Agilent J&W GC Column Installation Guide

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Trusted Answers

These instructions are suitable for the majority of the capillary columns that Agilent manufactures. There may be more specific conditioning, care and maintenance procedures for your new GC column (e.g., PLOT columns). Read all the information that comes with your new GC column to ensure that the column performs to expectations.

A fused silica capillary GC column (Figure 1) consists of:

- An amber-brown polyimide exterior coating that protects the tubing from breakage.
- The fused silica tubing.
- A stationary phase that is evenly coated onto the inner wall of the tubing. Common phases are silicon-based polymers (polysiloxanes), polyethylene glycols, and solid adsorbents.

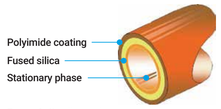

Figure 1. Capillary column

Maximize capillary GC column performance and lifetime by following these recommended guidelines for proper installation.

Tools for capillary column installation

- Column cutting tool such as a diamond-, carbide-, or sapphire-tipped pencil, or a ceramic cleaving wedge
- Supply of an appropriate nonretained compound
- Column test mixture (optional)
- Electronic flowmeter (optional)
- Electronic leak detector (optional)

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Inertness of the GC Flow Path is critical for accurate reliable analysis. Learn more:
www.agilent.com/chem/inert

1. Check traps, carrier gas, septum and liner

Check gas traps for expiration and replace if necessary. Install a new septum in the inlet. If needed, clean or replace the inlet liner and gold-plated inlet seal, especially after injecting dirty samples or when analyzing active compounds.

The liquid phase in the column is easily damaged by oxygen at temperatures above ambient. Use traps on the carrier gas lines to extend column lifetime and minimize background noise. A high capacity oxygen trap and an indicating oxygen trap are highly recommended. An indicating moisture trap before the oxygen trap prolongs the life of the oxygen trap and reduces background noise. An oxygen trap on the ECD makeup gas line is recommended.

2. Place nut and ferrule on the column, carefully cut column end

Place the column nut and ferrule over one end of the column. Typically, there is no front or back of the column, however, the posts of the column cage usually point towards the oven door. A few columns have a front and back end (e.g columns with guard column & particle trap). For these columns, it is critical to follow the front and back end indicator labels on the columns to ensure proper performance.

Cut the end of the column after nut and ferrule placement. Hold the section of column to be cut against a finger. In one motion, scribe the outside of the column using a suitable cutting tool. Do not cut completely through the tubing. Grasp the column on each side of the scribe mark and bend away from the mark. Inspect the column end with a magnifier. Ensure the cut is at a right angle to the tubing wall and free of chips, burrs, or uneven areas (Figure 2). Recut if necessary.

WARNING: Always use caution when cutting capillary tubing. WEAR SAFETY GLASSES.

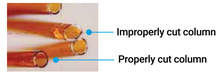

Figure 2. Cutting a fused silica capillary column

3. Install column into the inlet

Place the column on the GC oven column hanger. Make sure the column tubing does not touch the sides of the oven.

Unwind enough column to obtain a smoothly curved section of tubing connected to the inlet. Avoid tight bends as this stresses the tubing and could cause breakage. Make sure that column tags, sharp edges, or other items do not rub against the column.

The optimal insertion distance of the column into the inlet depends on the inlet type. Consult the GC instruction manual for the proper insertion depth and technique.

With the column at its proper position, finger tighten the column nut. When using Agilent's self-tightening nut finger tightening is all that is required. DO NOT use a wrench when using self-tightening nuts – this will strip the nuts and cause leaks. For standard nuts (not self-tightening) 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. Do not move the column while tightening the nut.

Ferrules, especially those made of graphite/polyimide, will change shape slightly upon heating. If the column was installed while the inlet and detector were cool, retighten the fitting. It is also good practice to make sure the column nuts are tight after conditioning the column.

4. Turn on carrier gas

Adjust the head pressure to obtain a reasonable flow rate of carrier gas (Table 1). These values are recommended as *starting points only.* The actual head pressure will depend on the carrier gas velocity or flows set in Step 9. Place the free end of the column in a small vial of hexane. A steady stream of bubbles should be visible. If bubbles are not seen, check the carrier gas supply, flow controllers, etc. for proper settings and for leaks. Wipe off any residual solvent before continuing.

Table 1. Approximate Column Head Pressure Settings for He Carrier Gas (psig)

Column length (m)	id (mm) 0.10	0.18	0.20	0.25	0.32	0.45	0.53
10	34-45	5-10					
12			10-15				
15					8-12	5-10	1-2
20	75-100	10-20					
25			20-30				
30					15-25	10-20	3-5 2-4
40		35-50					
50			30-60		15-25		
60					30-45	20-30	6-10 4-8
75						8-14	5-10
105					60-80		10-15

Carrier gas selection: High purity helium and hydrogen are the preferred carrier gases for capillary columns; nitrogen is not recommended. The use of the Hydrogen Sensor is recommended when using hydrogen as a carrier gas. A gas purity of 99.995% or better is recommended with oxygen being the most important impurity to avoid (less than 1 ppm oxygen).

