

Monday Afternoon Poster Session 7500-2400P

Inlet Artifacts in Capillary GC.MSD Analysis – Ghost Peaks from Splitless Injections on an HP/Agilent 6890 GC and the Impact of USC 21 CFR Part 11.

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Abstract.

Artifacts in GC analysis is of particular importance in pharmaceutical industry due to the compliance requirements under USC 21 CFR Part 11. The source of the vast majority of these “artificial results” has been the heated inlet. Although the purpose of the inlet is simply the vaporization of the sample and solvent for transferring the material into the GC column, in many cases this entire process creates artificial results.

The sources of artifacts may be from one or a combination of the following:

1. Thermal decomposition of the analyte(s).
2. Chemical reaction of components in analyte mixture.
3. Active surface effects of the inlet, liner and tubing.
4. Flow perturbations within the inlet.
5. Contaminated inlet.

In our laboratory experience, we have noted chronic artifacts in splitless injections on our HP/Agilent 6890 GC / 5973 MSD system. Analysis of deoxybenzoin has proven to be particularly problematic. Irreproducible ghost peaks above the minimum reporting limit continued to plague this method for years without resolution. The mass spectra of these ghost peaks are, however, identical to the nearest large, adjacent peak.

Recently, we have discovered the source of this artifact being a flow perturbation within the inlet, and have fabricated a modification to HP/Agilent’s Electronic Pressure Control (EPC) Module to remedy the problem. In this report, we will discuss our findings, our postulated reason for the artifacts, and show the vastly improved results after the remedy has been installed.