

SOLVENT CHANGE AND PURGE PROCEDURE DOCUMENT

A very common challenge for ViscoStar users manifests itself when switching solvents. Unlike other Wyatt Technology instruments, switching solvents in the ViscoStar is not a trivial process but rather requires a very specific series of steps to be taken in a certain order. The process is explained in section 4.2 of the ViscoStar User's Guide; however, some key points bear emphasis. Please see Section I below for a detailed explanation of the process.

Another common question regarding the ViscoStar concerns when and how to purge the device during regular use. Again, unlike other Wyatt Technology instruments, the purges should NOT be left on when the device is not in use. Rather, the device should be left in the configuration that continuously flushes the delay reservoirs. In other words, the device should be left in run-mode (all purges off). That being said, it is helpful to purge the transducers and purge valves periodically as a means of preventative maintenance. That procedure will be covered below as well in Section II.

IMPORTANT NOTE: Never attach fluid connections to the ViscoStar unless the device is powered up and has completed its boot cycle. The Transducer Protection System (TPS) requires that the device be fully booted up before it is active. Introduction of an alien solvent or air bubbles into the device can easily damage the differential pressure (DP) transducer if the TPS is not active.

Please see Figure 1 at the end of this document for a schematic of the ViscoStar.

I. VISCOSTAR SOLVENT CHANGE PROCEDURE

This procedure should be used whenever switching solvents in the ViscoStar.

- 1.) **Activate DP purge. Set flow rate to 0.2 mL/min. Let it flow for at least 15 minutes** This will purge the old solvent from the incoming tubing, both sides of the DP transducer, valves V2, and V3, the exit tubing, and the negative side of the IP transducer.
- 2.) **Activate both the Inlet Pressure (IP) and the Differential Pressure (DP) purges. Let it flow for at least 15 minutes.** This will purge the positive side of the IP transducer and valve V1.
- 3.) **Set flow rate to 0.1 mL/min. Disable the IP and DP purge.**

This puts the system into “run” mode and begins to purge old solvent out of the delay columns. It will purge R3, R4, and the delay columns (R5, R6 and R7 in Figure 1). This is the final, time-consuming step. Because R4 fills with the new solvent immediately but R3 contains the old solvent until the delay columns are filled, there will be a large pressure differential across the DP transducer during this step. If the pressure differential is large enough (> 0.73 psi or < -0.73 psi), the Transducer Protection System (TPS) will activate. Recall that the TPS actuates both the IP and DP purge valves when activated. This protects the transducers from damage but effectively prevents any fluid from flowing through the bridge.

To speed up this step, set the main graph to display DP pressure. While monitoring the DP pressure, increase the flow rate slowly. Adjust the flow rate so that the DP pressure remains on scale (± 0.73 psi). If the transducer protection system activates, reduce the flow rate, and disable the IP and DP purge. Continue until the DP pressure returns to near zero. As this occurs, you can continue to increase the flow rate until you reach the working flow rate for your chromatography. At this point the old solvent has been purged from the system.

Note that the actual flow rate that the device can tolerate during this step is going to be a function of the viscosity difference between the old and new solvents.

The default ViscoStar delay column configuration has approximately 10 mL of free volume which must be replaced. However, only half of the fluid being introduced into the instrument flows through the delay columns due to the bridge split. Thus it typically requires at least 20 mL of total solvent to purge the delay columns. We recommend that you flow at least 3-4X that value through the device prior to performing any experiments to ensure full purge of any residual solvent from the delay columns.

- 4.) **Set the flow rate to the desired value for chromatography and let the system stabilize. Perform the ViscoStar periodic purge as described below. In each step actuate the valves several times to release any old solvent trapped in their dead spaces.** This is a redundant step that ensures that the IP transducer, IP purge valve, and IP purge tubing is filled with the new solvent.

II. VISCOSTAR PERIODIC PURGE PROCEDURE

To reiterate the above, the device should be left in run mode (all purges off) when the device is not in use. This procedure is appropriate if the device has been in run mode for an extended period of time. The purpose of this procedure is to ensure that the transducers, purge valves and purge tubing are clean and bubble-free. This procedure should be performed periodically (~1X per week) as a means of keeping the device in good working order.

- 1.) **Activate the Inlet Pressure (IP) purge. Let it flow for approximately 10 mL.** This will purge the IP transducer, IP purge valve, and IP purge tubing.

- Figure 1: ViscoStar Schematic

