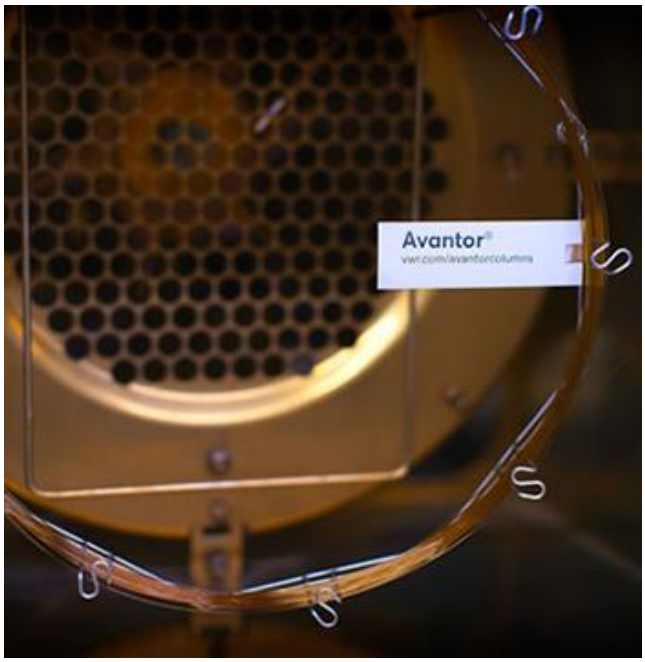
Chromatography Solutions

GC knowledge note # 001

GC Capillary Column Installation

INTRODUCTION

Correctly installing capillary GC columns into the column oven is a relatively straightforward process, however, incorrect installation can significantly impact the performance of the column. For a new column, a degree of initial preparation is required for correct installation to ensure optimal performance before its first use and to minimise stationary phase bleed during analysis. It is important that both the inlet and outlet of the column is correctly trimmed, with clean 90 degree burr free cuts. Poor quality trimming can result in degraded analytical performance and poor peak shape. Finally, it is also important to ensure that the correct ferrule is used for the application and to ensure that the overall instrument setup is correct. This knowledge note discusses the key steps involved to ensure optimum column performance and to maximise the column lifetime.



INSTRUMENTATION CHECK

It is worth verifying several key components of the system prior to column installation. At the inlet, the liner,

seals and column nuts should be checked and the inlet septum inspected prior to column installation. If the septum shows any sign of deterioration, coring or splitting, it should be replaced. Also verify that the septum material is correct for the intended application (for example, inlet temperatures >350 °C require high temperature septa).[1]

Damaged or degraded septa may cause gas leaks or deposition of septum fragments in the liner. At high temperatures, components may leach from the septa material and appear as extra peaks in the chromatogram. In the case of GC-MS, these will exhibit characteristic polysiloxane fragment ions (e.g. *m/z* 73, 147 and 281).[2] Gas purity traps should always be used on the carrier gas lines as this will help to minimise background noise and extend the column's lifetime. These should be checked for oxygen and humidity before commencing an analysis and replaced if necessary.

As a general point, keeping on top of routine maintenance and replacement of GC consumables at appropriate intervals for your application will reduce the need for time consuming troubleshooting and help to extend the column lifetime.

FERRULE SELECTION

A variety of ferrule materials are available for different applications and are typically manufactured from graphite, Vespel® or a composite of the two. Table 1 presents the characteristics of some common ferrule options.

TRIMMING THE COLUMN

New Avantor® Hichrom GC columns are sealed under an inert gas after manufacture and quality control testing. Prior to first use, 1-2 cm should be cut from each end of the column using a ceramic scribe or capillary cutter and a nut and ferrule positioned on each end of the column. An additional 1-2 cm should be trimmed from the column after positioning of the nut and ferrule to remove any ferrule fragments from the end of the capillary.

