

GC Troubleshooting Guide

Troubleshooting Basics



Take your system apart **logically** – don't just immediately reach for the tool box.

- Look at the symptoms:**
- Consider when and how the symptoms started (gradually or immediately).
 - Note any changes to the system.
 - Look for patterns.

Consider the possibilities – use troubleshooting tables to determine potential causes.

Isolate the problem – eliminate the possibilities one at a time. This allows you to determine the actual cause and to take preventative measures.

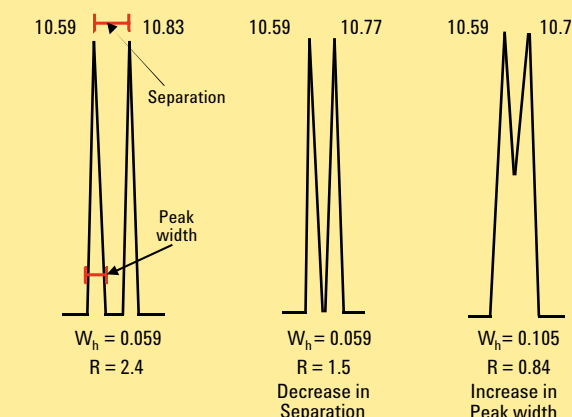
Don't overlook the obvious – gases, leaks, temperatures, etc.

Resolution Problems

1

Loss of Resolution

Resolution is a function of both separation (time between the peaks) and peak width.



Decrease in separation:

Possible cause	Solution	Comments
Different column temperature	Check 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 the column temperature.	Decrease column temperature and check for the appearance of a peak shoulder or tail.
Column contamination – resulting in a change in the column selectivity	Trim the column. Solvent rinse the column.	Remove ½-1 meter from the front of the column. Only for bonded and cross-linked phases.

Increase in peak width:

Possible cause	Solution	Comments
Change in carrier gas velocity	Check carrier gas velocity.	A change in retention time also occurs.
Column contamination	Trim the column. Solvent rinse the column.	Remove ½-1 meter from the front of the column. Only for bonded and cross-linked phases.
Inlet liner contamination	Clean or replace liner.	
Change in the injector	Check the injector settings.	Typical areas: split ratio, liner, temperature, injection volume.
Change in sample concentration or solvent	Try a different sample concentration.	Peak widths increase at higher concentrations.
Improper solvent effect, lack of focusing	Lower oven temperature. Choose different solvent for better solvent/sample polarity match. Use a retention gap.	For splitless injections.

Baseline Problems

1

Excessive Column Bleed

Column bleed is defined as the rise in the baseline as the column approaches its upper temperature limit. If there are peaks present, excessive noise, baseline wander, or baseline rise at lower oven temperatures it is not column bleed. In such cases there is some cause other than column degradation such as contamination.



Possible cause	Solution	Comments
Thermal damage to column	Remove column from detector and bake-out overnight, reinstall and condition as usual.	Use GC maximum temperature function.
Oxygen damage to column	Columns damaged by oxygen will usually need to be replaced although an overnight bake-out may be attempted.	Perform periodic leak checks. Change septa regularly. Use high quality carrier gases. Install and maintain oxygen traps.
Chemical phase damage to column	Remove ½ to 5 meters from the front of the column.	Perform sample prep to remove inorganic acids and bases from the sample. Install guard column and trim frequently. If acids or bases must be used, choose HCl or NH ₄ OH, or an organic alternative.

2

Erratic Baseline (wander, drift)



Possible cause	Solution	Comments
Inlet contamination	Clean the injector, replace liner, gold seal.	Try a condensation test; gas lines may also need cleaning. Take steps to prevent sample backflash (reduce injection volume, lower inlet temperature, use larger volume liner).
Column contamination	Bake-out column. Solvent rinse the column.	Limit bake-out to 1-2 hours. Only for bonded and cross-linked phases. Check for inlet contamination.
Incompletely conditioned column	Fully condition the column.	More critical for trace level analysis.
Un-equilibrated detector	Allow the detector to stabilize.	Some detectors may require up to 24 hours to fully stabilize.
Change in carrier gas flow rates during the temperature program	Normal in many cases.	MS, TCD and ECD respond to carrier gas flow rate changes.
Contaminated gases	Use appropriate purifier to remove contaminants.	More of a problem for detector gases.
Column and inlet liner misaligned	Check installation of column end and inlet liner, adjust if necessary.	Causes a baseline change after a large peak.
Large leak at septum during injection and for a short time thereafter	Replace septum. Use smaller diameter needles.	Causes a baseline change after a large peak. Common with large diameter needles.
Sample decomposing	Remove inlet liner and check cleanliness. Use new, deactivated liner or replace glass wool and packing.	Causes a baseline rise before or after a peak.