WARNING: Hydrogen forms explosive mixtures with air at concentrations of 4 to 10% hydrogen. Its high diffusivity minimizes this possibility, but the danger should not be discounted.

5. Install the column into the detector

Follow the installation precautions in Steps 2 and 3 for the detector side while installing the column into the detector. Confirm all detector gas flows with an accurate flow-measuring device.

6. Inspect for leaks

Inspect the GC system for leaks before heating the column for the first time. An electronic leak detector is the most reliable way to check the inlet and detector fittings. **Do not use Snoop®.** If use of a liquid is desired, try a 50/50 mixture of isopropanol/water.

7. Confirm carrier gas flow and proper column installation

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 velocity or flow values to be set.

Always consult the Column Performance Summary Sheet that accompanies the column for inner diameter information.

Note: For GC columns with smart keys, column dimensions and performance summary is automatically entered in.

Confirm carrier gas flow as described in Step 4 or by injecting a nonretained compound. Recommended nonretained compounds are in Table 2.

Table 2. Nonretained compounds[1]

Detector	Compound
FID	Methane, butane
TCD	Methane, butane, argon, air
ECD	Methylene chloride[2], SF_6, CF_2CL_2
NPD	Acetonitrile[2,3]
PID	Ethylene, acetylene
MS	Methane, butane, argon, air

1 Most of these compounds are significantly retained on PLOT columns.

2 Do not inject liquid. Use a very dilute headspace injection.

3 Acetonitrile is retained by most columns at temperatures below 100° C. Heat the column to 100° C or higher to set linear velocity.

Procedure: With the column temperature at 35 to 40° C, rapidly inject 1 to 2 µL of a nonretained compound with the split/splitless inlet *in the split mode.* If using Megabore direct or cool on-column modes, dilute the nonretained compound so that the sample will not saturate the detector.

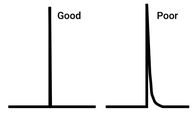

Figure 3. Methane peaks

A very sharp and symmetrical peak should be obtained (Figure 3); a small amount of peak tailing may be observed with splitless injection. 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, syringe and PC are connected and are functioning properly. If the nonretained compound is tailing, there may be a leak in the inlet, poor column installation, or an excessively low split ratio. Reinstall the column and check the inlet for leaks. A nontailing peak is required before continuing.

8. Condition the column

CAUTION: Heating a column without carrier gas flow or while oxygen is present in the carrier gas stream will rapidly and permanently damage the column.

Purge the column with carrier gas for 15 minutes. Heat the column to its upper temperature limit or a temperature 10 to 20° C above the highest operating temperature of the method, whichever is lower. Do not exceed the column upper limit or column damage will result.

After the column has reached the conditioning temperature, observe the baseline. It will rise for 5 to 30 minutes, then drop for another 30 to 90 minutes. A flat baseline should be obtained 1 to 3 hours after reaching the conditioning temperature. If the baseline does not stabilize after 2 to 3 hours or does not remain constant, stop the conditioning process.* An unstable baseline can be caused by a leak in the carrier gas line or inlet area, or by system contamination. Fix either problem before continuing.

Polar stationary phases and thick films usually require longer times to stabilize than nonpolar phases and thinner films. PLOT columns require special conditioning procedures. Refer to the column information sheet for the appropriate procedure.

Some detectors, such as ECDs and MSDs, may stabilize faster if the column is not connected to the detector during column conditioning. If the column is conditioned with the detector end disconnected, a small portion of the column end may be damaged. Remove 10 to 20 cm of the exposed column end before connecting the column to the detector.

* Thick-film and PLOT columns may take longer.

9. Accurately set the carrier gas velocity

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

$$\mu = L/t_r$$

where
μ = Average linear velocity (cm/sec)
L = Column length (cm)
t_r = Retention time nonretained peak (sec)

Recommended average linear velocities:
Helium: 30 to 40 cm/sec
Hydrogen: 50 to 80 cm/sec

GCs without EPC: Carrier gas velocity changes with oven temperature as carrier gas viscosity changes. Always set linear velocity at the same temperature for a given analysis (often the initial oven temperature). Inject 1 to 2 µL of the appropriate non-retained compound and calculate the linear velocity using the retention time of the peak and the equation above. Adjust the column head pressure until the desired average linear velocity is obtained.

GCs with EPC: The linear velocity can be entered into the PC software or the GC keypad and remains constant. The correct column dimensions must be entered into the PC software or the GC keypad for the system to accurately set the linear velocity (unless the columns have smart keys). Refer to the dimensions listed on the Column Performance Summary Sheet for the most accurate value of internal diameter. Length can be estimated by counting the loops on the column cage and multiplying by 0.54 m for standard cage columns or 0.40 m for 5-inch cages. Contact Agilent Technical Support for additional information.

