Agilent

Agilent 8860 Series Gas Chromatograph

Troubleshooting



Notices

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Concepts

This manual provides lists of symptoms and corresponding tasks to perform should you experience errors associated with GC hardware or chromatographic output, GC Not Ready messages, and other common issues.

Each section describes a problem and provides a bulleted list of possible causes for you to troubleshoot. These lists are not intended for use in the development of new methods. Proceed with troubleshooting under the assumption that method(s) are working properly.

This manual also includes common troubleshooting tasks as well as information needed prior to calling Agilent for service.

How to troubleshoot using this manual

Use the following steps as a general approach to troubleshooting:

1. Observe the symptoms of the problem.
2. Look up the symptoms in this manual using the Table of Contents or the **Search** tool. Review the list of possible causes of the symptom.
3. Check each possible cause or perform a test that narrows the list of possible causes until the symptom is resolved.

Using the GC Diagnostic Features

The browser UI includes Home screens that display current temperatures, flows, and similar data, including plots of output signals or other signals useful for troubleshooting.

In addition:

- Go to Diagnostics to view any issues detected by the GC. For some issues, the GC may provide automated troubleshooting. The GC also provides various automated diagnostic tests, available from Diagnostics > Diagnostic Tests.
- The System Health Report available from the Diagnostics tab, lists the current status of all GC components and also includes information needed when contacting Agilent for support (instrument firmware version, serial number, and so forth). The report also lists recent self-diagnostic results.
- Go to Logs and view the Run Log and System Log.

Configurable Items to Always Keep Current

Certain configurable items in the GC must always be kept current. Failure to do so will lead to reduced sensitivity, chromatographic errors, and possible safety concerns.

Inlet and detector configuration

When changing from one method to another, be sure to consider whether the inlet and detector consumable parts need to be changed as well. Failure to install appropriate consumable parts can lead to unexpected results.

Liners: The appropriateness of liner type varies depending on the GC inlet mode, for example split mode versus splitless mode, and the analysis.

FPD+ filters: The FPD+ filters require different gas flows to function properly. Set the flow according to the FPD+ filter installed (phosphorus versus sulfur) and make sure the correct filter is installed.

Column configuration

Update the GC's column configuration every time a column is trimmed or changed. Also verify that the data system reflects the correct column type, length, id, and film thickness. The GC relies on this information to calculate flows. Not updating the GC after altering a column causes incorrect flows, changed or incorrect split ratios, retention time changes, and peak shifts.

Automatic Liquid Sampler configuration

Keep the Automatic Liquid Sampler (ALS) configuration up-to-date to ensure proper operation. ALS items to keep current include installed syringe size and solvent and waste bottle usage.

Gas configuration

WARNING

Always configure the GC appropriately when working with hydrogen. Hydrogen leaks quickly and poses a safety concern if too much of it is released into the air or into the GC oven.

Reconfigure the GC every time the gas type is changed. If the GC is configured to a gas other than what is actually being plumbed, incorrect flow rates will result.

To check the gas type configured for a component, go to **Settings** > **Configuration**, then select the desired component (the inlet or detector, for example).

To View the Run Log and System Log

The GC maintains internal event logs. Use these logs to troubleshoot problems, especially when a message no longer appears on the display.

To access the logs, go to **Logs** and select the **Run Log** or **System Log**.

Run Log

For each run, the run log records deviations from the planned method. This log is overwritten at the start of each run. The run log information can be used for Good Laboratory Practice (GLP) standards and can be uploaded to an Agilent data system.

System Log

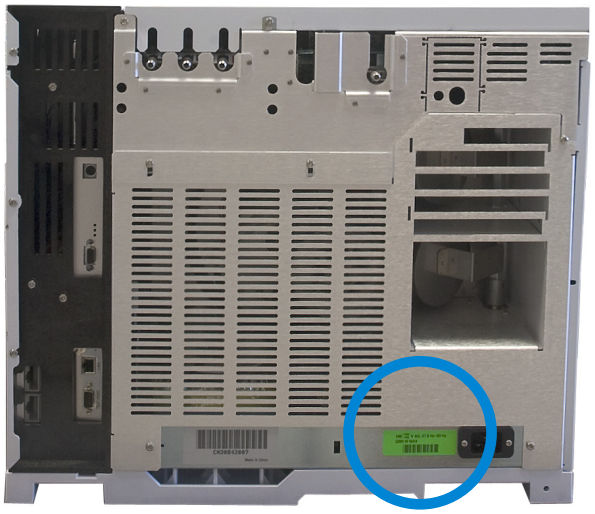
The system log records events such as shutdowns, warnings, faults, and GC state changes (start run, stop run, and so forth) that occur during GC operation. The System Log includes entries for system startup, firmware updates, and sequences.

Information to Provide When Contacting Agilent Support

If possible, generate a System Health Report using the Browser UI. This report contains most of the information listed below.

Gather the following information before contacting Agilent for service:

- Symptoms
- Problem description
- Hardware installed and parameters/configuration when the error occurred (sample, supply gas type, gas flow rates, detectors/inlets installed, and so forth)
- Any messages that appear
- Results of any troubleshooting tests you have run
- Instrument details. Obtain the following information:
  - GC serial number, which can be found on the bottom right corner of the GC.
  - GC firmware revision located in **Settings** > **About**
  - GC power configuration (located on a sticker on the bottom of the rear of the GC)



To obtain service/support contact numbers, see the Agilent Web site at **www.agilent.com/chem**.

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Plunger Errors

If the ALS reports a front or back plunger error, check the following possible causes:

- The syringe plunger is sticking or is not securely connected to the plunger carrier.
- The plunger solenoid is binding.
- The plunger carrier encoder is inoperable.
- The autoinjector plunger carrier mechanism will not move.
- The plunger does not move freely due to sample residue or wear. Install a new syringe, making sure to prime the syringe with solvent before installing.

Procedure

1. Remove the syringe and check it for plunger stickiness or binding.
2. Replace the syringe if necessary. Refer to the documentation for the 7693A or 7650A as applicable.
3. Check the viscosity of the sample against the viscosity parameter.
4. Reset the viscosity parameter if necessary.
5. Restart the sequence.
6. If the error occurs again, obtain Agilent service.

Alignment Light on 7693A/7650A Injector Tower is On

If the Align Mode light is on, first verify that the turret is properly installed. Then, perform the alignment procedure as described in the 7693A Automatic Liquid Sampler Installation, Operating and Maintenance manual or the 7650A Automatic Liquid Sampler Installation, Operation and Maintenance manual.

Syringe Needle Bends During Injection into Inlet

WARNING

When troubleshooting the injector, keep your hands away from the syringe needle. The needle is sharp and may contain hazardous chemicals.

Refer to your ALS documentation for additional information.

- Check that the GC septum nut is not too tight.
- Check that the syringe is installed correctly into the syringe carriage.
- Check that the needle support and guide are clean. Remove any residue or septum deposits. Install a new needle support foot if necessary.
- If using the cool on-column inlet, check that the correct insert for the syringe is installed.
- Check that you are using the proper syringe. The combined length of the syringe barrel and needle should be approximately 126.5 mm.
- Check that the sample vial dimensions meet specification.
- Check that the crimp cap is properly installed. Refer to your sampler documentation.

NPD Fails Leakage Current Test

A failed leakage current test usually indicates misassembly, contamination, or a damaged part.

If you have just performed maintenance on the NPD, first verify that the detector was reassembled properly before troubleshooting detector problems.

1. Replace the ceramic insulators. Retest.
2. Turn off the bead and view the output (leakage current).
3. Remove the bead and store in a safe place.
4. Remove the three screws that secure the lid in place, then remove the lid.
5. Examine the interconnect spring. Make sure that the interconnect spring is not damaged, bent, or dirty. The interconnect spring should be touching the bottom of the collector.
6. If the interconnect spring is not damaged or dirty, and the detector output signal is still high, contact Agilent for service.

FID Fails Baseline Test

If you have just performed maintenance on the FID, first verify that the detector was reassembled properly before troubleshooting detector problems.

If the FID fails the baseline test:

- Ensure the purity and quality of the gas.
- Replace dirty/expended chemical traps.
- Bakeout the detector.

FID Does Not Ignite

- Verify that the Lit Offset is ≤ 2.0 pA.
- Ensure that the FID temperature is high enough for ignition (>150 °C). Agilent recommends >300 °C.
- Check that the FID ignitor glows during ignition sequence. (See **"To Verify FID Ignitor Function During Ignition Sequence"** .)
- Try increasing the supply pressures to the FID flow module. This makes the flame easier to light without changing the setpoints.
- Increase hydrogen flow and decrease or turn off makeup gas flow until ignition occurs, then reduce them toward the method values. Experiment for the best values.

  Increasing hydrogen flow and decreasing makeup flow will help the FID ignite more easily. If it will light under these modified conditions, the cause could be a partially clogged jet, a weak ignitor, or a leak at the column fitting.
- Check for a plugged or partially plugged jet. (See **"To Check for a Plugged FID Jet"** .)
- Measure the FID flow rates. Actual flow rates should be within +/-10% of the setpoint. The hydrogen:air ratio greatly impacts ignition. Nonoptimal flow settings can prevent flame ignition. (See **"To Measure a Detector Flow"** .)
- There could be a large leak in the system if the flame still will not light. Large leaks result in measured flow rates being different from actual flow rates, causing nonideal ignition conditions. Thoroughly leak check the whole system, especially the column fitting at the FID. (See **"Checking for Leaks"**).
- Check the column flow rate. (See **"To Measure a Column Flow"** .) Hydrogen flow should be greater than the sum of the column flow and makeup flow.
- Check for leaks at the FID column fitting.
- Ensure that the FID temperature is high enough for ignition (>150 °C).
- If the analysis permits, substitute nitrogen for helium as makeup.
-

FID Ignitor Does Not Glow During Ignition Sequence

WARNING

Keep body parts at a safe distance from the FID chimney while performing this task. If using hydrogen, the FID flame will not be visible.

1 Remove the detector top cover.

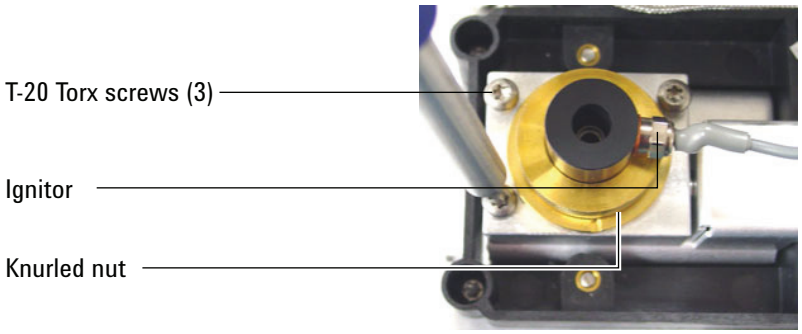
2 Turn the FID flame **On.**

3 Observe the ignitor plug though the FID chimney. The small hole should glow during ignition sequence.

If the test fails, check for the following possible causes:

- The ignitor may be bad; replace the ignitor.
- Detector temperature is set to < 150 °C. Agilent recommends operating the FID at > 300 °C.
- The ignitor is not making a good connection to the ground:
  - The ignitor must be tightly screwed into the FID castle assembly.
  - The three T-20 Torx screws that hold the collector assembly in place must be tight.
  - The brass knurled nut that holds the FID castle assembly in place must be tight.

