittings. Air-leak problems are easily corrected by replacement or adjustment of these parts.

REMOVING AIR

You may be fortunate enough to be able to remove the air bubbles by opening the purge valve on the high-pressure side of the pump, increasing the flow rate, and waiting for a few minutes for the bubble to clear the system. More often, you will have to loosen the compression fitting on the high-pressure side of the outlet check valve, and in persistent cases you will need to loosen the check valve itself. Opening one of these points on the high-pressure side of the pump while the pump is pumping slowly (about 1 ml/min) will usually force the bubble from the pump head. Retighten the appropriate fitting when you see the bubble escape. Some manufacturers supply a syringe that facilitates pressurizing the low-pressure side of the pump to help clear bubbles from the pump heads. Standard laboratory safety precautions, such as eye and skin protection, should be taken. When you finish, wipe up any spilled solvent. You should also avoid twisting the head assembly when working with the check valves or fittings, because this may result in broken pistons.

Air entrapment is most likely to occur upon startup, in solvent changeover, and with improperly degassed solvents. It may occur once a day or once a week, but with nearly all reciprocating pump systems, air entrapment will happen. With a little practice, you will be able to clear air from the system quickly and with minimum downtime during your workday. The methods discussed here apply to most pumps, but you should consult your operator's manual for specific recommendations for your particular pump.

CHECK-VALVE FAILURE

The second most common point of failure with reciprocating pumps is the check valve. The symptoms are similar to those associated with air in the pump head: erratic or low flow. Check-valve problems, unfortunately, are often the most difficult to diagnose. You should be familiar with troubleshooting aids designed for your particular pump. Some manufacturers provide sample pressure traces that can help you diagnose check-valve problems by using a chart recorder and the pressure output from the pump. By far the easiest method for isolating and eliminating a check-valve problem is to substitute a questionable valve with one known to work well. This valve can be a used valve taken from another pump in the lab, or it can be a new valve that is kept as a backup valve. Unfortunately, few labs keep a supply of extra check valves on hand because of their cost. If you are able to isolate a faulty valve by substitution, you can easily solve the problem.

If you do not have spare valves for substitution, cleaning the valve in question is the next best choice. The valve is removed from the pump, placed in a beaker of acid, sonicated for several minutes, and rinsed several times with water. Workers find that 10% to 50% nitric acid is effective for cleaning, but consult your operator's manual for specific recommendations. Avoid using halogenated acids, which can attack the stainless-steel check-valve body. Also avoid using nitric acid if low-wavelength UV detection is used, but if you do use nitric acid, rinse the valve extensively. Remember that most cleaning solutions can cause severe burns.

Sometimes the check valves deteriorate to the point — or trap so many contaminants — that they can no longer be cleaned. You should replace or rebuild such a check valve. Replacement with a new or factory-rebuilt valve is the best choice. Some vendors sell kits for valve rebuilding. If you choose this route, follow the vendor's instructions carefully and work in a dust-free environment. Amateur valve rebuilding can have a very low success rate, and this is not surprising when you consider that manufacturers assemble valves in surgically clean rooms to ensure proper function. Be careful to prevent a mixup of the inlet and outlet check valves, because reversing them upon installation will make the pump work in reverse. Furthermore, in some cases improperly installed valves may result in piston breakage. Some manufacturers mark the flow direction with an arrow on the valve or use valves of different size or thread to prevent mixup. You should scribe an arrow on the valve body if one is not there already.