10. Bleed tests

After the column is conditioned, run a blank (no injection) chromatogram using existing method temp 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. See Figure 4.

Column: DB-5, 30 m x 0.25 mm I.D., 0.25 µm
Agilent P/N: 122-5032
Carrier: Helium at 40 cm/sec
Oven: 50 to 325° C at 10° C/min 325° C for 5 min
Inlet: Split 1:100, 250° C
Detector: FID, 300° C Nitrogen makeup gas at 30 mL/min

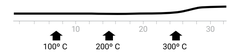

Figure 4. Bleed profile

There should be no peaks in the blank chromatogram. Peaks indicate a contamination problem, usually in the inlet area. As a column degrades with normal usage, the magnitude of the baseline rise will increase. If the baseline rise occurs at a much lower temperature than previously obtained, the column and/or GC is most likely contaminated or damaged.

11. Run test mix

Inject existing method or GC column test mix to further measure system performance. The column test mixture used by Agilent to determine column quality is recommended. The Performance Summary Chromatogram included with each column can be easily duplicated if the same conditions and test mixture are used. Failure to duplicate the chromatogram for a new column indicates an installation, operation, or instrument problem. The problem must be corrected before proceeding with sample analysis.

Column use considerations

For maximum operating life, keep the column temperature below 100° C when a column is installed but is not in use for analysis.

Column storage: Seal the column ends with GC septa and return to the original box. Upon reinstallation, cut column ends to insure that no small pieces of septum have been left in the column.

Chemical compatibility: Bonded and cross-linked stationary phases are not damaged by water or organic solvent injection. Inorganic acids (HCl, H2SO4, H3PO4, HNO3, etc.) and bases (KOH, NaOH, etc.) should not be injected into capillary columns. Rapid damage to the stationary phase will occur. If chemical damage does occur, removing the front 1/2 to 1 meter of the column will often restore column performance.

Rinsing columns: Do not solvent rinse the following non-bonded columns: DX-1, DX-4, SE-30, SE-54, HP-101, HP-17, Carbowax 20M, HP-20M, Cyclodex-B, CycloSil B, HP-Chiral β, CP-Cyclodextrin, CP-Chirasil Val, HP-88, CP-Sil 88, CAM, Select Silanes, CP-Volamine and CP-TCEP.

Do not solvent rinse PLOT columns. All other Agilent standard and cross-linked WCOT columns are solvent rinseable.

Retention gaps: The stationary phase of non-bonded columns is easily disrupted during the injection process. Attach a 3 to 5 meter retention gap to the front of the column. This minimizes the amount of stationary phase damage, especially with on-column and splitless inlets.

Temperature limits: Columns have both lower and upper temperature limits. Lower limits usually are at a phase change. Operation below this limit gives poor separation and peak shape problems, but will not damage the column.

Two upper limits are often given. The lower is the isothermal limit; the column can be held at this temperature for a prolonged time without harm. The higher one is a programming limit. The column can be heated to this limit for a short time (< 10 minutes).

Heating the column above the upper limits will significantly reduce column life. Set the GC oven maximum temperature at or below the column limit.

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Ultra Inert Inlet Liners

Agilent Ultra Inert GC Liners prevent surface adsorption providing robust, reproducible and reliable trace-level analysis of active compounds. The non-stick liner O-rings are certified pre-cleaned, conditioned to eliminate out-gassing of contaminants critical for trace analysis.

UltiMetal Plus Flexible Metal Ferrules

The novel, flexible design allows each ferrule to gently compress around the column, preventing column breakage or leakage. Stainless steel construction holds its shape during temperature cycling to maintain a leak-free connection without re-tightening while chemical deactivation provides an inert surface critical for analysis of active analytes at trace levels.

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A unique, proprietary process gives the most consistent, smooth and inert surface to seal the inlet and prevent leaks or sample degradation; critical when working with active compounds or high-sensitivity analyses.

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Agilent Self Tightening Column Nut

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Gas Management

Agilent Gas Clean Purification System

Filters carrier gas impurities reducing risk of column damage, sensitivity loss and improving trace analysis. For more information please visit **agilent.com/chem/gasclean**

ADM Flow Meter

An incorrect mix of gases can cause peak tailing, ghost peaks, retention time shifts, loss of resolution and baseline noise. ADM Flow Meters are ideal for measuring gas streams with composite gas composition. Learn more: **agilent.com/chem/admflowmeter**

G3388B Electronic Leak Detector

Gas leaks can cause detector noise and baseline instability, shorten column life, and waste expensive carrier gas. Agilent's Leak Detector is an easy way to quickly identify leaks in your system.

Column Installation Pre-Swaging tools

Ensure GC reproducibility by verifying proper length of column into the fittings, time after time. For more information visit **agilent.com/chem/betterGCconnections**

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