GC Troubleshooting Guide

Your guide to solving common problems and staying productive

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

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Condensation Test

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

- 1. Leave the GC between 40 to 50 °C for more hours.
- 2. Run a blank analysis (i.e., start the GC, injection) using the normal temperature and instrument settings.
- 3. Collect the chromatogram for this blan
- 4. Immediately repeat the blank run when is completed. Do not allow more than 5 elapse before starting the second blan
- 5. Collect the chromatogram for the seco and compare it to the first chromatogra
- 6. If the first chromatogram contains a la of peaks and baseline instability, the inc gas line or the carrier gas is contamina
- 7. If both chromatograms contain few pea baseline drift, the carrier gas and incon lines are relatively clean.

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And other

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	Ghost Peaks	Possible Cause	Solution	Comments
	or Carryover	Contaminants introduced with sample	Sample or solvent cleanup	Contaminants in sample process or solvent
gas (e.g., ghost	Clean Blank Run (no injection)	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 backflush (reduce injection volume lower inlet temperature, use larger volume liner)
8 or	Ghost Peaks	Septum bleed	Replace septum	Use a high-quality septum appropriate for the inlet temperature
but with no		Contamination of sample before introduction to the GC	Check sample handling steps for potential contamination sources: sample cleanup, handling, transfer, and storage	Usually occurs after changing a gas cylinder
e conditions		Semivolatile 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 to 2 hours. Only for bonded and cross-linked phases
nk run.				
n the first one 5 minutes to	Excessive Baseline Noise	Possible Cause Injector contamination Column contamination	SolutionClean the injector; replace liner, gold sealBake out the column	Comments Try a condensation test; gas lines may also need cleaning Limit the bake-out to 1 to 2 hours
ik run. And blank run	mmmmmmMMMMMMMMmmmmmmmmm		Solvent rinse the column	Only for bonded and cross-linked phases Check for inlet contamination
ond blank run ram.		Detector contamination	Clean the detector	Usually the noise increases over time and not suddenly
arger amount		Contaminated or low-quality gases	Use better grade gases; also check for expired Gas Clean filters	Usually occurs after changing a gas cylinder
coming carrier		Column inserted too far into the detector	Reinstall the column	Consult GC manual for proper insertion distance
ated.		Incorrect detector gas flow rates	Adjust the flow rates to the recommended values	Consult GC manual for proper flor rates
eaks or little ming carrier gas		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
		Old detector filament, lamp, or electron multiplier	Replace appropriate part	
leos:		Septum degradation	Replace septum	For high temperature applications use an appropriate septum
1003.	Decelie a le atabilite	Possible Cause	Solution	Comments
sit	Baseline Instability or Disturbances	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 to 2 hours
		Unequilibrated detector	Allow the detector to stabilize	Some detectors may require up to 24 hours to fully stabilize
		Incompletely conditioned column Change in carrier gas flow rate	Fully condition the column Often normal	More critical for trace-level analyses MS, TCD, and ECD respond to
olication:		during the temperature program		changes in carrier gas flow rate
		Possible Cause	Solution	Commente
	Fronting Peaks	Column overload	Solution Reduce mass amount of the analyte to the column. Decrease injection volume, dilute sample, increase split ratio	Comments Most common cause for fronting peaks
		Improper column installation	Reinstall the column in the injector	Consult the GC manual for the proper installation distance
level of ay productivity,	Symmetrical Fronting Overload	Injection technique	Change technique	Usually related to erratic plunger depression or having sample in the syringe needle. Use an autosampler
reliability and		Compound very soluble in injection solvent	Change solvent. Using a retention gap may help	More critical for trace-level analyses
provide the		Mixed sample solvent	Change sample solvent	Worse for solvents with large differences in polarity or boiling points
	Teiling Deales	Possible Cause	Solution	Comments
y	Tailing Peaks	Column contamination	Trim the column	Remove 0.5-1 meter from the front of the column
Agilent level on a			Solvent rinse the column	Only for bonded and cross-linked phases Check for inlet contamination
Agilent JaW GC Columns		Column activity	Irreversible. Replace the column	Only affects active compounds
		Solvent-phase polarity mismatch	Change sample solvent to a single solvent	More tailing for the early eluting peaks or those closest to the solvent front
		Solvent effect violation for	Use a retention gap	3 to 5 meter gap is sufficient
		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	Utilize inert flow path consumable components (agilent.com/chem/inert)	Most common for amines and carboxylic acids



