

Technical Article

It's a Matter of Degrees, but Do Degrees Really Matter?

An Observation of GC Inlet Temperature Profile and Inlet-to-Inlet Temperature Variability

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- For some manufacturers, only a portion of the GC inlet is actually at the temperature setpoint; a significant thermal gradient exists both above and below this zone.
- The thermal profile of one GC inlet can vary from other similar inlets—and vary dramatically between different styles.
- Removal or damage to GC insulation can have a large effect on the inlet's thermal profile.



Injecting a liquid sample into a hot GC inlet is a dynamic and complex event. Of the many parameters that affect the success of an injection, inlet temperature is one of the most significant. Raising or lowering the inlet temperature setpoint can have a profound effect on how much sample is transferred onto the column depending on sample volatility and thermal sensitivity. But, once the inlet temperature is set, how much of the inlet is actually kept at that setpoint? Moreover, how might thermal profiles change between inlets?

Temperature Varies Within and Between Similar Inlets

The motivation for this work came from a question about the actual temperature of an O-ring installed in an Agilent split/splitless inlet at a given inlet temperature setpoint. (See Figure 1 to identify the components of a GC inlet.) Instead of just measuring the temperature inside a liner near the O-ring's location, we used a thermocouple to measure temperature along the entire length of the liner at a constant inlet temperature setpoint of 250 °C. The resulting thermal profile confirmed that a temperature gradient exists within the inlet.*

In previous work (www.restek.com/hotseptum), we also discussed this gradient within GC inlets and noted that inlet thermal profiles can vary greatly between manufacturers, but would they vary between similar inlets from the same manufacturer? We checked another similar inlet to compare the thermal profiles and found that the second inlet exhibited a different thermal profile from the first. After measuring several more Agilent GC inlet temperature profiles, we found inlet-to-inlet variation in all cases, even in ostensibly identical inlets (Figure 2).

* For these experiments, we only measured the thermal profile of the liner inside the inlet, not the entire inlet.

Figure 1: Considering how little of the GC inlet is actively heated by the heating element, it's no surprise a temperature gradient exists—especially if insulation is missing from the top or bottom.

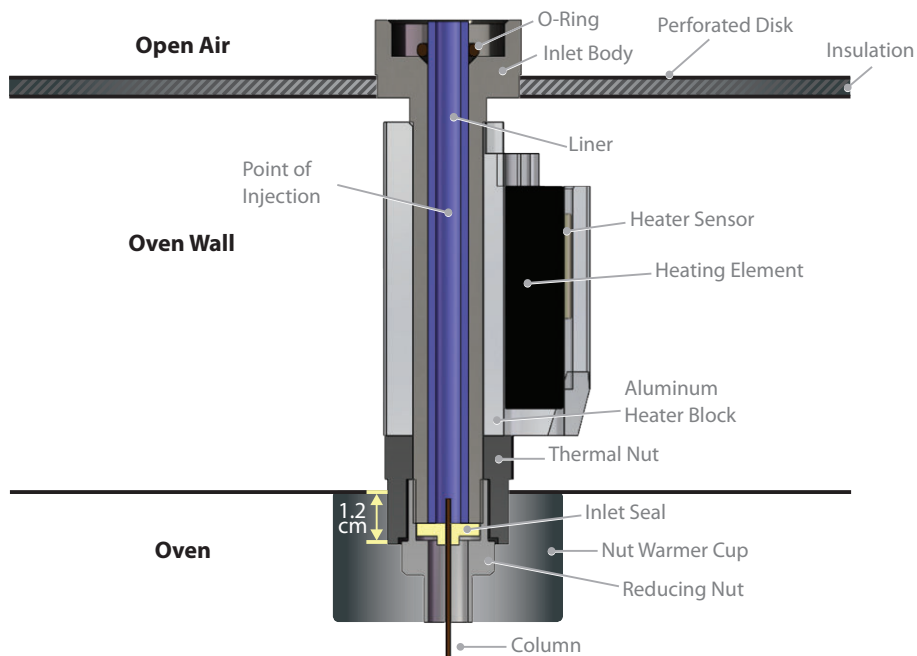
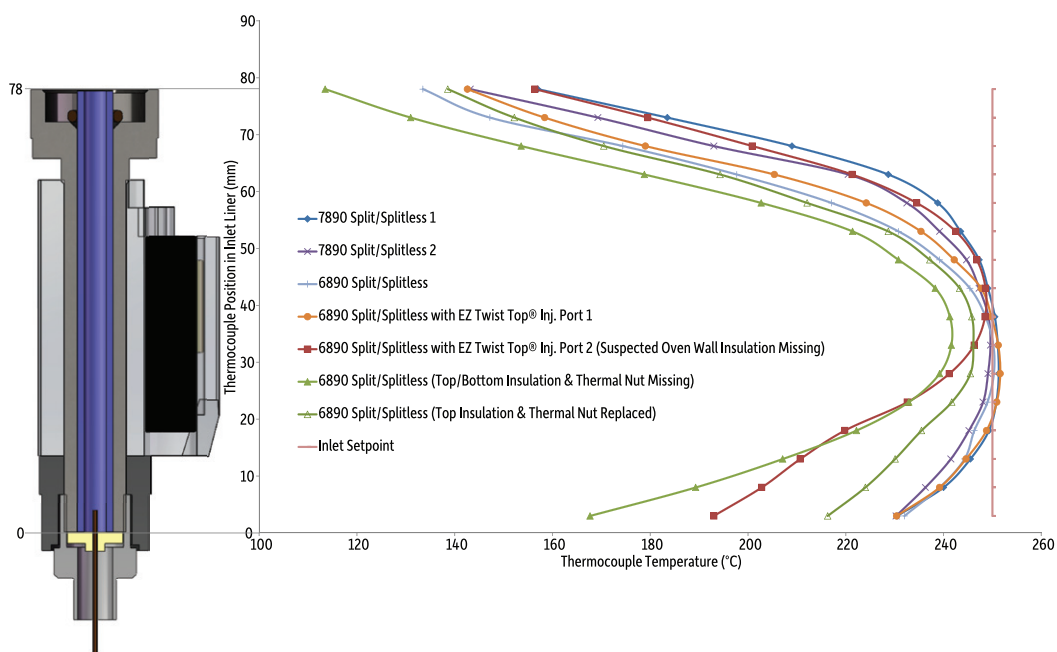


Figure 2: A temperature gradient exists within a GC inlet, and temperature profiles can vary between similar inlets. These variations increase dramatically with the absence of insulation.

