GC Troubleshooting Tips

BASIC STEPS

Follow these basic troubleshooting steps to isolate problems related to the sample, injector, detector, and column. Check the obvious explanations first and change only one thing at a time until you identify and resolve the problem.

Check the Obvious:

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

Identify the Cause:

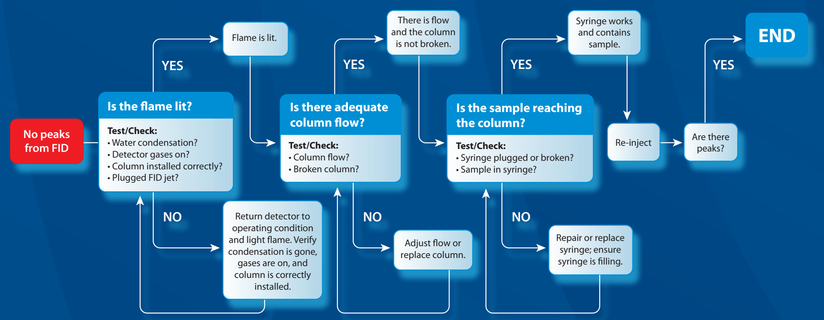
- Define the problem clearly; for example, "Over the last 4 days, only the phenols in my sample have been tailing."
- Review sample and maintenance records to identify trends in the data or problem indicators, such as area counts decreasing over time or injector maintenance not being performed as scheduled.
- Use a logical sequence of steps to isolate possible causes.

Document Work and Verify System Performance:

- 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.

EXAMPLE TROUBLESHOOTING SEQUENCE

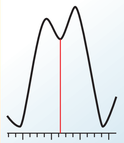
An analyst observed that no peaks appeared during a GC-FID analysis. The flowchart below shows a logical progression of steps that can be used to identify the cause and correct the problem.



SYMPTOMS AND SOLUTIONS

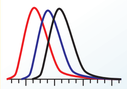
Good chromatography is critical to obtaining accurate, reproducible results. Coelutions, asymmetric peaks, baseline noise, and other issues are common challenges in the GC laboratory. These analytical problems and others can be overcome by troubleshooting your separations using the tips below.

Poor Resolution



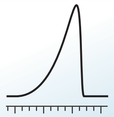
Causes	Solutions
Non-selective stationary phase	• Choose appropriate stationary phase and column dimensions.
Poor efficiency	• Optimize carrier gas linear velocity and GC oven temperature program.
Sample overload	• Adjust sample concentration or amount on column.
Incorrect analytical conditions used	• Verify temperature program, flow rates, and column parameters.

Poor Retention Time Reproducibility



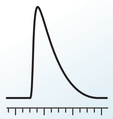
Causes	Solutions
Leaks	• Leak check injector and press-fit connections. • Replace critical seals (i.e., septa, O-rings, inlet disc, etc.)
Analyte adsorption	• Maintain inlet liner and GC column. • Use properly deactivated liners, seals, and columns.
Resolution/integration issues	• Avoid sample overload.
Incorrect column/oven temperature program	• Verify column temperature and oven temperature program.
Incorrect or variable carrier gas flow rate/linear velocity	• Verify the carrier gas flow and linear velocity. Repair or replace parts if neccessary.
Poor control of oven temperature programming	• Confirm GC oven program falls within instrument manufacturer's recommendation.
Incorrect oven equilibration time	• Extend GC oven equilibration time.
If manual injection, delay between pushing start and actual injection	• Use autosampler or standardize manual injection procedure.

Fronting Peaks



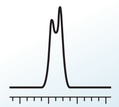
Causes	Solutions
Incompatible stationary phase	• Choose appropriate stationary phase.
Column overloading	• Reduce amount injected, dilute sample. • Increase column inner diameter and/or film thickness.

Tailing Peaks



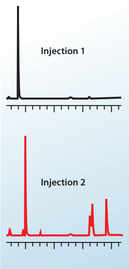
Causes	Solutions
Adsorption due to surface activity or contamination	• Use properly cleaned and deactivated liner, seal, and column. • Trim inlet end of column. • Replace column if damaged.
Adsorption due to chemical composition of compound	• Derivatize compound.
Leak in system	• Check for leaks at all connections, replace critical seals if needed.
Installation issues	• Minimize dead volume. • Verify that the column is cut properly (square). • Verify correct installation distances.

