Taking the Pressure Off Your Backpressure Regulator: How to Improve Ruggedness and Lengthen Lifespan

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INTRODUCTION

The automated backpressure regulator (ABPR) is a weak point in any SFC system. Like pump seals and check valves, any disruption of smooth operation can not only change flow or pressure but can dramatically affect chromatography. In bulk preparative SFC, any change in chromatography can destroy an automated stacked injection run, so a stable backpressure regulator is critical.

Our ABPRs use the Thar needle and seat design. The device adjusts the fit of the needle in the compressible seat, repeatedly closing and opening slightly as it attempts to maintain pressure. As the device wears, it adjusts the needle pressure too frequently, resulting in an unacceptable pressure fluctuation. Lowering the pressure setting requested stabilizes the fluctuation, implying that the wearing of the needle/seat is worse at higher settings. Taking the load off the ABPR might improve lifetime and performance.

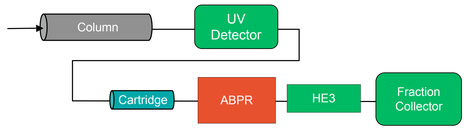
We tested this hypothesis using a fixed backpressure cartridge (Upchurch P/N U-469 [holder] and P-796 [1000 psi cartridge]).

U-469

P-796

INSTALLATION

The cartridge may be installed in-line, either on the inlet or outlet of the ABPR. Installation on the inlet side was chosen as it facilitated study of the pressure drop due to the cartridge itself. Moreover, installation on the outlet requires that the cartridge be heated to avoid excessive icing.

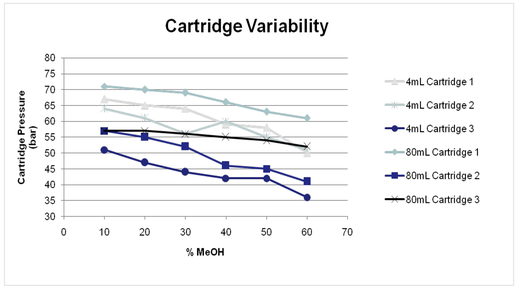


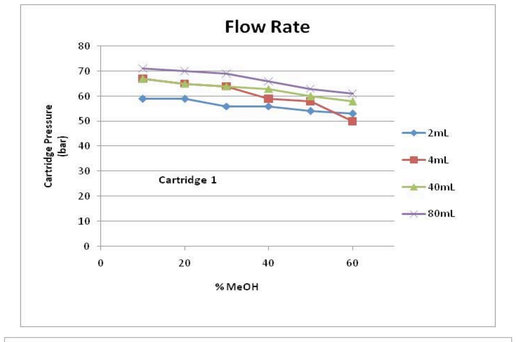
OPERATION

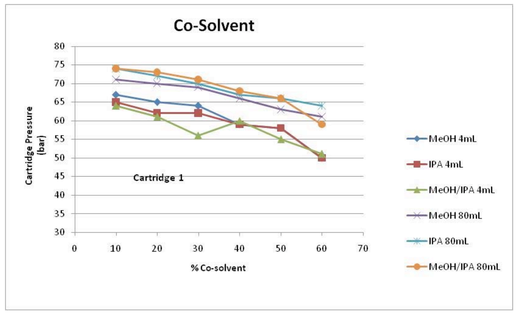
The Thar system reads the column pressure drop as the difference between the pressure sensors at the CO_2 pump head and the ABPR. The cartridge and the column are both restrictions placed between these points, so the pressure drop is the sum of the two. The ABPR pressure setting must therefore be adjusted downward by the cartridge's pressure value.

