

## *Liquid Chromatography Problem Solving and Troubleshooting*

### **Question:**

I have observed a white residue on the fittings at the outlet check valves of my HPLC pumping system. I am using a buffer in the mobile phase, so I assume the residue is salt. I wipe the salt off every month or so. The performance of the system is okay, but I have been told that I have a leak and that I should fix it. Do I have a leak?

### **Answer:**

The solvent delivery system is a key component for attaining good chromatography and should provide a constant, precise, pulse-free flow of mobile phase through the HPLC. Modern pumps incorporate single or dual piston design, syringe delivery or a diaphragm delivery format. Problems with the pumps are manifested in erratic retention times, noisy baselines, or spikes on the baseline due to pulsations. Leaks in the pump usually occur at inlet or outlet fittings or at the piston seal(s). Leaks at the fittings are easy to spot. However, leaks at the seal and check valve problems may require a "pressure ramp test," which should be described in the operation manual.

That being said, let me turn to your situation. First, refer to your instrument manual and do a pressure ramp test on your pump to determine if there is a "leak." If the system passes the ramp test, the next question is a philosophical one: When is a leak not a leak? Obviously the source of the salt is the evaporated mobile phase, and the only way for the mobile phase to be there is if it came out of the fitting around the sealing ferrule. However, is it a leak if it does not compromise the performance of your pumping system and chromatograph?

This leads to my second recommendation. If there are salt crystals on a fitting, clean them off and observe whether there is a leak. Placing a piece of tissue paper (e.g., a Kem Wipe®) next to the fitting during normal operation of the pump will confirm a leak—even a slow one. Look for evidence of a leak by observing if a blot of solvent occurs on the tissue. If there is evidence of a leak, fix it. If a "leak" cannot be observed this way and your pump performance is satisfactory, in my opinion, it is not appropriate to tighten or repair the fitting/connector at this time. Sometimes salt crystals form on a fitting as a result of mobile phase "weep"—a very, very slow leak. Because the weep is so very slow, the mobile phase evaporates rather than remaining visible. The result is a residue that appears over a time frame of a month or two. However, watch the system and monitor daily whether a leak develops over time.

If a reciprocating pump is being used, it is reasonable to assume that because there was salt formation on the fitting, a residue is likely forming on the piston. If this is true, it is a good idea to flush the back side of the piston and the support bushing as preventative maintenance to remove the salt. Unremoved salt on a piston can, in certain instances, act as a sandpaper-like surface and decrease the seal life. Many HPLCs have a seal wash option to automatically wash the piston/seal area.

The purpose of *Chromatography Problem Solving and Troubleshooting* is to have selected experts answer chromatographic questions in any of the various separation fields (GC, GC-MS, HPLC, TLC, SFC, HPTLC, open column, etc.). If you have questions or problems that you would like answered, please forward these to the *Journal* editorial office with all pertinent details: instrument operating conditions, temperatures, pressures, columns, support materials, liquid phases, carrier gas, mobile phases, detectors, example chromatograms, etc. In addition, if you would like to share your expertise or experience in the form of a particular question accompanied by the answer, please forward to JCS Associate Editor, *Chromatography Problem Solving and Troubleshooting*, P.O. Box 48312, Niles, IL 60714. All questions/answers are reviewed to ensure completeness. The *Journal* reserves the right not to publish submitted questions/answers.

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