  Perform FID maintenance if these parts are corroded or oxidized.



Corrosion in FID Collector and Ignitor Glow Plug

Agilent recommends inspecting the collector and ignitor glow plug for corrosion while performing maintenance on the FID.

The FID combustion process results in condensation. This condensation, combined with chlorinated solvents or samples, causes corrosion and sensitivity loss.

To avoid corrosion, keep the detector temperature above 300 °C.

FPD+ Does Not Ignite

- Check that the FPD+ temperature is high enough for ignition (> 150 °C).
- Check FPD+ flow rates and that they match the type of filter installed in the FPD+. The hydrogen:air ratio greatly impacts ignition. Nonoptimal flow settings can prevent flame ignition.

- Measure the actual detector flows.
- Check that the FPD+ ignitor operates.
- During the ignition sequence, display the air flow rate. The air flow rate should go to 400 mL/min while trying to ignite to flame. If not, there is insufficient air supply pressure.
- Check the **Lit offset** value. The typical **Lit offset** value is 2.0. If it is zero, autoignition is turned off. If it is too large, the software will not recognize that the flame is lit and will shut the detector down.
- If the flame still will not light, there could be a large leak in the system. This results in measured flow rates being different from actual flow rates, causing non-ideal ignition conditions. Thoroughly leak check the whole system. (See **"Checking for Leaks"** .)
- Try increasing the supply pressures to the FPD+ flow module. This makes the flame easier to light without changing the setpoints.
- Check the **Lit offset** value. If it is zero, autoignition is turned off. If it is too large, the software will not recognize that the flame is lit and will shut the detector down.
- Under some operating conditions, the flame will light more easily with the vent tube removed. After lighting the flame, reinstall the vent tube.
- Check cable connections to coupling, coupling connection to glow plug, glow plug tightness.

NPD Adjust Offset Process Fails

- Inspect the jet to see if it is clogged.
- Measure the actual detector flows. If the hydrogen or makeup flows are zero or much lower than the displayed flow, suspect a plugged jet.
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- If the process still fails, there could be a large leak in the system. This causes measured flow rates to be different from actual flow rates. Thoroughly leak check the whole system, especially the detector column fitting. (See **“Checking for Leaks”** .)

NPD Bead Will Not Ignite

- Verify that the flow settings are correct and appropriate.
- If the process still fails, there could be a large leak in the system. This causes measured flow rates to be different from actual flow rates. Thoroughly leak check the whole system, especially the detector column/adapter fitting. (See **"Checking for Leaks"** .)
- Check for fault messages. Press **Diagnostics**, and check the GC logs. You can also read the bead current.
- Check the condition of the bead.
- Inspect the jet to see if it is clogged. (See **"To Check for a Plugged NPD Jet"** .)
- Measure the actual detector flows. (See **"To Measure a Detector Flow"** .) If the hydrogen or makeup flows are zero or much lower than the displayed flow, suspect a plugged jet.

FPD+ Temperature Will Not Become Ready

If the FPD+ emission block temperature will not become ready:

- Check the oven temperature. If the oven temperature is high (over 325 °C) for a long time, set the emission block temperature to its highest value (165 °C).
- Check the transfer line temperature. If the transfer line is set to a very high temperature (near 400 °C), set the emission block temperature to at least 150 °C.

Shutdown (Defective) Device

By default, the GC monitors the status of all configured devices (inlets, detectors, valve box heaters, valves, oven heater, EPC or EPR modules, and so forth) and becomes ready when all of them reach setpoint. If the GC senses a problem in one of these devices, the GC will either never become ready, or may go into a shutdown state to protect itself or prevent a safety hazard. However, there may be a time when you do not wish to have the readiness state of a device prevent a run from starting. An important example is when an inlet or detector heater is defective. Normally, this fault prevents the GC from becoming ready and starting a run. However, you can set the GC to ignore this problem so that you can use the other inlet or detector until the device is repaired.

Not all devices can be ignored. You can ignore the readiness state of inlets, detectors, EPE or EPR modules, or the oven. The readiness state of other devices and components can never be ignored, for example, injection devices such as a switching valve or automatic liquid sampler.

To ignore the status of a device:

1. Turn off the device's heater and gas flows as applicable. (Make sure that it cannot create a safety hazard.)
2. Go to **Settings** > **Configuration**, then select the component and disable **Readiness**. The same task may be performed from a data system method editor.

You can now use the GC until the device is repaired.

CAUTION

Do not ignore the readiness state of a device that is being used unless you do not care if it reaches setpoint.

Be sure to re-enable Readiness after the device is repaired. Otherwise, its state (temperature, flow, pressure, and so forth) will continue to be ignored, even if using this device in the analysis.

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Retention Times Not Repeatable

- Replace the septum.
- Check for leaks in the inlet, liner (as applicable), and column connection. (See **"Checking for Leaks"**.)
  - **"To Perform an Inlet Leak Check"**
  - **"To Perform a Pressure Decay Leak Test"**
- Check for sufficient carrier gas supply pressure. The pressure delivered to the GC must be at least 40 kPa (10 psi) greater than the maximum inlet pressure required at final oven temperature.
- Run replicates of known standards to verify the problem.
- Verify that you are using the correct liner type for the sample being injected.
- Consider if this is the first run. (Has the GC stabilized?)
- If using an FID or NPD and retention times increase (drift), check the jet for contamination or replace the jet.

Peak Areas Not Repeatable

Check the ALS syringe operation. (See the Troubleshooting section of the sampler user manual)

- Replace the syringe.
- Check for leaks in the inlet, liner (as applicable), and column connection. (See **"Checking for Leaks"**.)
- Check sample level in vials.
- Run replicates of known standards to verify the problem.
- Consider if this is the first run. (Has the GC stabilized?)

Contamination or Carryover

If your output has contamination or unexpected peaks, do the following:

Isolate the source

1 Perform a solvent blank run using a new, pure source of solvent. If the contamination disappears, the problem may be either in the sample or solvent-related.

2 Perform a blank run (remove the syringe from the injector and start a run). If the contamination disappears, the problem is in the syringe.

3 Remove the column from the detector and cap the detector fitting. Perform another blank run. If the contamination disappears, the problem is in the inlet or column. If the contamination remains, the problem is in the detector.

Check possible causes—all inlet and detector combinations

Inlet, sampler, sample, gas supply

- Check the septum type and installation. The vial septum may be dissolving in the sample. Be sure the vial septum is resistant enough to the solvent you are using. Also ensure the vial septum is flat. If the vial septum is not flat, the needle tends to core the septum and drop pieces into the sample, causing contamination and ghost peaks.
- Perform complete inlet maintenance: Replace all consumable parts and bake out the inlet.
- Check for sample carryover from previous runs. Make several no-injection blank runs and see if the ghost peaks go away or get smaller.
- Check the septum purge flow. If it is too low, the septum may have collected contamination or condensate may be clogged in the purge line. For COC inlets, set the septum purge flow to at least 15 mL/min. Measure the flow. See **"To Measure a Column Flow"**".
- Check all gas trap indicators and dates.
- Verify the gas purity. Perform replicate runs, several with a short interval between them, then several with a large interval. If the contamination peaks are larger for the runs made with the longer interval, suspect contaminated gas: the contamination has more time to deposit onto the column and liner.
- Check for supply tubing and fitting contamination.
- If you suspect that there is contamination in the inlet, perform the bakeout procedure.
- Verify the operation of the inlets. Clean the inlet and replace the contaminated inlet parts.
- Check the solvent level in the ALS wash bottles.
- Replace the ALS syringe if necessary.
- Check the sample injection volume. Verify that the ALS is injecting enough sample into the inlet. Use the Solvent Vapor Volume Calculator to determine how much of the sample should be injected.

- Ghost peaks are sometimes caused by contaminated sample vials. Try new or clean vials to see if ghost peaks disappear.
- Some samples change with heat or ultraviolet light. Check the sample stability.

Column, method

- Perform column maintenance: Bake out contaminants, remove the contaminated length of column near the inlet, and reverse and bake out the column as needed.
- If you suspect that there is contamination in the column, perform the bakeout procedure.
- Verify that the oven program temperature and time are sufficient for the samples being injected. Ghost peaks that are broader than adjacent sample peaks could be from a previous run.
- Inspect the column for contamination. High molecular weight samples that contain residues may cause the syringe, the inlet liner, or the first few inches of column to become contaminated.
-

Detector, detector gas supply

- Check all gas trap indicators and dates.
- Verify the gas purity. Perform replicate runs, several with a short interval between them, then several with a large interval. If the contamination peaks are larger for the runs made with the longer interval, suspect contaminated gas: the contamination has more time to deposit onto the column and liner.
- Check for supply tubing and fitting contamination.
- If you suspect that there is contamination in the detector, perform the bakeout procedure.
- Verify the operation of the detectors. Replace the contaminated detector parts.

Larger Peaks Than Expected

- Check each configured column's dimensions against the actual column dimensions. (See **"Configurable Items to Always Keep Current"**.)
- Check the autosampler injection volume. In the normal injection mode, the sampler uses fast injection to deliver a representative amount of the sample. Fast injection minimizes needle fractionation. Chromatograms from manual injection or slower auto injection devices show higher levels of low molecular weight materials versus higher molecular weight materials because the volatiles boil out of the needle faster than the higher weight materials.
- Check the vial caps. Loose vial caps can cause selective loss of lighter materials from a sample. The cap should not rotate easily if installed properly.
- Check configured syringe size. Some syringe sizes are specified at half-capacity. If the maximum syringe volume is marked at half-height on the barrel, not at the top of the barrel, enter **twice** the labeled volume when configuring the syringe size.

Peaks Not Displayed/No Peaks

- If using an autosampler:
  - Ensure that there is sample in the vial.
  - Verify that the ALS plunger carriage is secured to the syringe plunger.
  - Check that the syringe is installed correctly and draws sample.
  - Check that the syringe is installed correctly and draws sample.
  - Verify that the turret is loaded correctly and injections are not from out-of-sequence vials.
  - Watch to see that the sample is pulled into the syringe.
- Verify the detector in use is assigned to a signal.
- Check the column for proper installation.
- Ensure that the column is not plugged. (See **"To Measure a Column Flow"**.) Perform column maintenance.
- Check for leaks. (See **"Checking for Leaks"**.)
- Check the flow settings, then measure the actual detector flows. (See **"To Measure a Detector Flow"**.)
- Some samples change with heat or ultraviolet light. Check sample stability.
- Check the sample level in the vial.
- If the sample is viscous, try the following:
  - Increase the viscosity delay time.
  - Dilute the sample in an appropriate low-viscosity solvent.
  - Turn the tower fan off.

If the problem is with the detector, see **Table 1**.

Table 1 Detector troubleshooting

Detector	Solution
FID, FPD+	• Verify that the electrometer is turned on. • Verify that the flame is still lit.
TCD	• Verify that the filament is turned on. • Ensure that the reference gas is not set to zero. (The filament will not turn on with zero reference gas flow.)
NPD, ECD	Verify that the electrometer is turned on.

