Technical Note 20684

GC Column Installation, Conditioning, Storage, and Maintenance

Peter Morgan, Thermo Fisher Scientific, Runcorn, Cheshire, UK

Key Words
Column, installation, conditioning, storage, maintenance

Abstract
To achieve optimum performance from your GC column, it must be installed and prepared for use correctly. Here we discuss how to achieve the optimum performance and peak shape from your column.

Introduction

To achieve the optimum performance from a GC system, correct column handling procedures must be followed. Incorrectly installing a new GC column can severely compromise performance and, in the worst case, permanently damage the stationary phase. This will be seen as poor peak shape and reduced column lifetime.

In this guide we discuss:

- Correct installation of the column into the instrument
- Best practice procedures for conditioning new columns
- Long and short term column storage procedures
- Basic column maintenance

Thermo SCIENTIFIC

Column Installation

All new Thermo Scientific™ GC columns are supplied with their ends sealed. This is performed either by inserting the column end into a septum or by fusing the end and covering with a metal cap as shown in Figure 1. The purpose of this is to make an air-tight seal at both ends of the column, preventing air or moisture from the atmosphere from entering the column. If air or moisture are able to enter the column, oxidation of the stationary phase will occur, severely affecting column performance and lifetime.

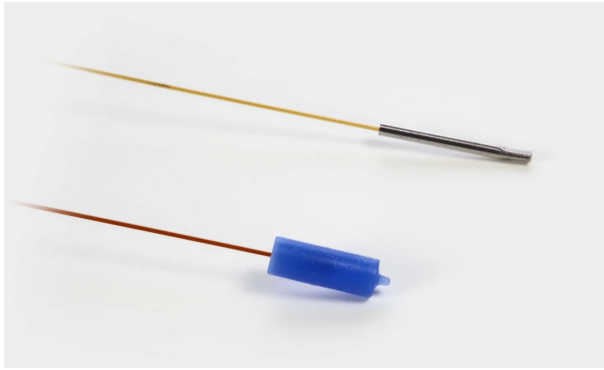

Figure 1: The two types of column seal used on Thermo Scientific GC columns. The top column uses a metal end cap found on the Thermo Scientific™ TRACE™ range of columns. The lower column is a Thermo Scientific™ TraceGOLD™ column sealed with a septum.

During installation approximately 3 cm (1 inch) of column must be removed from both ends to remove any septum material that may be blocking the column. This ensures the column is open and ready for use.

To install the column follow the protocol given below:

1. After taking out the old column, remove any graphite fragments present in the injector and detector ports. Now is also a good time to replace the Gold Inlet Base Seal (if present on your system) and change the inlet liner and septum.
2. Make sure the end caps/septum have been removed from both ends of the new column as previously discussed (cutting of the column may be required to do this).
3. Trim a minimum of 2 cm from both ends of the column and check that a clean square cut has been made. See Figure 2.

Figure 2: Cutting a fused silica column using a ceramic tile. For further details refer to the "GC Column Cutting Mini Guide" [1].

4. Install the front end of the column into the GC inlet*. It is essential to make sure the correct insertion depth is used for your system and injection type**.

   First tighten the inlet nut finger tight followed by one-third of a turn with a spanner. Over-tightening of nuts can lead to damaged ferrules or the build up of graphite within the inlet/detector, which may block the column.

Figure 3: In this photo the font/inlet end of the column in on the right and wound on the top of the cage. The rear/detector end is seen on the left.

5. Turn on the carrier gas and check there is a good flow. This can be tested by dipping the detector end into some methanol and checking for bubbles.
6. Install the detector end of the column into the detector.
7. Check for the presence of leaks using a leak detector such as a Thermo Scientific™ GLD Pro™ Gas Leak Detector (P/N 66002-001). If a leak is found from a fitting, try tightening the inlet or detector nuts gently to achieve a better seal. If this does not resolve the leak, try removing the connection and re-installing with a new ferrule.
8. Check that the correct column dimensions are entered into the GC software. If incorrect values are entered the instrument may use inappropriate flow rates, which will compromise performance.
9. Allow the column to purge for twenty minutes to expel any oxygen that has entered the column.

