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 microchannel 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 stainless steel micro union (mass < 1 gm) that uses a double ended metal ferrule to make a finger tight union between two capillary columns or a stainless steel micro union (mass < 1 gm) that uses a double ended metal ferrule to make a finger tight union between two capillary columns. Using these metal ferrules can be made to make a finger tight union between two capillary columns or a stainless steel micro union (mass < 1 gm) that uses a double ended metal ferrule to make a finger tight union between two capillary columns. 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. These technologies will be illustrated by providing case studies from experiences developing multi-column GC approaches.

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, tar and other semi-volatile matter can be flushed back out of the injection port while the oven remains at a relatively low temperature. This increases column lifetime dramatically.

Heart Cutting with SilFlow™ Dean’s Switch

Heart cutting is regularly used in multidimensional GC, wherever 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. 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. Figure 5 and 7 illustrate the Backflush set-up for the 3 Port SilFlow™ using 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 non-specific interactions in the detector (alumina-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 odourants.

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 connections, simplifying connections in the GC.
- Multi-channel fabricated devices simplify column splitting, backflushing and heart cutting.

Acknowledgements

1) M. Okawa (Shibak – Japan) Micro Union comparison with Glass Press Fit

For data on the 3 Port SilFlow™ work.