Baseline Rise During Oven Temperature Program

- Inspect the column for bleed.
- Check for leaks/oxygen in carrier gas supply. Oxygen can damage bonded phase capillary columns.
- Check gas supply oxygen trap indicator or date.
- Make solvent blank runs to evaluate baseline without sample.
- Make “no injection” blank runs (remove the syringe from the injector and start a run) to evaluate baseline without solvent.
- Check for contamination. (See **Contamination or Carryover**.)
- Consider the effect of column film thickness on bleed. Try using a column with a thinner film.
- Check for leaks at the column fittings. (See **“Checking for Leaks”**.)
- Prepare and use a column compensation profile.

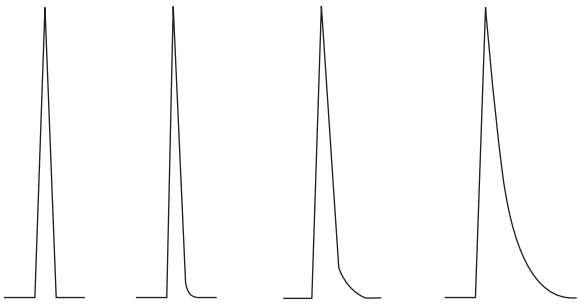
Poor Peak Resolution

- Set column flow to optimum linear velocity.
- Install and use deactivated consumable parts in the inlet (for example, a liner).
- Perform column maintenance: Bake out contaminants, remove the contaminated length of column near the inlet, and reverse and bake out the column as needed.
- Check column installation at both ends.
- Select a higher resolution column.

Peak Tailing

The figure below shows an example of tailing peaks. When troubleshooting tailing peaks, consider:

- Which peaks are tailing?
- Are the tailing peaks active compounds, all compounds, or are there trends (such as early eluters or late eluters)?



- Check the column for severe contamination.
  - If using a capillary column, remove 1/ 2 to 1 meter from the front of the column.
  - For bonded and cross-linked phases, solvent rinse the column.
  - Check for inlet contamination. Tailing will sometimes increase with compound retention. Clean the inlet and replace contaminated inlet parts. (See the Maintenance manual.)
- Consider the column stationary phase (active column). This only affects active compounds. An active column usually produces tailing that increases with retention time.
- Cut off
  - 1 meter from the front of the column.
  - Replace the column.
- Verify that the column was cut and installed properly.
  - Re-cut and reinstall the column into the inlet and replace the ferrules. Make a clean, square cut using a reliable tool.
  - Confirm the installation is leak free. If there is a leak at the column fitting, you will see more tailing for early eluting peaks. (See **“Checking for Leaks”**.)
- Consider the type of adapter, liner, and inlet seal being used. One or all of these may be contaminated or active.
  - Use a new, deactivated liner. This only affects active compounds.
  - Clean or replace the inlet seal.
- Check adapters (if installed) and liner for solid particles. If solid particles are visible, clean or replace.
- For capillary splitless injection, consider compatibility between the solvent and column.
  - Use a different solvent. This will help in instances where there is more tailing for the early eluting peaks or those closest to the solvent front.

  - Use a 3 to 5 meter retention gap.
- Verify that the injection technique is adequate. This is usually related to erratic plunger depression or having sample in the syringe needle.
- Verify the inlet temperature.
  - If the temperature is too high, tailing is generally worse for early eluters. Decrease inlet temperature by 50 °C.
  - If the temperature is too low, tailing usually increases with retention. Increase inlet temperature by 50 °C.
- Check for dead volume in the system. Check for correct column installation at both ends.
  - If peak tailing decreases with retention time, reduce dead volume in transfer line connections, fused silica unions, and so forth.
  - A column installed too high into a detector or inlet can create dead volume areas.
- Inspect any transfer lines for cold spots. Cold spots cause tailing that usually increases with retention time.

NPD Peak Tailing

For NPD, do the following:

- Verify that you are using the correct bead for the sample being run. If you are analyzing phosphorus, install a black bead. White beads can cause peak tailing when phosphorus is being analyzed.
- Verify that the correct jet is installed. Use an extended jet.
- Replace the ceramic insulators.

Peak Boiling Point or Molecular Weight Discrimination Poor

If you have trouble with peak boiling point or molecular weight discrimination (inlet discrimination), do the following:

- Check the inlet for contamination. Clean and change the liner if necessary. Replace all inlet consumable parts. See the Maintenance manual.
- Adjust the inlet temperature.
- Run standards against a known method to determine expected performance.

For any inlet operating in split mode with any detector

- Check liner type. Use a liner optimized for split analysis—one that contains glass wool or other surface area packing to allow complete sample vaporization.
- Increase the inlet temperature and verify that the insulation cup is installed and contains insulation.
- Check column cut and installation into the inlet.

For any inlet operating in splitless mode with any detector

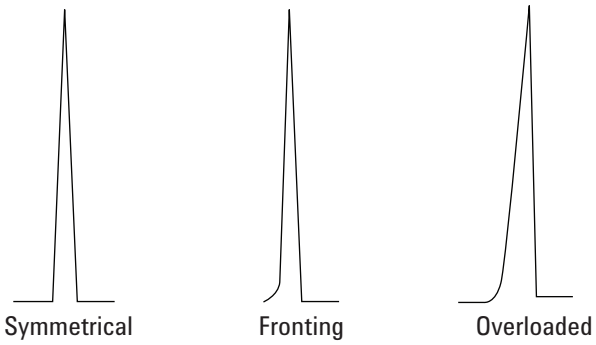
- Check the inlet for leaks. (See **“Checking for Leaks”**.)
- Check liner type. Use a liner optimized for splitless analysis (deactivated, large volume).
- Verify that the oven starting temperature is less than the solvent boiling point.
- Check column cut and installation into the inlet.
- Check that the solvent vapor volume does not exceed the liner capacity.
- Check for appropriate purge delay time. (Liner volume/column flow)

Sample Decomposition in Inlet/Missing Peaks

- Lower the inlet temperature.
- Check for air or water in the carrier gas; verify gas purity and functionality of traps.
- Verify that the liner is appropriate for the sample being run.
- Perform complete inlet maintenance: Replace all consumable parts and bake out the inlet.
- Install a deactivated liner (if liner is used).
- Check for leaks at the septum, liner, and column fittings. (See **"Checking for Leaks"**.)
- Install an Agilent Direct Connect liner.
- Use a pulsed pressure method for quicker sample transfer to column.
- Bake out the inlet.
- Clean the inlet.

Peak Fronting

The figure below shows examples of the three types of peaks: symmetric, fronting, and overloaded.



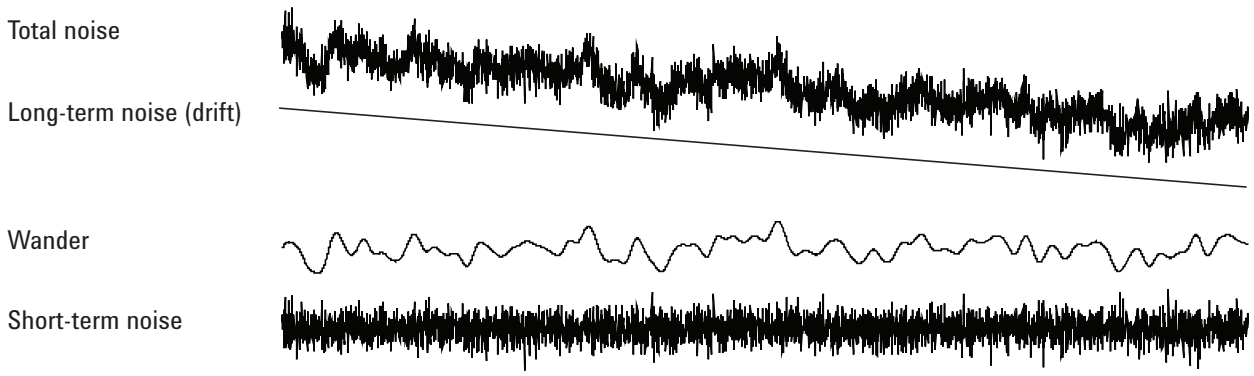
If peak fronting or overloading occurs, try the following:

- Verify that the injection volume is appropriate. Column overload is the most common cause for fronting peaks.
  - Decrease injection volume, dilute the sample, and/or increase the split ratio.
  - Change the column type or film thickness.
- Ensure that the column is installed properly. If not, reinstall the column in the inlet.
- Verify that the appropriate injection technique is being used. This is usually related to erratic plunger depression or having sample in the syringe needle.
- If using capillary splitless injection, consider the compound solubility in the injection solvent.
  - Change the solvent.
  - Use a retention gap.
- Check purity of sample solvent. For solvents with large differences in polarity or boiling points, a mixed sample solvent can cause peak fronting. Change the sample solvent.

Noisy Detector, Including Wander, Drift, and Baseline Spikes

Noise should be measured under "normal" operating conditions, with a column connected and carrier gas on. FID electrometer noise or drift (flame off), for instance, will not provide much indication of how the detector will perform in practice because major sources of noise are not included in this measurement. Noise typically has a high frequency component (electronic in origin) and lower frequency components that are referred to as wander and drift.

Wander is random in direction but at a lower frequency than the short-term electronic noise. Long-term noise (drift) is a monotonic change in signal over a period that is long compared to the wander and electronic noise (see below). Terms like "short" and "long" are relative to the width of the chromatographic peaks. In general, one should measure noise over a period of time that is about 10 times the peak width at half height (or 10 times the area/height ratio for a Gaussian peak). Measuring for longer times can over-estimate noise; shorter times may underestimate noise.



Noisy baseline

A noisy baseline or high detector output can indicate leaks, contamination, or electrical problems. Some noise is inevitable with any detector, although high attenuations can mask it. Since noise limits useful detector sensitivity, it should be minimized.

- For all detectors, check for leaks at the column fittings. (See **"Checking for Leaks"**.)
- For the FID, see **To Isolate the Cause of FID Noise**.
- For the TCD, verify data collection at £ 5 Hz.

If noise appears suddenly on a previously clean baseline, do the following:

- Consider recent changes made to the system.
- Bakeout the inlet.

Bakeout can reduce septum bleed and other contaminants. New septa may contribute noise from bleed of low molecular weight material. If noise decreases when inlet temperature is lowered, this is a likely cause. Use only high quality septa and store them where they cannot become contaminated.

- Verify the purity of carrier and detector gases. If a tank was replaced recently and the old one is still available and still has some gas in it, try the older tank to see if noise decreases.

  If the new gas is so badly contaminated that it saturates traps, changing to the old one may show little improvement until the traps are replaced or regenerated. This problem is most common with nitrogen carrier gas. Deal with a reliable gas supplier.
- For the TCD, check for ambient air pressure fluctuations at the GC. Air currents from a fan or air conditioner blowing across the GC may interfere with gas exiting the detector. This is a possible, though not very likely cause of noise since detectors are well protected. Switching off the air current source or shielding the detector exit identifies this problem. Install the TCD outlet restrictor (G1532-60070).
- Loose connections in the detector or its signal path generate noise.
- Verify proper reassembly after recent maintenance.
- Inspect the detector for contamination.

If noise increases gradually to an unacceptable level, check the following possible causes:

- Bakeout the detector.
- Inspect the detector for contamination. Replace parts as needed.
- Inspect the column and inlet for contamination.
- Inspect the FID or NPD jet for contamination.
  - **To Check for a Plugged FID Jet**
  - **To Check for a Plugged NPD Jet**
- Verify that the FPD+ photomultiplier tube (PMT) is properly installed. If it is not, light leaks and ultimately noise will result.

