Gas Chromatography

TROUBLESHOOTING GUIDE

SHIMADZU

Excellence in Science

Contents

Introduction

Instrument downtime is often costly and time consuming, but frequently the problems can be resolved quickly with some troubleshooting knowledge.

This Gas Chromatography Troubleshooting Guide is designed to assist chromatographers assess common GC problems. Shimadzu have included how to effectively troubleshoot and fix these issues to allow you to get your system back up and running and continue your analyses.

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.

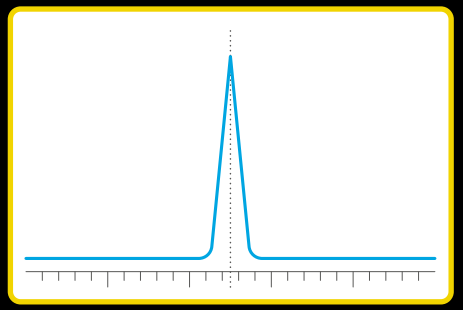
Document Everything:

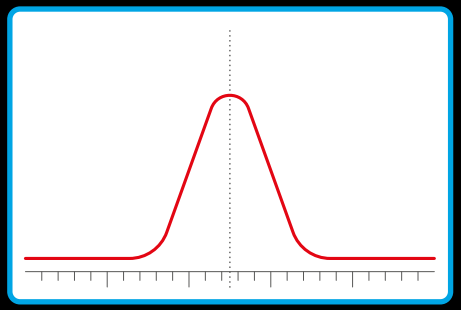
- Document all troubleshooting steps and results; this may help you identify and solve the next problem faster.
- Always inject a test mix and compare to previous data to ensure restored performance.

Still having problems?

Still struggling? Let us know!!!
gc_gcms_support@shimadzu.co.uk

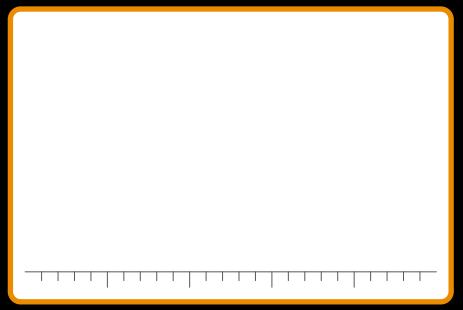
Broad Peaks





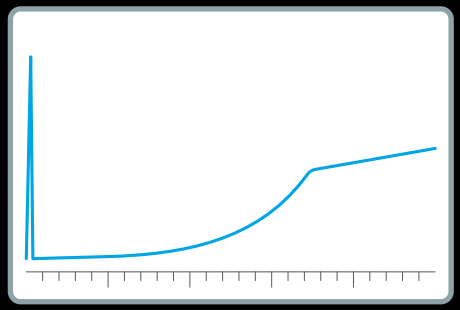
Causes	Solutions
High dead volume	- Minimise dead volume in the GC system; verify proper column installation, proper connectors, proper liners, etc.
Low flow rates	- Verify inlet and detector flow rates and adjust if needed. - Verify make-up gas flow and adjust if needed.
Slow GC oven program	- Increase 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.

No Peaks



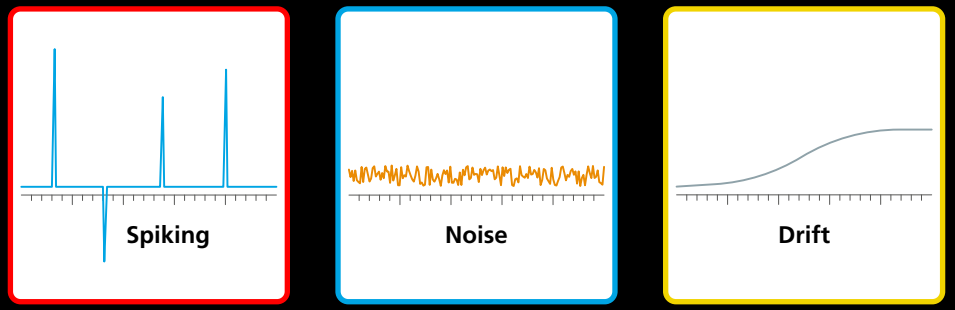
Causes	Solutions
Injection problems	- Blocked syringe; clean or replace syringe. - Verify there is sample in the syringe. - Injecting into wrong inlet; reset autosampler. - Verify carrier gas is flowing.
Broken column	- Replace column.
Column installed into wrong inlet or detector	- Reinstall column.
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.

