

# Why Don't More People Use Guard Columns in Capillary Gas Chromatography? An Investigation into the Advantages and Disadvantages of Available Techniques

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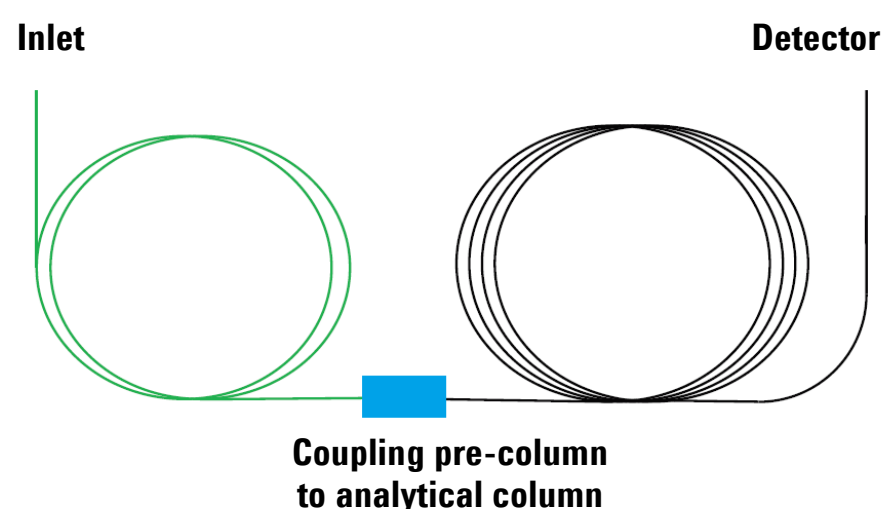
40<sup>th</sup> ISCC, Riva de Garda  
Poster E.13



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## Introduction

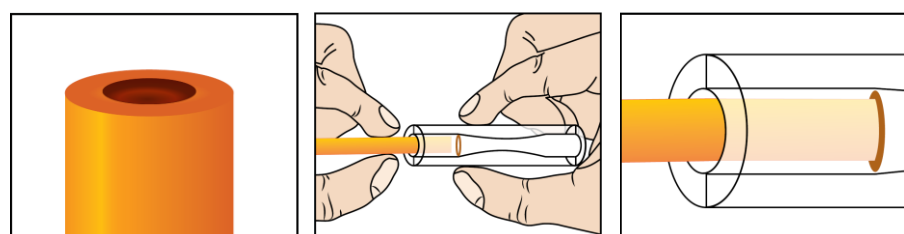
The addition of capillary tubing preceding the analytical column, either chemically passivated or stationary phase coated, may be used as a retention gap to improve peak shape or as a sacrificial guard column to protect the analytical column from matrix contamination. The supposition that the guard column protects the analytical column is based upon the idea that reactive or thermally labile matrix components will tend to degrade the stationary phase at the beginning of the column leaving the rest less affected.



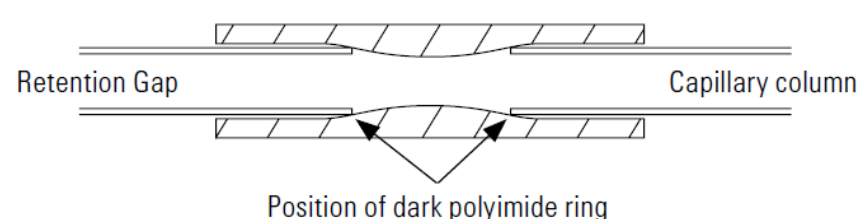
Many techniques are available for implementing guard columns. These include the use of press-fit connectors, low dead-volume unions, and integrated guard columns. In this paper, the advantages and disadvantages of each technique are explored in detail in an effort to reveal the reason why the guard columns are not widely applied in capillary gas chromatography.

## Press-fits

Installation of a press-fit is, by some users, classified to be cumbersome and requires some experience to make. First, the column needs to be inspected after cutting to ensure the column end is flat and burr-free. Second, the column should be pushed into the press-fit quite firmly. The connection must be inspected, showing a complete brown ring around the column indicating a leak-free connection. In addition, polyimide sealing resin may be applied to the connection. After a few power cycles leakage could occur, the connection should be inspected again.



