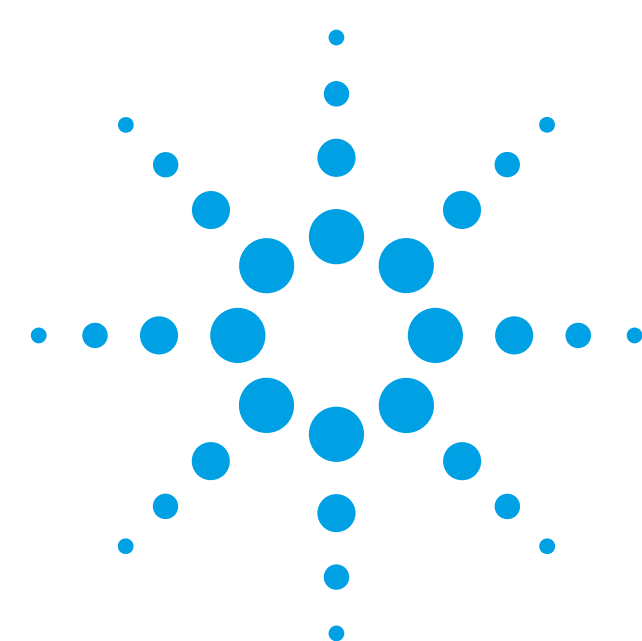


GC Troubleshooting Guide

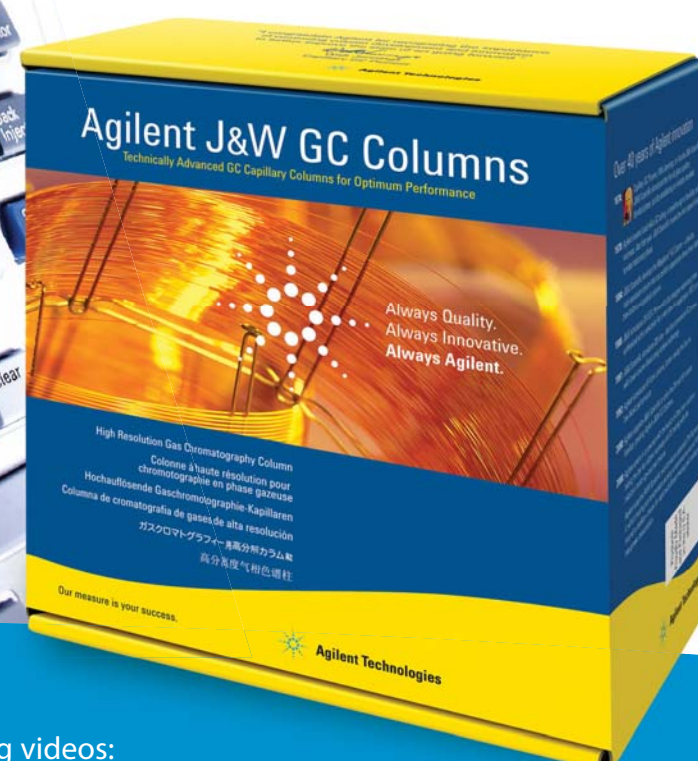
Your guide to solving common problems and staying productive



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Checking the Basics

A surprising number of problems involve fairly simple and often overlooked components of the GC system or analysis. Many of these items are transparent in the daily operation of the GC and are often taken for granted ("set it and forget it"). The areas and items to check include:

- Gases: pressures, carrier gas average linear velocity, and flow rates (detector, split vent, septum purge)
- Temperatures: column, injector, detector, and transfer lines
- System parameters: purge activation times, detector attenuation and range, mass ranges, etc.
- Gas lines and traps: cleanliness, leaks, and expiration
- Injector consumables: septa, liners, O-rings, and ferrules
- Sample integrity: concentration, degradation, solvent, and storage
- Syringes: handling technique, leaks, needle sharpness, and cleanliness
- Data system: settings and connections

Condensation Test

Use this test whenever injector or carrier gas contamination problems are suspected (e.g., ghost peaks or erratic baseline).

1. Leave the GC at 40-50°C for 8 or more hours.
2. Run a blank analysis (i.e., start the GC, but with no injection) using the normal temperature conditions and instrument settings.
3. Collect the chromatogram for this blank run.
4. Immediately repeat the blank run as soon as the first one is completed. Do not allow more than 5 minutes to elapse before starting the second blank run.
5. Collect the chromatogram for the second blank run and compare it to the first chromatogram.
6. If the second chromatogram contains a substantially larger amount of peaks and baseline instability, the incoming carrier gas line or the carrier gas is contaminated.
7. If the second chromatogram contains few peaks or very little baseline drift, the carrier gas and incoming carrier gas lines are relatively clean.