3

Noisy Baseline

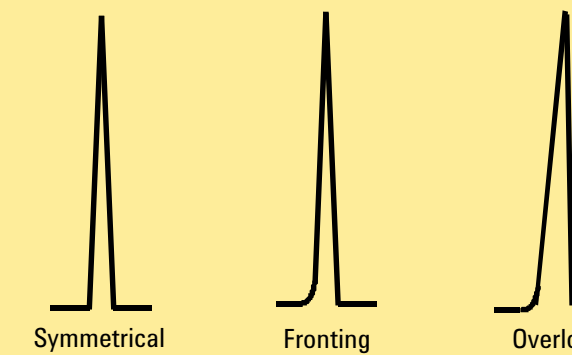


Possible cause	Solution	Comments
Inlet contamination	Clean the injector, replace liner, gold seal.	Try a condensation test; gas lines may also need cleaning.
Column contamination	Bake-out the column. Solvent rinse the column.	Limit the bake-out to 1-2 hours. Only for bonded and cross-linked phases. Check for inlet contamination.
Detector contamination	Clean the detector.	Usually the noise increases over time and not suddenly.
Contaminated or low quality gases	Replace spent gas purifier. Use purifiers to remove contaminants. Use better grade gases.	More of a problem for detector gases.
Column inserted too far into the detector	Reinstall the column.	Consult the GC manual for the proper installation distance.
Incorrect detector gas flow rates	Adjust the flow rates to the recommended values.	Consult the GC manual for the 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, NPD bead	Replace appropriate part.	
Septum degradation	Replace septum.	For high temperature applications use an appropriate septum.

Peak Problems

1

Fronting Peaks

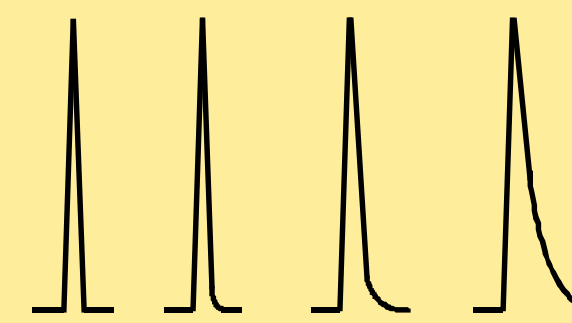


Possible cause	Solution	Comments
Column overload	Reduce mass amount of the analyte to the column. Decrease injection volume, dilute sample, increase split ratio.	Most common cause for fronting peaks.
Improper column installation	Reinstall the column in the injector.	Consult the GC manual for the proper 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 a retention gap may help.	
Mixed sample solvent	Change sample solvent.	Worse for solvents with large differences in polarity or boiling points.

2

Tailing Peaks

Hint: Which peaks are tailing? Are they active compounds, all compounds, or are there trends (such as early eluters or late eluters)?

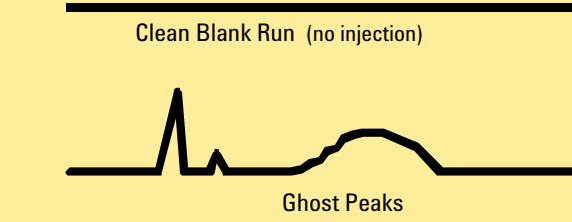


Possible cause	Solution	Comments
Severe column contamination	Trim the column. Solvent rinse column.	Remove ½-1 meter from the front of the column. Only for bonded and cross-linked phases. Check for inlet contamination. Tailing will sometimes increase with compound retention.
Active column	Cut off 1 meter from the front of the column. Replace column.	Only affects active compounds. Usually produces tailing that increases with retention.
Improper column installation, leak, or column end poorly cut	Re-cut and reinstall the column into the inlet. Replace ferrule. Confirm installation is leak free.	Make a clean, square cut using a reliable cutting tool. Consult the GC manual for the proper installation distance. More tailing for early eluting peaks.
Contaminated or active liner and gold seal	Use new, deactivated liner. Clean or replace gold seal.	Only affects active compounds.
Solid particles in liner	Clean or replace liner.	
Needle hitting and breaking packing in inlet liner	Partially remove packing from liner or use without packing.	
Solvent/column not compatible	Use a different 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.
Split ratio too low	Increase split ratio.	Flow from split vent should be ≥ 20mL/min.
Solvent effect violation for splitless or on-column injections	Decrease the initial column temperature to 10-25 °C below solvent boiling point.	Peak tailing decreases with retention.
Poor injection technique	Change technique.	Usually related to erratic plunger depression or having sample in the syringe needle. Use an autosampler.
Inlet temperature too high	Decrease inlet temperature by 50°C.	Tailing generally worse for early eluters.
Inlet temperature too low	Increase inlet temperature by 50°C.	Tailing usually increases with retention.
Dead volume in system	Reduce dead volume. Transfer line connections, fused silica unions, etc.	Peak tailing decreases with retention.
Cold spots (condensation)	Eliminate cold spots. Commonly at transfer lines.	Tailing usually increases with retention.
Overloading of PLOT columns	Reduce the amount injected onto column.	