When trimming the column, it is essential to ensure that good, 90 degree, burr free cuts are made to the capillary column. Figure 1 shows examples of good and bad cuts. To make a good cut, the outer polyimide coating of the capillary should be scored 1-2 cm from the end using the edge of a ceramic wafer/scribe (Figure 2A) or sapphire

Table 1: Ferrule materials and characteristics for capillary GC columns.[3]

Material	Typical temperature limit	Characteristics	Notes
Graphite	450 °C	High temperature limit, no bleed. Easily deformed and forms a reliable seal. Limited reusability.	General purpose for FID, NPD and ECD. Cannot be used for GC/MS due to air permeability.
Vespel®/graphite (85:15)	350 °C	Composite ferrule. Long lifetime, reliable leak free connections. Not reusable.	General purpose for GC, recommended for GC-MS applications. Ferrule hole must match column outer diameter exactly for correct seal. May require re-tightening after initial temperature cycle.
Vespel® (100% polyimide)	350 °C	Non-reusable, long lifetime. Non-porous to oxygen. Tends to shrink through repeated heating cycles.	May leak and require re-tightening after temperature cycling. Used for isothemal connection to the GC-MS interface, where insulated from oven temperature changes. Can fuse to fused silica GC columns

knife. These typically have two edges, the smoother of the two edges should be used to score the capillary. Once scored, hold the capillary just below the score line and gently push or flick the end of the capillary downwards, to avoid any fragments entering the column away from you.

Alternatively, a capillary column cutter (Figure 2B and C) can be used, where the capillary is inserted into the cutter, secured using the securing disc and a cutting disk rotated around the capillary to cut.

Once trimmed, carefully inspect the cut end using a magnifier to ensure that it is has a clean cut perpendicular to the capillary and that no burrs or jagged edges are present. If the cut is not clean, the capillary should be re-cut until a clean cut is obtained. If a good quality cut is not made, peak shape issues may be apparent, such as split or tailing peaks.

For previously used capillary columns, remove any capillary caps from the ends of the column and position the nut and ferrule on the column as above and trim 1-2 cm from the column. If the column previously showed loss of efficiency or poor peak shape, a longer length of capillary may be removed to attempt to recover performance, however, a record of trimmed column length should be maintained for reference and so that accurate column lengths can be configured in the GC operating software.

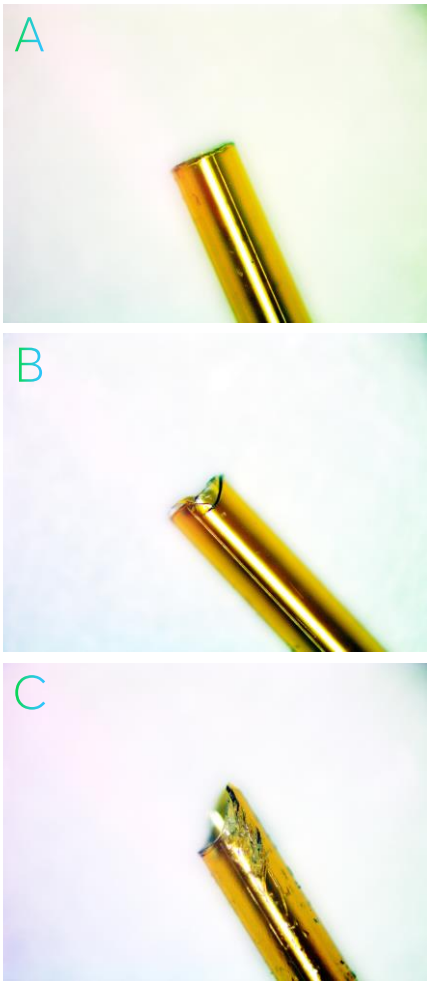


Figure 1: Images showing A. good quality, 90 degree, burr free cut and B&C. poor quality cuts made to a capillary GC column.

