The Wrench

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System Backpressure

We've all seen it. Turn on the pump and the pressure in the system starts to rise. 300psi...800psi...1500psi...3000psi...3800psi...system shut down.

It seems every chemist eventually encounters this situation, where the system shuts down because the max pressure has been exceeded or a connection begins to leak. This article addresses how to diagnose and solve a system pressure problem.

If a method or system has been operating normally prior to when a pressure increase occurs, it is generally due to a clog or restriction in the flow path. The first step to solving the problem is to find out where the restriction is located. If a new method is being developed you may need to go back to the drawing board and look at what types of solvents you are using, what kind of samples are being injected, mobile phase composition, etc. In this article we will focus on systems with problems that occur while running established methods that have not been problematic.

Remove the column from the system and join the column inlet and outlet lines with a union. Turn the pump back on. If the pressure rises in the system without a column in place the clog is in the system. If the pressure is low and stable then your column is the culprit.

Let's take a look at a system clog first. To locate the problem you want to work from the back off the flow path towards the front. With the pressure still high, begin loosening each connection from the waste line up to the pump. While loosening each connection check to see at what point the pressure in the system drops. The section of the instrument that is just past where the pressure drops is where the restriction lies. If the clog is in a length of tubing simply remove that section and replace it with one of similar length and i.d. Another area that can see clogs is in the 6 port valve of the autosampler. This may require replacing the rotor seal. While the valve is apart it is a good idea to run cleaning wire through each of the holes to remove any pump seal or other material that may have lodged there. Finally, pre-column or autosampler frits are another common source of high backpressure. If these areas are where the problem is located, simply replace the frit. One last side thought about frits. Although it is recommended to work from the back of the system towards the front, frits are major culprits for clogs. They are just past the pump seals and so can become fouled with seal material. They are also right in line to create a perfect place for salting out of buffers used in conjunction with organic solvents. Finally, they are relatively simple to check and replace, so it may be worth taking a quick look at them right at the start.

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If the column is clogged the easiest solution is to simply replace it with a new column of the same type and dimensions from the same vendor. Alternatively, you can try and salvage the column by flushing it. To get started, attach the outlet end of the column to the inlet end of the HPLC system. The outlet end should not be connected to the system, but simply drained to waste (you do not want to hook the column into the system, because anything you manage to flush off will immediately now be going to the detector). Once the system is set up you can begin flushing the column with a series of solvents of different strengths/polarities. The recommended solvents and order of flushing are dependent on whether you had been running normal phase, or reverse phase, the use of buffers, the type of material the column is made of, and type of sample injected onto the column. For any procedure you want to flush with at least 10 column volumes of each solvent. The column volume can be calculated by the equation $Vol = \pi * r^2 * L$. Typically, the progression is through a series of solvents of increasing strength with care being taken to ensure that each subsequent solvent is miscible with the previous solvent. The flow rate during this process can be set at 20-80% of the normal column flow rate. Outlined below is a select series of washes for the regeneration of a bonded silica, reverse phase column.

HPLC Grade Water
Methanol
Acetonitrile
Isopropanol/Acetonitrile
Isopropanol
Methylene Chloride
Hexane
Isopropanol
Mobile Phase

In this series the water is used to remove any remaining buffer from the system or column before switching to a high organic solvent environment. Isopropanol is used as a transition solvent. The Methanol and Acetonitrile are organic solvents to remove polar compounds, while the Methylene Chloride and Hexane work to dissolve non-polar contamination. For other types of columns, modes of separation, or types of samples, column manufacturer web sites can be an excellent resource for troubleshooting and column care.

Once the column has been returned to its mobile phase, flip the column back around and reconnect the inlet end of the column to the tubing from the autosampler and hook the outlet end back into the detector. A test run should be performed to ensure that pressure, as well as retention times and theoretical plates are back within an acceptable range. For help with backpressure or other HPLC system problems please give us a call at 800-682-6480.

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