GC Troubleshooting Guide

Your guide to solving common problems and staying productive

Comments

Usually related to erratic plunger

direction by approximately the

Measure the carrier gas velocity

with an increase in split ratio or

Only affects active compounds

Decrease column temperature

a peak shoulder or tail

phases

Typical areas: split ratio, liner, temperature, injection volume

Peak widths increase at higher

For splitless injection

and check for the appearance of

Most severe for split injections

with an unretained compound

same amount

sample dilution

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 between 40 to 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 when 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 first chromatogram contains a larger amount of peaks and baseline instability, the incoming carrier gas line or the carrier gas is contaminated.
- 7. If both chromatograms contain few peaks or little baseline drift, the carrier gas and incoming carrier gas lines are relatively clean.

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

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

Locate supplies and parts with ease: agilent.com/chem/partsfinder

Find the correct GC column for your application: selectgc.chem.agilent.com

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Ghost Peaks or Carryover



Solution	Comments
Sample or solvent cleanup	Contaminants in sample process or solvent
Clean the inlet and 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 anlet temperature, use larger volume liner)
Replace septum	Use a high-quality septum appropriate for the inlet temperature
Check sample handling steps for potential contamination sources: sample cleanup, handling, transfer, and storage	Usually occurs after changing a gas cylinder
	Sample or solvent cleanup Clean the inlet and replace liner, gold seal, and septum Replace septum Check sample handling steps for potential contamination sources: sample cleanup, handling,

Limit bake-out to 1 to 2 hours.

Only for bonded and cross-linked

Consult the GC manual for the

Worse for solvents with large

Remove 0.5-1 meter from the

Only for bonded and cross-linked

Check for inlet contamination

Only affects active compounds

peaks or those closest to the

3 to 5 meter gap is sufficient

Peak tailing decreases with

20 mL/min or higher

carboxylic acids

Flow from split vent should be

More tailing for the early eluting

Most common for amines and

More tailing for the early eluting

Comments

front of the column

differences in polarity or boiling

Excessive Baseline Noise



Baseline Instability

or Disturbances

Fronting Peaks

Possible Cause	Solution	Comments
Injector contamination	Clean the injector; replace liner and gold seal	Try a condensation test; gas lines may also need cleaning
Column contamination	Bake out the column. Implement backflush to avoid contamination.	Limit the bake-out to 1 to 2 hours
	Solvent rinse the column	Only for bonded and cross-linked phases
		Check for inlet contamination

carrier gas, or carrier gas lines.

Implement backflush to avoid

than sample peaks with similar

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 Clean filters	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

Contum degradation	Donloop contum	For high tomporature applications
Old detector filament, lamp, or electron multiplier	Replace appropriate part	
Leak when using an MS, ECD, or TCD	Create leak-free column unions with an UltiMetal Plus Flexible Metal ferrule or a Self-Tightening column nut	Usually at the column fittings or injector
Incorrect detector gas flow rates	Adjust the flow rates to the recommended values	Consult GC manual for proper flor rates
the detector		insertion distance

	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. Implement backflush to avoid contamination.	Limit a bake-out to 1 to 2 hours
	Unequilibrated detector	Allow the detector to stabilize	Some detectors may require up to 24 hours to fully stabilize
	Incompletely conditioned	Fully condition the column	More critical for trace-level

	column		analyses
_ [Change in carrier gas flow rate during the temperature program	Often normal	MS, TCD, and ECD respond to changes in carrier gas flow rate
	Possible Cause	Solution	Comments
	Column overload	Reduce mass amount of the analyte to the column. Decrease injection volume, dilute	Most common cause for fronting peaks

	injector	proper installation distar
Injection technique	Change technique	Usually related to erratic depression or having sa in the syringe needle. Us autosampler
Compound very soluble in	Change solvent. Using a retention	More critical for trace-lev

Change sample solvent

Implement backflush to avoid

Irreversible. Replace the column

single solvent

temperature

Use a retention gap

Decrease the initial column

Increase the split ratio

Reinstall the column

Utilize inert flow path

consumable components

(agilent.com/chem/inert)

sample, increase split ratio

Reinstall the column in the

Tailing Peaks	Possible Cause	Solution
3	Column contamination	Trim the column.

