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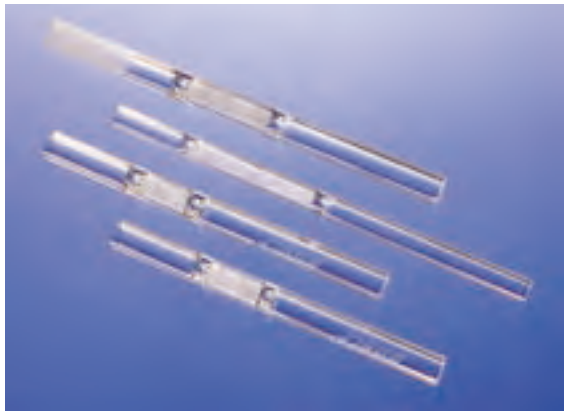
TECHNICAL ARTICLE

0.2% RSD's?

It's Now a Reality with SGE's FocusLiner

Optimize the accuracy and reproducibility of your chromatography by understanding syringe and liner interactions.



Many papers have been written about this phenomenon but the majority of these have not addressed the fundamental issue of dispensing very small samples from the syringe during the injection process. This is particularly important when using high speed autosamplers where the injection process is rapid and little interaction occurs between the syringe needle and the hot injection port.

Work has been undertaken to better understand the results of various autosampler syringe and inlet liner combinations by studying peak shape, chromatographic precision and reproducibility. The results of this work has lead to the development of an inlet liner to optimize autosampler injection.

The Syringe

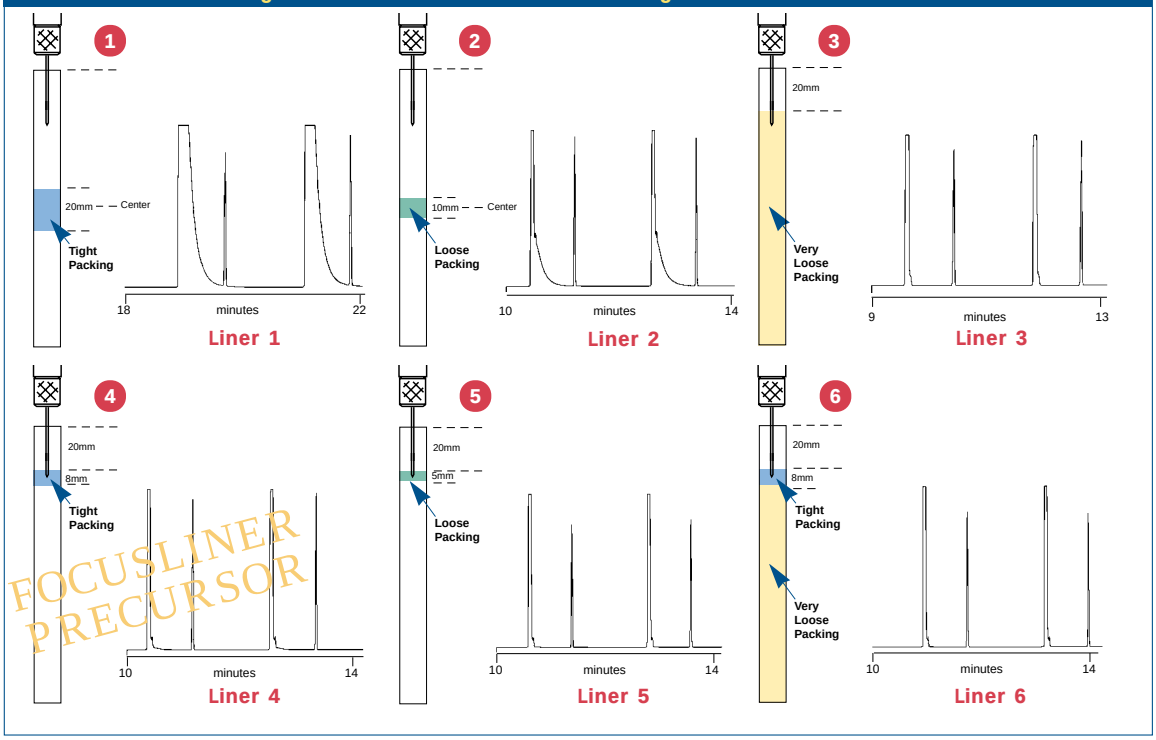
The small injection volume and the low sample exit velocity from the needle of a 0.5µL plunger-in-needle syringe make it ideal for this study. Without needle tip wiping, a sample leaving the tip of this syringe has the tendency to form droplets and wet the needle tip rather than forming a jet of nebulized liquid from the needle tip. Effective tip wiping with deactivated quartz wool will improve both peak shape and injection reproducibility.

Figure 1 illustrates the packing positions of the high density quartz wool used in each of the six liners studied.

