A Comparison of Liner Geometries and Their Effect on Gas Chromatographic Performance.

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Abstract

When using Split/Splitless injection ports, analysts have a wide variety of liner geometries to choose from. Split liners are designed with mixing chambers and tortuous flow paths to fully vaporize the sample into a homogeneous vapor cloud before it reaches the split point. Splitless liners usually are designed as straight tubes, with alternate designs, such as a gooseneck restriction, which help contain the sample cloud in the injector and minimize the breakdown of compounds sensitive to catalytic decomposition from contact with metal inlet parts. The residence time of the sample in a splitless liner is dependent on liner geometry, gas velocity, and sample vaporization time.

Introduction

With so many inlet liner designs on the market today, how do you determine which one is best suited for your analysis? Each liner geometry offers the analyst a unique sample flow, through the liner onto the analytical column, through cups, cyclos, and packings designed in the inner bore of the liner. How does each design affect sample flow? How does the internal volume of the liner affect chromatography?

We will look at these questions to determine which is the best liner for your analysis.

Basic Liner Characteristics

When choosing a liner for your analysis you should always consider the expansion volume of the sample that you are introducing into the liner. Expansion volumes can vary with the solvent that you are using.

Back flash can occur when the expansion volume of the solvent in the sample is greater than that of the expansion volume of the liner. This can cause poor peak area reproducibility, tailing solvent peaks and ghost peaks.

Backflash
Liner Volumes

	Theoretical*	Effective
1.0mm ID	= 59µL	30µL
2.0mm ID	= 236µL	118µL
3.0mm ID	= 530µL	265µL
4.0mm ID	= 942µL	471µL

* Liner volume actually available for vaporization with carrier gas present is ≤ ½ theoretical!

From Grob, Split and Splitless Injection, 3rd ed.

Backflash

Solvent Expansion Volumes

Injection volume (liquid)	Expansion volume (vaporized)				
	H_2O	CS_2	CH_2Cl_2	Hexane	Isooctane
0.5µL	~~710µL~~	212µL	200µL	98µL	78µL
1.0µL	~~1420µL~~	423µL	401µL	195µL	155µL
2.0µL	~~2840µL~~	~~846µL~~	~~802µL~~	390µL	310µL
5.0µL	~~7100µL~~	~~2120µL~~	~~2000µL~~	~~975µL~~	~~775µL~~

*Based on liner ID of 4mm, injection port temperature of 250°C and 10psig head pressure.

~~X~~ = Too large

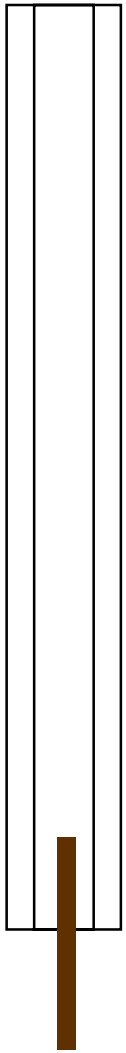
Splitless Liners

Splitless liners are designed to hold the sample in the liner between 0.5 and 2.0 minutes. A large surface area for sample vaporization is not a factor in splitless injections.

It is common to use packing materials whenever dirty samples are analyzed. Liners packed with wool help promote sample vaporization, as well as trap non-volatile residue to prevent column contamination which ultimately increases column life.

Splitless Liner Designs

Straight



Benefits:

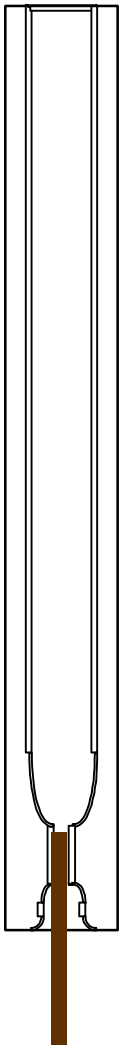
Low cost.

Drawbacks:

Prone to high molecular weight distribution.

Sample exposed to metal surface below liner.

Gooseneck



Benefits:

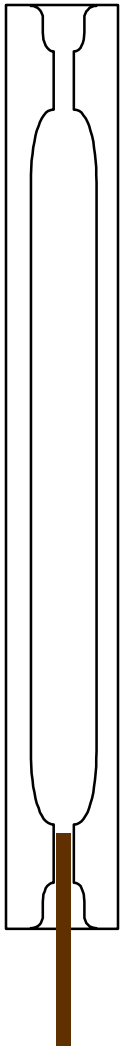
Decrease sample contact with metal inlet parts.

Improves sample transfer to column.

Drawbacks:

More sample backflash than with the double gooseneck.

Double Gooseneck



Benefits:

Decrease sample backflash.

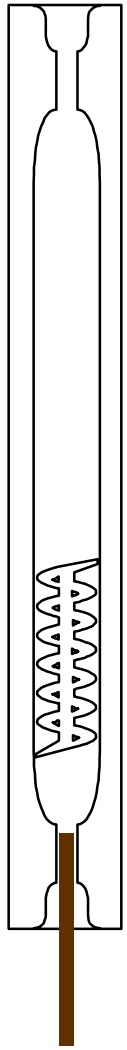
Decrease injection port discrimination.