When using an MS detector it is good practice to condition the column without the column installed in the detector. This is to prevent the column bleed exhibited during conditioning from contaminating the ion source. This will improve sensitivity and increase maintenance intervals.

*Although GC columns are symmetrical, by convention the end on top when the cage is laid down with the writing facing up is installed into the inlet and the bottom into the detector, see Figure 3. This is essential if using an integrated guard column to ensure the un-coated end is inserted into the inlet.

** It is recommend you use a measuring tool to make sure this is done correctly, otherwise poor peak shape and injection reproducibility may be seen. Refer to your instrument manual for correct insertion depths, the Thermo Scientific TRACE GC version is shown in Figure 4.

Figure 4: Thermo Scientific Tri-Scale Ruler (P/N 60180-783) for measuring column insertion depth on the TRACE GC.

Column Conditioning

All Thermo Scientific GC columns are pre-conditioned; however, it is essential to re-condition them prior to use. If re-conditioning is not performed, high, unstable baselines may be seen, which may not improve with subsequent conditioning.

GC columns have two maximum temperatures stated. The lower value is the isothermal maximum, i.e. the temperature the column can tolerate for extended periods of time. The higher value is the temperature the column can tolerate for short period of time (less than ten minutes) without sustaining damage. This is the maximum temperature for temperature gradient methods. For column conditioning use the isothermal temperature as the maximum.

To condition a column, first install the column into the instrument. Then either refer to the column documentation or follow the steps below:

1. Heat the column from 50 °C to the maximum temperature at 5 °C/minute and hold for 1 hour.
2. If using a non-MS detector monitor the baseline during this time. If the baseline is still gradually falling after 1 hour consider extending the hold until a stable baseline is achieved. If using longer columns (> 30 m) or with thicker stationary phases (> 0.5 µm) conditioning may take longer due to the increased amount of stationary phase present.
3. If using an MS detector the column can be installed into the detector at this point
4. After conditioning run a blank sample and check for a stable baseline
5. To check for correct installation run a standard and check peak shape. Tailing or the presence of anomalous peaks indicate incorrect column installation.

Column Storage

If a column is no longer required, it may be stored for short periods inside the instrument. For long term storage, remove the column and place it in the column box.

If storing in the instrument, a low flow of carrier gas must be maintained constantly with the split flow switched on. If the carrier gas or split flow are switched off, back diffusion of air will occur through the split and septum purge lines. This air will enter the column and cause damage. It is also recommended to keep the oven temperature at 60 °C or greater to prevent the build-up of moisture in or around the oven.

When removing the column for storage follow the procedure below:

1. Remove the column from the detector.
2. Insert the detector end into an inlet septum or a Thermo Scientific Capillary Column End Cap*
3. Turn off the carrier gas and remove the column from the inlet.
4. Quickly insert the inlet end into a septum or a Capillary Column End Cap.

Following this process minimizes the opportunity for air to enter the column. When re-installing the column it may be necessary to repeat column conditioning. To assess this, inject a blank and inspect the baseline.

Figure 5: Thermo Scientific Capillary Column End Caps (P/N 260EC111)

*Capillary Column End Caps work in a similar way to a "press-fit" column union but are closed at one end (see Figure 5). The advantage of these is that the column will not require trimming prior to re-installation as is necessary when sealing with a septum. This is of particular use when using Vespel® or metal ferrule types that may be difficult to remove. These can then be re-used in the original position.

Column Maintenance

Column Trimming

GC columns require regular maintenance to preserve optimum performance. Samples analyzed by GC often contain involatile material that becomes permanently retained by the column. Also some compounds can degrade the stationary phase. Both of these problems will be seen as deteriorating peak shape, usually in the form of tailing peaks and unstable baselines.

Fortunately the involatile material and stationary phase damage mostly occurs at the head of the column, i.e. the inlet end. To restore the performance of the column it is necessary to remove the damaged portion of the column.