Split Peaks



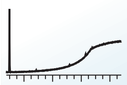
Causes	Solutions
Mismatched solvent/stationary phase polarity	• Adjust solvent or stationary phase to allow wetting.
Incomplete vaporization	• Add surface area, such as wool, to the inlet liner to enhance vaporization. • Use proper injector temperature.
Sample loading capacity exceeded	• Inject less sample (dilute, use split injection, reduce injection volume).
Fast autosampler injection into open liner	• Use wool or slow injection speed.

Carryover/Ghost Peaks



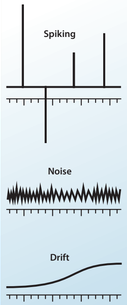
Causes	Solutions
Contaminated syringe or rinse solvent	• Replace rinse solvent. • Rinse or replace syringe.
Backflash (sample volume exceeds liner volume)	• Inject a smaller amount. • Use a liner with a large internal diameter. • Increase head pressure (i.e., flowrate) to contain the vapor cloud. • Use slower injection rate. • Lower inlet temperature. • Increase split flow. • Use liner with packing. • Use pressure-pulse injection.
Last analysis ended too soon	• Extend analysis time to allow all components and/or matrix interferences to elute.

High Bleed



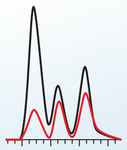
Causes	Solutions
Improper column conditioning	• Increase conditioning time and/or temperature.
Contamination	• Trim column and/or heat to maximum temperature to remove contaminants. • Replace carrier gas and/or detector gas filters. • Clean injector and detector.
Leak in system and oxidation of stationary phase	• Check for oxygen leaks across the entire system and replace seals and/or filters. • Replace column.

Unstable Baseline (Spiking, Noise, Drift)



Causes	Solutions
Carrier gas leak or contamination	• Leak check connections and replace seals if needed. • Replace carrier gas and/or detector gas filters.
Injector or detector contamination	• Clean system and perform regular maintenance.
Column contamination or stationary phase bleed	• Condition, trim, and rinse column.
Septum coring/bleed	• Replace septum. • Inspect inlet liner for septa particles and replace liner if needed.
Loose cable or circuit board connections	• Clean and repair electrical connections.
Variable carrier gas or detector gas flows	• Verify flow rates are steady and reproducible; may need to replace or repair flow controller. • Leak check system.
Detector not ready	• Allow enough time for detector temperatures and flows to equilibrate.

Response Variation

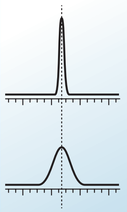


Causes	Solutions
Sample issues	• Check sample concentration. • Check sample preparation procedure. • Check sample decomposition/shelf life.
Syringe problems	• Replace syringe. • Check autosampler operation.
Electronics	• Verify signal settings and adjust if needed. • Repair or replace cables or boards.
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, seal, 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.

No Peaks

Causes	Solutions
Injection problems	• Plugged syringe; clean or replace syringe. • Verify there is sample in the syringe. • Injecting into wrong inlet; reset autosampler. • Verify carrier gas is flowing.
Broken column	• Replace column.
Column installed into wrong injector or detector	• Re-install column.
Detector problems	• Signal not recorded; check detector cables and verify that detector is turned on. • Detector gas turned off or wrong flow rates used; turn detector on and/or adjust flow rates.

Broad Peaks



Causes	Solutions
High dead volume	• Minimize dead volume in the GC system; verify proper column installation, proper connectors, proper liners, etc.
Low flow rates	• Verify injector and detector flow rates and adjust if needed. • Verify make-up gas flow and adjust if needed.
Slow GC oven program	• Increase GC oven programming rate.
Poor analyte/solvent focusing	• Lower GC oven start temperature.
Column film is too thick	• Reduce retention of compounds by decreasing film thickness and length.
Sample carryover	• See Carryover/Ghost Peaks solutions.

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