High Baseline
(Column Bleed)



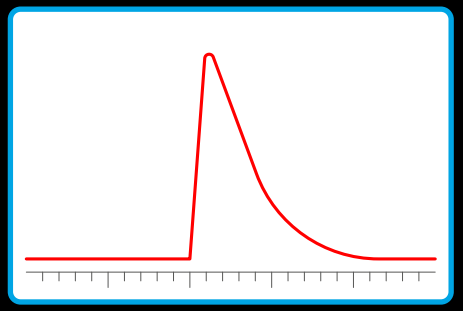
Causes	Solutions
Improper column conditioning	- Increase conditioning time and/or temperature.
Contamination	- Trim column and/or heat to maximum temperature to remove contaminants. - Replace carrier gas and/or detector gas filters. - Clean injector and detector.
Leak in system causing oxidation of stationary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

Unstable Baseline
(Spiking, Noise, Drift)



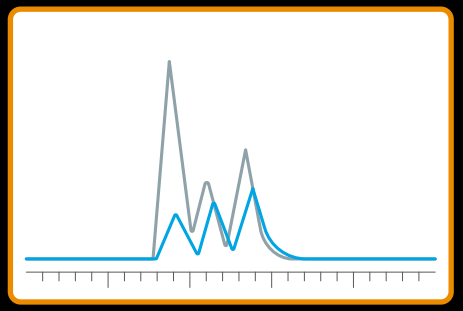
Causes	Solutions
Carrier gas leak or contamination	- Leak check connections and replace seals if needed. - Replace carrier gas and/or detector gas filters.
Inlet or detector contamination	- Clean system and perform regular maintenance.
Column contamination or stationary phase bleed	- Condition, trim, and rinse column.
Septum coring/bleed	- Replace septum. - Inspect inlet liner for septa particles and replace liner if needed.
Leak or poor quality gases	- Check GC and gas lines for leaks and confirm gas supply purity is adequate. If necessary, install gas filters.
Variable carrier gas or detector gas flows	- Leak check system and check AFC/APC functionality.
Detector not ready	- Allow enough time for detector temperatures and flows to equilibrate.

Tailing Peaks



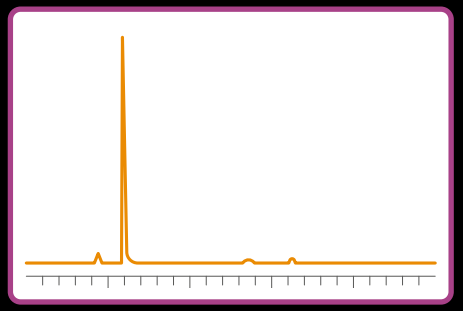
Causes	Solutions
Adsorption due to surface activity or contamination	– Use properly cleaned and deactivated liner and column. – Trim inlet end of column. – Replace column if damaged.
Adsorption due to chemical composition of compound	- Derivatise compound.
Leak in system	- Check for leaks at all connections,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

Changes in Response

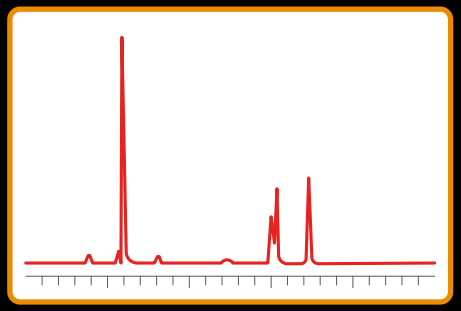


Causes	Solutions
Sample issues	- Check sample concentration. - Check sample preparation procedure. - Check sample decomposition/shelf life.
Syringe problems	- Replace syringe. - Check autosampler operation.
Electronics	- Verify signal settings and adjust if needed.
Dirty or damaged detector	- Perform 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.