Many analysts believe that shortening the column by trimming will reduce the resolving power of the column. Although this is true, it will only have a significant effect on resolution if large sections are removed (> 20% of the total column length); however, this is unlikely to occur within the lifetime of the column. For a more detailed explanation please refer to appendix A.

The length of column that must be removed depends upon the amount of damage that has been sustained. Begin by removing approximately 15 cm. If only a small improvement is seen by removing 15 cm then removing a further 30 cm will often restore performance.

For instructions on how to trim a GC column please refer to the "GC Column Cutting Mini Guide".

"Baking Out" the Column

Another technique to remove high boiling point contaminants from the column is by heating the column to a high temperature. This involves heating the column to its isothermal maximum temperature and holding for several hours. If this temperature is above the boiling point of the contaminants they should then elute from the column. The time required at the high temperature is dependent upon the concentration and nature of the contaminants.

Leak Checking

A major source of column damage is from air leaks. If a system is not leak tight then oxygen is able to enter the column, rapidly degrading the stationary phase. For this reason it is essential to regularly check the system for the presence of leaks. Repeat the procedure whenever a new connection is made, such as after column trimming.

If using an MS detector, leak checking is easy as the tuning software page normally includes a leak detection function. If a non-MS detector is used, or a leak is found on the MS software, it is then easy to check for leaks from gas line fittings and column connections using a leak detector such as that in Figure 6.

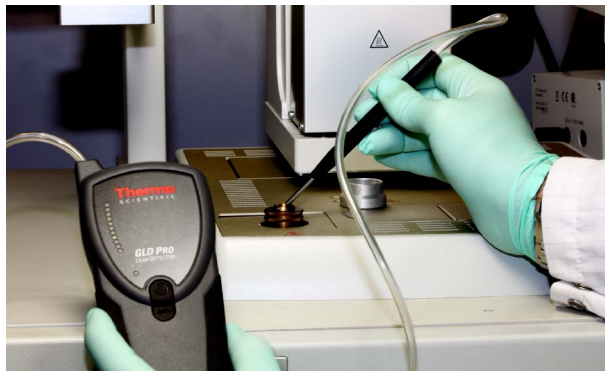

Figure 6: Thermo Scientific GLD Pro Gas Leak Detector (P/N 66002-001). Leak detectors such as this are highly sensitive and have a visual and audible alarm to signal when a gas leak is detected.

Conclusion

By following the steps given above you can be confident that the performance and lifespan of your new GC column has been maximized.

Reference

[1] Thermo Scientific Technical Note 20778

Appendix A

The effect of Column Trimming on Peak Resolution

As mentioned in the Column Trimming section some GC users believe that column trimming may have a detrimental effect on peak resolution. However, if we consider that resolution is not directly proportional to the column length, but determined by the square root (√) of the efficiency. Therefore halving the column length will halve the efficiency, but resolution will only be reduced by the square root of this.

For example, if we have an efficiency of 50,000 for a 30 m column. The same 15 m column will have an efficiency of 25,000. This column has 16.6 plates per cm.

The √50,000 = 223, and the √25,000 = 158. If the 30 m column gives us a resolution value of 2 between two peaks the 15 m column will give us a resolution value of 1.4 not the resolution value of 1 that you would logically expect by halving the length of the column.

If we take the same 30 m column and the same two compounds with a resolution value of 2 and remove 30 cm of the column (1% of the total column length) the efficiency will be reduced by 500 plates to 49,500. These 49,500 plates would give us a resolution of 1.98. This would not result in any noticeable loss of resolution.

If we were to remove 3 m (10% of the total column length) the expected resolution would be 1.8. Again this is not a significant loss of resolution.

To demonstrate this, an experiment was performed with a 30 m 5% phenyl methylpolysiloxane column analyzing a mix of 16 PAHs. The two compounds in this mix, benzo(b)fluoranthene and benzo(k)fluoranthene, are known to only partially resolve on this stationary phase.