SEAL DETERIORATION

The third most common point of pump failure is the deterioration of piston seals. The problem is usually noticed when the mobile phase begins to drip from the pump head, although pressure diagnosis can isolate early wear. Pump seals wear out in six months to a year of normal use in most pumps. You should keep a spare set on hand at all times because there is no way to predict when you will need them. Seal life can be reduced by use of buffered mobile phases, which can deposit abrading salt crystals on the piston. Damaged pistons and certain solvents also reduce seal life. Some vendors recommend different seal types for different solvents. One of the solvents used most often, tetrahydrofuran, may deteriorate seals prematurely. Pump seals should be replaced according to the method recommended in your operator's manual. The following general precautions apply. Use care in removing the pump head because twisting the head upon removal may cause breakage of glass or sapphire pistons. Pump seals have two sides, so be sure to observe which side is showing when you remove the old one — a seal put in backwards will not work. A screw makes a good tool for removing the old seal if care is taken not to scratch the pump head. It is usually worth the additional cost to purchase the seal installation tool for your particular pump. Although the tool is rarely essential, its use reduces seal damage on installation and usually speeds the operation. Throw the old seals away — more than one chromatographer has wasted an afternoon by installing an old seal thought to be new.

PISTON BREAKAGE

The last common problem with the liquid ends is piston breakage. Most pumps in use today use sapphire pistons, although some older models may have glass or metal pistons. The symptom of a broken piston is no mobile phase flow and perhaps scraping sounds in the vicinity of the pump head as well. Piston breakage is almost always caused by operator error. Pistons rarely, if ever, break spontaneously or wear out. Pistons are most often broken when pump heads are removed or replaced, so take extra care during these operations. Applying too much torque to the pump head while tightening the check valves or fittings may also result in broken pistons. In older pumps, the pistons were often fixed firmly to a driving plunger and even slight misalignment would result in a broken piston. Fortunately, most newer pumps have pistons mounted in a slightly flexible mount, which allows for a certain amount of error during disassembly and reassembly of the heads. Once again, you should carefully follow the manufacturer's instructions for piston replacement. Pump heads should be cleaned thoroughly, and seals should always be replaced when a piston is replaced.

OTHER PROBLEMS

Other hydraulic problems with pumps are those that are common to the rest of the system — namely leaks. Leaks are most often fixed by tightening the offending fitting or replacing it if necessary. Other parts of the pump assembly such as mixers, proportioning valves, and pressure transducers are usually specially designed for a particular brand of pump. These parts are not discussed here. You should consult your operator's manual to help solve problems involving these parts.

Electronic problems with LC pumps are generally best left to service personnel. Pump motors can stall under too much load or from bearing failure. Motors can overrev if an optical coupler or other type of regulator fails, resulting in excessive flow rates. Stepping motors can lose one or more of the electric driving phases, resulting in lower-than-normal flow. Other sources of problems are electronic or software failure in the motor-driving circuitry. In most cases, the most expedient way to address electronic problems is to perform a cursory examination for loose wires or other obvious causes. Next, perform any troubleshooting procedure recommended in the operator's manual. Finally, call the manufacturer's service department for consultation on the best way to solve the pump problem.

PREVENTION OF PROBLEMS

As with other parts of the system, pump problems can be minimized by good laboratory habits. Use only HPLC-grade, filtered solvents. Flush any buffers from the pump at the end of each working day. Follow the manufacturer's recommendations for lubrication and other preventative maintenance of your pump. Make regular entries in your logbook that will help you easily determine whether the pump is functioning abnormally. With your logbook and the operator's manual, you will be able to solve most pump problems quickly. Do not hesitate to contact the pump manufacturer if you have problems, because many times a suggestion from the service department will allow you to fix the problem yourself.

A NOTE TO READERS

We are planning to prepare question-and-answer troubleshooting columns for future issues. We would appreciate submission of questions or suggestions of troubleshooting topics that are of particular interest to you. Please write directly to the authors or in care of *LC* magazine. • John W. Dolan, IBM Instruments, 40 West Brokaw Rd., San Jose, CA 95110; • Vern V. Berry, Dept. of Chemistry, 352 Lafayette St., Salem State College, Salem, MA 01970. For your convenience, a brief question or comment can be written on the bottom line of the Reader Service Card contained in this issue.

REFERENCES

(1) J.W. Dolan and V.V. Berry, *LC* **1**, 470 (1983).■

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