GC Troubleshooting Series
Part One: Ghost Peaks

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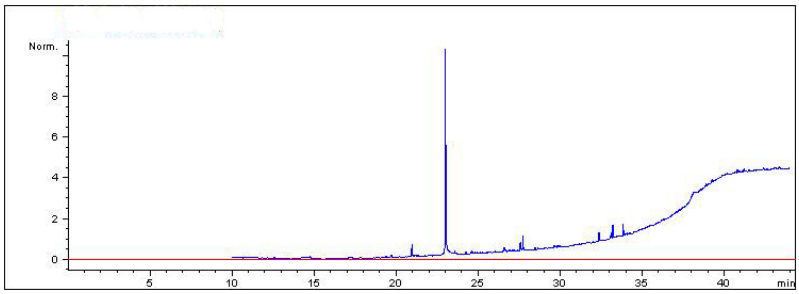
The terms "injector" and "inlet" are often used interchangeably by chromatographers. We may do it, too, on occasion. But usually, when we say "inlet" we are referring to the part of the GC where the sample is introduced., not the injection port.

This video series is focused exclusively on the split/splitless inlet.

Ghost peaks indicate contamination somewhere in your process.



A good place to start looking for the cause of a ghost peak is sample preparation. From there, look at the following five basic areas, which follow the sample flow path: The flow from the gas tank through the inlet of the GC, through the column, the detector, and out to the data system. Thinking in those terms helps keep it related to what areas are coming into contact with your sample, and in what order.

Make sure you're using high quality, highly clean sample prep materials. Visit www.agilent.com/chem/sampliQ to learn about Agilent's high quality SPE products.

Prioritized list for ghost peaks:

- Check your sample prep process for contamination points
- Purge your gas lines
- Change your solvent, or use higher quality solvent
- Change your liner, gold seal, septa (see details, beginning p. 3); perform condensation test to help diagnose issue
- Correct Back-flash: change liner volume, injection volume, solvent type, inlet temperature, or pressure-pulse your EPC
- For semi-volatile contamination, bake-out your column longer after each analysis, or periodically

See more about gas management, including information about Agilent's renewable gas filter, which provides three types of filtration in one easy-to-change cartridge, at www.agilent.com/chem/renewable.

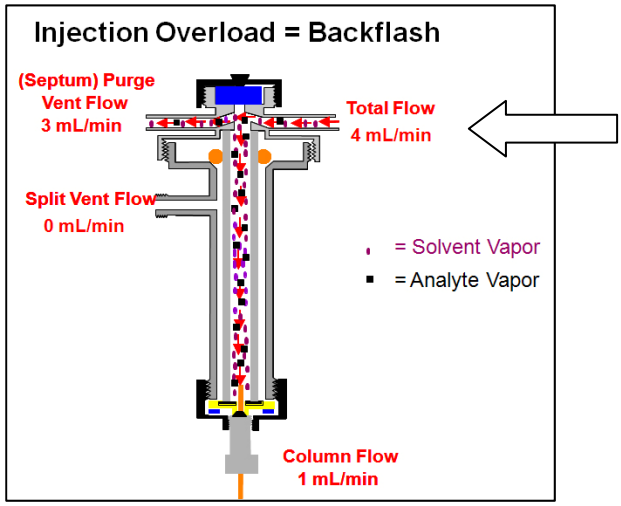
Steps to run a condensation test (to help identify the source of your ghost peaks issue):

1) Do a blank run after a long period of non-use (overnight or a weekend)
2) GC oven should be at the starting temperature during this time.
3) If you see ghost peaks when you run a blank run, immediately run it again through a temperature cycle.
4) If the peaks are larger in this second run, then there is contamination prior to the column -- in the inlet or lines.

If you don't see ghost peaks or they don't get bigger or smaller, you likely have another type of contamination or carryover (syringe, sample, etc.)

Backflash:

Backflash occurs when the vapor volume exceeds the liner volume. The vapor can condense on a cooler part in the top of the injector or injector lines because it isn't as hot there. This contaminates the system.



If you have a persistent backflash problem, consider cleaning or replacing the insert assembly – the tubing from the EPC module to the inlet – including the carrier line.

To clean the weldment assembly, dip a long swab soaked in solvent -- hexane is a good option. Run the swab along the inside of the weldment.

Take steps to prevent backflash after correcting for it:

- Reduce injection volume
- Lower inlet temperature
- Consider a larger volume liner
- Pressure pulse the injection (if possible)
- Change solvents

To help address backflash issues, download the free vapor volume calculator at www.agilent.com/chem/flowcalculator.

More information about Agilent BTO Septa, go to http://www.chem.agilent.com/btosepta.

Sample Carryover:

When ghost peaks are broader than sample peaks, it indicates a potential sample carry-over issue.

To correct, extend your column bake-out at the end of each analysis, and/or perform periodic column bake-outs for higher boiling semi-volatile contamination removal.

Limit bake-out to 1 – 2 hours and take care not to exceed the thermal limits of the column (check the column spec sheet).

