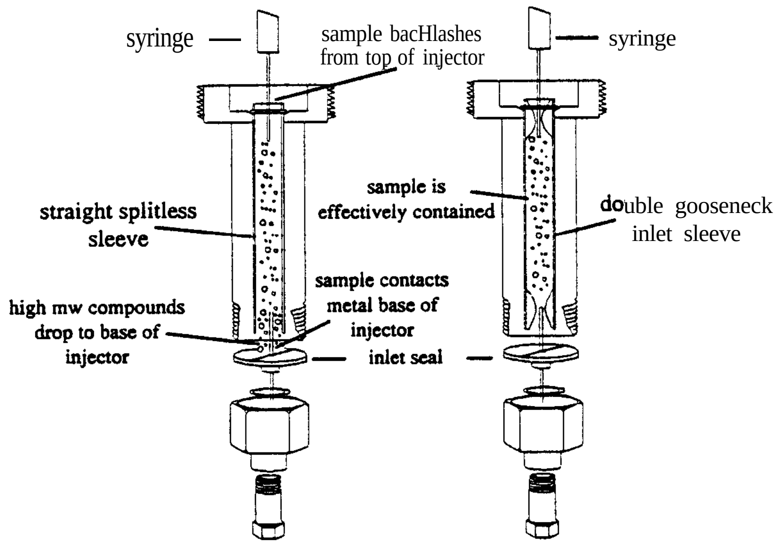
Hints for the Capillary Chromatographer

Double Gooseneck Sleeves Improve Splitless Results

The splitless injection mode is the most widely used injection technique for trace analysis in capillary gas chromatography. Straight inlet sleeves are commonly used because a high surface area for rapid vaporization is not necessary. Little emphasis was placed on inlet sleeve design because of the long sample residence time in the injector. The recent development of a new sleeve, called the double gooseneck, has shown a significant advantage **over** open sleeve designs.

Figure 1 - Double gooseneck sleeves have chromatographic advantages over straight splitless sleeves.



Double gooseneck sleeves effectively contain the sample, enhancing the response of high molecular weight compoutids, as well as increasing sensitivity by reducing breakdown.

Which end is up?

Single gooseneck sleeves for the HP 5890 GC were introduced to prevent backflash ougf the top of the injector and mimic the design and function of Varian inlet sleeves. While we thought it was obvious that the gooseneck went at the top of the inj&tor, several analysts installed it backwards with the gooseneck at the bottom. After trying to convince one chromatographer that he installed it backwards, he convinced us we had it wrong. When the gooseneck was installed at the base of the injector, the breakdown of sensitiv+?ompounds was greatly reduced. In ad@iuon, the response factors for higher molecular weight compounds were enhanced.. We still thought a gooseneck at the top of the injector was important too, hence the double gooseneck was invented!

How **does the double gooseneck sleeve work?**

Sample injection volumes greater than 2~1 tend to backflash out of the top of the injector and exit the septum purge. The top gooseneck keeps the sample contained in the sleeve, reducing the injector backflash. The bottom gooseneck serves a similar purpose. As the sample is injected, it is sprayed toward the bottom of the injector with the higher molecular weight compounds falling below the column inlet. This problem is accentuated with a rapid autosampler injection. Samples residing near the base of the injector are eventually drawn out when the splitiess purge is turned on. In addition, the sample stream is forced into contact with the metal inlet seal or injection port fittings, causing adsorption of active compounds. The bottom gooseneck chamfer contains the sample and reduces sample adsorption. The double gooseneck design effectively serves as a containment chamber and directs the majority of the sample onto the column, preventing it from being lost out of the top or bottom of the injector.

Reduced Breakdown **and Adsorption of Sensitive Compounds**

Sensitive compounds are adsorbed and degraded by exposure to hot metallic sites and poorly inert surfaces in the injection port. Sample exposure **to** the top of the injector is of little importance because the septum purge removes backflash vapors. Most of the catalytic effects occur at the base of the injector since the sample contacts this area for long periods of time. This problem is particularly pronounced in HP 5890 GCs because the column end sits only a few millimeters above the surface of the metal disk During the purge off mode, the sample is in intimate contact with the hot metal surface for up to two minutes.

To confirm the effectiveness **of the** double gooseneck sleeve, endrin, a chlorinated pesticide, was injected into a conventional4mm ID splitless sleeve packed with wool and into a double gooseneck sleeve. Endrin, a sensitive compound, is prone to breakdown on active or contaminated surfaces. The decomposition products of endrin, endrin ketone and endrin

Table 1 - Double gooseneck sleeves minimize degradation and adsorption of active compounds in an HP 5890 injector.

