GC COLUMN CONDITIONING & TROUBLESHOOTING TIPS

Condition Column Before Use

To flush out residual contaminants at elevated temperature and make it fit for reliable use.

Procedure:

1. Connect the column to the GC inlet. Do not connect the column to the detector.
2. Set the GC oven to 40°C and purge column at this temperature for 10 - 40 minutes.
3. Set the temperature gradient to 10°C/min.
4. Program the oven either to 20 °C above the final temperature called for in the analysis or to the column's maximum ISOTHERMAL temperature — whichever is lower.
5. Once the upper temperature limit has been reached the column should be conditioned for the correct amount of time based on the dimensions and phase type.
6. With carrier gas still flowing, cool the oven, install a fitting and ferrule onto the detector end of the column, connect the column to the detector, and repeat steps 2-4.

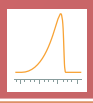
Flow rates and time for purging capillary GC column

Column ID (mm)	Minimum Flow Rate (mL/min)	Minimum Purge Time (min)
0.53	5.0	10
0.32	1.5	20
0.25	1.0	25
0.18	0.8	30
0.10	0.5	40

Conditioning time for capillary GC column

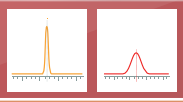
Length (m)	Film Thickness (µm)	Conditioning Time for Phase Type (min)		
		Non-polar	Mid Polar	Polar
< 30 m	< 0.5	15	20	30
	0.5 – 1.0	30	40	45
	> 1.0	60	60	60
30 – 60 m	< 0.5	30	40	60
	0.5 – 1.0	45	60	90
	> 1.0	60	80	120
> 60 m	< 0.5	60	80	80
	0.5 – 1.0	90	120	120
	> 1.0	120	160	160

PEAK FRONTING



Causes	Solutions
Incompatible stationary phase	• Choose appropriate stationary phase
Column overload	• Reduce amount injected, dilute sample or increase split ratio. • Increase column inner diameter and/or film thickness

BROAD PEAK



Causes	Solutions
High dead column	• Minimize dead volume in GC systems; check proper column installation and other connections (septa & glass inserts).
Low flow rates	• Check and optimize injector, detector and make-up gas flow rates.
Slow GC oven program	• Increase GC oven temperature ramping rate.
Poor analyte / solvent focusing	• Lower GC oven start temperature.
Column film is too thick	• Reduce compound retention by reducing film thickness and length.
Sample carryover	• *See carryover / ghost peaks section.*

SPLIT PEAK

Causes	Solutions
Mismatch solvent/stationary phase polarity	• Change solvent or stationary phase to allow wetting.
Incomplete vaporization	• Add surface area (e.g. wool) to glass insert. • Use proper injector temperature.
Sample overloading	• Inject less sample (dilute, split injection, reduce injection volume).
Fast autosampler injection into open glass insert	• Use wool or slower injection speed

POOR RETENTION TIME REPRODUCTIVITY

Causes	Solutions
Leaks	• Check for leaks from any column connections. • Replace parts (septa, O-rings, glass insert) if necessary.
Analyte adsorption	• Maintain glass insert and GC column. • Use properly deactivated glass inserts and columns.
Resolution / integration issues	• Reduce sample amount (avoid overload).
Incorrect oven temperature program	• Check and optimize oven temperature program.
Incorrect carrier gas flow rate	• Check and optimize carrier gas flow rat • Repair or replace parts if necessary.
Poor control of temperature program	• Confirm GC oven program falls within instrument manufacturer's recommendation.
Insufficient oven equilibration time	• Extend GC oven equilibration time.
Delay between pushing start and actual injection (manual injection)	• Use autosampler or standardize manual injection procedure.

NO PEAK

Causes	Solutions
Injection problems	• Check carrier gas flow. • Clean or replace clogged syringe. • Check if sample is in syringe. • Injecting into wrong inlet, reset autosampler.
Broken column	• Replace column.
Column installation problem	• Re-install column.
Detector problems	• Check if detector is turned on. • Check detector gas and flow rates.

Basic Steps

Follow these three steps To isolate where the problems is. Check the obvious explanations first and change only one thing at a time!

