

GC Troubleshooting

Before you start any troubleshooting, it is essential to observe safe laboratory practices. Know the chemical and physical properties of any solvents used and have the appropriate Material Safety Data Sheets (MSDSs) readily available. All electrically powered instruments should be shut down and unplugged before starting. Eye protection should also be worn.

The following table lists common GC problems encountered, the possible causes and solutions for your quick reference.

Symptom	Cause	Recommended Solutions
Baseline Related Problems		
Baseline Drifting	Accumulation of stationary phase.	Remove the end section of the column.
	Carrier gas cylinder pressure too low to allow control.	Replace the carrier gas cylinder. Increase the pressure.
	Drifting carrier gas or combustion gas flows.	Check the gas controllers.
	Accumulation of impurities in the column.	Check impurity levels in the gas source. Use correct gas purity. Replace or install appropriate Gas Filters (see page 3-080).
Baseline Falling	Carrier gas leak in the system.	Perform a leak test. Check the tightness of the connections on the carrier gas line.
	Column is baking out.	Allow enough time for the column to stabilize.
Baseline Falling Away Slowly After a High Initial Value	Purge valve left closed during acquisition.	Alter the GC program. See your GC user manual for details.
	Inadequate purge flow rate.	Increase the purge flow rate.
	Purge valve left closed for too long.	Shorten the purge time.
	Solvent tail peak.	Increase the solvent delay. Shorten the purge time.
	Pre-filters are dirty. (when using a quadrupole MS detector)	Contact your service representative.
Baseline Rising	Accumulation of impurities in the column.	Check impurity levels in the gas source. Use correct gas purity. Replace or install appropriate Gas Filters (see page 3-080).
	Contaminated detector.	Check the detector and clean it.
	There is bleeding from the GC column.	Condition column. Change the column.
	Air is leaking into the system.	Trace and repair the leak.
Baseline Rising Under Temperature Program Control	Column contaminated.	Recondition the column.
Baseline High Standing Current	Carrier gas flow rate too high.	Reduce the carrier gas flow.
	Column contaminated.	Recondition the column.
	Contaminated gases.	Replace gas cylinders. Replace the gas filters.
	Excessive column stationary phase bleeding.	Check the oven temperature, ensuring that it doesn't exceed the column upper limit. Recondition the column. Replace the column.
	Loose connections.	Ensure that all interconnections and screw connections are tight.
Baseline Irregular Shape: Dip After Solvent Peak	Detector contaminated.	Bake out the detector. Clean the detector.
Baseline Irregular Shape: S-shaped	Excessive column bleed during column temperature programming.	Reduce the upper column temperature. Bake out the column. Install a high temperature column.
	Oxygen contamination is decomposing the stationary phase.	Install oxygen filters in the carrier gas line. Check the pneumatic and inlet systems for leaks. Use correct gas purity with low oxygen content.
Baseline High Frequency Noise	Contaminated detector.	Isolate the detector from the electronics. If noise disappears, clean the collector.
	Combustion gas flow too low or too high.	Check the detector gas flows.
	Column contaminated.	Condition the column.
	Contaminated detector gas supply.	Check the gas purity and install appropriate filters.
	Detector temperature higher than column maximum temperature.	Reduce the detector temperature to the column temperature upper limit.
Baseline Spiking	Loose column fittings.	Tighten fittings accordingly.
	Column too close to flame. (when using an FID)	Lower the column to the correct position (2-3mm below the tip of the jet).
	Dirty jet or detector.	Isolate the detector from the electronics. If the spiking disappears, clean the jet and the collector.
	FID temperature too low. (when using an FID)	Increase the FID temperature to at least 150°C.