Sleeve type	endrin breakdown	
	clean seal	dirty seal
Splitless with Wool)	6.0%	12.8%
Double Gooseneck	2.0%	2.4%

Endrin degradation *doubles when a* contaminated inlet seal is installed in the injector with conven-*tional straight splitless sleeves, confirming thut the sample is contacting the hot catalytic metul surface. 7'he endtin breakdown data further illustrates that the double gooseneck effectively iso-* *lates the sample from the base of the injector and minimises catalytic effects*

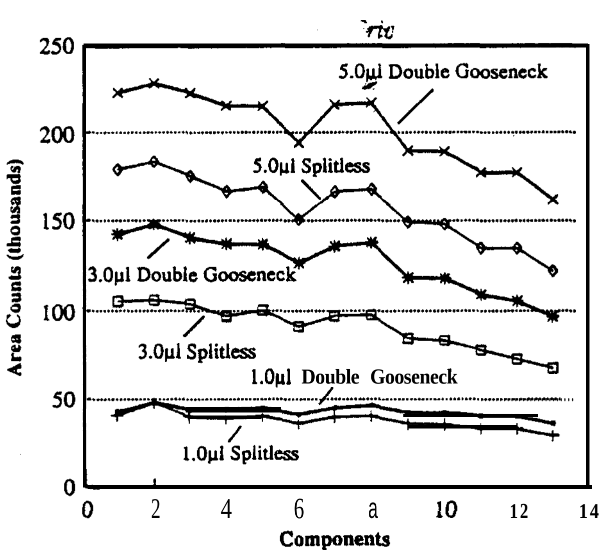
aldehyde were measured on each system. Table I shows the breakdown on the splitless sleeve with wool was three times higher than the double gooseneck sleeve. When the inlet seal was intentionally contaminated, the breakdown on the splitless sleeve doubled, while the breakdown on the double gooseneck sleeve did not significantly increase. This illustrates how effective the double gooseneck sleeve is in isolating the sample from the metal parts of a split/splitless injector.

Reduced Discrimination and Injector Backflash

Discrimination commonly occurs in split and splitless injectors and is indicated by a reduction of the area count for high molecular weight compounds. Sleeve design plays an important role in reducing discrimination. Thirteen polynuclear aromatic hydrocarbons (PNAs) were injected into a straight splitless sleeve packed with wool and then into a double gooseneck sleeve without wool. The area counts for high molecular weight PNAs are approximately 20% greater for the double gooseneck than with the splitless sleeve (Figure 2). This data indicates that the double chamfer prevents the high molecular weight discrimination and enhances complete sample vaporization.

The data in Figure 2 further illustrates that the double gooseneck reduces sample backflash from the top of the injector. The area counts for a 3ul injection are three times that of the lpi injection into a double gooseneck sleeve. However, the straight splitless sleeve only shows double the area counts even though three times more sample was injected. The 5u1 injections show that even the double gooseneck cannOt contain this large sample cloud, but it does deliver over 30% higher area counts than the straight splitless sleeve. Therefore, the top restriction of a double gooseneck is effective in reducing injector backflash when larger samples are injected.

Figure 2 - Double Gooseneck sleeves reduce discrimination of higher molecular weight compounds and reduce sample backflash when large volumes are injected.



List of Components

1. naphthalene
2. acenaphthylene
3. acenaphthene
4. fluome
5. phenanthrene
6. anthmcenc
7. fluomnthenc
a. p-cymene
9. benzo(a)anthracenc
10. chrysene
11. benzo(b)fluoranthene
12. benzo(k)fluomnthene
13.

The area counts for the higher molecular weight ***PNAs are greater for the double-goose sleeve than the straight splitless sleeve indicating less mass discrimination***

Injecting Dirty Samples

Splitless inlet sleeves are commonly packed with fused silica or borosilicate glass wool to trap dirty sample residue and decrease column maintenance requirements. However, packing double gooseneck sleeves with wool is difficult because of the small 0.8mm orifice. Cycle double goosenecks use glass screws in place of wool to fulfill this requirement. The glass screw traps dirt before it can enter the column and decreases the overall maintenance requirements for the system.

Improved Splitless Sleeve Design

Double gooseneck splitless sleeves show reduced breakdown and less mass discrimination over conventional straight splitless sleeve designs. They should be used with sensitive compounds such as endrin and high boiling compounds that are prone to mass discrimination.

If there's a topic you'd like to see covered in "Hints for the Capillary Chromatographer", write to:

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