LC TROUBLESHOOTING

Pump Problems

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You must be resourceful to repair a pump whose parts are no longer in production.

This month we look at a reader's problem with an erratically functioning liquid chromatographic (LC) pump. The general problem and the remedies discussed apply to most brands of pumps available today. In addition, we have to deal with the restrictions of working with a pump that is no longer commercially available. A brief response is also given to a reader's question regarding passivation of an LC system.

FLUCTUATING PRESSURE

Q: My LC pump does not function reliably. It pumps for a few minutes, then the pressure drops momentarily. The pressure fluctuation can be correlated with the delivery stroke of one pump head, but the same head does not consistently exhibit the problem. At other times, the pump does not work at all because the minimum pressure (15 psi) cannot be maintained, apparently because both pump heads are malfunctioning simultaneously. The pump is more likely to work when pumping pure methanol or acetonitrile than when pumping organic solvent–water mixtures. I replaced the pump seals and observed no improvement. I flushed the pump with 20% nitric acid to clean the check valves, but this process has not improved the performance. When I remove the check valves, I can shake them and hear them rattle, so the ball is moving in the valve. Unfortunately, the pump is no longer commercially available, so parts are hard to obtain. Do you have any suggestions about how to fix this problem?

JWD: There are several possible problem sources, including pump starvation, degassing problems, air leaks, and faulty check valves. The order in which you perform the following tests is not very important; however, I prefer to do the easy tests first.

DEGASSING

If the mobile phase is not degassed properly, air bubbles can form on the low-pressure side of the pump or inside the pump, causing symptoms such as those you observe. The easy way to check for degassing problems is to test the pump performance with thoroughly degassed mobile phase. Degas the mobile phase by sparging for several minutes with helium, then reducing the helium flow to a trickle. Prime the pump and see whether the problem is solved. If degassing fixes the problem, continue to operate the system using this method. Many LC pumps will not operate reliably unless the mobile phase is thoroughly degassed.

PUMP STARVATION

If degassing the mobile phase does not help, note whether the pump is starving for mobile phase. Because you have observed that your pump is more likely to pump low-viscosity mobile phases (for example, pure methanol) than higher viscosity mixtures, I suspect that this may be the source of your problem. Each mobile phase inlet line should have an inlet frit on the end. If one or more of these frits becomes restricted or blocked, the pump will draw a partial vacuum in the attempt to fill with solvent. This partial vacuum can cause bubbles to form in the pump head, and the pump will exhibit the symptoms you observe. Remove the inlet-line frits one at a time and see whether the pump performance is improved. If the problem is corrected, replace all of the frits with new ones (usually it is not productive to try to clean these frits). Frits are available from most LC supply houses in both 5- and 10-μm porosity. Use the 10-μm frits because they are less likely to get blocked. These frits are not substitutes for filtering the mobile phase but are meant only to keep the occasional speck of dust from entering the pumping system. For this reason, be sure to filter the solvents through a 0.5-μm or smaller membrane filter before placing them in the reservoirs.

An additional aid to prevent pump starvation is a slight head pressure on the mobile phase reservoirs. Commercial reservoirs are available that maintain the mobile phase under a slight positive pressure of helium. Alternatively, elevate the reservoir above the pump so that siphon action provides a little pressure. Be sure the reservoirs are not completely sealed; otherwise a partial vacuum can form in the reservoir as the mobile phase is pumped out, causing pump starvation.

AIR LEAKS

My next step would be to check for air leaks in the solvent-inlet lines and connections before the pump. Depending on the system design, there may be one or more connections in the inlet line. Tighten each fitting to make sure all are snug. Typically, these fittings are plastic, so take care not to overtighten them and strip the threads. If tightening fixes the problem, you should be back in business. If not, you may want to do a more thorough check for leaks. The easiest way is to connect the inlet line directly to the inlet check valve, bypassing any other connections or mixing manifolds. If the problem is solved in this configuration, you'll have to check each fitting for proper assembly and tightness and replace any fitting that is suspect. Most fittings for low-pressure connections are interchangeable, so it is not critical to carefully match brands of these fittings. If the threads on the nuts are the same (generally, 10-32 threads), you can safely assume that the fittings can be interchanged.