FIDs are susceptible to gradual buildup of deposits in the detector. In extreme cases, spiking occurs along with increased noise level.

Carbon (black) deposits may form from solvents that burn poorly (primarily chlorinated materials and aromatics). Avoid such solvents if possible. If you must use them, be prepared to clean the detector regularly.

Silicon dioxide (white) is formed when bleed from a silicone column is burned in the flame. To minimize this, use low column loadings, select phases with high temperature limits, condition columns thoroughly before use, and use the lowest possible oven temperature for the analysis.

To remove either type of deposit, disassemble the detector and scrub with a small brush. A solvent (almost anything will do) helps flush away the particles. Agilent recommends replacing dirty collector and insulator parts.

Other factors that can contribute to noise:

- Column installed too high into detector.
- Oven temperature exceeds column maximum recommended temperatures.

Baseline wander and drift

Baseline wander or drift can occur when a flow or temperature setting is changed. If the system has not stabilized at the new conditions before it starts a run, some baseline changes are to be expected. The following cases assume that sufficient stabilization time has elapsed since the last change in operating conditions.

If experiencing baseline wander, check for leaks, especially at the septum and at the column. (See **"Checking for Leaks"** .) If the leak is at the detector end of the column, retention times are stable from run to run but sensitivity is reduced. If it is at the inlet end, sensitivity is reduced and retention times are increased.

Also consider whether the oven temperature program is sufficient.

Baseline drift is most often seen during temperature programming. To correct baseline drift, do the following:

- Verify that column compensation is used and the profile is current. (To compensate for bleed.)
- Verify that the column is conditioned.
- Check column bleed while at operating temperature.
- Check the signal mode assigned to the column in the data system.
- Check the column compensation profile. It may be too little (upscale drift) or too much (downscale drift).

  This cause of drift is minimized by thorough column conditioning. Operating at a lower temperature reduces the drift but prolongs the analysis. Use of a chromatographically equivalent column with a higher temperature limit is also possible.

Baseline spiking

There are two types of spiking on the baseline output: cyclic and random. Spiking will not normally be noticed on the display; it will be noticed only on a plot or online trace.

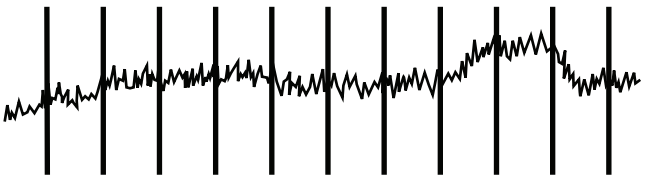


Figure 1. Cyclic spiking

Cyclic spiking can be caused by the following:

- An electric motor
- Building heating/cooling system

- Other electronic interferences in the lab

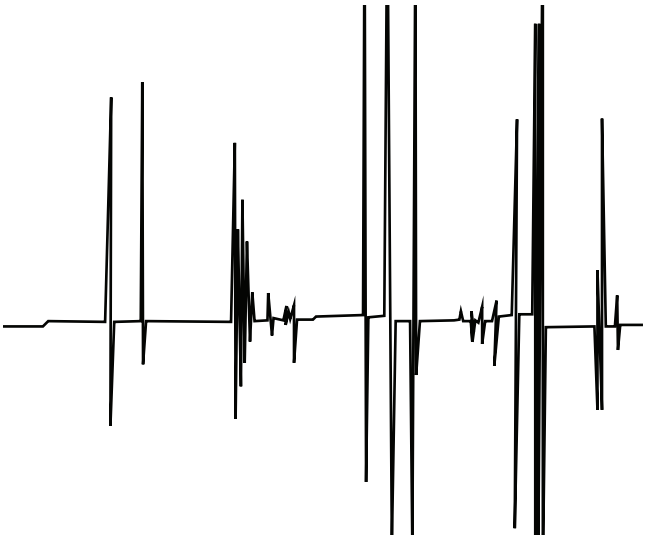


Figure 2. Random spiking

Spikes are isolated baseline disturbances, usually appearing as sudden (and large) upscale movements. If accompanied by noise, resolve the noise problem first since spiking may disappear at the same time.

-
- Check for a contaminated detector. In an extremely dirty FID, particles of carbon or silicon dioxide may break away and fall into the detection zone.
- For a packed column, check that the packed column exit is properly sealed with glass wool. This will cause packing particles to be blown into the detector. A defective or missing glass wool plug at the exit of a packed column will do this. This may happen with any detector but the flame detector is particularly susceptible because of the narrow bore of the jet.
- Check packed column installation. The bottom of the jet or the end of a transfer tube extends into the end of the column. If it touches a glass wool plug, spikes are produced.
- Check for the correct jet.
- Check that the detector temperature is not too low.

Electron Capture Detector (ECD) Noise and Sensitivity

CAUTION

Detector disassembly and/or cleaning procedures other than thermal should be performed only by personnel trained and licensed appropriately to handle radioactive materials. Trace amounts of radioactive 63 Ni may be removed during these other procedures, causing possible hazardous exposure to radiation.

WARNING

To prevent possible hazardous contamination of the area with radioactive material, the detector exhaust vent must always be connected to a fume hood, or otherwise vented in compliance with local regulations.

Performance problems associated with the ECD include (but are not limited to) loss of sensitivity (real, or perceived), high signal background, noisy baseline, and chromatographic peaks or humps that are not characteristic of the samples being injected.

If the problems are not accompanied by an increase in the signal output, as read from the front keypad of the GC, then suspect the detector only after the other parts of the chromatographic system have been checked.

Before starting extensive troubleshooting, consider first the nature of the problem:

1. If recent changes were made in the system, such as changing carrier or detector gases, performing inlet or column maintenance, or changing the column, investigate the possibilities that contamination or leaks were introduced.
2. If the problem has been chronic and is now acute enough to interfere with analysis, suspect contamination, column degradation, or ultimately a bad ECD cell.

See also:

"Display signal evaluation"

"Sensitivity"

Display signal evaluation

The first consideration is the signal value when the GC is in the idle state. The "idle" signal level is a function of the type and quality of the carrier and detector gases, as well as the flows and application. It may be different for different situations but as a general rule, the following values apply (**Table 2**). **Figure 3** shows typical ECD life cycle stages.

Table 2 ECD signal evaluation

ECD signal	Comments
<200	ECD is "healthy".
200 to 400	Slightly elevated, no cause for concern at this point. Signal still in "good" range.

Table 2 ECD signal evaluation (continued)

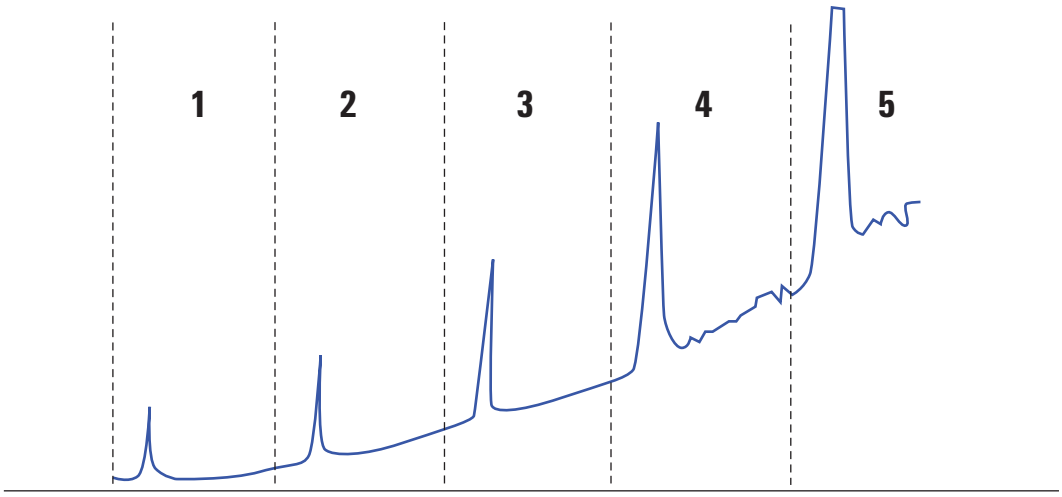
ECD signal	Comments
400 to 1000	System showing signs of contamination from gases, column, or samples. If the signal increases in response to increased oven temperature, suspect the column.
1000 to 2000	Suspect more severe contamination, follow the troubleshooting guidelines.
>2000	If the following procedures do not work, suspect the ECD cell.

NOTE

It may take 24 hours for the ECD baseline to completely stabilize, especially if you are starting with a cold system and want to assure high-sensitivity operation. For the most accurate results, run the detector at normal operating conditions for as long as possible (at least 2 hours and up to 24 hours) before running the frequency test.

If you will be injecting into an unused inlet, you must use low-bleed septa. Make sure to condition new septa before use in an inlet for several hours with 1 to 5 mL/min carrier flow.

Typical ECD Life Cycle Stages:



Stage 1: New cell

Background = 100 Hz. Cell meets Agilent signal-to-noise (S/N) standard.

Stage 2: 6 months – 1 year

Background = 180 Hz. Increased response.

Stage 3:1–2 years

Background = 350 Hz. More response, increasing noise, lower S/N.

Stage 4: 2–4 years

Background > 500 Hz. Noisy baseline, negative peaks. Thermal cleaning required.

Stage 5:4–10 years

Background > 1000 Hz. Huge response. Very noisy, cell contaminated. Requires cell exchange.

Figure 3. Typical ECD life cycle

Sensitivity

If the ECD is in the "good" signal range and sensitivity is the issue, the problems quite likely may be in the inlet or column. Consider the following issues:

1 If using an inlet with split and splitless modes, verify that the mode has not changed and that the split valve is functioning. This can be done by verifying that there is a corresponding change in the response by making split and then splitless injection, all other

method parameters remaining the same. If using split mode, verify the split flow using a flow meter.

2 Check all flow rates. See **"Measuring FID, TCD, ECD, and FPD+ flows"**.

3 Confirm that complete inlet maintenance has been performed. This includes trimming and re-installing the column.

4 Verify that the injecting device is working properly. If the method uses a sampling valve, make a direct syringe injection of concentration similar to that introduced by the valve. Rule out a problem with the valve.

5 Inspect the mixing liner in the makeup gas adapter. A small piece of graphite or sample contamination will reduce sensitivity.

Contamination (high baseline)

If the ECD has a baseline higher than expected for its age, check the following:

- Rule out all other possible causes. See **"Sensitivity"** above. Also check carrier and detector gas supplies, supply gas traps, and the column.
- Inspect the mixing liner in the makeup gas adapter. A small piece of graphite can contaminate results.
- If the detector baseline is > 500 Hz, regardless of detector age, bakeout the detector. Let it run for 1–2 days.

Low Peak Area or Height (Low Sensitivity)

- If using an inlet in split mode, check the split ratio.
- Check for leaks. (See **"Checking for Leaks"**.) To troubleshoot small leaks in a split/splitless inlet, see **To Manually Check for Leaks in a Split/Splitless Inlet**.
- Check the inlet for contamination. (See **"Contamination or Carryover"**.)
- Check each column and verify that it was cut and installed properly at each end.
- Verify that the column type is correct.
- Perform column maintenance: Bake out contaminants, remove the contaminated length of column near the inlet, and reverse and bake out the column as needed.
- Verify that the liner type is appropriate for the sample.
- Verify that the detector flow settings are correct.

