GC Troubleshooting Series: Column Installation

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What you'll need to have on hand to change a column:

- Column cutter
- Magnifying glass
- Methand or Butane (for testing)
- Electronic flowmeter
- Electronic leak detector

It may also be useful to have a test mix on hand to run when you are done installing your column.
At column change time, it's also a good time to:

- Check gas traps for expiration and replace if necessary.
- Change the inlet consumables: Install a new septum in the inlet. Inspect and change liner, if it is visibly dirty. Sometimes even though it looks clean the liner needs to be replaced because it has become active. Use a new O-ring when you replace the liner.

Agilent also recommends using traps on the carrier gas lines as an extra measure of protection against contaminants and therefore extend column lifetime and minimize background noise. Our gas management video covers more information about that. Agilent sells a number of gas purifiers and systems... go to http://www.agilent.com/chem/gasmanagement for more information.

Check out the gas management best practices video at www.agilent.com/chem/gasmgtvideo

Safety tip: Always wear safety glasses when cutting columns.

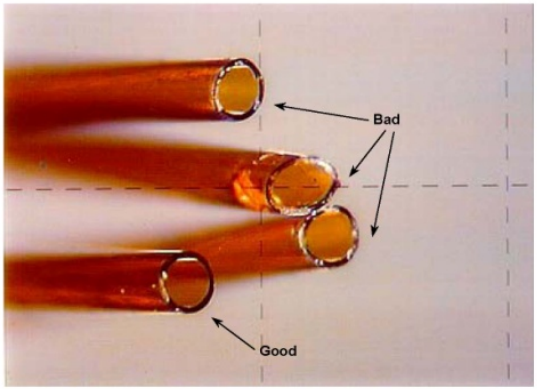
Steps to install a column into the inlet:

1) Place the column on the GC oven column hanger. Make sure tubing does not touch the sides of the oven.
2) Unwind enough column to obtain a smoothly curved section of tubing to connect to the inlet. Avoide tight bends, as this stresses tubing and causes breakage.
3) Place the column nut and ferrule over one end of the column.
4) Cut the end of the column after the nut and ferrule placement so as not to leave any ferrule material at the end of the column.
5) Cleanly cut the column at a right angle to the tubine wall, so it is free of chips, burrs, excess polyimide, etc.
6) For split/splitless inlets, insert 4 – 6 mm above the ferrule.
7) Wipe the end of the column with a clean wipe and some methanol or acetone.

8) With the column at its proper position, finger tighten the column nut. Use a wrench to tighten an additional 1/2 turn. If the column can be moved in the fitting, tighten another 1/4 turn. Failure to achieve a leak-free seal will cause rapid and permanent column damage when you heat the column

You can tell the right edge of the ceramic wafer cutting tool by feeling the edges of the wafer. Place the smooth edge towards the column when you cut.

Examples of Column Cuts



To find Agilent 100% Vespel ferrules, and other high quality inlet supplies, visit www.agilent.com/chem/gcsupplies

Ferrules will change shape slightly upon heating, so it's a good practice to check and retighten the nut after conditioning the column.

Set the head pressure at the right rate for your columns. Settings vary. For a 30-meter column, using helium carrier gas, head pressure should be 15 - 25 psi for a 0.25 mm ID column. These recommendations are starting points only.

Installing the column into the detector: Follow the same basic steps as for the inlet installation (steps 3-7).

When installation is complete:

1) Confirm all detector gas flows with an accurate flow-measuring device (Learn about Agilent's Precision Flow Meter at www.agilent.com/chem/flowmeter).
2) Check for leaks in the system. The Electronic Leak Detector (http://www.agilent.com/chem/leakdetector) has audible and visual alerts for leaks as small as 0.01 ml/min.
3) Enter flow rate. Electronic pressure control (EPC) allows direct entry of carrier gas linear velocity or flow rate. It is critical that correct column dimensions are entered into the PC software or via the GC keypad for accurate velocity or flow values to be set. Check the *Column Performance Summary Sheet* that comes with your column.
1) Check the system with a non-retained compound: With the column temperature at 35 to 40° C, rapidly inject 1 to 2 µL of a non-retained compound with the split/splitless inlet *in the split mode*. If using Megabore direct or cool on-column modes, dilute the non-retained compound so that the sample will not saturate the detector. A very sharp and symmetrical peak should be obtained; a small amount of peak tailing may be observed with splitless injection.

Tip: Using a butane lighter is a quick-and-easy way to do this test.

If no peak appears, there may not be any carrier gas flow. Check the regulators, gas supply and flow controllers for the proper settings. Make sure that the detector, recorder, and syringe are functioning properly. If the non-retained compound is tailing, there may be a leak in the inlet, poor column installation, or an excessively low split ratio. Reinstall the column, check the settings and check the inlet for leaks. A non-tailing peak is required before continuing.

Conditioning the column:

Never heat a column without carrier gas flow or while oxygen is present in the carrier gas. This will rapidly and permanently damage the column.

Steps to condition a column:

1) Make sure all oxygen is purged from the system.
2) Heat the column to its upper temperature limite, or a temperataure 10 - 20° C above the highest operating temperature of the method, whichever is lower. Do not exceed upper column limit.
3) Observe the baseline. You may observe some artifacts (peaks) but they shouldn't be persistent.
4) After the column has reached the conditioning temperature, observe the baseline.
5) If it continues to rise as you hold temperature, check for leaks. If it rises and then falls, it may indicate the elution of some contamination.
6) A flat baseline is the goal. This should be able to be achieved between 15 minutes – over an hour of time.
7) If the baseline does not stabilize in 2 – 3 hours, stop the process and check for leaks and/or system contamination. Cool the instrument down before continuing.

For capillary columns, the average linear velocity (μ) is a better and more meaningful measure of the carrier gas than the volumetric flow rate.

Always set carrier gas average linear velocity at the same temperature for a given analysis (often the initial oven temperature).

It is a good idea to run a blank run when you are done conditioning your column. This can be useful for troubleshooting later on.

Use your standard temperataure program, or start at 40 to 50° C, ramp at 10 to 20° C/min and hold for 10 to 15 minutes at the conditioning temperature. Save this background trace for future comparisons.