



CAPILLARY COLUMN CONDITIONING TO MINIMIZE AND MONITOR COLUMN BLEED (Mass Spectral Detectors)

Column installation and conditioning is crucial for optimum performance of any analytical instrument. There are many techniques in use today. The following is one procedure that maybe used.

1. Cool all heated zones and vent the MSD.
2. Turn off the inlet flow, remove the old column, and cap the MS transfer line with a no-hole ferrule (Use nut part number 05921-21170 and ferrule part number 5181-3308).
3. Install the new column into the GC inlet assembly according to procedures appropriate to the type of injection port being used, and turn on the inlet flow. Using an electronic leak detector, verify that installation is leak free.
4. Trim the detector end of the column, and place the end into a beaker containing water or methanol. Verify the presence of bubbles. This will ensure that there is carrier gas flow through the column.

NOTE: Once the column is correctly installed in the GC injection port, it is important to remove all of the oxygen inside the inlet and column before increasing the temperature. If the next step is skipped, the oxygen can damage the phase and significant column bleed could result.

This document is believed to be accurate and up-to-date. However, Agilent Technologies, Inc. cannot assume responsibility for the use of this material.
The information contained herein is intended for use by informed individuals who can and must determine its fitness for their purpose.

Warning: Do not condition the column using Hydrogen. Hydrogen accumulation in the GC oven can result in an **Explosion. If planning to use Hydrogen as a carrier gas, first condition the column using ultra-pure (99.999% or better) inert gas such as Helium, Nitrogen, or Argon.**

5. Allow 6 column volumes of carrier gas to pass through the column before temperature is applied, or 15 minutes of time after column flow has been initiated.

6. Close the oven door, turn on the injection port temperature and ramp the GC oven to 20 degrees above final temperature specified in the analytical runs, but do not exceed the maximum temperature limit of the column. During initial conditioning, it is suitable to hold at the Program Temperature Maximum of the capillary column for the extended time of conditioning.

NOTE: The column is not yet installed into the MSD at this point.

7. Once the final temperature has been reached, hold that temperature for about 2 hours, or as specified by the column manufacturer, and then cool the oven.

8. Install the column into the MSD, pump down and equilibrate per normal procedures.

It is a good idea at this point to obtain a profile of the column background to use for future reference. Using a manual injection setting, run the method once or twice without actually making an injection, and print the resulting chromatogram(s). A good chromatogram should have low abundance. This can be relative, depending on the application. Refer to previous performance to determine an acceptable value for your application. There should be a lack of significant column bleed masses in the background spectra, typically 207 and/or 281, and no evidence of contaminant peaks. The profile can be used at a later date to determine if column performance has started to degrade. If you suspect the column is starting to bleed excessively, obtain another profile of the column background. If the 2 chromatograms have a similar profile, then the problem may not be the column. It could be the samples, inlet, and/or the tuning of the MSD contributing to the background. If the second profile is much higher than the first, especially at increasing oven temperature, the column phase may be deteriorating and you may need to consider column replacement. Also note the relative increase in bleed ions (e.g., 207 or 281) to confirm column bleed.