Simplifying Connections in the GC

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Abstract

Analytical chemists are increasingly turning to the use of multi-column capillary GC systems to address the increasing demand for speed, selectivity, and / or sensitivity of analysis. Electronic pressure control has reached a level that has permitted a revival of multi-column approaches. However, the success or otherwise of a GC multi-column approach often depends upon something seemingly mundane: column connections. Without proper care and sufficient skill, increasing the number of connections, unions and connecting conduits in the GC system, can increase the likelihood of leaks, unswept volumes, and active sites. Those using multi-column systems warmly welcome technologies that alleviate these problems. Here we discuss an approach using microfabrication processes to develop a number of robust solutions ideally suited to the harsh environment of the GC oven including:

- Stainless steel planar micro channel devices
- Leak free finger tight metal ferrules
- Metal surface deactivation.

These technologies will be illustrated by providing case studies from experiences developing
multi-column GC approaches.

Guard Column and GC x GC Connector

Metal ferrules are ideal for connecting capillary columns as they provide a leak free solution in thermal gradient experiments. Figure 1 shows a stainless steel micro union (mass < 1 gm) that uses a double ended ferrule to make a finger tight union between two capillary columns or a guard column. The micro union is a convenient connector as it uses finger tight fittings to connect tubing, making it user friendly. There is no impact on the chromatography when using a micro-union and their permanent connection makes them more reliable than glass pressfits (see Figure 2).

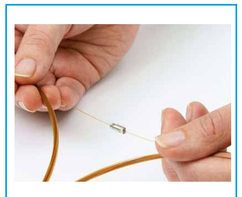

Figure 1: The SilTite™ µ-union. Finger tight installation, small thermal mass and low dead volume.

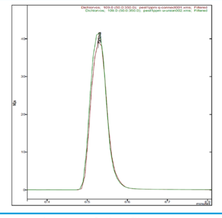

Figure 2: Evaluation of SilTite™ µ-union compared to glass press fits on pesticide analysis.

SilFlow™ Multichannel Devices

SilFlow™ is a diffusion bonded micro channel device, an innovation in design and fabrication resulting in an efficient and reliable micro fluidic platform that improves GC connectivity enabling maximum chromatography performance. Figure 10 shows the SilFlow™ device and finger tight tooling as installed.

Backflushing

Using BackFlush eliminates the need to "bake" heavy sample fractions off the capillary column. Oils, tars and other semivolatile matter can be flushed back out of the injection port while the oven remains at a relatively low temperature. This increases column lifetime dramatically.

4 Port SilFlow™

The 4 Port SilFlow™ is an ideal Backflush configuration as it enables two detectors to accurately identify when to trigger the Backflush event (Figure 3). Figure 4 demonstrates the analysis of ethanol, benzene and toluene in gasoline on a polar BPX90 capillary column. The sample was first run on a nonpolar BP1 to identify the time where the Backflush is intiated to prevent the non-volatile running onto the BPX90 column.

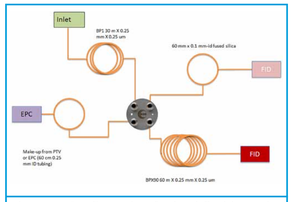

Figure 3: Configuration for backflushing using 4 Port SilFlow™ to FID.

Figure 4 A, separation of gasoline on A) B) BP1 (30 m x 0.25 mm x 0.25 µm) and BPX90 (60 m x 0.25 mm x 0.25 µm) with 2-Butanol IS. Figure 4 B, separation of gasoline on BP1 (30 m x 0.25 mm x 0.25 µm) and BPX90 (60 m x 0.25 mm x 0.25 µm) with Backflush triggered at 2 minutes - note the absence of the semivolatiles post 10 minutes.

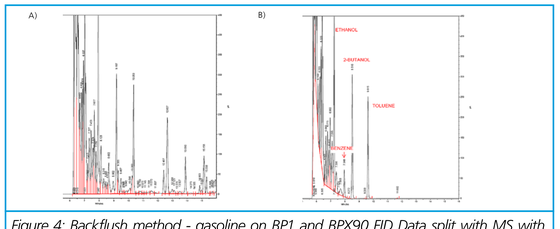

Figure 4: Backflush method - gasoline on BP1 and BPX90 FID Data split with MS with 2-Butanol IS.

3 Port SilFlow™

The 3 Port SilFlow™ is ideal for detector splitting, column splitting and also functions in Backflush mode. In natural gas analysis it is preferred to backflush if water is present - the following examples demonstrate mercaptans spiked into nitrogen. Figures 5 and 7 illustrate the Backflush set-up for the 3 Port SilFlow™ using two different detection systems - FID and SCD (Sulphur Chemiluminescence Detector) for the analysis of mercaptans in natural gas. Figure 6 highlights the excellent peak shape for the different mercaptans in the FID set-up. While slight peak tailing is obvious in Figure 8 for the low ppm levels, this is due to the non-specific interactions in the detector (alpha-alumina). Figure 9 shows a commercial natural gas sample run using the 3 Port SilFlow™ set-up highlighting the chemical inertness and detection of the sulphur odorants.

