Inlet liner geometry and the impact on GC sample analysis

Introduction

The function of the GC injection port or inlet is to vaporize a liquid sample and introduce a portion of that sample onto the GC capillary column so that an effective separation can take place. Today there are a multitude of GC inlet liner geometries and packing options available on the market. Coupled with the various injection modes that are available, choosing the optimal inlet liner for a given application is increasingly difficult or in most cases, ignored.

Choosing the correct liner design and packing can significantly impact analytical performance. The use of glass quartz wool in inlet liners is well documented. Quartz wool on the positive side helps volatilization, as long as it is properly positioned inside the liner. On the negative side, quartz wool even if fully deactivated can cause breakdown of very active analytes. Liner choice also affects molecular weight discrimination. The best inlet liner allows all compounds, regardless of boiling point, to load onto the column equally and in a sharp band. In some cases optimization of the inlet system can improve sensitivity. Conversely, choosing the wrong liner geometry can significantly decrease the reproducibility and quality of a given analysis.

Using a series of controlled injection parameters, we report the differences between various GC inlet liner designs for a group of analytes across a wide boiling point range.

Experimental

All experiments were performed on a Shimadzu GCMS QP2010, fitted with a single standard split/splitless inlet using a BPX50 (50% phenyl polysilphenylene siloxane) column (20 m x 0.18 mm x 0.18 µm).

The best way to show the result of mass discrimination is to analyze a series of compounds from low to high molecular weight (i.e. from high volatility to low volatility). For this reason, a 1 µL injection of 20 ng/µL of the components in Table 1 were analyzed.

Table 1. Sample components in the test mix. Solvent methylene chloride.

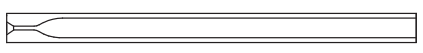
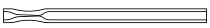
ID number	Name
1	naphthalene
2	2-methylnapthalene
3	1-methylnaphthalene
4	acenaphthylene
5	acenaphthene
6	fluorene
7	phenanthrene
8	anthracene
9	fluoranthene
10	pyrene
11	benzo(a)anthracene
12	chrysene
13	benzo(b)fluoranthene
14	benzo(k)fluoranthene
15	benzo(j)fluoranthene
16	benzo(a)pyrene
17	indeno(1,2,3-cd)pyrene
18	dibenzo(a,h)anthracene
19	benzo(g,h,i)perylene

Conditions

Inlet temperature	300°C
Transfer line temperature	300°C
Initial temperature	60°C, 1 min
Rate 1	35°C/min to 230°C
Rate 2	6°C/min to 240°C
Rate 3	50°C/min to 265°C
Rate 4	4°C/min to 320°C
Final temperature	320°C, 1 min
Detector	MS, 260°C
Scan	35-400 amu in 0.5 sec/scan
Injection mode	Splitless, High pressure (35 psi), 1 min

The different GC inlet liners for evaluation were chosen to demonstrate the impact of quartz wool, wool position and internal volume on liners and how they contribute to boiling point discrimination of analysis of samples.

Table 2. GC Inlet liner design parameters.

Part number	Geometry	Design	Volume
092071	Short taper no quartz wool		800 µL
092062	Quartz wool at fixed position into quartz wool injection		726 µL
092068	Bottom taper quartz wool at fixed position into quartz wool injection		660 µL
092058	Bottom taper quartz wool at fixed position onto quartz wool injection		660 µL
092329	Direct injection taper		600 µL

Results

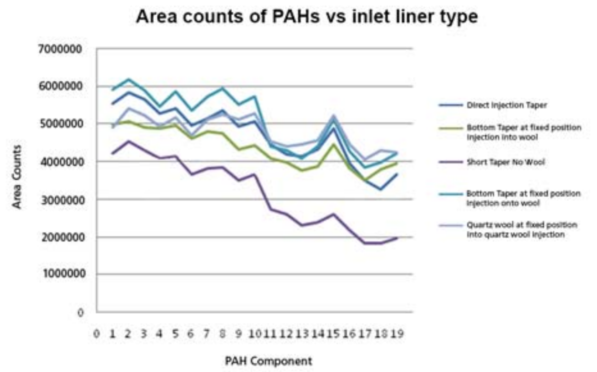


Figure 1. Area counts of the PAH components for each inlet liner geometry. Note that the peak area has more than doubled across the range of components between the inlet liner with the poorest response, compared with the top performing liners.

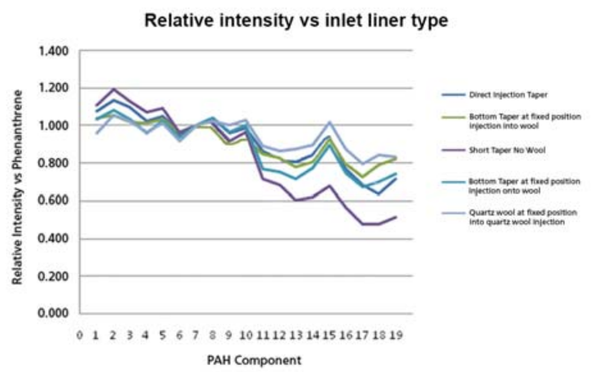


Figure 2. Relative intensity of versus the response for phenanthrene, for each inlet liner geometry. Note how the lack of wool contributes to a loss of response for the later eluting components.