Because the cartridge's pressure value is not directly measured when a column is in place, we measured the cartridge's contribution to the backpressure independently. We were interested in:

- The variation in pressure value from cartridge to cartridge
- The effect of changing the CO2 co-solvent (mobile phase viscosity)
- The effect of changing the mobile phase flow rate

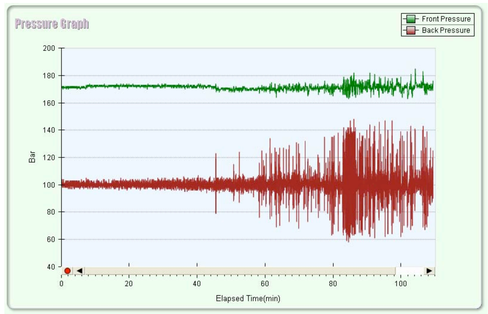


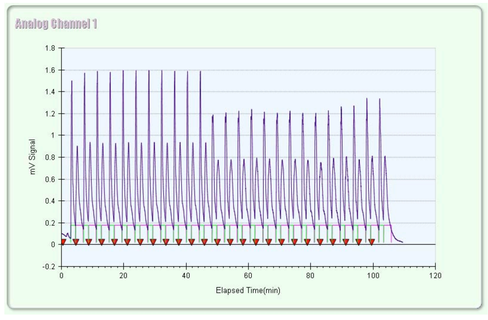




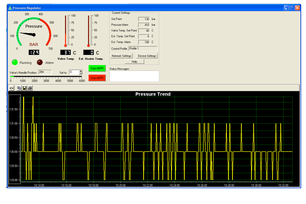
PERFORMANCE

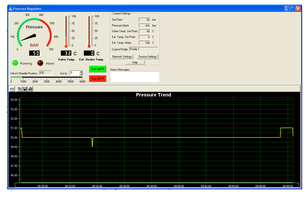
The figures below show the results of an ABPR failure on a stacked injection separation of a racemic mixture. The pressure traces show that the ABPR's sudden failure to self-regulate at about 45 minutes into the run causes a pressure fluctuation at the pump head as well (green trace). The pressure change affects the chromatography, resulting in broadened peaks and a change in the event timing needed to control the fraction collector.



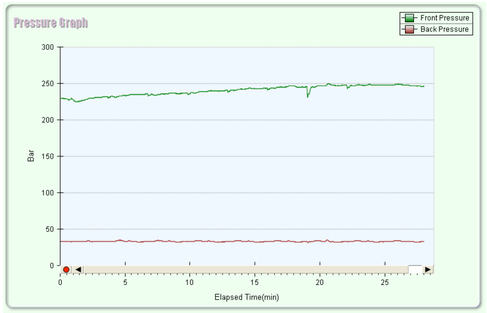


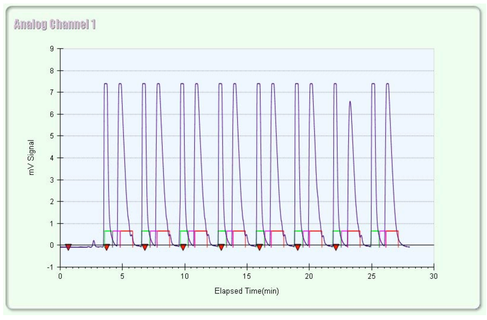
The cartridge, in removing part of the load previously borne entirely by the ABPR, stabilizes the device. The two traces below are ABPR pressure trend traces when operating normally (without the cartridge) at 130 bar, and with the cartridge in place (nominal setting 50 bar, true backpressure of 120 bar).





Below are pressure traces and chromatograms from a stacked injection run of the same sample, using the cartridge to supplement the ABPR. The ABPR was not serviced or recalibrated between the runs - the cartridge was simply installed in line and the pressure setting decreased by 70 bar.





CONCLUSIONS

1. The cartridge is an affordable and beneficial addition to the SFC system.
2. Each cartridge must be rated individually to determine its mean pressure value.
3. While the cartridges were more resistant to mobile phase flow at lower mobile phase concentrations, the variation was <15% over the normal range of co-solvent concentration.
4. The cartridges behaved similarly whether analytical or preparative scale flows were applied.
5. Changing the viscosity of the co-solvent had little effect.
6. The behavior of the ABPR is more stable using the backpressure cartridge to bear some of its load.
7. The ABPR appears to be less susceptible to wear (and need less frequent recalibration or parts replacements) when the cartridges are used.