HPLC and GC troubleshooting

Routine checks for common laboratory faults

These notes are for routine faults on HPLC and GC systems. They are not a repair procedure and they do not replace the instrument manual. Error text differs by brand and firmware, so act on the symptom you can see, not on a code you have not confirmed.

Work through an item once. Stop if you smell gas, if a hydrogen or leak shutdown is active, if a fitting or column is damaged, or if the fault is unchanged after the safe check. Call Mahut Technical Co. on +968 71130316 or email info@mahuttechnical.com.

Mahut is an independent service company in Muscat. This document does not authorize opening detectors, bypassing interlocks, or working on live gas leaks.

Each item states the symptom, the usual routine causes, the safe check, and the point at which you should stop and call.

HPLC

Pressure is higher than normal

Compare the reading with your usual pressure for the same column, flow, mobile phase, and temperature. A rise usually means a restriction: inline filter, guard column, column inlet frit, or plugged tubing. Buffer salts that have precipitated are a common cause.

Stop the flow before you open any fitting. Remove or bypass the guard column first and check the pressure again. If it is still high, stop the flow, remove the analytical column, and connect the inlet and outlet tubing with a union rated for the pressure. At the method flow, pressure with no column should fall to a low value set by the tubing. If it does, the restriction was the guard or the column.

If pressure stays high with the column out, isolate one section at a time, working back from the detector toward the pump. Do not backflush a column unless the column instructions allow it. Do not switch a salty aqueous system straight into a strong organic solvent; wash with a high-water mobile phase first.

Call Mahut if pressure is still high after the column is removed, if a fitting leaks or is damaged, or if you are not sure which solvent is in the pump.

Pressure is lower than normal

A low but steady pressure, with the same method, usually means a leak or less resistance in the path. Look for liquid at pump fittings, a loose column fitting, a worn pump seal, or a column that is not the one you think is installed. Also confirm the flow set-point and that the mobile phase composition has not changed.

Tighten a leaking fitting only until the leak stops. Over-tightening cuts ferrules and makes the next leak worse. If the pump runs and the pressure is near zero, treat it as “no pressure” below.

Call Mahut if the leak is inside the pump head, if seals have already been replaced once, or if tightening the visible fittings does not hold.

The pump does not build pressure

Confirm the pump is running and the flow is not zero. Check that the purge or prime valve is closed. Check that solvent is in the reservoir and that the inlet line and sinker frit are under the liquid. Open the prime path and draw solvent through until the line is full and free of air, then close it and try the method flow again.

A large leak downstream will also show as no pressure. Find it before you keep the pump running dry.

Call Mahut if the pump reports a fault and stops, if you cannot prime, or if there is no pressure and no visible leak after priming. Do not bypass a leak or pressure interlock.

Pressure fluctuates

Fast, regular ripple often comes from air in the pump head or a check valve that is not seating. Slower wander often comes from a small leak, a partially blocked inlet frit that starves the pump, poor degassing, or a change in temperature or solvent composition.

Purge each channel in use. Confirm the inlet frit is not blocked and the bottle is not empty. Watch one channel at a time if you use a gradient. If a single check valve or seal has just been disturbed, air is the first thing to remove before you replace more parts.

Call Mahut if purging does not settle the pressure, or if the ripple started right after a seal or valve change and priming does not clear it.

Baseline noise

Separate noise (a fast, jagged baseline) from drift (a slow climb or fall). On a UV detector, common routine causes are an aging lamp, a dirty flow cell, bubbles, or pump pulsation. On either HPLC or GC, a contaminated column or a bad mobile phase or gas can look similar.

For HPLC, degas the mobile phase and purge the pump. Confirm the lamp is on and note whether noise grew worse as lamp hours rose. A bubble will often spike and then recover; a dying lamp usually does not.

Do not open a flow cell or a detector that is not designed for user cleaning. Call Mahut if noise remains after degassing and a simple lamp-energy check, or if the detector will not ready.

No peak

Confirm that a run really started and that the data system was acquiring. Check that the vial had sample, the needle or syringe is not blocked, and the injection volume is the one you intended. Confirm the detector is on, the lamp is on for UV, and the wavelength or channel is the one the method uses.

Then confirm flow reaches the column and the detector, not a waste or purge path. A leak at the injector can lose the sample before it reaches the column. A sample that is not retained can sit in the void and be missed if the window is wrong; a sample that is fully retained never appears.

Call Mahut if the injector does not move, the detector stays off, or a standard that used to work produces no peak after the checks above.

Peaks are smaller than usual

Compare with a fresh standard, not with a sample of unknown history. Low peaks come from a partial injection (blocked syringe, leaking needle seat, air in the draw), a standard that has degraded, the wrong wavelength, a weak detector lamp, or a column that is retaining or degrading the analyte.

Change one item. A new standard that restores the height points to the sample. A new lamp, or a failed lamp-energy check, points to the detector. A height that is low only on some peaks points to the chemistry or the column, not to the injection volume.

Call Mahut if the autosampler errors, if lamp energy is failed, or if the loss appeared suddenly on every peak including a fresh standard.

Retention time has changed

If every peak moves in the same direction, check flow, column temperature, and mobile-phase composition first. A small error in organic content or buffer moves retention on most reversed-phase methods. Incomplete equilibration after a solvent change does the same.