In the past it was necessary to use press-fit connectors with dimensions to match the column outer diameter. Modern generic press-fit connectors are 'laser-milled' to provide highly reproducible taper angles throughout the length of the press-fit, ensuring a better sealing. Moreover, deactivated press-fits (Agilent Ultra Inert) are currently available to provide a robust and inert flow path.



Some benefits to using press-fit connectors are that they are relatively inexpensive, inert, and the sealing surface can be inspected visually. If used correctly, the press-fit provides a good leak-free connection.

## Conclusion & discussion

- Inert pre-column tubing has demonstrated benefits improving peak shape for splitless / on-column injections.
- They have also been shown to protect the analytical column stationary phase by retaining non-volatile matrix and saving on instrument downtime.

## Why use retention gaps?

An example demonstrating the effect of column contamination due to matrix injections is shown in the figure on the right. The figure shows the change in benzidine response and peak shape after the injection of multiple acidic soil extracts.

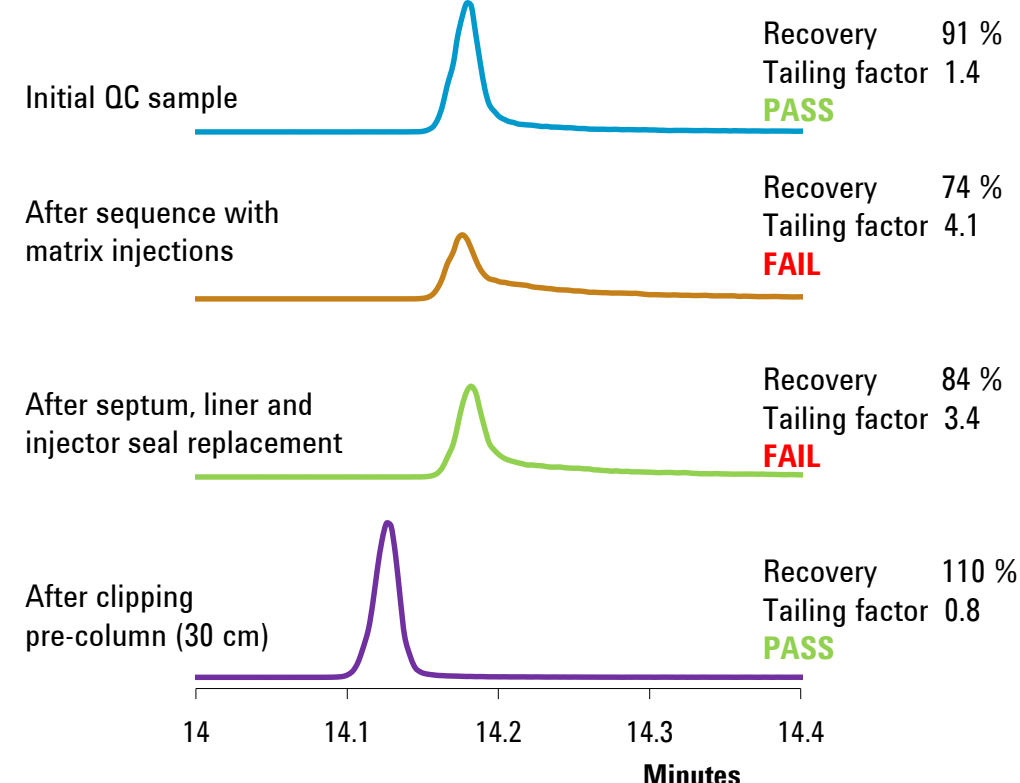
After inlet maintenance such as septum, liner and injector seal replacement benzidine recovery improved slightly, however not fully. Clipping the column (30 centimeter) shows a recovery. If retention gap were used in this case, it is anticipated that response and peak shape would be recovered without a change in retention time.

User feedback with benefits and disadvantages for using retention gaps are given in table below.