Results

Liner #1 and #2: The chromatograms obtained from Liner #1 and Liner #2 show severe solvent peak tailing. This is consistent with slow volatilization of

FIGURE 1. Liner Packing Positions and the resultant chromatograms



CHROMATOGRAPHIC CONDITIONS

Column:	**Column:** SGE, 25m x 0.32mm, BP1, 0.5µm film **Carrier Gas:** Helium **Flow Velocity:** 2.3mL/min **Oven Temperature:** 120°C
Injector:	**Injection:** Split/Splitless Injector **Liner packing positions:** (as specified) **Injector Temperature:** 200°C **Split Ratio:** 50:1
Detector:	FID @ 300°C
Autosampler:	Injection Volume = 0.1µL No. of injections per vial = 15 No. of pumps = 6
Sample:	(approx.) 0.02% C_{10} in C_7
Signal Parameters:	**Range: 0** **Attn:** 4
Data Handling:	Delta Chromatography System - calculations based on peak area.

the sample and/or slow transfer of the sample onto the column. These liners do not wipe the needle tip during injection.

Liner #3: Minor peak tailing was observed from Liner #3. On removal of this liner from the injection port, it was found that the needle had pushed the quartz wool beyond the point where it wiped the needle tip during injection.

Liner #4, #5 and #6: Acceptable chromatography was achieved with Liners #4 to #6. These were packed to ensure that the needle tip was wiped during injection. However, upon multiple injections, it was found that Liner #5 suffered the same problem as Liner #3, with the packing being pushed beyond the point where it wipes the needle tip. The quartz packing needs to be tight enough to avoid being pushed down the liner by the needle.

Boiling Point Discrimination

The temperature program chromatograms of a C8 to C32 hydrocarbon mixture injected onto Liner #1 and Liner #4 were also examined. The aim of this experiment was to determine if there is any detrimental boiling point discrimination by moving the liner packing from the center (Liner #1) of the injection port to a position where the needle tip is wiped (Liner #4). **Table 1** expresses the ratio of the four highest molecular weight components against C11. (Note: components were not in equal ratios). No significant variations were observed.

Figure 2. The SGE FocusLiner™

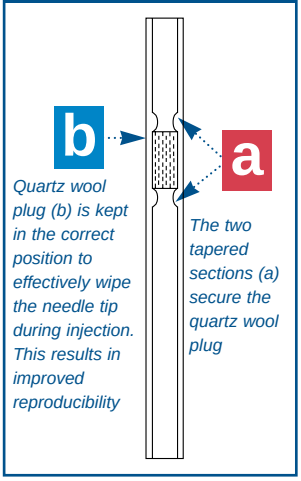


Table 1. Boiling Point Discrimination Results

Compound Ratio	Liner 4 Ratio C_x/C_{11} Packed to wipe needle	Liner 1 Ratio C_x/C_{11} Packed-center of liner
C_{22}/C_{11}	0.95	0.95
C_{24}/C_{11}	0.44	0.44
C_{28}/C_{11}	0.47	0.49
C_{32}/C_{11}	0.55	0.54

Reproducibility

Chromatographic reproducibility was checked on Liner #1 and Liner #4. The results are tabulated below:

Table 2: Reproducibility Test Results

Syringe	0.3µL(0.5B-0.63)	0.3µL(0.5B-0.63)
Liner no.	Liner 4	Liner 1
Tailing:	Good	Poor
Mean Area Count	898.55	671.45
Std. Dev. Area Count	5.16	85.49
%RSD (Area Count)	**0.57**	**12.73**

Conclusion

Severe peak tailing and poor reproducibility is often observed if the needle tip is not wiped during injection when using high speed autosamplers, particularly when injecting small (<2µL) volumes. It occurs when the sample being delivered from the syringe forms droplets that wet the syringe needle tip rather than forming a jet of nebulized liquid from the tip. With the rapid withdrawal of the needle from the injection port, the tip is wiped by the septa causing slow secondary vaporization near the cool septum cap.

By placing the quartz wool packing in a position in the liner where the needle tip is wiped during injection, peak shape and reproducibility are improved.

SGE's "FOCUSLINER™" (**Figure 2**) is now available with the quartz wool positioned for optimum needle tip wiping. The quartz wool in this liner is held in position by means of two tapered sections in the liner. The tapered sections are located to ensure the needle tip penetrates the secured quartz wool plug and consequently wipes any residue liquid sample from the needle tip while providing sufficient surface area for the volatilization of the liquid sample.

Only SGE's FocusLiner provides the levels of reproducibility and confidence that is needed for split injection analyses.

ORDERING INFORMATION - FOCUSLINER™

Instrument	*Injection Mode*	*Dimensions*	*Part No.* (Pkt 5)
Carlo Erba 8000	Split	5mm ID x 8mm OD x 105mm	**092045**
Hewlett Packard	Split	4mm ID x 6.3mm OD x 78.5mm	**092002**
Perkin Elmer Autosystem	Split	4mm ID x 6.2mm OD x 92mm	**092092**
Shimadzu 14A/B	Split	3.4mm ID x 4.8mm OD x 99mm	**092065**
Shimadzu 17A	Split	3mm ID x 5mm OD x 95mm	**092062**
Varian 1075/1077	Split	4mm ID x 6.3mm OD x 72mm	**092022**
Varian 1078/1079	Split	3.4mm ID x 5mm OD x 54mm	**092037**