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Performance Note

MicroPro Pump: Pressure Decay Study

Introduction

Syringe pumps are effective at delivering very low flow rates without pulsation. When using syringe pumps in a chromatographic application, it is important to factor into consideration the requirement that the syringe refill at some point. For optimum reproducibility, this is usually done after the end of each run, so the system starts at the same point in the syringe cylinder at the beginning of each injection.

Consequences of Refilling

When the syringes in the µPro refill, a high pressure valve is turned to isolate the syringes from the downstream injector, column and detector. The syringes refill and are then switched back on-line with the downstream devices. At the point of switching back on-line, the syringes are at atmospheric pressure, while the downstream devices may still be at substantial pressures. The pressure difference between the syringes and the downstream devices can have deleterious effects on the column (e.g., "unpacking" the column if it is not protected by a filter on the top of the column, or decreasing the column's efficiency) in special circumstances.

Because of the potentially negative effects of these pressure differences, it is useful to analyze pressure decay in various systems. A good understanding of pressure decay will allow users to configure their methods to avoid the potential negative effects of pressure differences.

Pressure Decay On-Line

Figure 1 illustrates an experiment using a binary gradient µPro system with 2mL syringes, a 15µL mixer, connecting tubing and an additional pressure transducer. The data indicates the required time for the system pressure to decay varies from approximately 4 minutes (with a 1mm x 100cm column) to 40 minutes (with a .32mm x 150cm column).
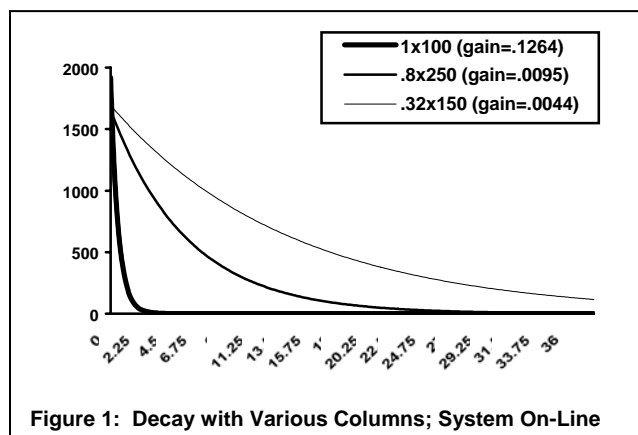

Figure 1: Decay with Various Columns; System On-Line

The data indicates that some adjustments may need to be made to user methods to prevent the potential negative effects, particularly when using packed capillary columns.

Pressure Decay Off-Line

A primary factor in the amount of time required is the volume of the system. When the system includes the 4mL volume associated with the two syringes, the time required is much greater than when the syringes are isolated from the downstream devices. Figure 2 illustrates the amount of time required for pressure decay with a 2mL syringe binary system, with the syringes isolated from the downstream devices.
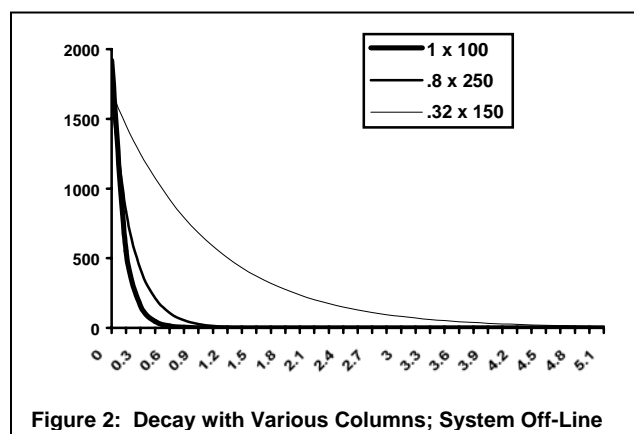

Figure 2: Decay with Various Columns; System Off-Line

In order to measure the pressure decay of the downstream devices, a pressure transducer must be installed. The pressure transducer has a relatively significant volume associated with it, and therefore, the actual pressure decay times can be expected to be a bit less in actual operation.

Possible Solutions to Pressure Differences

There are several possible ways to deal with the pressure differential.

- Add a series of steps at the end of the method to run the system at a negative flow rate to depressurize the system. The disadvantage of this way of dealing with the pressure difference is the risk of pulling mixed fluids back into the system and negatively impacting the next gradient run.

- Add a series of steps at the end of the method to run the system at a nominal flow rate until the system, including syringes is depressurized. The disadvantage of this way of dealing with the pressure difference is this can take a significant amount of time, depending on system volumes and pressure, as illustrated in Figure 1.

Slow Refill

During the refill operation of the syringes, the syringes are isolated from the downstream devices. By having the syringes refill at a rate which occupies a time equal to the natural decay of the downstream devices, the pressure difference normally associated with bringing the syringes back on line can be avoided. Slower refill rates will accomplish the goal of eliminating any pressure shock, in a relatively short period of time, without additional hardware.

Calculating Decay Time

It is possible, by analyzing the pressure decay data, to establish a relationship between the gain value and the time required for pressure to decay.

The gain value is the µPro's measure of the system resistance when the characterization routine is done, and is an expression of the relationship between pressure and flow rate. There are several variables involved in determining the gain value, primarily column ID and length, column material particle size, solvent composition, and column temperature.