### Experimental

Agilent 7890B GC: 1 µl pulsed splitless injection at 300°C (60 psi for 0.5 min). Agilent J&W DB-5ms UI column: 30 m x 0.25 mm x 0.5 µm column at 2 mL/min constant flow [40°C (2min) at 20°C/min to 260°C – 6°C/min to 315°C]. Agilent 5977A MSD: source 230°C, quad temperature 150°C, scan mode full scan m/z 35 to 550. QC sample: benzidine 25 µg/mL in dichloromethane. Matrix sample: sedge peat extracted in methanol.

### Benzidine in QC sample



### Benefits of guard columns

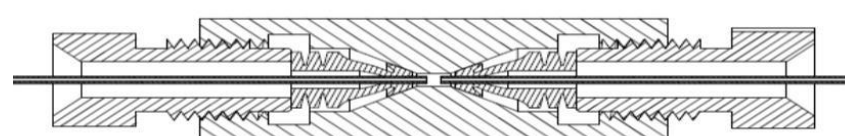
- Extends column lifetime, results in less downtime
- Reduces cost per analysis
- Sample focusing for better peak shape (on-column and splitless injections)
- Different polarity pre-columns available
- Inert versions available (Ultimate Plus fused silica tubing)
- No/less retention time shift compared to analytical column clipping or installation new column

### Reasons not to use pre-columns

- Installation and use requires some experience
- Adds extra connections, potential leakage and dead volume
- Added complexity, requires additional method optimization, understanding of maintenance intervals and integration in standard operation procedure
- Advantages and disadvantages for the multiple retention gap approaches unknown

## Low dead volume connectors

A more reliable, robust and high-quality connection compared to press-fits is achievable by using low dead volume connectors. As an example Agilent Ultimate Union kits results in a leak-free connection of the pre-column.



Although these low dead volume connectors also require some skills for correct installation, they eliminate time consuming curing step using polyimide glue compared to using press-fits.

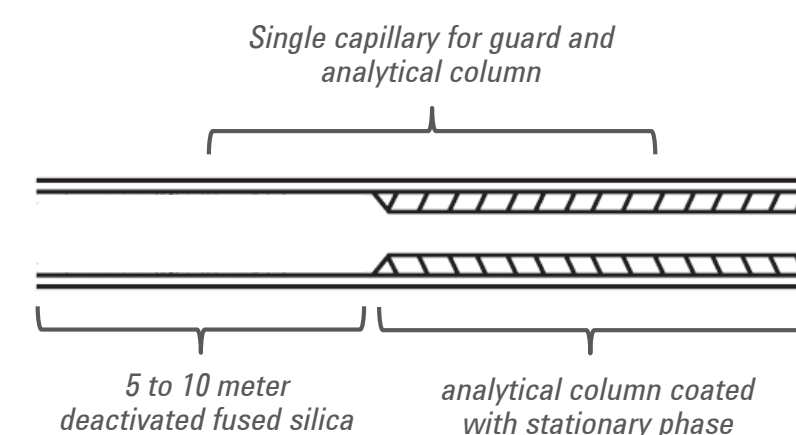


Some other advantages are:

- Strong, reliable and reusable mechanical seal based on Agilent proprietary MicroFluidics technology
- No need to inspect after temperature cycles like with press-fits or retighten as with other metal union designs
- minimal dead volume to prevent peak broadening
- One size fits fused silica column/tubing id 100 – 530 µm.
- Available in deactivated or non-deactivated versions

## Integrated guard columns

Integrated guard columns are an alternative to using press-fit and low dead volume connections. These columns contain a 5 to 10 meter section of deactivated fused silica preceding the analytical column.



The benefit of this technology is that it eliminates the need for a connection ensuring a leak free transition from retention gap to column. The use of an integrated guard column does not require any skill in assembly and no possibility of unswept or dead volume.

Disadvantages of integrated guard columns include limited availability of stationary phases and column dimensions. Also, once the retention gap is removed through multiple trimmings, the column may still be viable but the protection afforded by the retention gap is no longer present.

## References

Please check [www.agilent.com](http://www.agilent.com) for more information.



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