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 ensure that no small pieces of septum have been left in the column.

Chemical compatibility

Bleeder or cross-linked stationary phases are not damaged by water or organic solvent injection. Inorganic acids (HCl, H2SO4), H2O2, 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 to 1 meter of the column will often restore column performance.

Rinsing columns

Do not rinse rinse the following non-bonded columns:
- DX-1, DX-2, DX-4, SE-30, SE-52, SE-54, Carbosieve, DMCS-250,
- Cyclodex B, Cyclodex™ B-HP 28, HP-101, HP-17, and HP-CR-8
- All other Agilent standard and cross-linked WCOT columns are solvent rinsable.

Retention limits

The stationary phase of nonbonded 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 inlet methods.

Temperature limits

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

Two upper limits are often given. The lower one is the isothermal limit; the higher one is a programming limit. The column may be heated to this limit for a short time (< 10 minutes). The higher limit is a programming limit. The column can be heated to this limit for a short time (< 10 minutes). Heating the column above the upper limit will significantly reduce column life. Set the GC oven maximum temperature at or below the column limit.

They may be small...

but Agilent inlet supplies can have a big impact on your results.

Agilent inlet supplies are engineered with the same reliability you expect from Agilent instruments. And they are designed to work with your new Agilent GC or GC/MS system. What’s more, Agilent inlet supplies can have a big impact on your results.

Polyethylene glycols, and solid adsorbents.

Toxicity

A fused silica capillary GC column (Figure 1) consists of:
- An amber-brown polyimide exterior coating that protects the tubing from breaks.
- A stationary phase that is evenly coated onto the inner wall of the tubing. Common phases are silicon-based polymers (polysiloxanes), polyphenylene glycols, and solid adsorbents.

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
- Tools for capillary column installation

Figure 1. Capillary columns.

A fused silica capillary GC column can be more specific conditioning, care and maintenance procedures for your new GC column (e.g., PLOT columns). Be certain to read all the information that comes with your new GC column to ensure that the column performs to expectations.

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For a complete listing of Agilent Columns and Supplies

Visit www.agilent.com/chem/GC supplies or call 800-227-9770 (in the U.S. or Canada). Or contact your local Agilent Representative or Agilent Authorized Distributor.

The story behind Agilent J&W advanced GC Columns

In 1920, Agilent Technologies, the inventor of fused silica GC tubing, merged with J&W Scientific, the creator of the first GC stationary phase made from cross-linked silicone polymers.

Now, thanks to this partnership, you can find both the renowned HP and DB column families under one name. All brought to you by Agilent Technologies – a company with over 40 years of gas chromatography experience.

The best low-bled columns for sensitivity and performance.

Column bleed can decrease spectral integrity, reduce sensitivity, and shorten column life. But Agilent J&W columns have the widest range of low-bled standard and stationary phases featuring superior strength and high upper temperature limits – especially for time trap MS users.

Better precision for better results.

Agilent J&W columns adhere to strict retention factor (k) specifications, providing consistent retention and separation. They also feature narrower retention indices 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 stringent testing ensures reliable quantitative and qualitative results – even for your most challenging compounds. For example, we measure peak height ratios for both acids and bases to ensure performance for the widest range of compounds. We also measure 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.

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3. Install column into the oven

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 these stress the tubing and could cause breakage. Make sure that column tags, sharp edges, or other items do not rub against the column.

The optimal installation 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. 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/Vespel™, 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 listed in Step 3.

Table 1. Approximate Column Head Pressure Settings for the Carrier Gas (psi)

<table>
<thead>
<tr>
<th>Column I.D. (mm)</th>
<th>L.B. (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>30-45</td>
</tr>
<tr>
<td>0.12</td>
<td>15-30</td>
</tr>
<tr>
<td>0.18</td>
<td>10-20</td>
</tr>
<tr>
<td>0.20</td>
<td>5-10</td>
</tr>
<tr>
<td>0.25</td>
<td>3-5</td>
</tr>
<tr>
<td>0.32</td>
<td>2-4</td>
</tr>
<tr>
<td>0.45</td>
<td>1.5-2.5</td>
</tr>
<tr>
<td>0.53</td>
<td>1.2-2.0</td>
</tr>
</tbody>
</table>

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, use 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.

Always consult the Column Performance Summary Sheet that accompanies the column for accurate inner diameter information. Conform carrier gas flow as described in Step 4 by injecting a nonretained compound. Recommended nonretained compounds are listed in Table 2.

Table 2. Recommended Compounds

<table>
<thead>
<tr>
<th>Detector</th>
<th>Compound</th>
</tr>
</thead>
</table>
| TCD        | Methylene chloride, hydrogen, argon, air, He, N₂, CO₂, CO, CH₄, CH₃OH, N₂O, CO₂, 
| ECD        | Methylene chloride, argon, air, He, N₂, CO₂, CO, CH₄, CH₃OH, N₂O, CO₂, 
| FID        | Methylene chloride, argon, air, He, N₂, CO₂, CO, CH₄, CH₃OH, N₂O, CO₂, 
| NPD        | Methylene chloride, argon, air, He, N₂, CO₂, CO, CH₄, CH₃OH, N₂O, CO₂, 

Column properties:

- Carrier gas linear velocity can be entered into the PC software or the GC keypad
- Carrier gas flow rate can be obtained from the software or via the GC keypad
- Linear velocity or flow rate can be set to the desired value
- The linear velocity can be entered into the PC software or the GC keypad
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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. For either problem before continuing.

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 non-retained compound and calculate the linear velocity using the retention time of the peak and the equation below. Adjust the column head pressure until the desired average linear velocity is obtained.

\[ \mu = L / t_r \]

where

\[ \mu \] = Average linear velocity (cm/sec)
\[ L \] = Column length (cm)
\[ t_r \] = Retention time nonretained peak (sec)

Recommended average linear velocities:

- Hydrogen: 10 to 48 cm/sec
- Hydrogen: 10 to 48 cm/sec
- Hydrogen: 10 to 48 cm/sec
- Hydrogen: 10 to 48 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. 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 in standard gauge columns or 0.46 in 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. Start at 40 to 50 °C, ramp to 10 ± 2 °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.

11. Run test mix

Inject a test mixture 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. This problem must be corrected before proceeding with sample analysis.

Figure 4. Methane peaks.

Figure 3. Methane peaks.

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Oven: 50 to 325 °C at 10 °C/min
Carrier: Helium at 40 cm/sec
Detector: Nitrogen make-up gas at 30 ml/min

Figure 1. Bleed profile.

Figure 1. Carrier gas velocity settings.