Symptom	Cause	Recommended Solutions
Peak-Related Problems		
Peaks Broadening	Column flow too high.	Reduce the flow to slightly above optimum.
	Column flow too low.	Increase the flow to slightly above optimum.
	Split flow too low in split injection.	Increase the flow to 40-50mL/min.
	Column performances degraded.	Test the column at the optimum flow rate.
	Dirty injector.	Clean or replace the liner.
	Stationary phase accumulated in the outlet.	Remove the last two coils from the column.
	Detector base body temperature too low.	Increase the temperature to 5°C below the column maximum.
	The sample is overloading the column.	Reduce the amount and/or concentration of the sample.
Double Peaks	Injection speed too low.	Inject more rapidly in a smooth motion.
	Wrong autosampler injection speed or mode.	Use a higher speed.
Peak Fronting	Column or detector overloaded.	Decrease the injected amount. Decrease the analyte concentrations. Increase the split ratio.
	Column temperature too low.	Increase the temperature.
	Stationary phase too thin.	Use a thicker-film column.
	Poor injection technique.	Repeat, with better injection technique.
Ghost Peaks	Contaminated carrier gas.	Replace the cylinder. Replace the filter (see page 3-080).
	Contamination from laboratory glassware.	Ensure the glassware is clean and contamination-free.
	Decomposition of injected sample.	Decrease the injection port temperature. Use the on-column injection technique.
	Dirty injection solution.	Carry out adequate clean-up of sample prior to injection.
Broad Ghost Peaks	Contaminated inlet or pneumatics.	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
	Incomplete elution of previous sample.	Increase the final oven program temperature or total run time. Increase the column flow rate.
Irregular, Chair-shaped Peaks	Solvent flooding of column.	Increase the initial oven temperature. Reduce the injection volume (On-column). Install a retention gap (On-column).
No Peaks After Solvent Peak	Carrier gas flow too high.	Reduce the carrier gas flow rate.
	Combustion gas flow incorrect.	Check the combustion gas flow.
	Detector contaminated.	Bake out or clean the detector.
	FID flame extinguished by solvent peak.	Check the detector temperature and that flame is lit.
	Too much sample injected.	Inject less sample.
	Incorrect column position in S/SL injector (too high).	Check the column position.
No Peaks at All	Clogged syringe needle.	Replace or repair the syringe.
	Column broken or disconnected.	Check the column and connections.
	Defective electrometer or amplifier.	Check electrometer or amplifier and associated connections. Replace if required.
	Defective recording device.	Replace the recording device.
	FID flame is out.	Clean FID jet, check detector gas flows and re-light flame.
	Incorrect column position in S/SL injector (too high).	Check the column position.
Sample Peak Tailing	Column degradation causing activity.	Inject a test mixture and evaluate the column.
	Column/oven temperature too low.	Increase the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
	Column contaminated at inlet.	Trim first 10-20cm from column and re-install in injector.
	Glass wool or inlet liner causing activity.	Replace with fresh silanized wool and a clean inlet liner.
	Inlet temperature too low.	Increase the inlet temperature.
	Poor or obstructed column connections.	Remake the column inlet connection.
	Wrong stationary phase.	Replace the column according to the column manufacturer's literature.
	Solvent Peak Tailing	Incorrect column position in inlet.
Initial oven temperature too high (On Column).		Reduce the initial oven temperature.
Septum purge flow too low and/or split/splitless vent flow too low.		Check and adjust the septum purge and vent flows.
Too large injection size.		Reduce the injection size.

GC Troubleshooting *continued*

Symptom	Cause	Recommended Solutions
Unresolved Peaks	Carrier gas flow rate too high.	Reduce the carrier gas flow rate.
	Column deteriorated.	Replace the column.
	Column temperature too high.	Lower the column oven temperature.
	Column too short.	Use a longer column.
	Incorrect column choice.	Install a suitable column.
	Injection technique is not adequate.	Choose a correct injection technique.
Discrete High-intensity Contaminant Peaks	Bleed from the GC column.	Condition or change the column.
	Bleed from the septum.	Replace the septum.
	Sample vial septa are contaminating the sample.	Discard sample. Store samples upright, in a refrigerator. Use Teflon™ faced septa, with the Teflon facing downwards (i.e. towards the sample).

Results-Related Problems

Low Reproducibility of Peak Area	Concentration not compatible with the dynamic range of the detection system.	Ensure that the sample concentration is suitable for the detection system.
	Inappropriate injection technique.	Try a different injection technique.
	Injection parameters inappropriate.	Check the injection temperature. Check the flow rates.
	Non reproducible sample injection technique.	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
	Leaking syringe or septum.	Check and replace the syringe at regular intervals. Check and replace septum at regular intervals.
	Leaks at the injection.	Check the column connections. Run a leak check.
	Poor injection technique.	Carefully meter the injected amount. Use a clean, good-quality syringe.
	Poor split flow or ratio control.	Monitor the flow. Replace the in-line filter.
Poor Sensitivity Increased Retention Time	Carrier gas flow rate too low.	Increase the carrier gas flow rate. Locate and remove possible obstructions in the carrier gas line. Check the injector/column ferrules.
Poor Sensitivity with Normal Retention Time	Oven or injector parameters are not optimized.	Adjust the oven parameters. Adjust the injector parameters.
	Leaks in the GC carrier gas line.	Run a leak test and correct leaks.
	Syringe leaks during injection.	Replace syringe or piston seals, if applicable.
	Split injection temperature too low.	Increase the temperature of the injector.
	Column is in poor condition, or wrong column type used.	Condition the columns. Change the column.
Retention Times Decreasing	Stationary phase deteriorated by oxygen and/or water.	Use a carrier gas free of oxygen and water. Replace or install appropriate gas filters (see page 3-087 to 3-089).
	Stationary phase loss due to column bleeding.	Reduce the column temperature.
Retention Times Increasing	Increasing carrier leakage.	Check the septum and column connections.
	Carrier gas supply running out.	Replace the bottle.
Low Reproducibility of Retention Times	Drifting or unstable pneumatic controller.	Monitor the column pressure or flow. Check and replace the controller if necessary.
	Poor injection technique.	Start the run at consistent time after injection.
	Sample size is too large.	Reduce the injected amount and/or volume.
	Unstable column temperature.	Check the main oven door and cooling flap. Monitor the column temperature.
Retention Times are Inconsistent	GC column is in poor condition.	Condition the column. Change the column.
	Insufficient equilibration time set on GC.	Increase equilibration time.
	Poor injection.	Repeat with better injection technique.
	Oven temperature programmed to rise too quickly.	Reduce oven temperature ramp rate.
	Air is leaking into the system at the injector seal or the carrier gas manifold.	Trace and repair the leak.