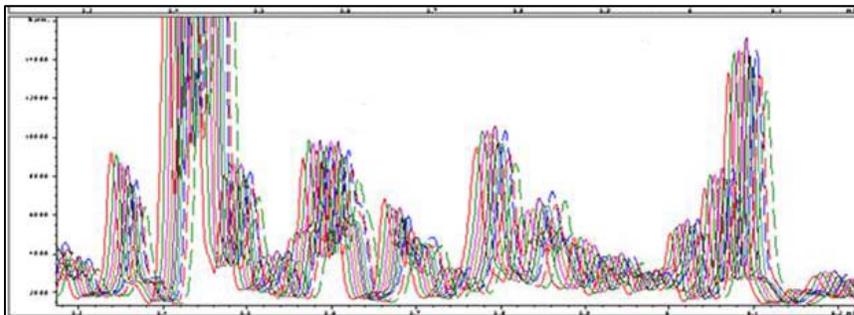




## GC Troubleshooting Series

### Part Six: Retention Time Shifts



#### Possible Cause: Changes in Column Dimensions vs. Previous Column

If your issue occurs after changing a column, it is probably due to changes in column length. The old column may have been trimmed over time to a significantly shorter length.

If your GC has an electronic pneumatic control (EPC), enter the actual column dimensions in to the software. Check the performance summary sheet that comes with your column for the exact dimensions.

The best way to verify that you have the right flow rate, whether you have an EPC or not, is to inject something of known retention at a given temperature and make sure it comes out at the right retention time.

#### Possible Cause: Leak in injector or Blockage in Gas Line

Check for leaks in your injector and blockages in your gas line.

If available, use Agilent Lab Advisor to perform the leak check. Open the software, select the GC, and run the Prep Run Leak Check. The test does not require capping fittings; run the test using the analytical setup to quickly check for leaks after performing maintenance. Agilent Lab Advisor automatically runs the test and presents the results. The prep run leak check finds leaks at the column fitting, septum nut, weldment nut, split vent trap housing and gold seal fitting.

If the test indicates a leak, tighten the column fitting, septum nut, insert weldment nut, split vent trap housing, and gold seal fitting. Rerun the test. If the test still fails, perform basic GC inlet maintenance: replace the liner, septum and o-ring (see demos at [www.agilent.com/chem/gcinletcare](http://www.agilent.com/chem/gcinletcare)).

The automation provided by Agilent Lab Advisor can be performed at the GC if the software is not available. Also, it is useful to monitor the total flow directly at the GC display while tightening fittings. To perform the test manually:

1. Confirm that the column dimensions (length and id) are entered accurately into the GC column configuration. Use the following formula to determine column length: Diameter of column basket (m)  $\times$  Pi  $\times$  Number of turns of column. For 6890 or 7890A columns, use diameter of 0.20 mm

2. Set the inlet to splitless mode.
3. Set purge flow to 50 mL/min.
4. Set column flow to 7 mL/min.
5. Set the septum purge flow to 3 mL/min. Allow the inlet pressure to stabilize. The purge valve is open and total flow should be 60 mL/min. Total flow = column flow + split vent flow + septum purge flow = 7 + 50 + 3 mL/min. You may have to adjust the inlet pressure setpoint to achieve this column flow. If there is a leak, the total flow will be higher than 10 mL/min by the amount of the leak (>15 mL/min indicates a definite leak or uncalibrated flow sensor.). Note that the flow sensor is less stable and accurate at flows below 10 mL/min.
6. Press [Prep Run] (press it twice if the GC is Not Ready) to shut off the purge valve (split vent flow) and change the inlet configuration.
7. Check the inlet total flow. It should now read approximately 10 mL/min if there are no leaks. Total flow = column flow + split vent flow + septum purge flow = 7 + 0 + 3 mL/min.
8. While monitoring the total flow, tighten the column fittings, gold seal fitting, split vent trap housing, septum nut, and insert weldment nut.

If the leak remains, do basic inlet maintenance: replace the septa, liner and o-ring(demos available at [www.agilent.com/chem/gcinletcare](http://www.agilent.com/chem/gcinletcare))

Open the split vent trap and check the O-ring seating. Replace the split vent trap if needed (see [www.agilent.com/chem/splitventtrap](http://www.agilent.com/chem/splitventtrap)).

#### Possible Cause: Large Change in Sample Concentration

Large changes in sample injection concentration can cause retention time shifts. Check inlet flows, injection settings and syringe sizes to make sure nothing has changed.

You may need to dilute the sample to get it to your original concentration range.

#### Possible Cause: Solvent Incompatibility

You will typically see this on splitless injections due to Reverse Solvent Effect. This occurs when compounds eluting around the major component or solvent are competing with being in the solvent and the stationary phase. A kind of "tug-of-war" is happening in the column. Try a retention gap or guard column to correct this.

#### Possible Cause: Inaccurate Purge Time

Check your injection purge time to be sure it hasn't changed.

#### Potential Cause: Column Temperature

Check all your parameters – flow rate, temperature settings, hold times, ramp times – against your original method.

Injecting a standard or component of known retention is the best way to verify you have the correct flow, if all other settings are correct.