If the spacing between peaks changes, not just the clock time, selectivity has changed. That is more often pH, a different column, or a contaminated column than a flow-rate error.

Make up mobile phase again by volume, confirm the column name against the method, and allow the system to equilibrate at the starting composition. Call Mahut if flow and temperature are correct, the fresh mobile phase matches the method, and retention is still wrong.

Peaks move from one injection to the next

This is different from a one-time shift after a method change. Wandering retention during a sequence usually means the column is not equilibrated, the column oven is not stable, premixed solvent is evaporating, or the proportioning valve is not delivering a repeatable composition.

Keep the solvent bottles closed. Give gradient methods the same equilibration time on every injection. Confirm the oven is on and at the set point.

Call Mahut if retention wanders while temperature and a freshly prepared mobile phase are stable, especially if pump pressure wanders with it.

Bubbles in the solvent path

Bubbles in the inlet line are air drawn in before the pump. Usual causes are an empty bottle, a loose inlet fitting, a frit sitting above the liquid, or solvent that was not degassed. Purge each channel until the outlet stream is continuous. A vacuum degasser, helium sparge, or the instrument degasser is appropriate. Do not shake solvent bottles. Be careful with flammable solvents and do not degas them in a domestic ultrasonic bath as a routine practice.

Bubbles that appear only after the pump, with noisy pressure, point to a leaking check valve or seal rather than the bottle.

Call Mahut if purging fills the line and bubbles return at once, or if you suspect a leaking pump seal.

GC

No peak

Confirm the data system acquired the run and the detector is ready. On an FID, a flame that is out produces no peaks; check that hydrogen and air are present and that lighting the flame is the procedure your instrument allows. Do not keep hydrogen flowing while you hunt for a leak.

Check the syringe. A plugged plunger or needle injects nothing. Confirm the column is connected to the inlet and the detector, the oven program actually reaches a temperature that elutes the analytes, and the split ratio is not so high that a dilute sample disappears.

A broken column or a column installed in the wrong detector also yields no peak. Do not force a column into a fitting.

Call Mahut if the flame will not stay lit, the autosampler does not inject, or a previous good standard is absent after a sound injection and a confirmed flame or detector-ready state.

Peaks are smaller than usual

Small peaks with a normal retention time often mean less sample reached the column or the detector is less sensitive. Check for a partial syringe plug, a leaking septum, a leaking column nut, a split ratio set too high, or a contaminated inlet liner. An exhausted or wrong liner is a frequent cause after dirty samples.

If only late or active peaks shrink, the column or the inlet may be active or the analytes may be decomposing. If every peak shrinks, including a solvent peak on an FID, look at injection volume and detector gases before you blame the column.

Call Mahut if you see an inlet leak you cannot stop by a septum and column-nut check, or if detector gases are correct and sensitivity stays low.

Inlet or detector pressure error

An inlet pressure fault usually means the instrument cannot hold the setpoint. Routine causes are the gas supply turned down or empty, a septum leak, a column ferrule leak, a column that is broken or not installed, a plugged split vent trap, or column dimensions in the method that do not match the column in the oven. Electronic pressure control calculates flow from those dimensions. Wrong length or inner diameter produces the wrong pressure.

A detector pressure fault points at makeup gas, FID hydrogen or air, or the detector gas fitting, depending on the detector. Read the message. Do not swap gases to “see what happens.”

If the system shuts down on a leak, or if you smell gas, ventilate, stop, and call Mahut. Never use a flame to find a leak. Hydrogen collecting in an oven or a closed room is a fire hazard.

Gas generators and cylinders

A generator or cylinder fault often appears first as a pressure error or a detector that will not ready. Check the cylinder pressure and that the changeover valve is on the live cylinder. Confirm the regulator outlet pressure matches what the instrument expects. Confirm the gas identity at the connection. Do not adapt a fitting to force a different gas onto an inlet.

Generator alarms for purity, water, or compressor shutdown should be cleared on the generator before you adjust the chromatograph. An exhausted moisture or hydrocarbon trap more often ruins the baseline than it removes all pressure. Replace traps on their service interval.

Call Mahut or the gas supplier if a regulator, a generator compressor, or a leak-shutdown will not reset. Do not open generator covers that are interlocked.

Baseline noise

A quiet baseline that rises smoothly at the top of the oven program can be normal column bleed. A sudden noisy or spiking baseline is not. Routine causes are septum particles in the liner, a contaminated liner, poor gas purity, an exhausted trap, an unstable FID flame, or a dirty jet. Electrical noise and an oven leak are less common and are not a user cleaning task.

Lower the oven to the normal start temperature and see whether the noise collapses. If it does, the column or the inlet contamination is likely. If the noise is present with the detector gases on and the oven cold, look at the detector and the gas quality.

Call Mahut if the FID will not hold a stable flame, if noise remains after a new liner and septum and a confirmed gas supply, or if you suspect a leak inside the oven.

When to stop and call

Call +968 71130316 when:

- You smell gas, or a hydrogen or leak shutdown is active
- Pressure stays wrong after the column is isolated or the gas supply is confirmed
- A fitting, column, or syringe is damaged
- The detector, pump, or autosampler reports a fault you cannot clear with the checks above
- A fresh standard still fails after one careful pass through the relevant checks

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