Assuming characterization has been done, you can see the gain value by accessing SETUP, then PRE-PRESSURIZATION, then CHARACTERIZATION on the local display. Press ENTER through the fields for TARGET PRESSURE, etc. to display the screen showing the GAIN VALUE.

Figure 3 shows the relationship between the gain value and the time required to decay 95% of the system pressure (with the syringes off-line, as they are during refill). Figure 4 expands the view of a narrower range of gain values and times.

Once you know the gain value, it is possible to calculate the amount of time required to achieve 95% decay of system pressure (with the syringes off-line) by dividing the gain value into a constant, which is 0.015 ($t^{95}=0.015/Gain$).

When you calculate the amount of volume dispensed from the syringes during a run, you can determine the refill rate required to allow the system to decay 95% and enter that value in the REFILL RATE field. Since the above calculations are only a reasonable approximation, you may wish to enter a slightly slower refill rate.

An Example

Assuming the use of a run time of 20 minutes at a flow rate of 10 µL/min, the dispensed volume will be 200 µL. With a gain value of .005, the time to 95% pressure decay should be 3 minutes. Consequently, to refill at a rate which will not bring the syringes back on-line until the pressure has decreased 95% a refill rate of 66 µL/min is required. Since the calculations are only a good approximation, we might enter a refill rate of 50 µL/min.

Transitional Liquid Delivery System

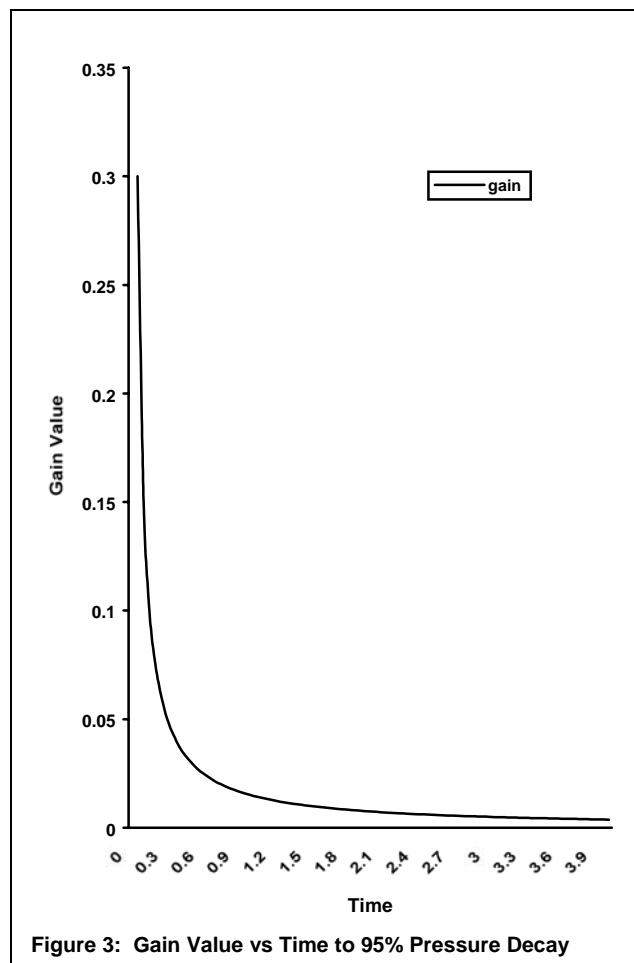Another way to eliminate pressure shock is to add a Transitional Liquid Delivery (TLD) syringe to the system.
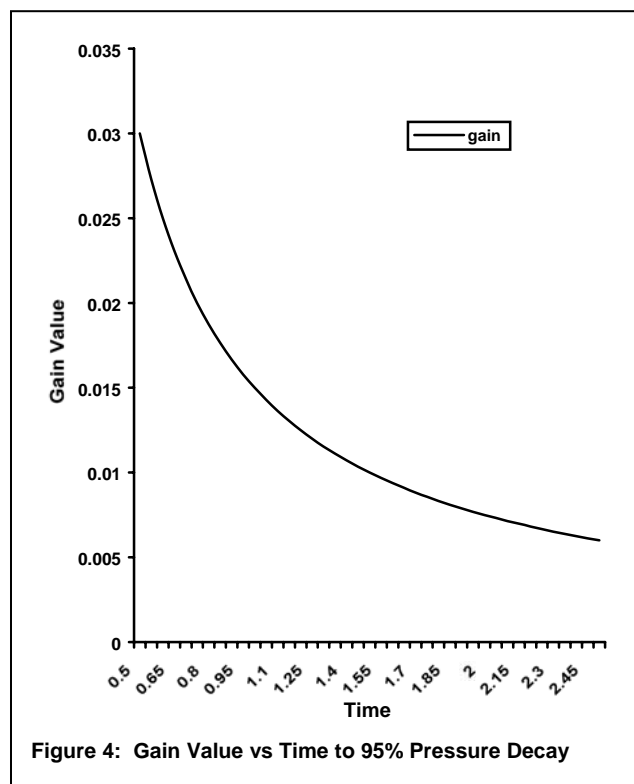

Figure 3: Gain Value vs Time to 95% Pressure Decay

Figure 4: Gain Value vs Time to 95% Pressure Decay

While the gradient syringes are refilling, the TLD syringe delivers solvent to the system, keeping the system under pressure.