Comments

injector

points

distance

Usually related to erratic plunger depression or having sample in

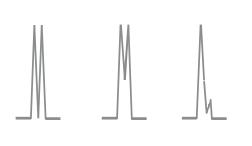
the syringe needle. Use an auto

Worse for solvents with large

differences in polarity or boiling

Usually a large error in the insertion

Split Peaks



pin	r cans		
		K	

Possible Cause

Injection technique

Mixed sample solvent

Poor column installation

Sample degradation in the

Poor sample focusing

Possible Cause

concentration

Septum leak

Leak in the injector

Blockage in a gas line

Change in carrier gas velocity

Change in column dimension

Large change in compound

injector

Solution

Change technique

a single solvent

Reinstall the column

Use a retention gap

Check the carrier gas velocity

Verify column identity

Try a different sample

Leak check the injector

concentration

Replace septum

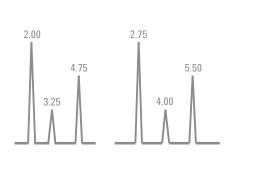
Solution

Change in column temperature Check the column temperature

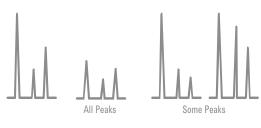
Change sample solvent to

Reduce the injector temperature

Retention Time Shift



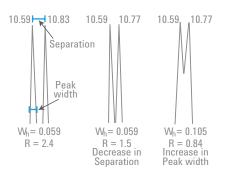
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bongo	in	Dook Cizo	
Janue		Peak Size	



Loss of Resol

or phase

lack of focusing



	Sample solvent incompatibility	Change sample solvent Use a retention gap
ak Size	Possible Cause	Solution
	Change in detector response	Check gas flows, temperatures, and settings
Some Peaks		Check background level or noise
	Change in the split ratio	Check split ratio
	Change in the purge activation time	Check the purge activation line
	Change in injection volume	Check the injection technique
	Change in sample concentration	Check and verify sample concentration
	Leak in the syringe	Use a different syringe
	Column contamination	Trim the column
		Solvent rinse the column
	Column activity	Irreversible
	Coelution	Change column temperature or stationary phase
	Change in injector discrimination	Maintain the same injector parameters
	Sample flashback	Use Agilent Vapor Volume Calculator to adjust injection size, liner volume, inlet temperature, or solvent
	Decomposition from inlet contamination	Clean the injector; replace liner, gold seal
lution	Possible Cause	Solution
	Decrease in concretion	

Comments **Decrease in separation** Differences in other peaks will be Different column temperature Check the column temperature visible Differences in other peaks will be **Different column dimensions** Verify column identity, measure the carrier gas velocity visible Coelution with another peak Change column temperature Decrease column temperature a peak shoulder or tail Increase in peak width Check the carrier gas velocity Change in carrier gas velocity also occurs Column contamination Trim the column front of the column Solvent rinse the column phases Check the injector settings Change in the injector

Change in sample concentration Try a different sample concentration Improper solvent effect,

Lower oven temperature, better For splitless injection solvent, sample phase polarity match, use a retention gap

If the temperature is too low, peak broadening or tailing may occur Change to an on-column injection Requires an on-column injector For splitless and on-column injection Comments All peaks will shift in the same direction by approximately the same amount Not all peaks will shift by the same amount

> Measure the carrier gas velocity with an unretained compound May also affect adjacent peaks. Sample overloading is corrected with an increase in split ratio or sample dilution A change in peak size usually also occurs

Clean or replace the plugged line More common for the split line; also check flow controllers and solenoids Check for needle barb

For splitless injection

All peaks may not be equally

Comments

affected May be caused by system contamination and not the detector All peaks may not be equally affected For splitless injection Injection volumes are not linear Changes may also be caused by degradation, evaporation, or variances in sample temperature or pH Sample leaks passed the plunger or around the needle; leaks are not often readily visible Remove 0.5 to 1 meter from the front of the column Only for bonded and cross-linked

phases Only affects active compounds Decrease column temperature

and check for the appearance of a peak shoulder or tail Most severe for split injections

Less solvent and higher flow rates are most helpful

Only use deactivated liners and glass wool in the inlet

and check for the appearance of A change in the retention time Remove 0.5 to 1 meter from the Only for bonded and cross-linked Typical areas: split ratio, liner, temperature, injection volume Peak widths increase at higher

concentrations