Check the Basics:

- Power supply
- Electrical connections
- Signal connections
- Syringe condition
- Sample preparation
- Analytical conditions
- Temperature settings
- Gas purity
- Gas flows

Identify the Cause:

- Define the problem clearly; for example, "Over the last four days, only the phenols in my sample have been tailing."
- Review sample and maintenance records to identify trends in the data or problem indicators, such as area counts decreasing over time or inlet maintenance not being performed as scheduled.
- Use a logical sequence of steps to isolate possible causes.

Document Everything:

- Document all troubleshooting steps and results; this may help you identify and solve the next problem faster.
- Always inject a test mix and compare to previous data to ensure restored performance.

Still having problems?

Still struggling? Let us know!

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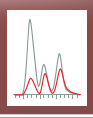
CHANGES IN RESPONSE



Causes	Solutions
Sample issue	• Check sample concentration. • Check sample preparation procedure. • Check sample decomposition/shelf life.
Syringe problems	• Replace syringe • Check autosampler operation.
Electronics	• Verify signal settings and adjust if needed
Dirty or damaged detector	• Perform detector maintenance or replace parts
Flow/temperature settings wrong or variable	• Verify Steady flow rates and temperatures , then adjust settings and/or replace parts if needed
Adsorption/reactivity	• Remove contamination and use properly deactivated liner and column
Leaks	• Check for leaks at all connections and repair connections as needed.
Change in sample introduction/injection method	• Verify injection technique and change back to original technique. • Check that split ratio is correct. • Verify that the splitless hold time is correct.

PEAK TAILING

Causes	Solutions
Leak	• Check for leaks, replace parts (septa, O-rings, glass insert) if needed.
Column installation issue	• Minimize dead volume. • Check that column is cut properly (square). • Check column installation distance to inlet and detector.
Adsorption due to surface activity or contaminant	• Use clean and deactivated glass insert, septa and column. • Trim inlet end of column. • Replace column.
Adsorption due to chemical nature of compound	• Derivatize compound.

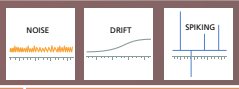
HIGH BLEED

Causes	Solutions
Improper column conditioning	• Increase conditioning time and/or temperature.
Contamination	• Clean injector and detector. • Change carrier gas and gas filters. • Trim column and/or heat column to maximum temperature to remove contaminants.
Oxidation of stationary phase	• Check for oxygen leaks across system and replace septa, glass inserts, ferrules. • Replace column.

POOR RESOLUTION

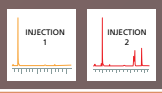
Causes	Solutions
Non-selective stationary phase	• Choose appropriate stationary phase and column dimension.
Poor efficiency	• Check and optimize carrier gas linear velocity and GC oven temperature program.
Sample overload	• Optimise sample concentration or amount on column by increasing split ratio.
Incorrect conditions	• Check and optimise temperature program, flow rates and column parameters.

UNSTABLE BASELINE



Causes	Solutions
Carrier gas leaks or contamination	• Check for leaks and replace parts (septa, O-rings, glass insert) if necessary • Change carrier gas and gas filters.
Injector or detector contamination	• Clean injector and detector.
Column contamination or bleeding	• Condition, trim and rinse column. • Replace column.
Septum coring/bleed	• Replace septum, change glass insert.
Variable gas flow	• Check system for leaks. • Check flow rates are steady and reproducible.
Detector not ready	• Allow enough time for detector flow and temperature to equilibrate.
Loose cable or circuit board connection	• Clean and repair electrical connections

CARRYOVER / GHOST PEAK



Causes	Solutions
Contaminated syringe or rinse solvent	• Clean or replace syringe • Replace rinse solvent
Backflush (sample volume exceed glass insert volume)	• Inject lower sample volume. • Increase split flow. • Use glass insert with packing • Use bigger inner diameter glass insert. • Increase head pressure. • Use slower injection rate. • Lower inlet temperature. • Check solvent expansion volume.
Last analysis ended too soon	• Extend analysis time to allow all component / matrix interferences to elute.