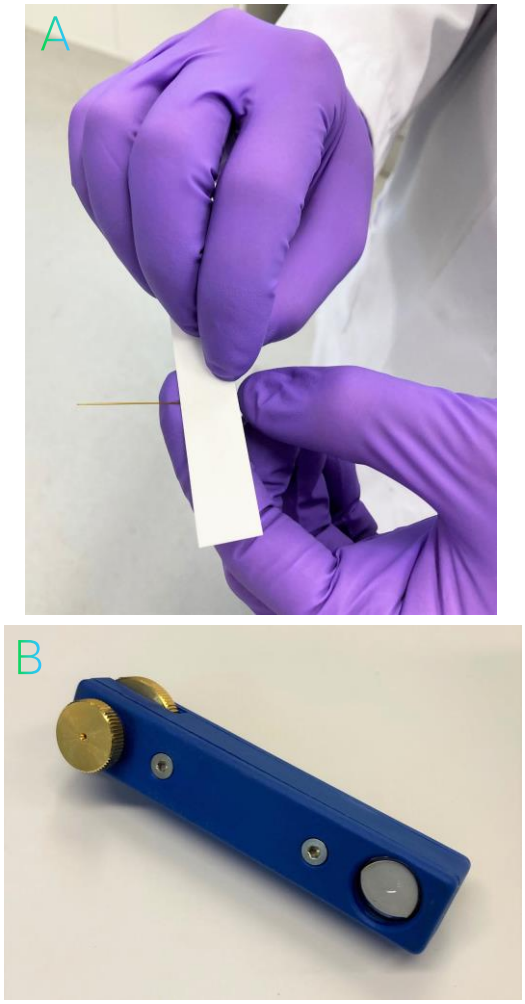


Figure 2: A. scoring a GC capillary column using a ceramic wafer and B. a capillary column cutter.

INSTALLING THE COLUMN

Before installing the column, ensure that the heated zones of the GC are cool and carefully place the column cage on the hanger inside the column oven. For GC-MS systems, please refer to the manufacturer guidance to ensure that the system is suitably cooled and vented before de-installing and installing a column. When installing, avoid any sharp bends or stress of the capillary column, or contact with sharp surfaces. Begin by connecting the front end of the column into the GC inlet, at the instrument manufacturers recommended insertion distance and finger tighten the nut, followed by a further 1/4 of a turn using a spanner. Additional tightening may be required after the first temperature cycle, however, care should be taken not to overtighten the fitting as cross threading may occur.

It is essential that the insertion distance of the capillary column into the inlet/injector is correct (A in Figure 3) as this will vary between instrument manufacturers and if not measured correctly, will result in poor column performance. Note that the insertion distance may be specified as either the distance from the end of the ferrule to the end of the capillary or the injector end of the column nut to the end of the capillary. The column can be purged for a short duration (1-2 minutes) to remove any particulates that may have entered the column during trimming. When using hydrogen as the carrier gas, use caution as hydrogen is explosive; follow all necessary safety precautions when using hydrogen, do not allow the oven to fill with hydrogen and ensure that all heated zones of the GC instrument are cool. The column outlet can now be installed into the detector (check the detector type and instrument/detector manufacturer for the correct capillary insertion length into the detector). Once installed, check for the presence of leaks using a leak detector. If leaks are found, try gently tightening the inlet and outlet nuts. If this doesn't resolve the leak, re-install the column using new ferrules.

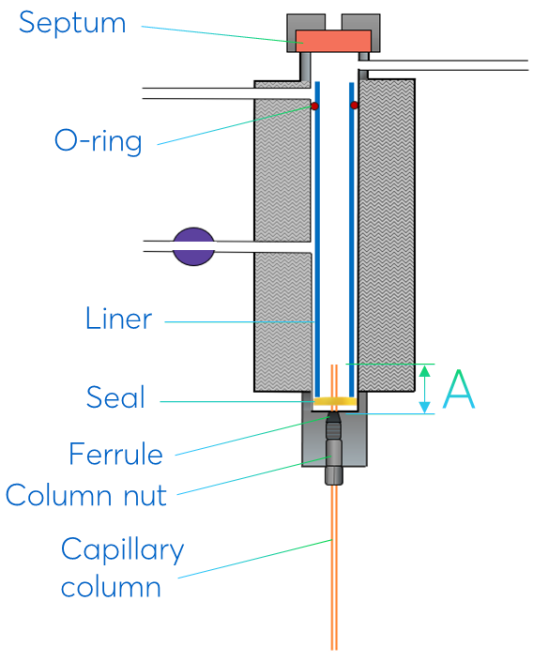


Figure 3: Schematic diagram of column installation at the GC inlet. It is important to ensure that the insertion distance (A) is measured accurately, according to the GC instrument manufacturers recommendation. Note that the insertion distance may be specified as either the distance from the end of the ferrule to the end of the capillary or from the injector end of the column nut to the end of the capillary.