Discussion

Addition of wool

The addition of quartz wool clearly impacts the performance of the inlet liner regardless of geometry (see Figures 1 and 2) – this is exacerbated for the high boiling point analytes where the inclusion of wool improves recovery as well as the relative response.

Optimal geometry

Four geometries delivered good recoveries of the PAHs; the optimal geometries based on recovery of the high boiling point PAHs were the liners where the wool was in a fixed position and the sample was injected into the wool regardless of presence of a taper.

Fixing wool position

Introducing a focused zone to secure the quartz wool has previously shown to benefit reproducibility (less than 1% compared with 5-10% without the fixed wool position)[1]. This is due to the sample being injected into the quartz wool, and the needle tip being wiped clean during the injection process, (see Figure 3).

Figure 3. The two tapered sections of an inlet liner secure the quartz wool plug effectively wiping the needle tip during injection. This results in improved reproducibility.

The reduction in analyte degradation is due to the cold solvent effect. As the sample is injected into the hot liner the evaporating solvent cools the quartz wool around the analytes. After the solvent has evaporated and as the quartz wool reheats, the analytes dissolve in the gas phase as they reach volatility. They then pass in laminar flow down the column inlet with minimal contact with the liner wall.

Position of wool

While much has been discussed previously about the function of quartz wool at a fixed position to ensure the needle tip has been wiped, some inlet liner geometries have the sample being injected on top of the wool rather than into the wool. Comparing two inlet liners of this geometry with different quartz wool placement, shows this effect for the range of analytes.

The raw chromatogram suggests an equivalent response (see Figures 4 and 5) for both injecting into the wool and on top of the wool. However, close analysis of the peak areas demonstrates an increased yield for an injection into the wool (see Figure 1).

When analyzing active components it is considered better to inject onto the wool, as penetrating the wool can create active sites.

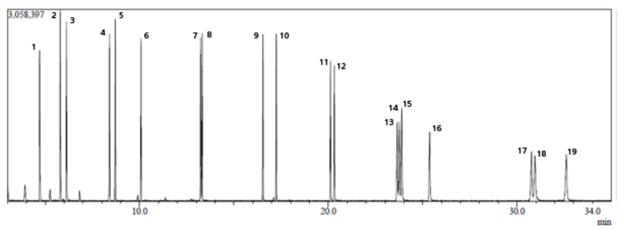


Figure 4. PAH test mix analyzed using a bottom taper and two tapers fixing quartz wool position (part number 092058) where the sample is injected onto the quartz wool.

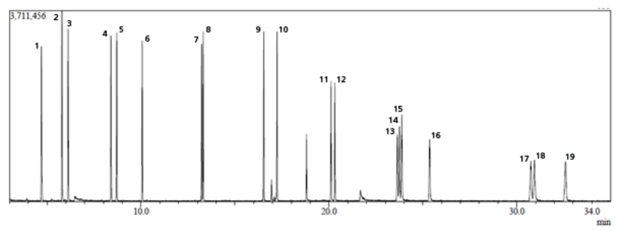


Figure 5. PAH test mix analyzed using a bottom taper and two tapers fixing quartz wool position (part number 092068) where the sample is injected into the quartz wool.

Direct injection

The direct injection tapered liner uses a direct inject technique to ensure full on column injection - effectively bypassing any quartz wool or cooling effect associated with a taper. This inlet liner does demonstrate relatively even loading of the analytes onto the column (see Figure 6).

The direct injection tapered liner is an excellent choice to improve loading without the use of wool as it has similar loading capabilities to a fixed wool liner.

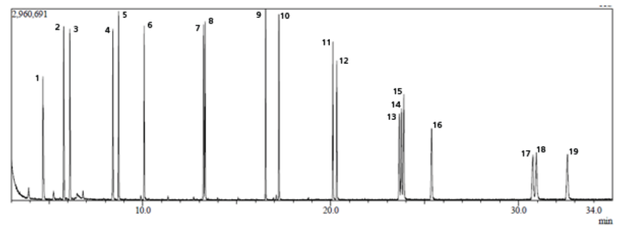


Figure 6. PAH test mix analyzed on a direct inject liner (part number 092329). Demonstrating excellent recoveries in all components.

Conclusion

The geometry of the inlet liner impacts the analytical performance and outcome. The bottom taper quartz wool at fixed position is ideally suited to evaluate a large boiling point range of analytes, without compromising the resolution.

For those analyses where very sensitive or active samples are being evaluated, and the presence of wool can adversely affect the result, the direct injection tapered liner yields excellent recoveries.

References

1. D. DiFeo, A. Hibberd, G. Sharp, Reducing Mass Discrimination by Optimization of the Liner Quartz Wool Position.

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