  Measure the actual detector flows. If an actual flow does not match the GC display, check for contamination and restrictions, for example a plugged jet. See the following:

  - **To Measure a Detector Flow**
  - **To Check for a Plugged FID Jet**
  - **To Check for a Plugged NPD Jet**
- Check the supply gas purity.
- Check all trap indicators and dates.
- Verify that the method parameters are correct.
- Some samples change with heat or ultraviolet light. Check sample stability.
- Check configured syringe size. Some syringe sizes are specified at half-capacity. If the maximum syringe volume is marked at half-height on the barrel, not at the top of the barrel, enter **twice** the labeled volume when configuring the syringe size.
- If the drop in peak area or height happened gradually due to baseline rise, rather than a sudden change, check for detector contamination. Bakeout the detector.

If using an FID:

- Verify that the correct jet is installed.
- Check for a dirty jet.
- Check for contaminated detector parts.
  - **To Check for a Plugged FID Jet**

If using an ECD:

- Replace the fused silica indented mixing liner.
- Replace and reinstall column.

If using an NPD:

- Check the detector for contamination.
- Replace ceramic insulators.
- Replace the bead.

If using an FPD+:

- Verify correct column installation.
- Check that the correct filter is installed and is clean.
- Check the flow rates.
- Check the makeup gas type.

To Resolve Low Sensitivity with an FID

In normal use, the FID can develop deposits on the collector, insulators, jet, and so forth. To reduce contamination buildup, Agilent recommends using the detector at 300 °C or higher. However, even with normal use deposits develop in the jet (usually white silica from column bleed or black, carbonaceous soot). These deposits reduce sensitivity and cause chromatographic noise and spikes. Jets require periodic cleaning or replacement. The following procedure checks for causes of low sensitivity by frequency of occurrence.

For sensitivity loss associated with noise, wander, or drift, also see **"Noisy Detector, Including Wander, Drift, and Baseline Spikes"**.

1 Check detector flow settings.

   The general rule is 1:1 flow ratio of hydrogen to (column + makeup gas).

2 Measure the actual detector flow rates. See **"To Measure a Detector Flow"**. If the actual hydrogen, makeup, and capillary column flows are lower than the display, the jet is becoming plugged. See **"To Check for a Plugged FID Jet"**. Replace the jet.

3

4 Check that column is properly installed. Reinstall it. Make sure the column is installed all the way to the tip and withdrawn 1–2 mm (column id > 100 µm).

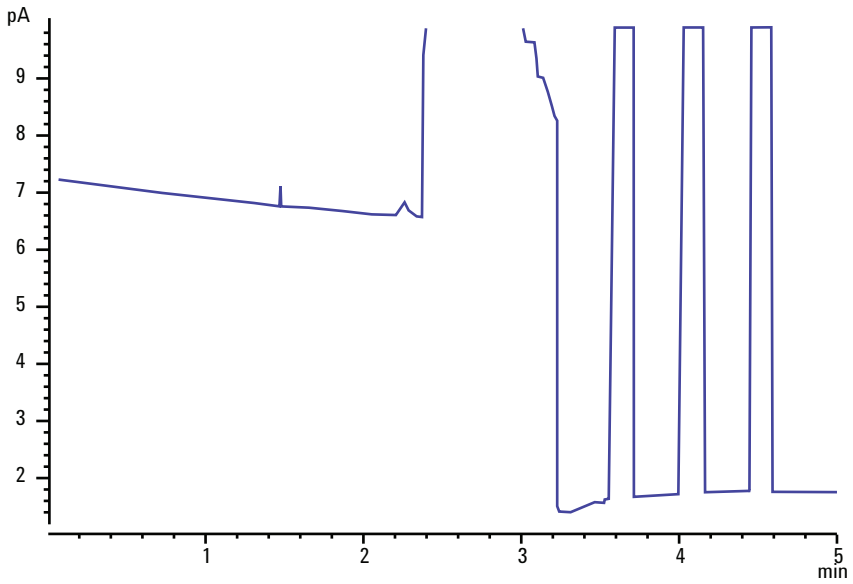
5 Check inlet parameters that control venting, such as split ratio and splitless purge delay time. Make sure the sample is not being inadvertently vented.

6 Perform inlet maintenance (replace all consumable parts) and pressure test the inlet when completed. See the Maintenance manual and **"Checking for Leaks"**.

7 Perform complete FID maintenance. Disassemble the FID and clean or replace all parts.

FID Flame Goes Out During a Run and Attempts to Reignite

The following is an example chromatogram showing a flameout from a large solvent peak.



After a flameout, the GC will try to ignite the flame three times. The GC tries to reignite whenever the detector output falls below the **Lit offset** setpoint, regardless of whether or not the flame was extinguished. (In a very clean system, the baseline output may be less than 2 pA.)

If the FID flame goes out during a run, do the following:

- See if an aromatic solvent peak or water extinguished the flame.
- Check for a plugged jet.
- Verify that the gas flow settings are correct. Verify that **Lit offset** is set appropriately.

If the FID flame attempts to reignite but is already lit, do the following:

- Verify that the FID **Lit offset** setting is appropriate for the run (typically < 2.0 pA).
- Check to see if an aromatic peak or water extinguished the flame.
- Check for a partially plugged jet. Measure actual hydrogen, air, and makeup flows at the detector. (See **"To Measure a Detector Flow"**.) Replace the jet as needed.
- Check for leaks at the detector column fitting. (See **"Checking for Leaks"**.)

FID Baseline Output Above 20 pA

- Verify the purity of the carrier and detector gas supply.
- Inspect the column for column bleed. Lower the oven temperature to ambient. If the detector output drops significantly, suspect a contaminated or bleeding column or contaminated carrier gas. Confirm column bleed by turning off column flow (with oven cool) and checking the detector output.
- Check the gas supply trap indicators/dates and ensure that the traps are not expended.
- Verify that the detector was reassembled properly after recent maintenance.
- Inspect the detector for contamination. Bake out the detector.
- Check that the FID leakage current is < 2.0 pA. (See **"To Measure FID Leakage Current"**.)
-

FID Baseline Output at Maximum (~8 Million)

If the FID output seems to be stuck at a very high value (up to 8 million counts), check for a shorted collector.

1. Check if the interconnect spring has been bent. Remove the collector assembly and visually inspect the spring.
2. Disassemble the collector assembly and visually check for rust buildup on any parts. Replace parts as needed. To avoid this problem, operate the detector at >300 °C.
3. Check for carbonization in the detector due to injection or aromatic or chlorinated solvents. To avoid this problem, operate the detector at >300 °C. Reassemble and install the collector and operate the detector using higher air and hydrogen flows (air must be 450 mL/min, hydrogen at 35 mL/min).

FPD+ Flame Goes Out During a Run and Attempts to Reignite

If the flame goes out during a run, do the following:

- Check the GC system for leaks, especially at the detector column fitting. (See **"Checking for Leaks"**.)
- FPD+: Verify the transfer line temperature is set ≥ 200 °C.
- Ensure that condensation in the vent tube is not dripping back into the detector. The flexible plastic vent tube must run from the detector into a container, without sagging, in order to properly drain water condensate. Keep the open tube end out of the water in the container.

If the FPD+ flame goes out and then reignites, do the following:

- Verify that the **Lit offset** setting is lower than the normal baseline.
- Check for leaks. (See **"Checking for Leaks"**.)
- Check the flow settings, then measure the actual detector flows. (See **"To Measure a Detector Flow"**.)
- Certain environmental conditions, such as:
  - Strong electromagnetic fields
  - Large ambient temperature swings
  - Large atmospheric pressure swings

  can cause an artificially low signal in the GC, incorrectly indicating that the flame has gone out. As a result, the run aborts and the GC tries to relight an already-lit flame.

You can verify that the flame is lit by holding a cold, shiny surface (such as a mirror or a wrench) over the exit tube. Condensation on the surface indicates that the flame is lit.

Reset the **Lit offset** to 2.0.

FPD+ Quenching/Repeatability

Hydrocarbon quenching occurs when a high concentration of carbon dioxide from a hydrocarbon peak is in the flame at the same time as the sulfur species. Part of the light emitted by the sulfur species is absorbed by some CO2 species.

Self-quenching occurs at high concentrations of the heteroatom species. Some other ground state (inactivated) species reabsorbs the emitted photon, preventing it from reaching the PMT.

To resolve hydrocarbon quenching:

- The column should provide good separation of the compounds, those that contain sulfur or phosphorus as well as those that do not but may absorb light.
- Optimize the chromatographic separation such that hydrocarbon peaks are resolved from sulfur or phosphorus peaks.

  1 Run the analysis first on a FID in order to see all the peaks (the FPD+ ignores hydrocarbons).

  2 Run the analysis on the FPD+.

  3 Modify the method so that the peak of interest is separate from the rest of the peaks.

FPD+ Output Too High or Too Low

- Verify that the correct filter is being used. Do not use a phosphorus filter with sulfur-optimized flows or a sulfur filter with phosphorus-optimized flows.
- Check the position of the column as installed in the detector.
- Check the gas purity.
- Verify that the flows are optimized for the filter being used. Monitor the FPD+ output. The table below provides examples of detector output when the filter installed in the detector and the gas flows in use do not match.

Table 3

	Outputs	
Gas flows optimized for	**With sulfur filter**	**With phosphorus filter**
Sulfur	30 to 50	10 to 12 (low)
Phosphorus	240 to 250 (high)	30 to 50

Besides having a mismatch between the filter installed and a particular set of gas flows, check the FPD+ signal output with the flame lit:

- If the output is 0.5 to 3.0, check that the flame is ON.
- If the output is 0, check if the electrometer is turned OFF or the signal cable is disconnected.
- If the output < 30, the flame may be in the wrong position. Check detector flows, column flow, and column position. See the following:
  - **To Measure a Column Flow**
  - **To Measure a Detector Flow**

FPD+ Low Peak Areas

- Check the flow settings, then measure the actual detector flows. (See **"To Measure a Detector Flow"**.)
- Perform complete inlet maintenance: Replace all consumable parts and bake out the inlet.
- Perform column maintenance: Bake out contaminants, remove the contaminated length of column near the inlet, and reverse and bake out the column as needed.
- Verify that the column is installed properly.
- Consider the filter type (sulfur or phosphorus).
- Check the system for leaks. (See **"Checking for Leaks"**.)
- Verify that the method settings are appropriate.
- Check the flow rates.
- Check the makeup gas type.

FPD+ Large Peak Width at Half-Height

If the FPD+ produces peaks that are abnormally wide at half the peak height, do the following:

- Check the actual injection volume; reduce if necessary.
- Verify that the liner is not reacting with the sample.

FPD+ Baseline Output High, > 20 pA

- Check the supply gas purity.
- Check all trap indicators and dates.
- Check the detector for contamination.
- Check for light leaks at the photomultiplier tube (PMT); tighten the PMT if it is loose.
- Perform complete inlet maintenance: Replace all consumable parts and bake out the inlet.
- Perform column maintenance: Bake out contaminants as needed.