Inlet Maintenance

Good basic GC Inlet Maintenance Tips:

1) Create a maintenance method to help you streamline this process
2) Frequently clean the inlet and replace the liner and o-ring [usually once each week, depending on usage]
3) Less frequent: replace gold seal and septa

The frequency of inlet maintenance you'll need will depend on the amount and the types of samples you're running.

Setting up a maintenance method:

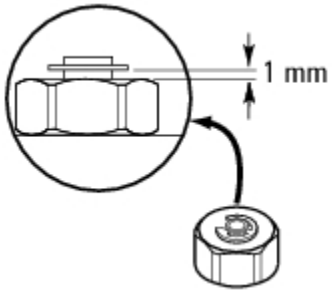
1) Cool and depressurize the inlet
2) Create a copy of the analytical method and change the name to include "IM" for "Inlet Maintenance".
3) Under Instrument Parameters, set the over temperature to 40°C and apply.
4) Set the Inlet temperature to "off" (uncheck box) and the inlet pressure to 0 psi.
5) Change the detector settings as needed.

Save the method, and load it the next time you do inlet maintenance.

Replacing the inlet septum, liner and o-ring

Supplies you'll need to keep on hand for routine inlet maintenance: tweezer, septa, liners, inlet wrench (which came with your GC).

1) Loosen the Remove the septum retainer nut.
2) Use tweezers to remove the septum from the retainer nut. Do not gouge or scratch the interior of the septum head. Firmly press the new septum into the fitting.
3) Install the septum retainer nut finger-tight. Tighten it until the C-ring is about 1 mm above the nut, as in diagram:



4) Over-tightening the septum nut can cause contamination from the syringe coring the septum and depositing the pieces in the inlet.
5) Slide the locking tab forward (counterclockwise). Lift the septum assembly straight up and away from the inlet to avoid chipping or breaking the liner. Loosen the O-ring from the sealing surface with tweezers. Grasp the liner with tweezers and pull it out.
6) Inspect the surface of the gold seal for graphite or rubber septum contamination. If required, replace the gold seal.
7) Clean the inlet if there is visible or suspected contamination. Clean O-ring residue from sealing surface. Slide a new O-ring onto the replacement liner. Return the liner to the inlet, pushing it all the way in until the liner contacts the gold seal.
8) Line up the tab on the bottom of the septum assembly with the slot on the insert assembly and push down to connect. Slide the locking tab to the back.
9) Turn on the inlet. Allow the inlet and column to purge with carrier gas for 15 minutes before heating the inlet or the column oven.

Agilent recommends **Certified Non-Stick Bleed and Temperature Optimized (BTO) Inlet Septa.** They are plasma-treated to eliminate sticking and coring, and help to keep your inlet clean from external contamination. Agilent's septa are blister-packed for cleanliness and convenience.

- 11 mm BTO septa, 50 /pk: part number 5183-4757; 100/pk: part number 5183-4757-100
- 5 mm BTO septa through-hold for on-column injection in glass jar, 50/pk: part number 5183-4758

For more information about other Agilent certified GC and GC/MS supplies, check out www.agilent.com/chem/gcsupplies

Replacing the Gold Seal: Agilent part number: 5188-5367 -- Gold-plated seal (standard application)

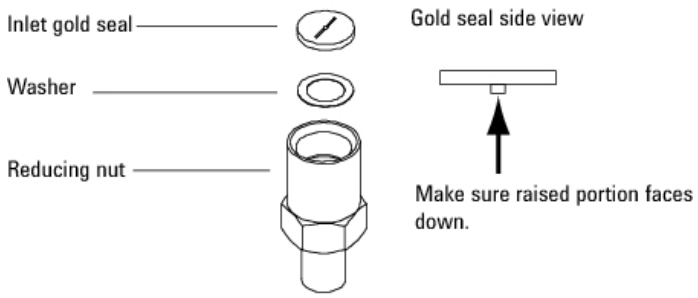
Enhance your system's inertness with **Certified Gold Seals.** A unique, proprietary manufacturing process gives you the most consistent, smooth and inert surface to seal the inlet and prevent leaks or sample degredation.

The gold seal is also called the "inlet base seal".

For tools, you'll need one ¼-inch and one ½-inch open-end wrench. During the procedure, avoid handling the seal with your bare hands. Oils from your skin can cause extra peaks on your chromatogram.

1) The gold seal is at the bottom of the inlet where the column is connected. To get to the gold seal, we must remove the liner and the column.
2) First remove the liner.
3) Using the ¼-inch wrench, remove the column from the inlet. Cap the open end to prevent air or contamination from entering the column. Also remove the insulation cup around the base of the inlet.
4) Loosen and remove the reducing nut.
5) Remove the washer and seal inside the reducing nut.
6) Put a new washer in the reducing nut and place the new gold seal on top of it (raised portion facing down – see diagram, below)
7) Replace the reducing nut and tighten securely with a wrench.
8) Replace the inlet liner.

9) Install the insulation cup and the column.
10) Turn on the inlet. Allow the inlet and column to purge with carrier gas for 15 minutes before heating the inlet or the column oven.
11) Restore the analytical method.



Tip: When you put in the new gold seal, **be sure not to overtighten the reducing nut!**

Record your maintenance in a logbook, or LabAdvisor software.