View the Agilent GC troubleshooting videos: www.agilent.com/chem/gctroubleshooting

Download the Agilent GC Pressure and Flow iPhone app www.agilent.com/chem/GCapp

For Agilent Technical Support please visit www.agilent.com/chem/techsupport

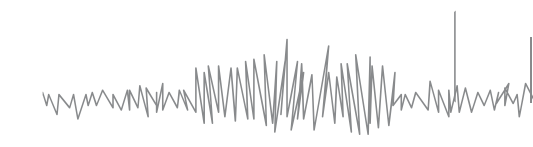
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Ghost Peaks or Carryover



Possible Cause	Solution	Comments
Contaminants introduced with sample inlet contamination	Sample or solvent cleanup Clean the injector, replace liner, gold seal, and septum	Contaminants in sample process or solvent Try a condensation test; gas lines may also need cleaning. Take steps to prevent sample backflush (reduce injection volume, lower inlet temperature, use larger volume liner).
Septum bleed	Replace septum	Use a high quality septum appropriate for the inlet temperature
Contamination of sample prior to introduction to the GC	Check sample handling steps for potential contamination sources: sample gas cylinder cleanup, handling, transfer, and storage	Usually occurs after changing the column. Check for contamination in the inlet, carrier gas or carrier gas lines.
Semi-volatile contamination (peak widths will be broader than sample peaks with similar retention)	Bake-out column. Solvent rinse the column. Check for contamination in the inlet, carrier gas or carrier gas lines.	Limit bake-out to 1-2 hours. Only for bonded and cross-linked phases.

Excessive Baseline Noise



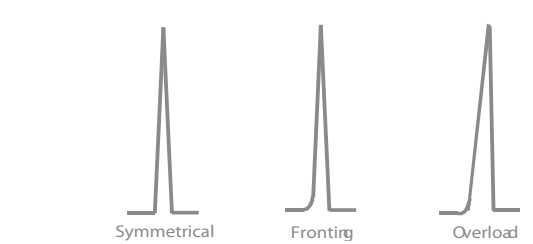
Possible Cause	Solution	Comments
Injector contamination	Clean the injector; replace liner, gold seal	Try a condensation test; gas lines may also need cleaning
Column contamination	Bake-out the column Solvent rinse the column	Limit the bake-out to 1-2 hours Only for bonded and cross-linked phases Check for inlet contamination
Detector contamination	Clean the detector	Usually the noise increases over time and not suddenly
Contaminated or low quality gases	Use better grade gases; also check for expired gas traps or leaks	Usually occurs after changing a gas cylinder
Column inserted too far into the detector	Reinstall the column	Consult GC manual for proper insertion distance
Incorrect detector gas flow rates	Adjust the flow rates to the recommended values	Consult GC manual for proper flow rates
Leak when using an MS, ECD, or TCD	Find and eliminate the leak	Usually at the column fittings or injector
Old detector filament, lamp or electron multiplier	Replace appropriate part	
Septum degradation	Replace septum	For high temperature applications use an appropriate septum

Baseline Instability or Disturbances



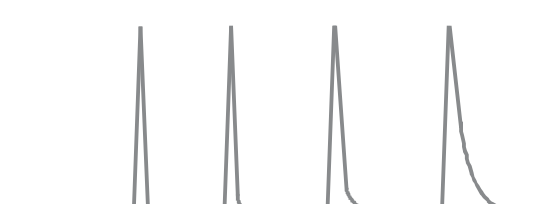
Possible Cause	Solution	Comments
Injector contamination	Clean the injector	Try a condensation test; gas lines may also need cleaning
Column contamination	Bake-out the column	Limit a bake-out to 1-2 hours
Unequilibrated detector	Allow the detector to stabilize	Some detectors may require up to 24 hours to fully stabilize
Incompletely conditioned column	Fully condition the column	More critical for trace level analyses
Change in carrier gas flow rate during the temperature program	Normal in many cases	MS, TCD and ECD respond to changes in carrier gas flow rate

Fronting Peaks



Possible Cause	Solution	Comments
Column overload	Reduce mass amount of the analyte to the column. Decrease injection volume, dilute sample, increase split ratio.	Most common cause for fronting peaks
Improper column installation	Reinstall the column in the injector	Consult the GC manual for the proper installation distance
Injection technique	Change technique	Usually related to erratic plunger depression or having sample in the syringe needle. Use an autosampler.
Compound very soluble in injection solvent	Change solvent. Using an on gap may help.	More critical for trace level analyses
Mixed sample solvent	Change sample solvent	Worse for solvents with large differences in polarity or boiling points

Tailing Peaks



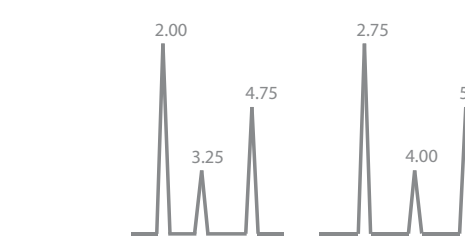
Possible Cause	Solution	Comments
Column contamination	Trim the column Solvent rinse the column	Remove 0.5-1 meter from the front of the column Only for bonded and cross-linked phases Check for inlet contamination
Column activity	Irreversible. Replace the column	Only affects active compounds
Solvent-phase polarity mismatch	Change sample solvent to a single solvent Use a retention gap	More tailing for the early eluting peaks or those closest to the solvent front 3-5 meter retention gap is sufficient
Solvent effect violation for splitless or on-column injections	Decrease the initial column temperature	Peak tailing decreases with retention
Too low of a split ratio	Increase the split ratio	Flow from split vent should be 20 ml/min or higher
Poor column installation	Reinstall the column	More tailing for the early eluting peaks
Some active compounds always tail	None	Most common for amines and carboxylic acids