FPD+ Chromatographic Output Showing Clipped Peaks

If you have an application at the upper limit of the dynamic range (especially with sulfur), you may have to desensitize your instrument. Replace the 1000-1437 sulfur filter with filter part number 19256-80000. Then set the detector gas flows to the values used in the phosphorus checkout method. This raises the baseline but with some loss in the signal-to-noise ratio.

There should be no problems with either solution and the use of hydrogen. However, please note the following warning.

WARNING

Hydrogen gas is flammable and potentially explosive. Be sure to turn off the hydrogen gas at the source until connections are made. Also, leak test connections, lines and valves before operating or servicing the instrument.

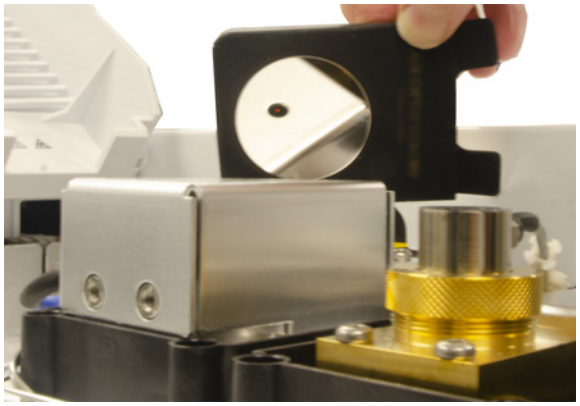
NPD Solvent Quenching

If the baseline does not recover after a solvent peak, try the following:

- Turn hydrogen off/on around the solvent peak.
- Use nitrogen as the makeup gas.
- Set the total column flow and makeup gas to less than 10 mL/min.
- Increase the air flow by 10 mL/min.
- Increase the detector temperature to 325 °C.
-

NPD Response Low

- Perform complete inlet maintenance: Replace all consumable parts and bake out the inlet.
- Perform column maintenance: Bake out contaminants as needed. Verify correct column installation.
- A large concentration of solvent has extinguished the hydrogen/air plasma. Run the makeup gas at a flow rate of 5 mL/min.
- Verify that there is hydrogen coming from the external supply. Check that flow and pressure are turned on at the keyboard. The hydrogen flow rate should be between 1.0 and 5.5 mL/min. Measure the actual gas flow at the detector. (See **"To Measure a Detector Flow"**.)
- Check for a partially plugged jet. See **To Check for a Plugged FID Jet**.
- Verify that the bead is activated. Use a mirror to look through the vent hole on the detector lid to see if the bead is glowing orange. If the bead is not glowing, check the detector background signal. Turn the bead off to establish a reference level, and then turn on the bead and look for a sudden sharp increase in output, which indicates that ignition occurred. If 4 V are being supplied to the bead but it is not igniting (viewable from **Settings** > **Service Mode** > **Detectors** > **Signal**), the bead is probably burned out. Replace the bead.



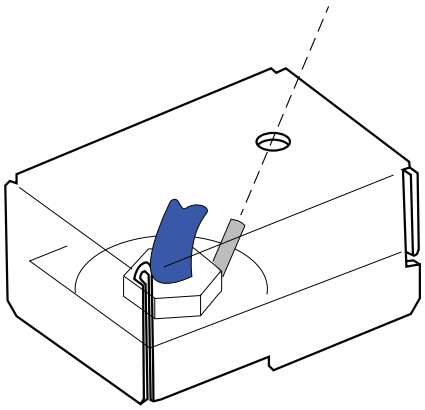


Figure 4. Lit blos bead

WARNING

The emissions from the detector are hot enough to cause burns. Use proper protection such as safety glasses or a mirror when checking to see if the bead is glowing.

- Replace the insulators/collector.
- Check for liquid phase contamination (polar phases).

NPD Baseline Output > 8 million

- The collector is shorted to the detector housing. Disassemble the collector and insulators and reinstall.

NPD Adjust Offset Process Not Functioning Properly

- Inspect the jet to see if it is clogged. (See **"To Check for a Plugged NPD Jet"**)
- Measure the actual detector flows. (See **"To Measure a Detector Flow"** .) If the hydrogen or makeup flows are zero or much lower than the displayed flow, suspect a plugged jet.
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- If the process still fails, there could be a large leak in the system. This causes measured flow rates to be different from actual flow rates. Thoroughly leak check the whole system, especially the detector column fitting. (See**"Checking for Leaks"** .)
-

NPD Low Selectivity

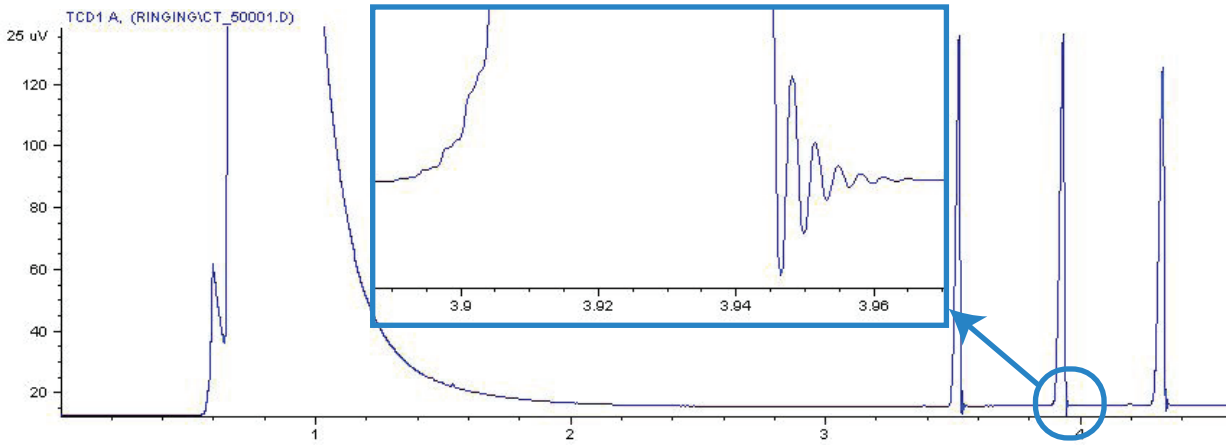
(High hydrocarbon response relative to nitrogen or phosphorus.)

- Verify that the hydrogen flow is correct (< 3 mL/min).
- Inspect the bead; it may be defective or expended.
- Replace the collector and insulators.

Negative Peaks Seen with TCD

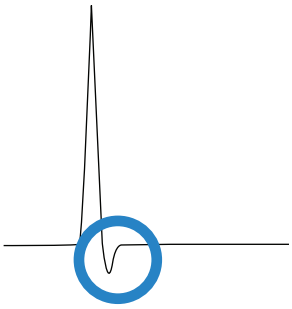
- Verify that the correct gas type is being used.
- Check for a leak in the system, especially at the detector column fitting. (See **“Checking for Leaks”** .)
- Consider thermal conductivity of analytes relative to carrier.
- Consider sensitivity to analytes.
- Check the flow settings, then measure the actual detector flows. (See **“To Measure a Detector Flow”** ”.)

TCD Baseline Has Dampened Sinusoidal Noise Trailing Peaks (Ringing Baseline)



Wrong data rate is selected in the data system. For TCD, the data rate should be ≤ 5 Hz.

TCD Peaks Have Negative Dip on Tail



- Check for leaks at the detector column adapter fitting. (See **“Checking for Leaks”** .)
- Upgrade the detector to a passivated filament.

4 GC Not Ready Symptoms

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This section includes faults and symptoms that will occur when the GC is on but cannot perform analyses. This is indicated by a “Not Ready” warning, by fault messages, or by other symptoms.

GC Never Becomes Ready

Normally the GC becomes ready after flows and temperatures reach setpoint. If the GC does not become ready after a long period of time:

- Select the status/control tray, or the a component under **Method** to see which setpoints or conditions are not ready.
- Check for a sampler problem.
- Check for a data system problem.
- If performing manual injections in splitless or gas-saver mode, you may need to select to prepare the inlet for the injection. Do this, for example:
  - To toggle the inlet purge valve before a splitless injection
  - To prepare for a pulsed injection
  - To turn off gas saver.

  For more information on , see the Agilent 8860 GC Operation Manual.

Flow Never Becomes Ready

If the gas flow never becomes ready, check for the following:

- Check the supply gas for sufficient delivery pressure.
- Check the restrictors installed in the Aux EPC module (if applicable).
- Check the configured gas type. The configured gas type must match the actual gas plumbed to the GC.
- Check for leaks in the gas delivery plumbing and the GC. (See**"Checking for Leaks"** .)

Oven Temperature Never Cools Down/Cools Very Slowly

If the oven does not cool down or cools down very slowly:

WARNING

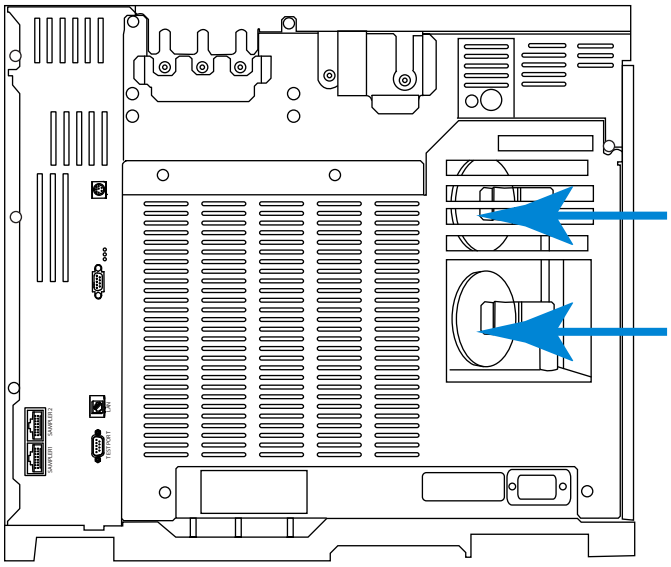
The exhaust coming from the back of the GC is very hot. Keep hands and face away from the exhaust vent.

- Check oven flapper operation.

  1 Decrease oven temperature by at least 20 degrees.

  2 Verify that the oven flaps in the back of the GC are **open**. Listen to verify that the fan is operating. The figure below illustrates the location of the two oven flaps.

  If the flaps are not operating smoothly, contact Agilent for service.



-

Oven Never Heats Up

- Select **Diagnostics** to check for errors to report to Agilent.

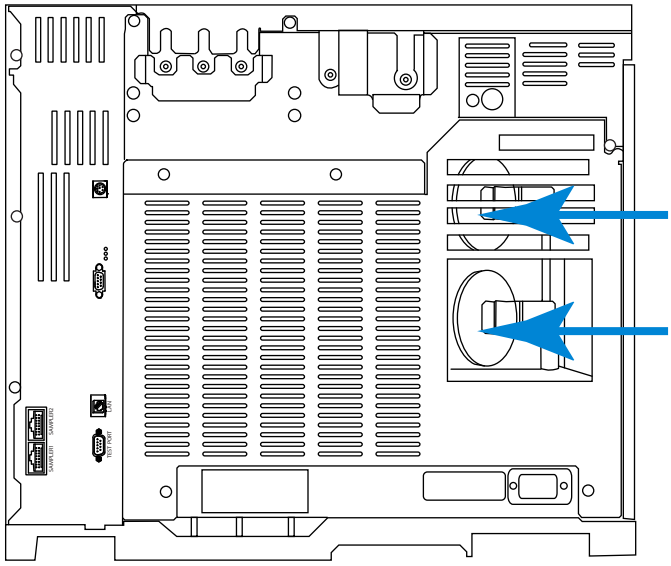
WARNING **The exhaust coming from the back of the GC is very hot. Keep hands and face away from the exhaust vent.**

- Power cycle the GC.
- Check oven flapper operation.