The initial resolution between these compounds was calculated as 0.95. The column was then trimmed and the mix re-analyzed until a significant drop in resolution was observed. It was not until 13 m had been removed (43% of the total column length) that an obvious visible reduction in resolution was seen. The final resolution was calculated as 0.75.

It was calculated that removing 13 m of the column should have given a 25% reduction in resolution. The results showed a 21% drop. Figure 7 shows the loss in resolution between these two compounds.

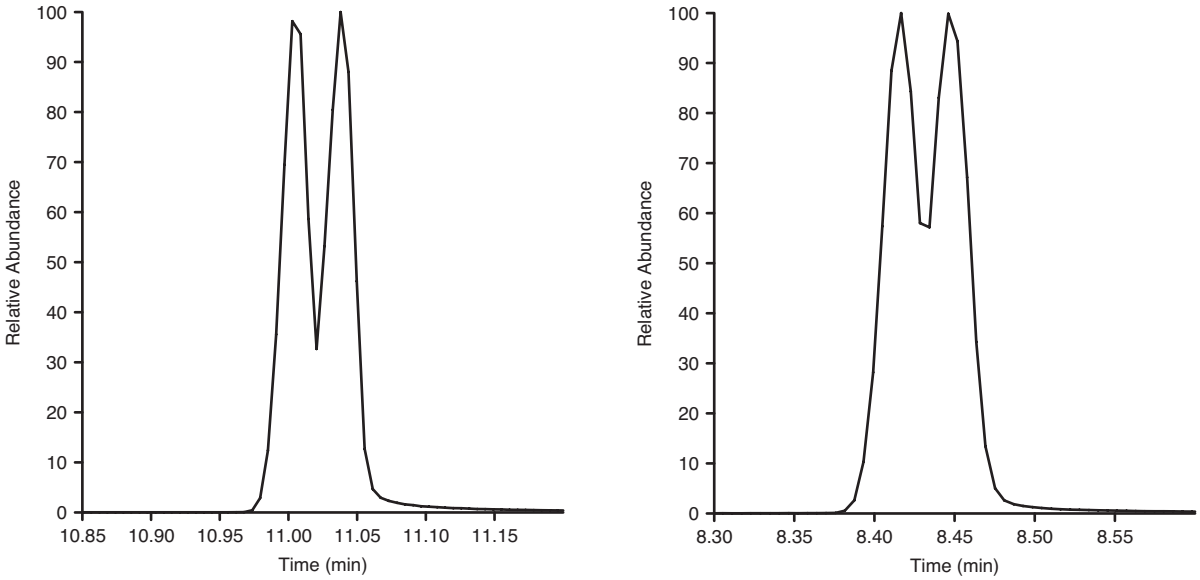

Figure 7: Resolution between benzo(b)fluoranthene and benzo(k)fluoranthene. The left chromatogram shows the resolution using new 30 m column, and the right chromatogram shows the resolution after removing 13 m of the column. This work was carried out using a Thermo Scientific TRACE TR-5MS 30 m x 0.25 mm x 0.25 µm column.

This shows that removing short sections will not significantly impact resolution. Considering that tailing peaks will quickly cause peak co-elutions it is clear that column trimming is an essential part of GC column maintenance.

thermoscientific.com/columnsforgc

USA and Canada +1 800 332 3331
France +33 (0)1 60 92 48 34
Germany +49 (0) 2423 9431 20 or 21
United Kingdom +44 (0)1928 534110
Japan +81 3 5826 1615

China +86 21 68654588 +86 10 84193588
+86 20 83145199 800 810 5118
India +91 22 6742 9494 +91 27 1766 2352
Australia 1 300 735 292 (free call domestic)
New Zealand 0800 933 966 (free call domestic)
All Other Enquiries +44 (0) 1928 534 050

Technical Support
North America +1 800 332 3331
Outside North America +44 (0) 1928 534 440

Thermo SCIENTIFIC
Part of Thermo Fisher Scientific

TN20684_E 05/13S