COLUMN CONDITIONING

For new columns, once the column is installed and has been checked for leaks, it should be purged with carrier gas for 20-30 minutes, at an intermediate flow rate within the columns flow limits, to remove any oxygen and avoid oxidising the column. It is now ready to be conditioned.

Avantor® Hichrom GC columns are pre-conditioned during quality control procedures. An additional short conditioning cycle is additionally recommended, using a 5-10 °C/min ramp to the columns upper isothermal temperature limit, or a temperature 10-20 °C higher than the highest operating temperature of your specific analytical method, and maintaining this temperature for 20-30 minutes until a flat baseline is established.

Carrier gas flow should be maintained at all times during conditioning when the column is above ambient temperature. Do not exceed the upper temperature limit of the column or phase damage will occur.

The temperature limits are shown on the column label, in some cases two maximum operating temperatures are specified. The lower temperature is the maximum for isothermal conditions and the higher temperature is the maximum for temperature-programmed conditions. Note that for some detectors (e.g. ECD & MS), conditioning the column before connecting to the detector is recommended (please refer to your instrument user manual for guidance). If an elevated/noisy baseline is

observed, additional column conditioning may be required.

TESTING COLUMN PERFORMANCE

Once the column has been conditioned and an acceptable baseline has been obtained, set instrument parameters to a suitable method and perform a test injection to assess column performance. Avantor® Hichrom GC columns are tested with the original Grob test mixture (or a dedicated mixture for custom columns) as a quality control test. Once installed, the column installation and performance can be verified by injecting the Grob test mix using the chromatographic test conditions specified on the columns Test Chromatogram.

COLUMN STORAGE AND CLEANING

For long term storage of the GC capillary column once removed from the GC system, the capillary ends should be flame-sealed or sealed using a suitable column cap or insertion into a septum and the column kept in its original box.

The use of retention gaps (a length of deactivated fused silica tubing) is highly recommended in the case of analysing "dirty" and/or non-volatile samples. Any non-volatile sample components or particulate matter will accumulate within the retention gap and therefore protect the column and extend the column's lifetime.

Deterioration in column performance can occur through contamination of the column, through the accumulation of non-volatile sample components and particulates on the column at the column inlet. This may be indicated by the adsorption and tailing of active analytes, excessive bleed or alteration of the retention characteristics of the column.[3] If this occurs, trim 1-2 cm from the inlet end of the column, which could recover the column's performance. In some cases, longer lengths of column may need to be removed.

If this does not recover the column performance, then the column can be re-conditioned (or "baked out") at 10-20 °C below the columns upper temperature limit for at least 1-2 hours. Carrier gas flow should be maintained at all times during this procedure. In extreme cases, re-conditioning overnight may be required.

CONCLUSION

Correct column installation is very important for achieving good quality, reproducible results in capillary GC. Column installation is relatively straightforward and trouble-free as long as key sequential steps are followed. This technical note has outlined the key steps in trimming, installing, conditioning and testing that are required to ensure that the column is correctly installed in the GC instrument for optimum performance. As an additional point, ensuring regular routine maintenance and replacement of GC consumables at appropriate intervals is carried out, along with regular inspection of the GC system as a whole (e.g. injector, detector, gas traps etc.) will reduce the likelihood of problems occurring and help to extend capillary column lifetime.

REFERENCES

1. "The LCGC Blog: GC Inlet Maintenance...have you really heard it all before?," 2014. [Online]. Available: https://www.chromatographyonline.com/view/lcgc-blog-gc-inlet-maintenancehave-you-really-heard-it-all.

2. D. Rood, "Gas Chromatography Problem Solving and Troubleshooting," *Journal of Chromatographic Science*(2001) **39**, 309-310.

3. E. F. Barry and R. L. Grob, Columns for Gas Chromatography Performance and Selection, New Jersey: John Wiley & Sons Inc, 2007.