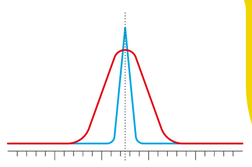
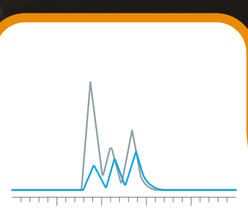
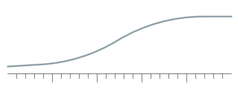
GCMS Troubleshooting Tips



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 ratess	- Verify carrier gas flow rate and adjust if needed.
Slow GC oven program	- Increase GC oven programming rate.
Poor analytes / solvent focusing	- Lower GC oven start temperature.
Column film is too thick	- Reduce retention of compounds by decreasing film thickness and length.
Simple carryover	- See Carryover/Ghost Peaks solutions.

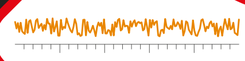




Drift

Changes in Response

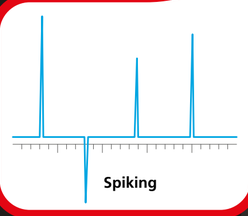
Causes	Solutions
Sample issues	- Check sample concentration. - Check sample preparation procedure. - Check sample decomposition/shelf life.
Syringe problems	- Replace syringe. - Check autosampler operation.
Electronics	- Verify signal settings and adjust if needed.
Dirty or damaged detector	- Clean the ion source and replace the electron multiplier if required. - Check and replace filaments if required.
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.



Noise

Unstable Baseline

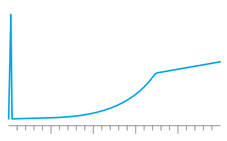
Causes	Solutions
Carrier gas leak or contamination	- Leak check connections and replace seals if needed. - Replace carrier gas and/or detector gas filters.
Inlet or detector contamination	- Clean the inlet and ion source. Schedule and perform regular maintenance.
Column contamination or stationary phase bleed	- Condition, trim, and rinse column.
Septum coring / bleed	- Replace septum. - Inspect inlet liner for septa particles and replace if needed. - Replace the solvent in the wash vials. - Perform a manual tune, the presence of m/z 73, 147, 207, 281 & 355 will indicate siloxane contamination from damaged septa.
Aging filament	- Switch to second filament and replace the first as soon as practicable.
Variable carrier gas or detector gas flows	- Leak check the system and check that the flow and pressure controllers are functioning correctly. - Check collision gas pressure.
Detector not ready	- Allow enough time for ion source and transfer line temperatures to equilibrate.



Spiking

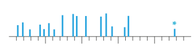
High Baseline

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 ion source.
Leak in system causing oxidation of stationary phase	- Check for oxygen leaks across the entire system and replace seals and/or filters. Perform a manual tune and look for the presence of m/z 18, 28 & 32. Large peaks will indicate a leak. - Check the column fittings on the inlet and MS transfer line. - Replace column. - Check the seal around the access door to the MS.



Undesired Fragmentation

Causes	Solutions
Ion source setting too harsh	- Check source temperatures are appropriate for analyte. - Check ionisation voltage is appropriate for analyte.
Collision energy too high / low	- Check and optimise collision cell gas pressure and collision energy.
Collision energy too high / low	- Change to the second filament and replace the first as soon as practicable.
Contaminated source	- Clean the ion source and perform a tune.



Basic Steps

Follow these steps to isolate where the problem 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
- MS vacuum
- Collision gas (level & pressure)
- Rough pump (oil level & gas ballast)

Identify the Cause:

- Clearly define the problem
- Review sample and maintenance logs to identify trends in the data or possible problem indicators
- Use a logical sequence of events to isolate possible causes

Document Everything:

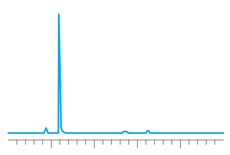
- Document all troubleshooting steps and results; this may help you identify and solve the next problem faster
- Always inject a test mix and compare to previous data to ensure restored performance

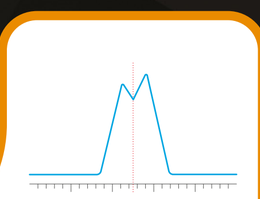
Still having problems with your instrument? Let us know at

gc_gcms_support@shimadzu.co.uk

Carry Over / 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 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.



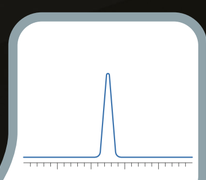


Poor Peak Resolution

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.
MS method settings	- Use different m/z when using selected ion monitoring (SIM) or a different transition in multiple reaction monitoring (MRM). - Set quadrupole to 'High' resolution for analytes affected in MRM mode.

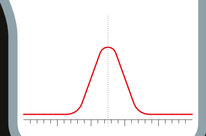
No Peaks

Causes	Solution
Injection problems	- Blocked syringe; clean or replace syringe. - Verify there is sample in the vial. - 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	- Ensure MS is an appropriate technique for the analyte(s) - Check the vacuum is sufficient and stable, perform a leak check. - Perform and check the MS tune. - Check the ion source temperatures and gas flows are as expected and stable. - Check the collision gas pressure is correct and stable. - If the method contains scheduled events, such as MRM, ensure the analyte elutes within the correct event window. - Check that the concentration of the analyte is above the limit of detection. - Blown filament, switch to second filament and replace the first as soon as practicable.



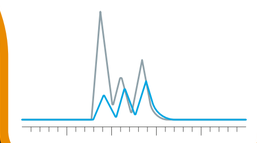
Changes in MS Resolution

Causes	Solutions
MS out of tune	- Perform and check tune.



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.
Detector saturation	- Reduce the injection volume or inject a lower concentration of sample. - Set quadrupoles to 'High' resolution for analytes affected in MRM mode.



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