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12. 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 low volume of water or organic solvent injection. Inorganic acids (HCl, H₂SO₄, H₃PO₄, HNO₃, and so forth) and bases (KOH, NaOH, and so forth) should not be injected into capillary columns. Rapid damage to the stationary phase will occur. If chemical damage does occur, removing the front ½ 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, 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 nonbonded columns is easily disrupted during the injection process. Attach a 2.5 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 minimum and maximum operational temperature limits. The lower limit normally indicates a phase change from liquid to solid state. Operation below this limit gives poor separation and peak shape problems, and might 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.

The story behind Agilent advanced GC columns

In 2000, Agilent, the inventor of fused silica GC tubing, acquired J&W Scientific, the creator of the first GC stationary phase made from cross-linked siloxane polymers. This partnership united the renowned DB and HP column families, creating Agilent J&W GC Columns. The innovative products that would follow include the Agilent J&W Ultra Inert, High Efficiency, LTM, and PAH GC columns. In 2010, Agilent acquired Varian, adding PLOT, Select, VF and CP-Sil to the most extensive and innovative GC column offering in the industry.

The best low-bleed columns for sensitivity and performance

Column bleed can decrease spectral integrity, reduce uptime, and shorten column life. But Agilent GC columns have the widest range of low-bleed and standard stationary phases featuring superior inertness and high upper temperature limits—especially for MS users performing trace level analyses.

Better precision for better results

Agilent GC columns adhere to tight retention factor (k) specifications, promoting consistent retention and separation. They also feature narrow retention indexes and a high number of theoretical plates per meter, ensuring narrow peaks and improving the resolution of closely eluting peaks.

The industry's tightest quality control specifications

Agilent's stringent testing ensures reliable qualitative and quantitative results—even for your most challenging compounds. For example, we measure peak height ratios for both acids and bases to ensure top performance for the widest range of compounds. We also monitor peak

symmetry and tailing for a broad scope of chemically active compounds.

As the world's leading provider of GC capillary columns, Agilent is uniquely positioned to offer you superior quality and unmatched service and support.

For additional column recommendations, chromatograms, and method parameters, go to: www.agilent.com/chem/myGCColumns

Maintain column performance with high quality GC supplies

Certified supplies

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.

Certified Gold Seals

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.

Visit: www.agilent.com/chem/gcsupplies

Agilent Self Tightening Column Nut

This unique stainless steel GC column nut delivers a leak-free connection without adaptors, or special tools. Learn more at: www.agilent.com/chem/stnut

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: www.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: www.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: www.agilent.com/chem/betterGCconnections



Helpful GC resources

Locate supplies and parts with ease: www.agilent.com/chem/partsfinder

To view the Agilent GC troubleshooting video series, visit: www.agilent.com/chem/gctroubleshooting

To learn more about making better GC connections, visit: www.agilent.com/chem/bettergcconnections

For Agilent Technical Support, please visit: www.agilent.com/chem/techsupport

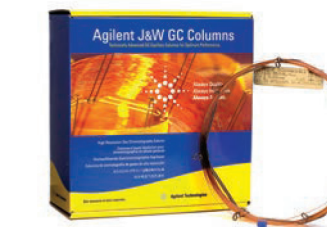
Agilent J&W Capillary GC Column

Installation Guide



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 (for example, PLOT columns).



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)

Be certain to 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.

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

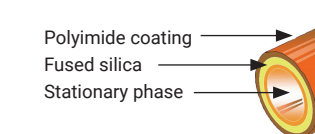


Figure 1. Capillary column.

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. An indicating filter that removes oxygen, moisture, and hydrocarbons such as the Agilent Gas Clean GC/MS filter is highly recommended for the carrier gas.

Agilent Gas Clean filters can also be added to detectors gas lines to improve sensitivity and lifetime.

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

Warning: Be careful! The oven and/or inlet may be hot enough to cause burns. If the inlet is hot, wear heat-resistant gloves to protect your hands.

Warning: Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.

Place the column nut and ferrule over one end of the column. Hang the column inside the oven with the cage posts pointing out towards the oven door. There is not a “front-back orientation” to the majority of Agilent J&W capillary columns. For certain columns that do have specific “front-back orientation”, (for example, with factory installed guard column, particle traps, and so forth) be certain to read, and follow specific installation guidelines provided with those columns.

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.

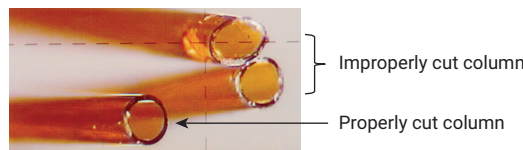


Figure 2. Cutting a fused silica capillary column.

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 ½ turn. If the column can be moved in the fitting, tighten another ¼ 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).

length (m)	Column id (mm)						
	0.10	0.18	0.20	0.25	0.32	0.45	0.53
10	35-45	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. Hydrogen is recommended for use with microbore GC columns (0.10 mm id and smaller). A gas purity of 99.995% or better is recommended with oxygen being the most important impurity to avoid (less than 1 ppm oxygen).

It is highly recommended to use gas purifiers such as the Agilent Gas Clean filter system, to ensure the highest quality gas while keeping gas lines clean and leak-free. Clean gases reduce risk of column damage, sensitivity loss, and instrument downtime.

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. The use of the Hydrogen Sensor is recommended when using hydrogen as a carrier gas.

5. Install the column into the detector

Follow the installation precautions in Step 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 accurate velocity or flow values to be set.

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

Always consult the column performance summary sheet that accompanies the column for accurate inner diameter information.

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 ₂ CL 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.

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.

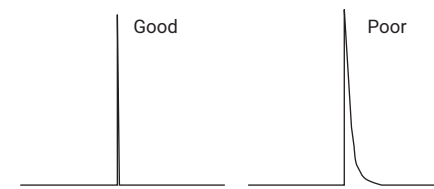


Figure 3. Methane peaks

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.

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 nonretained compound and calculate the linear velocity using the retention time of the peak and the following equation. Adjust the column head pressure until the desired average linear velocity is obtained.

$$\mu = L/t_r$$

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 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 test

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.

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.

Column	DB-5, 30 m x 0.25 mm id, 0.25 µm (p/n 122-5032)
Carrier	Helium at 40 cm/sec
Oven	50 to 325 °C at 10 °C/min, 325 °C for 5 minutes
Inlet	Split 1:100, 250 °C
Detector	FID, 300 °C, Nitrogen makeup gas at 30 mL/min

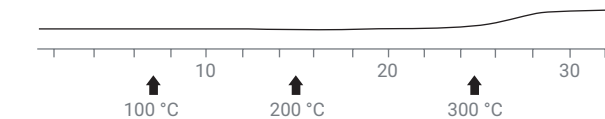


Figure 4. Bleed profile.

11. Run test mix

Agilent offers detector and column specific checkout standards to verify instrument and column quality. The Performance Summary Chromatogram included with each column uses their specific Agilent checkout mixes as proof of quality. It is highly recommended in conducting a test run by duplicating the method parameters and standards with your new column as referenced on the performance summary sheet. Failure to closely replicate this chromatogram for a new column could indicate an installation, operation, or instrument problem. The problem must be corrected before proceeding with sample analysis.