Drawbacks:

Cannot be packed with wool.

Difficult to clean.

Cyclo Double-Gooseneck



Benefits:

Extends column lifetime by trapping non-volatile residue.

Drawbacks:

Cannot be packed with wool.

Difficult to clean.

Straight Tube Design

The straight tube design is the most common splitless liner design. This liner is ideal for low molecular weight samples that are prone to thermal decomposition. If used for high molecular weight sample analysis, packing material such as glass wool or CarboFrit™ material is recommended to aid in sample vaporization.

Gooseneck Liners

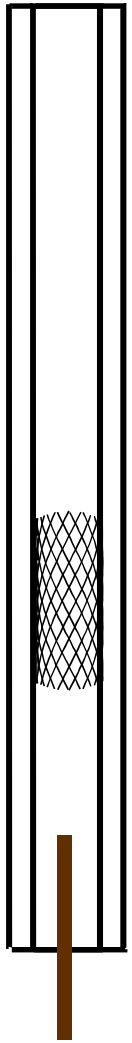
The gooseneck liner helps isolate the sample from the metal injection port parts situated at the base of the injector. This design funnels the sample onto the analytical column for increased splitless efficiency and decreased breakdown of highly active compounds, such as Endrin and DDT. The double gooseneck design helps to contain the sample cloud in the liner, for increased performance with larger sample introductions, but cannot be packed with wool.

Split Liners

Split liners are designed to help vaporize the sample by using mixing chambers and tortuous flow paths to help vaporize the sample before it enters the analytical column. Materials such as deactivated fused silica wool or beads, CarboFrit™ packing, and other packings are used to increase sample vaporization.

Split Injection Liner Designs

Split with wool or CarboFrit™



Benefits:

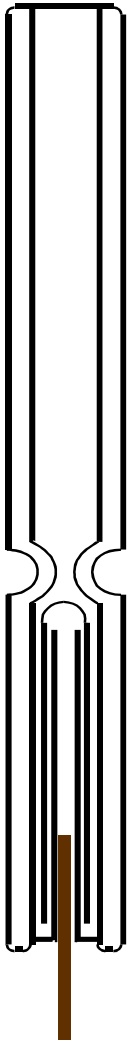
Low cost.

Reproducible performance.

Drawbacks:

Wool can be adsorptive, especially if fibers are broken.

Laminar Cup



Benefits:

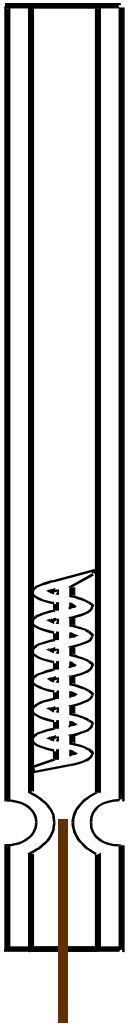
Best splitter for high molecular weight compounds.

High resolution.

Drawbacks:

Costly.

Cyclosplitter®



Benefits:

Ideal for dirty samples.

Allows many injections of dirty samples before cleaning is required.

Drawbacks:

Not recommended for large volume injections.

Straight Tube Design

The most common liner for split analysis is the straight liner with deactivated wool, which offers the analyst a wide variety of options. The wool has a high surface area for more sample evaporation to occur, and promotes a uniform vapor cloud to enter the split point. The drawback of this design is that the wool increases breakdown of highly active compounds. When using this liner, the wool needs to be changed frequently. The position of the wool inside the liner and the quantity of the wool used is critical to reproducibility.

Cup Splitter Designs

Cup splitter liners offer a more homogenous vaporization through increased sample residence time in the liner. The sample passes through a series of tortuous flow paths, which aids in sample vaporization. These liners are best suited for high molecular weight compounds.

The Cyclosplitter® liner incorporates a cylindrical glass screw in the sample pathway. The screw helps to mix and vaporize the sample. The increased surface area in the glass screw also helps to trap non-volatile residue, therefore, making it ideal for dirty samples.

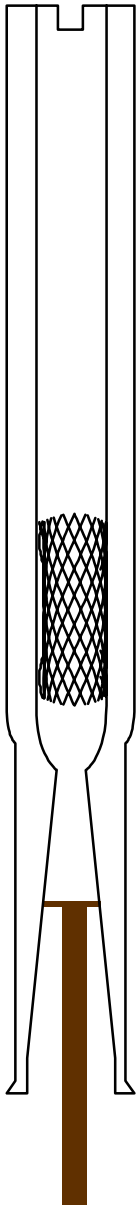
Uniliner® for Direct Injection

A Uniliner® is designed with a “press tight” fit between the glass surface of the liner and the analytical column. This prevents the sample from contacting metal inlet parts at the base of the splitless injection port. It also eliminates many problems associated with splitless hold times, such as reduced response and adsorption of high molecular weight compounds in the inlet, providing overall higher sensitivity.

