Agilent Technologies

Troubleshooting Chromatographic Contamination

Ghost Peaks/Carryover

Accurate quantitation and peak identification requires clean background on a GC blank run to insure that there are no peaks or baseline changes that interfere with sample peaks. This requires the following:

- Clean carrier/makeup gas and delivery tubing
- Clean solvent
- Syringe (It is important to use sufficient solvent washes in autoinjector to avoid cross contamination from sample to sample.)
- Inlet liner/septum
- Column
- Detector plumbing and base weldment

When diagnosing chromatographic problems it is very important to use tests that systematically isolate the problem to the detector, column or sample introduction components (syringe, inlet, liners, supply gases, etc.)

Typically, sharp, well resolved peaks that elute during a temperature program with no injection are from the carrier/inlet system. Broader "humps" in the baseline are usually from the makeup system or from sample peaks that did not elute from the previous run.

In almost every case of GC system contamination, the source is external to the GC. The three primary sources of contamination are:

- Impure Gases
- Contaminated consumables
- Sample contamination-typically from repeated injections or dirty samples that overload the injection port.

With any GC system, especially when using high sensitivity/selective detector, it is imperative to insure that the carrier/makeup and detector gases meet the Agilent published specification of 99.9995%. This represents a 5 PPM allowed impurity compared to the part per trillion for the 6890 micro ECD.

The gas delivery system must meet the following requirements:

- Stainless steel diaphragm tank regulators capable of sufficient supply pressure.
- All metal plumbing manifold comprised of clean 1/8" copper tubing (Agilent part number 5180-4196). The gas delivery system must be leak free.
- Gas traps are recommended for carrier and makeup gas supplies: All metal conditioned moisture trap, closest to the tank, all glass O2 indicating trap, closest to the GC and a hydrocarbon trap can be installed in between. Never use traps made of plastic or that use o-ring seals.

The first step in solving any problem is to define the problem accurately.

1. Problem definition: The presence of peaks in the chromatogram that are not expected to be in the method standard sample.

Review the chromatogram. Are there peaks eluting in and around the peaks of interest that might interfere with quantitation? It is a good idea to have on hand a chromatogram that is considered "normal" so that a comparison can be made, especially if the method is used for analyzing more complex matrices.

2. Solvent Evaluation—The next step is to determine the purity of the solvent used to prepare the standard. Analyze a vial of solvent from another source than that used to prepare the original sample and compare the background profile.

Is the solvent used in the blank run cleaner that the solvent used to prepare the standard? Do you observe fewer peaks in the chromatogram?

Are there still interference peaks showing up? (even small peaks at the same retention time.) At this point interference peaks could be coming from the carrier gas, sample/solvent, sryinge, injection port/liner, column or makeup gas weldment.

If the peaks are absent in the solvent blank, then the contamination was in the orginal sample.

3. **Eliminating the Solvent**—A system blank evaluation is performed by running the GC method, but making no injection at all. The easiest way to do this is to remove the syringe from the autoinjector. The purpose of this step is to determine what portion of the interference is coming from the solvent/syringe and what portion is from the injection port/liner/column/makeup gas weldment.

   By comparing the system blank to that of the solvent blank from step # 2:

   Is the interference still present?

   Are there peaks in the system blank, just reduced in size?

   If peaks are observed in the system blank the most likely source could be the injection port/ liner or a contaminated column.

   Additional tests:

   a. A series of replicate system blank runs (no injections) could be performed to see if the contamination peaks become smaller or are eliminated. This would isolate the source to the solvent/syringe or possibly the injection port.
   b. To determine if the ghost peaks are due to contamination in the carrier gas or the inlet EPC module, allow the GC to sit with the oven at the method starting temperature or cooler for an extended period of time (1/2 to 1 hour). If the peaks get significantly bigger on the next run, you will know that they are coming into the GC injection port and building up on the front of the column while the GC oven is cool.
   c. Perform Inlet maintenance. (See the inlet manual for instructions.)

d. Try a new, clean syringe for solvent blanks. (Also don't forge the possibility of contaminated glassware, pipettes, sample vials, etc.

4. **Eliminating the Injection Port/Column**-- The next step is designed to aid in isolating the source of contamination to the injection port/column or the detector base itself or makeup gas weldment. With an ECD or TCD, the makeup gas weldment can become contaminated from either poor quality gas or graphite/dirty samples. As the temperature in the oven increases, the makeup gas supply tube in the GC oven heats up and condensed material can be liberated into the detector. Normally makeup gas contamination shows up as a broad hump on the baseline.

   By removing the column from the detector and plugging/capping the base of the detector, the baseline can be evaluated with just makeup gas flow. Compare the system blank chromatogram (with the column installed) to this blank chromatogram (with the column removed), if interference peaks are still present the makeup gas or makeup gas purity would be suspect.

   If the peaks are absent, then injection port maintenance should be performed, if not already done. If using a capillary column the approximately 5-10 cm should be trimmed from the front of the column.

5. Replace the column—The last step in the process would be to install a good or new well conditioned column, after the injection port and detector has been ruled out.