Mixed sample solvent

Solvent effect violation for

Too low of a split ratio

Poor column installation

Some active compounds

always tail

splitless or on-column injection

	l.		contamination.
			Solvent rinse the column
7		Column activity	Irreversible. Replace the colum
		Solvent-phase polarity mismatch	Change sample solvent to a



Possible Cause

Column activity

Change in injector

Change in the injector

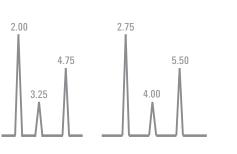
Improper solvent effect,

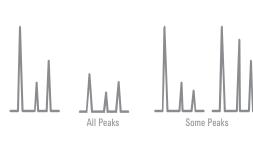
Change in sample concentration Try a different sample

discrimination

Coelution

Retention Time Shift





		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	If the temperature is too low, peal broadening or tailing may occur
	Change to an on-column injection	Requires an on-column injector
Poor sample focusing	Use a retention gap	For splitless and on-column injection
Possible Cause	Solution	Comments
Change in carrier gas velocity	Check the carrier gas velocity	All peaks will shift in the same

Solution

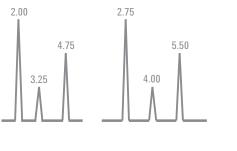
Change in column temperature Check the column temperature

Verify column identity

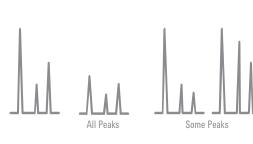
Try a different sample

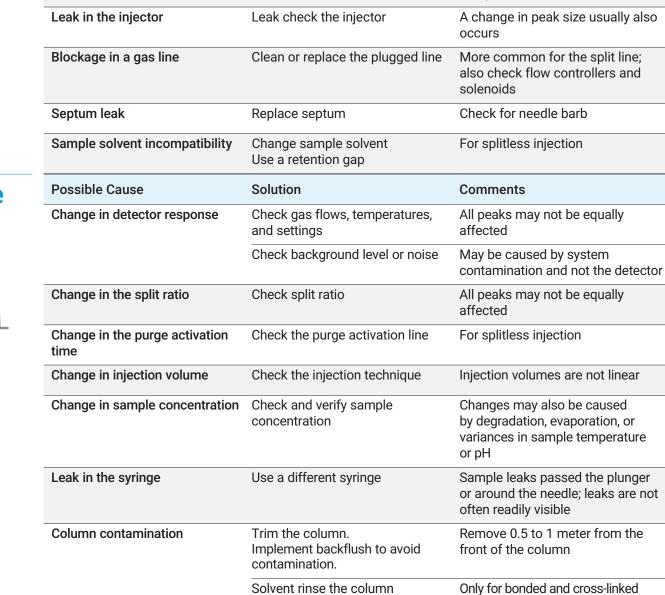
concentration

Change technique

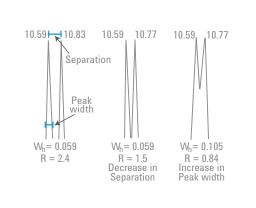


Change in Peak Size





Loss of Resolution



	Sample flashback	Use Agilent Vapor Volume Calculator to adjust injection size, liner volume, inlet temperature, or solvent	Less solvent and higher flow rates are most helpful
	Decomposition from inlet contamination	Clean the injector; replace liner and gold seal	Only use deactivated liners and glass wool in the inlet
	Possible Cause	Solution	Comments
	Decrease in separation		
	Different column temperature	Check the column temperature	Differences in other peaks will be visible
	Different column dimensions or phase	Verify column identity, measure the carrier gas velocity	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		
	Change in carrier gas velocity	Check the carrier gas velocity	A change in the retention time also occurs
	Column contamination	Trim the column. Implement backflush to avoid contamination.	Remove 0.5 to 1 meter from the front of the column
		Solvent rinse the column	Only for bonded and cross-linked

Check the injector settings

Lower oven temperature, better

solvent, sample phase polarity

match, use a retention gap

Irreversible

parameters

Change column temperature

Maintain the same injector

or stationary phase