Split Peaks



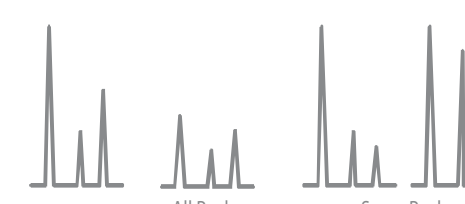
Possible Cause	Solution	Comments
Injection technique	Change technique	Usually related to erratic plunger depression or having sample in the syringe needle. Use an auto injector.
Mixed sample solvent	Change sample solvent to a single solvent	Worse for solvents with large differences in polarity or boiling points
Poor column installation	Reinstall the column	Usually a large error in the insertion distance
Sample degradation in the injector	Reduce the injector temperature	Peak broadening or tailing may occur if the temperature is too low
Poor sample focusing	Change to an on-column injection Use a retention gap	Requires an on-column injector For splitless and on-column injection

Retention Time Shift

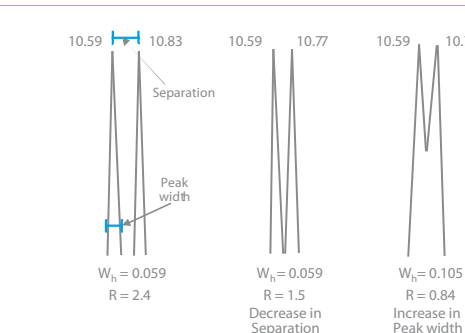


Possible Cause	Solution	Comments
Change in carrier gas velocity	Check the carrier gas velocity	All peaks will shift in the same direction by approximately the same amount
Change in column temperature	Check the column temperature	Not all peaks will shift by the same amount
Change in column dimension	Verify column identity	
Large change in compound concentration	Try a different sample concentration	May also affect adjacent peaks. Sample overloading is corrected with an increase in split ratio or sample dilution.
Leak in the injector	Leak check the injector	A change in peak size usually occurs also
Blockage in a gas line	Clean or replace the plugged line	More common for the split line; also check flow controllers and solenoids
Septum leak	Replace septum	Check for needle barb
Sample solvent incompatibility	Change sample solvent Use a retention gap	For splitless injection

Change in Peak Size



Possible Cause	Solution	Comments
Change in detector response	Check gas flows, temperatures and settings Check background level or noise	All peaks may not be equally affected May be caused by system contamination and not the detector
Change in the split ratio	Check split ratio	All peaks may not be equally affected
Change in the purge activation time	Check the purge activation line	For splitless injection
Change in injection volume	Check the injection technique	Injection volumes are not linear
Change in sample concentration	Check and verify sample concentration	Changes may also be caused by degradation, evaporation, or variances in sample temperature or pH
Leak in the syringe	Use a different syringe	Sample leaks passed the plunger or around the needle; leaks are not often readily visible
Column contamination	Trim the column Solvent rinse the column	Remove 0.5-1 meter from the front of the column Only for bonded and cross-linked phases
Column activity	Irreversible	Only affects active compounds
Coelution	Decrease column temperature or stationary phase	Decrease column temperature and check for the appearance of a peak shoulder or tail
Change in injector discrimination	Maintain the same injector parameters	Most severe for split injections
Sample flashback	Inject less, use a larger liner, reduce the inlet temperature	Less solvent and higher flow rates are most helpful
Decomposition from inlet contamination	Clean the injector; replace liner, gold seal	Only use deactivated liners and glass wool in the inlet



Loss of Resolution

Possible Cause	Solution	Comments
Decrease in separation	Check the column temperature	Differences in other peaks will be visible
Different column dimensions or phase	Verify column identity	Differences in other peaks will be visible
Coelution with another peak	Change column temperature	Decrease column temperature and check for the appearance of a peak shoulder or tail
Increase in peak width	Check the carrier gas velocity	A change in the retention time also occurs
Change in carrier gas velocity	Check the carrier gas velocity	
Column contamination	Trim the column Solvent rinse the column	Remove 0.5-1 meter from the front of the column Only for bonded and cross-linked phases
Change in the injector	Check the injector settings	Typical areas: split ratio, liner, temperature, injection volume
Change in sample concentration	Try a different sample concentration	Peak widths increase at higher concentrations
Improper solvent effect, lack of focusing	Lower oven temperature, better solvent/sample phase polarity match, use a retention gap	For splitless injection