4

Ghost Peaks

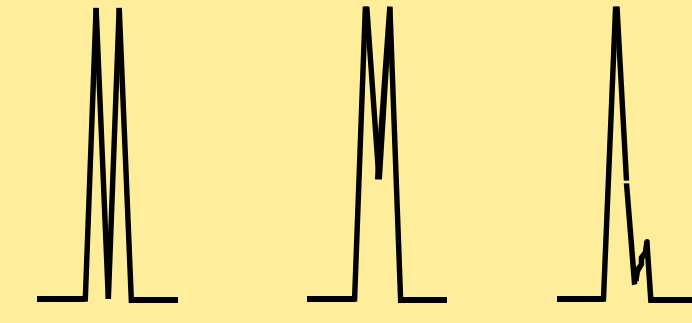
Ghost peaks (or "extra" peaks in a chromatogram) are not column bleed. When evaluating ghost peaks it is important to look at the peak width of the ghost peaks.



Possible cause	Solution	Comments
Contaminants introduced with sample	Sample or solvent cleanup.	Contaminants in sample process or solvent.
Inlet contamination	Clean the injector, replace liner, gold seal, and septum.	Try a condensation test; gas lines may also need cleaning. Take steps to prevent sample backflash (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 cleanup, handling, transfer, and storage.	
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.

3

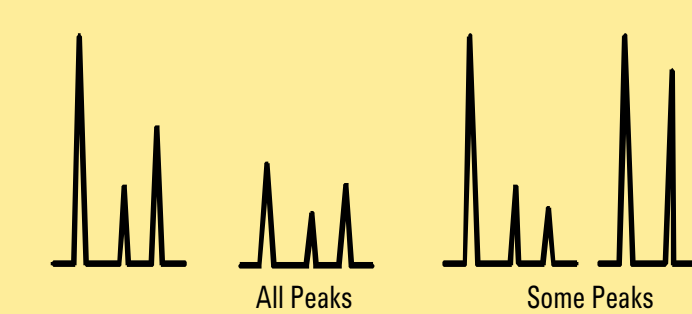
Split Peaks



Possible cause	Solution	Comments
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.
Mixed sample solvent	Change sample solvent.	Worse for solvents with large differences in polarity or boiling points.
Solvent/column not compatible	Use a different solvent. Use a retention gap.	
Poor sample focusing	Use a retention gap.	For splitless, on-column, and PTV injectors.
Sample degradation in injector (only some peaks show splitting)	Reduce inlet temperature. Derivatize sample to make compounds thermally stable. Change to an on-column injection.	Peak broadening or tailing may occur if the temperature is too low. Requires an on-column injector.
Severe detector overload	Reduce the amount of sample on column.	May only affect some peaks.

4

Changes in Peak Size

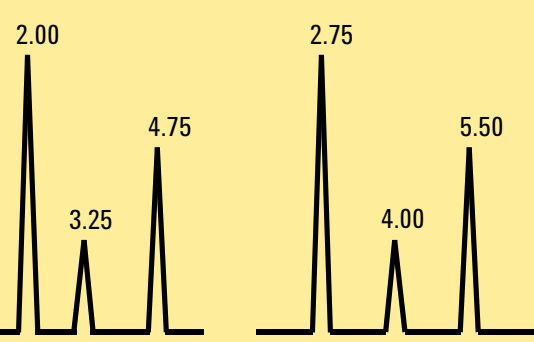


Possible cause	Solution	Comments
Change in detector response	Check gas flows, temperatures and settings. Check background level or noise.	All peaks may not be equally affected. May be caused by system contamination not the detector.
Change in the split ratio	Check split ratio.	All peaks may not be equally affected.
Change in the purge activation time	Check the purge activation time.	For splitless injections.
Change in injection volume	Check injection technique.	Injection volumes are not linear.
Change in injector discrimination	Maintain the same injector parameters: flows, temperatures, liners, etc.	Most severe for split injections. All peaks may not be equally affected.
Change in sample concentration	Check and verify sample concentration.	May be caused by degradation, evaporation or variances in sample temperature or pH.
Leak in the syringe	Use a different syringe.	Sample leaks past the plunger or around the needle; leaks are often not readily visible.
Column contamination	Trim the column. Solvent rinse the column.	Remove ½-1 meter from the front of the column. Only for bonded and cross-linked phases.
Column activity	Trim or replace the column.	Only affects active compounds.
Coelution	Change column temperature or stationary phase.	Decrease column temperature and check for the appearance of a peak shoulder or tail.
Sample backflash	Inject less, use a larger liner, and/or reduce the inlet temperature.	Less solvent and higher flow rates are most helpful.
Decomposition from inlet contamination	Clean the inlet, replace the liner, replace the gold seal.	Only use deactivated liners and glass wool in the inlet.
Loss of sample prior to introduction to the GC	Check sample handling: sample preparation, transfer and storage.	

Retention Time Problems

1

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 dimensions	Verify column identity.	
Large change in compound concentration	Try a different sample concentration.	May also affect adjacent peaks. Sample overloading is corrected with an increased split ratio, sample dilution or decreased injection volume.
Leak in the injector or column connection	Leak check the injector and column installation.	Usually accompanied by peak size change.
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 the septum.	Check for needle barb.
Sample solvent incompatibility	Change solvent. Use a retention gap.	For splitless injection.
Contamination	Trim the column. Solvent rinse the column.	Remove ½-1 meter from the front of the column. Only for bonded and cross-linked phases.