  1 Increase oven temperature by at least 20 degrees.

  2 Verify that the oven flaps in the back of the GC are **closed**. The figure below illustrates the location of the two oven flaps.

  If the flap is stuck open or if the flaps are closed and the oven still does not heat, contact Agilent.



3

Temperature Never Becomes Ready

To be considered ready, a temperature must be at setpoint ±1 °C for 30 s. If a temperature never becomes ready, do the following:

- Check for a missing insulation cup on an inlet or detector.
- Check for a very large temperature difference between the oven and inlet or detector.
- Check for missing insulation around the inlet or detector.

Cannot Set a Flow or Pressure

If you cannot set a flow or pressure using the split/splitless inlet, do the following:

- Check the column mode.
- Check that a capillary column is configured to the correct inlet.
- Check the configured column dimensions.
- Check that the flow is turned on.

A Gas Does Not Reach Adjusted Value or Setpoint Pressure or Flow

If an inlet does not reach its pressure setpoint or adjusted value, it will shut down in an amount of time determined by the type of inlet. (EPR equipped inlets will indicate a lack of readiness via a flashing asterisk.) Do the following:

- Check for sufficient gas supply delivery pressure. The pressure at the supply should be at least 10 psi greater than the desired setpoint.
- Check for leaks. (See **"Checking for Leaks"** .) A large leak may be present somewhere in the system. Use an electronic leak detector to find leaks, then correct them. Do not forget to check the column—a broken column is a very large leak.
- If using gas saver, be sure that the gas saver flow rate is high enough to maintain the highest column-head pressure used during a run.
- Check for an incorrectly installed column.
- Check for a defective inlet or detector pressure sensor.

If you are using a split/splitless inlet:

- Check the split ratio. Increase the amount of split flow.

A Gas Exceeds Adjusted Value, Pressure Setpoint, or Flow

If a gas exceeds its adjusted value, pressure, or flow setpoint, do the following:

If using a split/splitless inlet:

- Decrease the split ratio.
- Replace the split vent filter.
-
- Verify that the correct liner is selected (if liner is used).
- Verify the method pressure settings for the SS inlet are above the minimum viable settings available on the GC.
- Check the gold seal on the split/splitless inlet for contamination.

If using an FID or NPD:

- Check for a plugged jet. See **“To Check for a Plugged FID Jet”** or **“To Check for a Plugged NPD Jet”** .

Valves:

- Check for a misaligned rotor.If necessary, align the rotor.

The Inlet Pressure or Flow Fluctuates

A fluctuation in inlet pressure causes variations in the flow rate and retention times during a run. Do the following:

- Check if the gas purifier or gas generator is operating at or near capacity.
- Check the supply gas for sufficient delivery pressure.
- Verify that the supply pressure regulator is functioning properly. Systems with long supply tubing lengths may require a step-down regulator near the GC. Also, use an additional regulator to smooth out fluctuations caused by gas generators.
- Check for leaks. (See **"Checking for Leaks"** .) A large leak may be present somewhere in the system. Use an electronic leak detector to find leaks, then correct them. Do not forget to check the column—a broken column is a very large leak.
- Check for large restrictions in the inlet liner or split vent trap.
- Verify that the correct liner is installed. Some liners have large pressure drops caused by design or tight packaging.
- Check for extreme changes in room temperature during runs. Correct laboratory temperature problem or move the instrument to a more suitable location.
- Verify Auto Flow Zero feature is On.

Cannot Maintain a Pressure as Low as the Setpoint on a Split Inlet

If the GC cannot maintain a pressure as low as the setpoint, check for the following:

- Consider using a liner designed for split analysis.
- Method pressure parameter (or resultant pressure from a flow setting) is too low for the carrier gas type.
- Check for a plugged liner.
- Check for contamination in the split vent line. Contact Agilent service to replace, if necessary.
- For the split/splitless inlet, replace gold seal.

The Measured Column Flow Does Not Equal the Displayed Flow

If the actual column flow does not match the calculated flow displayed on the GC within 10%, do the following:

- Verify that the measured flows are corrected to 25 °C and 1 atmosphere.
- Verify that the correct column dimensions are configured accurately, including the actual (trimmed) column length.
- A short (<15 m) 0.58 to 0.75 mm id WCOT column is being used with a split/splitless capillary inlet. The total flow controller is set for a high flow rate, which creates some pressure in the inlet and causes column flow even with a setpoint pressure of zero. (In these situations, an actual pressure may be shown on the display, even with a zero setpoint.) With short, 530 to 750 mm columns, keep the total flow rate as low as possible (for example, 20 to 30 mL/min). Install a longer column with higher resistance (for example, 15 to 30 m).
- The split vent line or trap may be partly plugged, creating an actual inlet pressure higher than the setpoint pressure. Check for a restriction in the split vent line.
- Make sure that the auto flow zero is turned on. As applicable, zero the flow and pressure sensor for the flow module. If this does not solve the problem, replace the flow module.

FID Does Not Ignite

- Verify that the Lit Offset is ≤ 2.0 pA.
- Ensure that the FID temperature is high enough for ignition (>150 °C). Agilent recommends >300 °C.
- Check that the FID ignitor glows during ignition sequence. (See **"To Verify FID Ignitor Function During Ignition Sequence"** .)
- Try increasing the supply pressures to the FID flow module. This makes the flame easier to light without changing the setpoints.
- Increase hydrogen flow and decrease or turn off makeup gas flow until ignition occurs, then reduce them toward the method values. Experiment for the best values.

  Increasing hydrogen flow and decreasing makeup flow will help the FID ignite more easily. If it will light under these modified conditions, the cause could be a partially clogged jet, a weak ignitor, or a leak at the column fitting.
- Check for a plugged or partially plugged jet. (See **"To Check for a Plugged FID Jet"** .)
- Measure the FID flow rates. Actual flow rates should be within +/-10% of the setpoint. The hydrogen:air ratio greatly impacts ignition. Nonoptimal flow settings can prevent flame ignition. (See **"To Measure a Detector Flow"** .)
- There could be a large leak in the system if the flame still will not light. Large leaks result in measured flow rates being different from actual flow rates, causing nonideal ignition conditions. Thoroughly leak check the whole system, especially the column fitting at the FID. (See **"Checking for Leaks"**).
- Check the column flow rate. (See **"To Measure a Column Flow"** .) Hydrogen flow should be greater than the sum of the column flow and makeup flow.
- Check for leaks at the FID column fitting.
- Ensure that the FID temperature is high enough for ignition (>150 °C).
- If the analysis permits, substitute nitrogen for helium as makeup.
-

FID Ignitor Does Not Glow During Injection Sequence

WARNING **Keep body parts at a safe distance from the FID chimney while performing this task. If using hydrogen, the FID flame will not be visible.**

1 Remove the detector top cover.

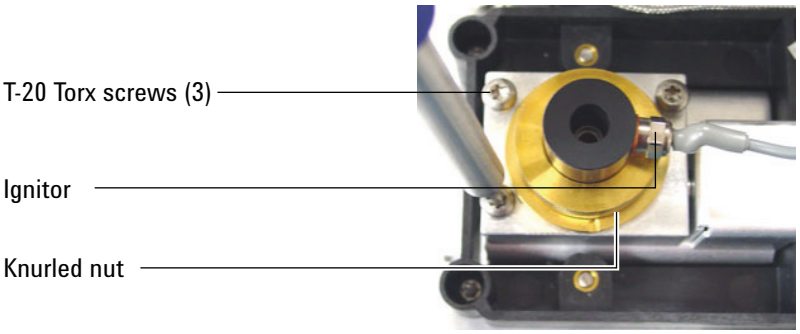
2 Turn the FID flame **On.**

3 Observe the ignitor plug though the FID chimney. The small hole should glow during ignition sequence.

If the test fails, check for the following possible causes:

- The ignitor may be bad; replace the ignitor.
- Detector temperature is set to < 150 °C. Agilent recommends operating the FID at > 300 °C.
- The ignitor is not making a good connection to the ground:
  - The ignitor must be tightly screwed into the FID castle assembly.
  - The three T-20 Torx screws that hold the collector assembly in place must be tight.
  - The brass knurled nut that holds the FID castle assembly in place must be tight.

  Perform FID maintenance if these parts are corroded or oxidized.



FID or NPD Measured Hydrogen and Makeup Gas Flows Much Less Than Setpoint

- Check for a clogged or partially clogged jet. A clogged jet creates backpressure. Since the flow module uses pressure control, the increased backpressure simulates proper flow. The actual flow rate will drop but the GC remains functional. See:

  “To Check for a Plugged FID Jet”

  “To Check for a Plugged NPD Jet”
- Check for a column fitting leak at the detector base.
- Replace the FID or NPD jet.

NPD Adjust Offset Process Fails

- Inspect the jet to see if it is clogged. (See To Check for a Plugged NPD Jet.)
- Measure the actual detector flows. (See To Measure a Detector Flow.) If the hydrogen or makeup flows are zero or much lower than the displayed flow, suspect a plugged jet.
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- If the process still fails, there could be a large leak in the system. This causes measured flow rates to be different from actual flow rates. Thoroughly leak check the whole system, especially the detector column fitting. (See **“Checking for Leaks”** .)
-

FPD+ Does Not Ignite

- Check that the FPD+ temperature is high enough for ignition (> 150 °C).
- Check FPD+ flow rates and that they match the type of filter installed in the FPD+. The hydrogen:air ratio greatly impacts ignition. Nonoptimal flow settings can prevent flame ignition.

- Measure the actual detector flows.
- Check that the FPD+ ignitor operates.
- During the ignition sequence, display the air flow rate. The air flow rate should go to 400 mL/min while trying to ignite to flame. If not, there is insufficient air supply pressure.
- Check the **Lit offset** value. The typical **Lit offset** value is 2.0. If it is zero, autoignition is turned off. If it is too large, the software will not recognize that the flame is lit and will shut the detector down.
- If the flame still will not light, there could be a large leak in the system. This results in measured flow rates being different from actual flow rates, causing non-ideal ignition conditions. Thoroughly leak check the whole system. (See **“Checking for Leaks”** .)
- Try increasing the supply pressures to the FPD+ flow module. This makes the flame easier to light without changing the setpoints.
- Check the **Lit offset** value. If it is zero, autoignition is turned off. If it is too large, the software will not recognize that the flame is lit and will shut the detector down.
- Under some operating conditions, the flame will light more easily with the vent tube removed. After lighting the flame, reinstall the vent tube.
- Check cable connections to coupling, coupling connection to glow plug, glow plug tightness.

Valve Not Ready

Troubleshooting depends on the type of valve.

Gas sampling valves

The GC is normally not ready whenever the inject time or load time has not elapsed. It becomes ready when the specified load or inject time has passed.