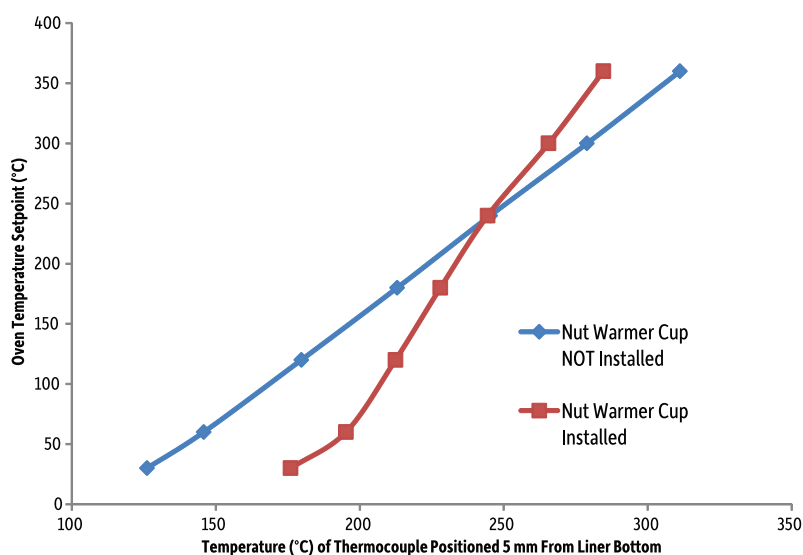


Insulation is Crucial to Minimizing Temperature Variation

We did observe one split/splitless inlet with significantly lower temperatures at the top and bottom. After investigating, we discovered that the top ring of insulation, which sits just below the perforated disk of the Agilent 6890 split/splitless inlet weldment, was missing. Some of the insulation at the bottom of the inlet, along with the thermal nut, was also not installed. Simply placing insulation in the top cavity and installing the thermal nut caused the temperature of the inlet liner to more closely match the other inlets (Figure 2). This test was a valuable reminder of the need to carefully reconstruct the inlet whenever the insulation is disturbed.

When insulation is missing at the top of the inlet, the inlet loses heat to the open air; however, when insulation is missing at the bottom, the GC oven influences the temperature in both directions (Figure 3). Because column installation can be more challenging with the insulated nut warmer cup installed, analysts may be tempted to leave it in a drawer, but the effect on your inlet temperature can be significant.

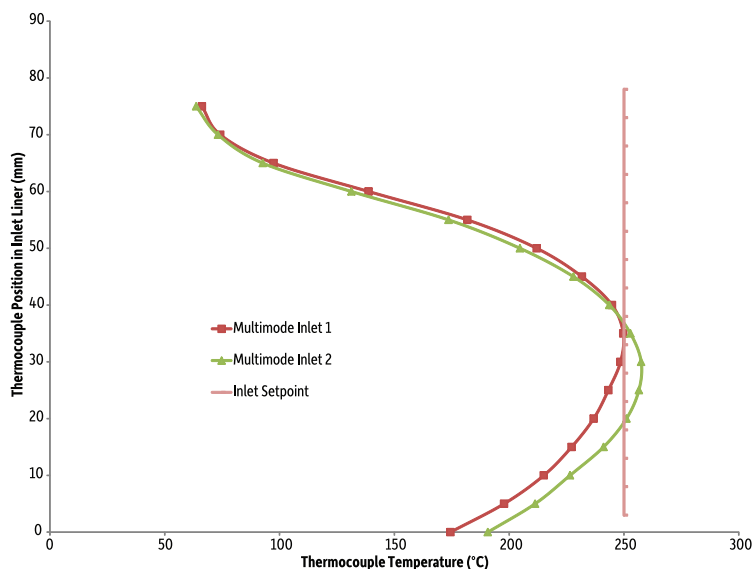
Figure 3: Installing the nut warmer cup can help minimize the effects of oven temperature on the actual temperature of the inlet. (Inlet shown below was set to a constant 250 °C.)



Temperature Can Vary Drastically Between Dissimilar Inlets

The newly introduced Agilent Multimode Inlet (MMI) is said to be capable of performing both hot split and hot splitless injections like a normal split/splitless inlet. But, when we measured the thermal profiles for two MMI inlets, it was interesting to note how different the MMI thermal profiles were from a split/splitless inlet—a drop of over 190 °C from the setpoint to the top of the inlet as opposed to around 100 °C for the split/splitless inlets (Figure 4). This variation shows that changing equipment may also change your results, even if the equipment is nominally able to do the same analysis.

Figure 4: The Multimode Inlets (MMIs) we measured experienced almost twice the temperature drop (190 °C) of a standard split/splitless inlet between the inlet setpoint to the top of the liner.



The Effects of Inlet Temperature Variations on Chromatography

As demonstrated here, thermal gradients exist within a single GC inlet, and temperature profiles can vary between similar, as well as between dissimilar, inlets. These variations affect the vaporization of a liquid sample (and, thus, the overall success of the analysis).

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