Drilled Uniliner® design

The drilled Uniliner® is ideal for use with EPC equipped GC systems. The hole equalizes pressure and maximizes sensitivity. The drilled Uniliner® with the hole near the bottom is recommended for analysis in which compounds of interest could be affected by a tailing solvent peak. The drilled Uniliner® with the hole near the top is recommended for analysis where compounds of interest elute away from the solvent peak.

Direct Injection Liners



Open-top Uniliner® w/ wool

Benefits:

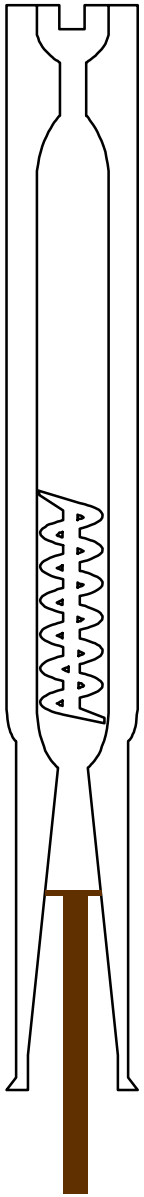
Easy to clean.

No contact with metal inlet parts.

Simulates on-column sample introduction.

Drawbacks:

Wool can be adsorptive, especially if fibers are broken.



Cyclo Uniliner®

Benefits:

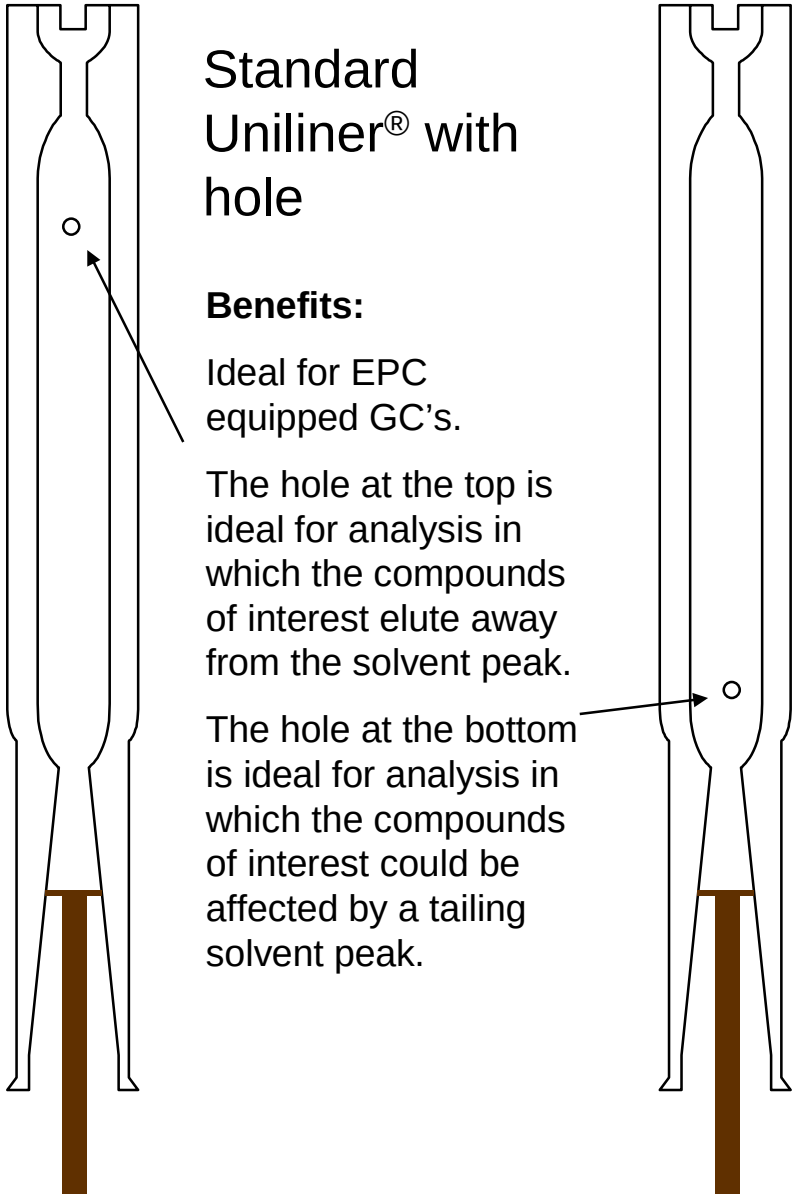
Excellent vaporization for high and low molecular weight samples.

Traps non-volatile residue.

Drawbacks:

Cannot be packed with wool.

Difficult to clean.



Standard Uniliner® with hole

Benefits:

Ideal for EPC equipped GC's.

The hole at the top is ideal for analysis in which the compounds of interest elute away from the solvent peak.

The hole at the bottom is ideal for analysis in which the compounds of interest could be affected by a tailing solvent peak.

Summary

When choosing a liner for your analysis, there are several key factors that will affect how the sample travels through the liner and onto the column. Choose a liner for the type of injection you will be performing and the type of compounds that are in the sample. Each liner offers a unique sample pathway and is designed to work best with a particular type of injection mode. Be sure to review all the variables, so your results are accurate and your downtime is decreased.