Carryover/Ghost Peaks



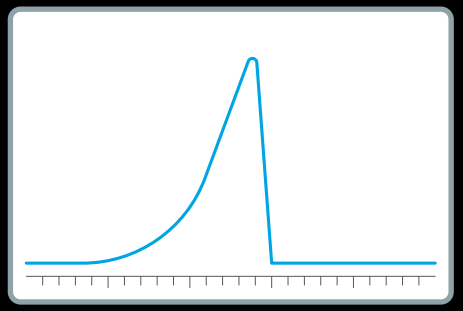
Injection 1



Injection 2

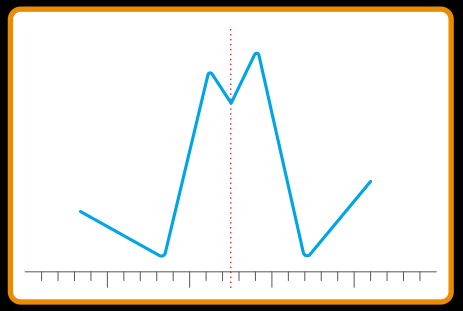
Causes	Solutions
Contaminated syringe or rinse solvent	- Replace rinse solvent. - Rinse or replace syringe.
Backflash (sample volume exceeds liner volume)	- Inject a smaller amount. - Use a liner with a large internal diameter. - Increase head pressure (i.e., flowrate) to contain the vapour cloud. -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

Fronting Peaks



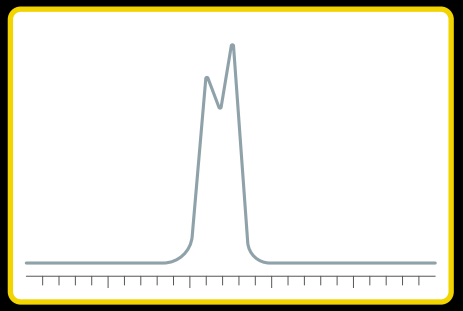
Causes	Solutions
Incompatible stationary phase	- Choose appropriate stationary phase.
Column overloading	- Reduce amount injected, dilute sample or increase split ratio. - Increase column inner diameter and/or film thickness.

Poor Peak Resolution



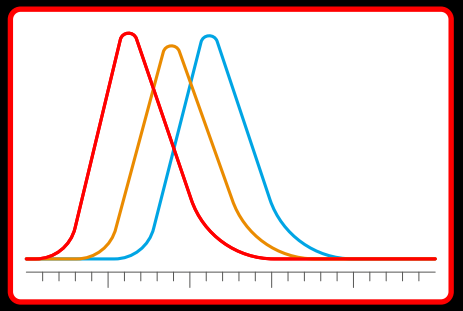
Causes	Solutions
Non-selective stationary phase	- Choose an appropriate stationary phase and column dimensions.
Poor efficiency	- Optimise carrier gas linear velocity and GC oven temperature program.
Sample overload	- Adjust sample concentration or amount on column by increasing split ratio.
Incorrect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

Split Peaks



Causes	Solutions
Mismatched solvent/ stationary phase polarity	- Adjust solvent or stationary phase to allow wetting.
Incomplete vaporisation	- Add surface area, such as wool, to the inlet liner to enhance vaporisation. - Use proper inlet 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.

Retention Time Variability



Causes	Solutions
Leaks	- Leak check inlet and any column connections. - Replace septa, O-rings, etc.
Analyte adsorption	- Maintain inlet liner and GC column. - Use properly deactivated liners and columns.
Resolution/integration issues	- Avoid sample overload by diluting sample or increasing split ratio.
Incorrect column/oven temperature program	- Verify column temperature and oven temperature program.
Incorrect or variable carrier gas linear velocity	- Verify the carrier gas linear velocity. - Repair or replace parts if necessary.
Poor control of oven temperature programming	- Confirm GC oven program falls within instrument specifications.
Incorrect oven equilibration time	- Extend GC oven equilibration time.
If manual injection, inconsistencies between pushing start and injection procedure	- Use autosampler or standardise manual injection procedure.

Shimadzu Scientific Instruments
7102 Riverwood Drive, Columbia, MD 21046
Phone: (800) 477-1227 / (410) 381-1227
www.ssi.shimadzu.com