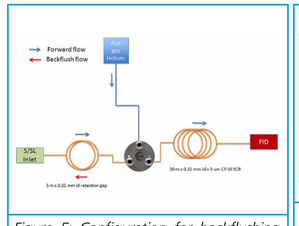

Figure 5: Configuration for backflushing using 3 Port SilFlow™ to FID.

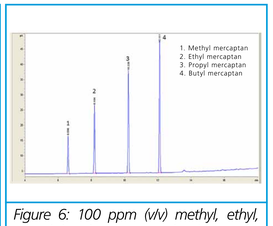

Figure 6: 100 ppm (v/v) methyl, ethyl, propyl, butyl mercaptans in nitrogen by GC/FID.

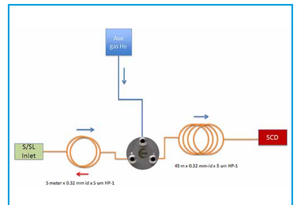

Figure 7: Configuration for backflushing using 3 Port SilFlow™ to SCD.

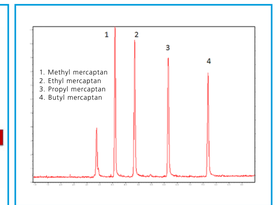

Figure 8: 1 ppm (v/v) of methyl, ethyl, propyl and butyl mercaptan in nitrogen GC/SCD.

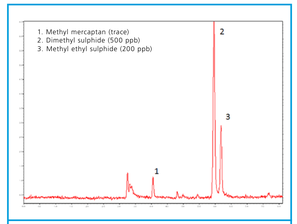

Figure 9: Commercial Alberta Natural Gas.

GC: Agilent 6890N with split/ Splitless inlet at 250 °C, Focus liner, split ratio 5:1 Detector: Agilent Dual-plasma sulphur chemiluminescence detector Reactor temperature: 850 °C Hydrogen: 50 mL/min Air: 35 mL/min Reactor vacuum: 310 torr Ozone reactor vacuum: 10 torr, 5 psig air Oven profile: 40 °C (2min) – 15 °C/min – 200 °C - 5min

Heart Cutting with SilFlow™ Dean's Switch

Heart cutting is regularly used in multidimensional GC where a time slice of the elute from a separation in the first dimension is directed onto a second capillary column of an alternate stationary phase (the second dimension). Figures 10 and 11 demonstrate a simple example where the co-eluting peaks of Octenyl Acetate and Linalool L were "heart cut" from the non-polar BPX5 onto the polar BPX70 highlighting the separation of both these compounds from Lavender Oil.

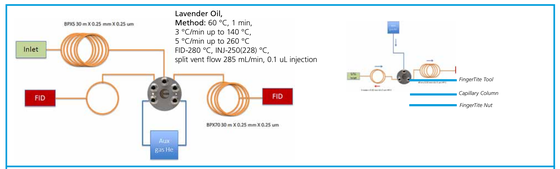

Figure 10: Configuration for heart cutting using Deans' Switch SilFlow™ to dual FID.

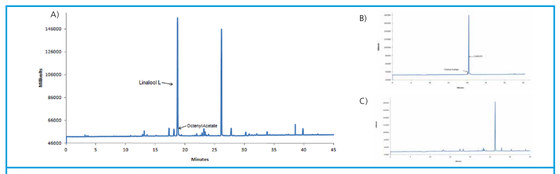

Figure 11: Lavender Oil heart cutting results.
A) Lavender oil separated on the first dimension BPX5 column - note the coeultion of octenyl acetate and Linalool L.
B) Separation of the BPX5 "heart cut" on BPX70, C) Separation of the remaining lavender oil components on BPX5.

Conclusion

- Stainless steel planar microchannel devices provide a versatile format for multidimensional analysis.
- Each of the metal devices described are optimized for low thermal mass.
- The chemical surface treatment of the channel devices and finger tight fittings do not impact chromatography – even for sulphur containing analytes.
- Finger tight fittings provide robust and easy to use column connectors, simplifying connections in the GC.
- Multi-channel fabricated devices simplify column splitting, backflushing and heart cutting.

Acknowledgements

1) Mr Okawa (Bruker - Japan) Micro Union comparison with Glass Press Fit

2) J Luong, R. Gras, R.A. Shellie, H. Cortes – manuscript in preparation. For data on the 3 Port SilFlow™ work.

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