Inlet Liner Geometry and the Impact on GCMS Sample Analysis

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Abstract

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.

Choosing the correct liner design and packing can significantly impact analytical performance. The use of glass wool in inlet liners is well documented. Glass wool on the positive side helps volatilization, as long as it is properly positioned inside the liner. On the negative side, glass wool, even if it is fully deactivated, can cause breakdown of very active analytes. Liner choice also affects molecular weight discrimination. The best 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 sensitivities three fold. Conversely, choosing the wrong liner geometry can significantly decrease the reproducibility and quality of a given analysis. In this poster, through a series of controlled injection parameters, we will evaluate the differences between various GC inlet liner designs for a group of analytes across a wide boiling point range.

Background

All experiments were performed on a Shimadzu GCMS QP2010, fitted with a single standard split/splitless inlet.

Previous work evaluating inlet liners of differing geometries analyzing a PAH mix, demonstrated that a bottom taper liner containing wool in a fixed position yielded improved recoveries of the analytes, as well as an even loading of the sample onto the column. Where highly active compounds are being analyzed, it is better to inject the sample onto the wool, rather than into the wool, or forego wool altogether and use a direct inject tapered inlet liner – this is because penetrating the wool can break the wool, causing active sites. This study looks into liner geometries best suited for highly active analytes.

Injection Parameters and GC Settings

ID Number	Name
1	Phenanthrene-D10 IS
2	Aniline
3	2,4-Dinitrophenol
4	4-Nitrophenol
5	2 Methyl-4,6-Dinitrophenol
6	Pentachlorophenol
7	Amino biphenyl
8	Benzidine
9	Benzo(b)fluoranthene
10	Benzo(k)fluoranthene

Table 1. GC Inlet Liner design parameters.

The active analytes used in this study are shown in Table 1.

Inlet temperature 250 °C
Transfer Line 300 °C
Initial temperature 50 °C
Initial hold 1 minute
Rate 1 10 degrees °C / minute
Rate 1 final temperature 300 °C

MS – Source temperature 260 °C
Scan – 35-400 amu in 0.5 sec / scan
High Pressure Injection (35 psi) Splitless for 1 minute

The different GC Inlet Liners for evaluation are shown in Table 2.

Inlet Liner Geometry	Design	Volume of Inlet Liner
Long Taper wool		575 µL
Bottom Taper wool at fixed position onto wool injection		660 µL
Direct Injection Taper with wool		600 µL

Table 2. GC Inlet Liner design parameters.

Results

Area Count

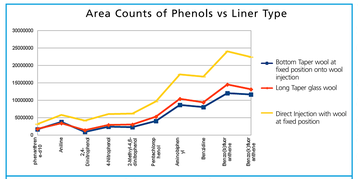


Figure 1. Area counts of the compounds for each Inlet Liner geometry.

The area count for the analytes are shown for each of the liner types evaluated (Figure 1). Interestingly, the response for the direct injection liner with wool at a fixed position yields almost double that of the other two geometries. Both long taper wool and bottom taper with fixed position glass wool perform similarly.

Relative Response Factor

Figure 2 demonstrates the relative response factor for the active analytes versus Phenanthrene-D10 for each of the liner geometries. For each liner geometry the results are the average from 17 separate injections. Overall, each of the geometries perform similarly where the largest discrimination between geometries was observed for 2,4-dinitrophenol, 4-nitrophenol, 2-methyl-4,6-dinitrophenol and pentachlorophenol. The most significant difference was for the recovery of 2,4-dinitrophenol between the three liner types – the direction injection with wool at a fixed position demonstrated a 144 % improved recovery over the bottom taper with wool at a fixed position and 68 % improved recovery over the long taper with wool.

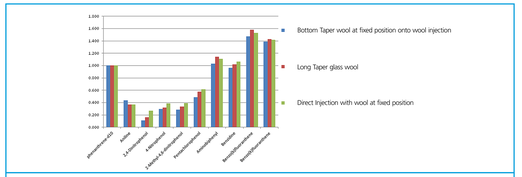


Figure 2. Relative intensity of each component normalized versus the response for Phenanthrene-D10 IS, for each Inlet Liner geometry.

Figure 3 represents the % RSD for the same data set shown in Figure 2. The variation in % RSD is greater than anticipated for a liner with wool in a fixed position due to several factors:
1) Injections were on top of the wool
2) Detector in MS scan mode
3) 1 µL injection from a 10 µL syringe.
Nevertheless, wool in a fixed position appears to minimize the variation observed from injection to injection compared to the long taper wool liner.

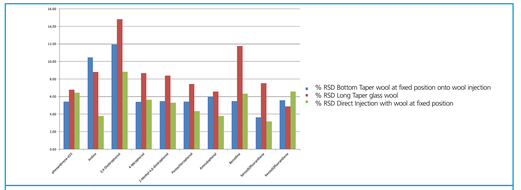
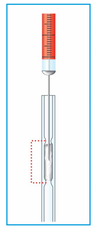


Figure 3. % RSD for each component and Inlet Liner geometry.

Discussion

Fixed Wool Position



Introducing a focused zone to secure the liner wool has previously shown to benefit reproducibility particularly for mass discrimination (% RSD less than 1 % compared with 5 – 10 % without the fixed wool position)[1]. This is due to the sample being injected into the wool, and the needle tip being wiped clean during the injection process (see Figure 4).

In this study we have focused on active analytes such as phenols and in particular the influence of the taper design and wool in a fixed position. All liners and wool were deactivated as certified by the manufacturer. In the GCMS studies reported here, the % RSD was higher than anticipated due to system variability and the susceptibility of the analyte to break down.

Figure 4. The two tapered sections of an Inlet Liner secure the wool plug effectively wiping the needle tip during injection. This results in improved reproducibility.

Conclusion

The geometry of the Inlet Liner impacts the analytical performance and outcome, particularly for GCMS. For those samples where very sensitive or active samples are being evaluated, combining the performance of a fixed position wool inlet liner with a direct injection yields the best and most sensitive analysis.

References

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