In rare cases, air can leak through a ruptured solvent-proportioning valve in low-pressure mixing systems. If you suspect this problem, bypass the proportioning manifold by connecting the manifold inlet and outlet lines with a union and retest the system. If the problem is isolated to the manifold, it is best to replace the entire proportioning manifold because the proportioning valves need to be carefully matched. If you can't get new parts from the pump manufacturer, check with the manufacturer of the proportioning valves (the name should be on the valves) for replacement parts.

CHECK VALVES

My last choice is to work with the check valves. I am more suspicious of prepump blockages or air leaks than of check valve malfunctioning, because the problem moves from one head to another and because your device will pump low-viscosity solvents better than higher viscosity ones. When a pressure problem such as you describe can be correlated with a single pump head, the check valves usually are the first suspect; but this isn't necessarily the case when the problem moves from head to head. The easiest way to track down such a problem is to replace the check valves in a stepwise manner until the problem valve is identified. Even though the pump is no longer in production, you may be able to get new or rebuilt check valves. I would call the manufacturer (ask for technical support) to see whether you can get parts from them or whether they know of a source for parts. Alternatively, several aftermarket

suppliers (see the *LC•GC* Buyers' Guide [1]) make replacement check valves. Talk to these suppliers to see whether they have a replacement part or one that can be substituted.

Another approach to tracking down the bad valve is to systematically move the check valves from one pump head to another. In your case, however, I don't think this procedure will work because the problem seems to move randomly from one pump head to another. If new check valves are not available, I would try a rigorous cleaning as the next step. Remove the check valves, taking care to mark the inlet and outlet valves with a scribe if they are not easy to tell apart. Place the valves in a beaker of nitric acid diluted by about half with water and sonicate them 30 min or more. (Take the normal safety precautions when working with nitric acid!) Then rinse the valves and sonicate them again using several rinses of HPLC-grade water. You can tell when the acid has been rinsed out by testing the wash solution until it reaches a constant pH. Reinstall the check valves, prime the pump with degassed methanol, and you should be back in business.

BE RESOURCEFUL

I hope that one of these remedies solves your problem. Your case is a dilemma that we all go through — when is the system no longer worth fixing? This question has different answers depending on the financial situation of your institution. If you are a scavenger and want to track down spare parts for your dinosaur, you have to be clever about where to look. I mentioned contacting the original manufacturer for help. Sometimes the manufacturer's service personnel or a service person from an independent service company will know the location of an unused system of the same model — you might be able to get it for parts by paying the cost of shipping. If you're really desperate, consult old instrument application notes or publications that reference work of other laboratories on your model of instrument. Then contact the authors to see whether they have parts. Somewhere, though, the road ends, and you'll have to relegate your faithful LC pump to the trash heap.

PASSIVATION

Q: I have read about using passivated stainless steel in LC pumps and fittings. What is passivation, and why would I want to select passivated parts?

JWD: Passivation of stainless steel parts was discussed in detail in a previous "LC Troubleshooting" column (2), so I will just touch on the highlights. Passivation is the process of using nitric acid to remove small impurities of iron that remain on machined surfaces. Although we refer to stainless steel as "stainless," it will corrode under the right conditions. Various stainless steel alloys are available, and 316 stainless steel, the most common alloy used in LC work, contains sufficient iron to allow corrosion. Chemical interactions with the nonpassivated surface can be of special importance when electrochemical detection is used or when biomolecules are being analyzed.

When newly machined parts are passivated, they must be degreased, then passivated, and finally rinsed before they can be used. The degreasing step can be eliminated for LC system components that have been in use. So to passivate your LC system, just remove the parts that will be damaged by acid treatment (for example, the column), pump 6 N nitric acid through the system, and then flush it out with water. If you plan to use this procedure, see the referenced article (2) for details and safety precautions.

REFERENCES

(1) *LC•GC* **8**(8), 589–594 (1990).
(2) R. Shoup and M. Bogdan, *LC•GC* **7**(9), 742–744 (1989).

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