5 Shutdown Symptoms

Column Shutdowns 92

Hydrogen Shutdowns 93

Thermal Shutdowns 94

Column Shutdowns

The GC monitors inlet and auxiliary gas streams. If a carrier gas (which can include an auxiliary flow module) is unable to reach its flow or pressure setpoint, the GC assumes that a leak exists. It will warn you with a beep after 25 seconds, and it will continue to beep in intervals. After about 5 minutes, the GC will shut down components to create a safe state. The GC:

- Displays **Front inlet pressure shutdown**.
- Turns off to avoid column damage.
- Opens oven flaps in the back of the oven halfway.
- Turns off all flows for the column. When viewed, their parameters flash **Off**. For example, the column flows for a split/splitless inlet would turn off.
- Turns off all other heaters. When viewed, their temperature parameters flash **Off**.
- Attempts to turn on a shut-down zone fail with an error message.
- Turns off the TCD filament.
- Turns off the FID or FPD+ ignitor, and the air and fuel gas flows.
- Turns off the NPD bead, and air and fuel gas flows.

To recover from this state.

1. Fix the cause of the shutdown. Verify the carrier gas supply. The GC requires a delivered gas pressure of 70 kPa (10 psi) higher than the highest pressure used in the run.
   - Check for a broken column near the inlet.
   - Check for leaks.
   - Replace the inlet septum.
   - Replace the inlet O-ring.
   - Check the supply pressure.
2. Select **Diagnostics**. Select the alert. Select **Clear Shutdown - On** to clear the alert and turn on all zones. Select **Clear Shutdown - Off** to clear the alert and turn on all zones except the shutdown zone.

Hydrogen Shutdowns

Hydrogen gas may be used as a carrier or as fuel for some detectors. When mixed with air, hydrogen can form explosive mixtures.

Hydrogen used in inlets and auxiliary gas streams

The GC monitors inlet and auxiliary gas streams. If a stream is unable to reach its flow or pressure setpoint and if that stream is configured to use hydrogen, the GC assumes that a leak exists. It will warn you with a beep after 25 seconds, and it will continue to beep in intervals. After about 5 minutes, the GC will shut down components to create a safe state. The GC:

- Displays **Hydrogen Safety Shutdown**.
- Closes the carrier supply valve to the inlet and closes and turns off both pressure and flow controls.
- Opens the split vent valves in the split/splitless inlet.
- Turns off the oven heater and fan and opens the oven flaps.
- Turns off all heaters (including any devices connected to the auxiliary heater controls, such as valve box heaters and transfer line heaters).
- Turns off the TCD filament.
- Turns off the FID or FPD+ ignitor, and the air and fuel gas flows.
- Turns off the NPD bead, and air and fuel gas flows.
- Sounds an alarm.
-

WARNING

The GC cannot detect leaks in the detector gas streams. For this reason, it is vital that the column fittings of the FID, NPD, and any other detectors that use hydrogen always be connected to a column or have a cap or plug installed and that hydrogen streams be configured so that the GC is aware of them.

To recover from a hydrogen shutdown state:

1. Fix the cause of the shutdown:
   - Replace the inlet septum. See the Maintenance manual.
   - Replace the inlet O-ring. See the Maintenance manual.
   - Check for broken column.
   - Check the supply pressure.
   - Check the system for leaks. See **Leak Check Tips**.
2. Power cycle the GC.
3. Select **Diagnostics**. Select the alert. Select **Clear Shutdown - On** to clear the alert and turn on all zones. Select **Clear Shutdown - Off** to clear the alert and turn on all zones except the shutdown zone.

Thermal Shutdowns

A thermal fault means that the oven or another heated zone is not within its allowable temperature range (lower than minimum temperature or higher than maximum temperature). Several things could cause this error:

- A problem with the electrical supply to the instrument.
- A malfunction of the zone control electronics.
- A shorted temperature sensor.
- A shorted heater.

To recover from this state:

1. Fix the cause of the shutdown:
   - Check for missing insulation.
2. Most thermal shutdowns can be cleared by shutting off the thermal zone.

6 Electronics Troubleshooting GC Power On and Communication Symptoms

GC Does Not Turn On 96

PC Cannot Communicate with GC 97

GC Does Not Turn On

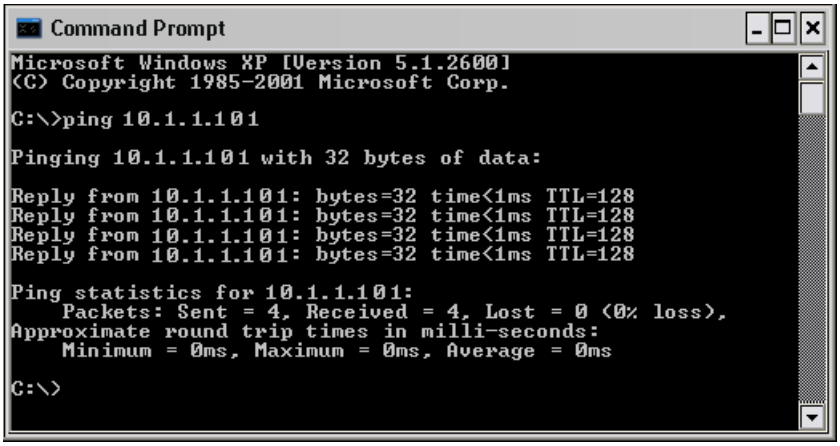
If the GC does not turn on:

- Check the power cord.
- Check the building's power.
- If the problem is at the GC, turn off the GC power. Wait 30 seconds, then turn the on the GC power.

PC Cannot Communicate with GC

- Run a **ping** test

  The MS-DOS **ping** command verifies communications across a TCP/IP connection. To use it, open the command prompt window. Type **ping** followed by an IP address. For example, if the IP address is 10.1.1.101, enter **ping 10.1.1.101**. If LAN communications are working properly, you will see a successful reply. For example:



If the ping test is successful, check the software configuration.

If the ping test is unsuccessful, do the following:

- Check the LAN cabling.
- Verify the IP address, subnet mask, and gateway addresses.
- Make sure all network devices (hubs, switches, and so forth) are turned on, properly connected, and working.
- Check for a defective LAN card in the PC.
- Check if some other software is connected to the GC. For example, only one software keypad can connect to a GC at a time. Agilent data systems also block connection by other data systems.

7 Checking for Leaks

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Leak Check Tips

When checking for leaks, consider the system in two parts: external leak points and GC leak points.

- **External leak points** include the gas cylinder (or gas purifier), regulator and its fittings, supply shutoff valves, and connections to the GC supply fittings.
- **GC leak points** include inlets, detectors, column connections, valve connections, and connections between flow modules and inlets/detectors.

WARNING

Hydrogen (H2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, a flow meter). Purge flowmeters with inert gas as needed. Always measure gases individually. Always turn off detectors to prevent flame/bead autoignition.

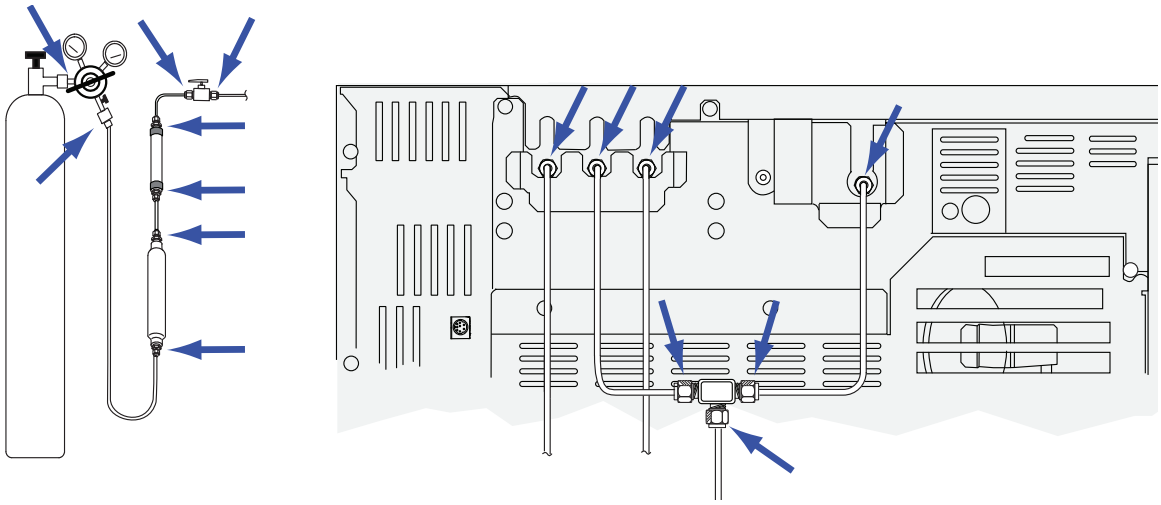
WARNING

Hazardous sample gases may be present.

1. Gather the following:
   - Electronic leak detector capable of detecting the gas type
   - 7/16-in, 9/16-in, and 1/4-in wrenches for tightening Swagelok and column fittings
2. Check any potential leak points associated with any maintenance recently performed.
3. Check GC fittings and connections that undergo thermal cycling, since thermal cycling tends to loosen some fitting types. Use the electronic leak detector to determine if a fitting is leaking.
   - Start by checking any newly made connections first.
   - Remember to check connections in the gas supply lines after changing traps or supply cylinders.

To Check for External Leaks

Check for leaks at these connections:



- Gas supply bulkhead fittings
- Gas cylinder fitting
- Regulator fittings
- Traps
- Shut-off valves
- T-fittings

Perform a pressure drop test.

1. Turn off the GC.
2. Set the regulator pressure to 415 kPa (60 psi).
3. Fully turn the regulator pressure adjustment knob counterclockwise to shut the valve.
4. Wait 10 min. If there is a pressure loss greater than 7 kPa (1 psi), there is a leak in the external connections.

To Check for GC Leaks

Check for leaks at these connections:

- Inlet septum, septum head, liner, split vent trap, split vent trap line, and purge vent fittings
- Column connections to inlets, detectors, valves, splitters, and unions
- Fittings from the flow modules to the inlets, detectors, and valves
- Column adapters
-

First, use the GC's built-in leak test to check for leaks at the inlet column fitting, septum, liner, split vent trap line, and so forth. See **"To Perform an Inlet Leak Check"**. Correct any leaks found using this test. If the GC still shows symptoms of a leak, check the other possible leak points.

If using Agilent Instrument Utilities, you can also use it to remotely run an inlet leak check (for selected inlet types).

If the inlet passes the inlet leak check but you still suspect a leak in the inlet, you can use the Instrument Utilities software to perform a pressure decay test for most inlets. (You can also perform a manual pressure decay test on any inlet.) Any inlet that passes the inlet pressure decay test can be considered leak free.

- **To Perform a Pressure Decay Leak Test**

Use an electronic leak detector to check column connections and tubing connections. See also:

- **To Manually Check for Leaks in a Split/Splitless Inlet**
- **Leaks in Capillary Flow Fittings**
-

Leaks in Capillary Flow Fittings

For capillary flow fittings, a leak usually indicates that the fitting has been overtightened. Unless the fitting is obviously loose, do not tighten it further. Instead, remove the connection, trim the column end, and install it again.

Also inspect the plate and connection for a broken column tip.

To Perform an Inlet Leak Check

The GC provides a real-time, built-in leak check for all inlets. This check is most useful for finding inlet leaks during and after inlet maintenance. While not as thorough or sensitive as a complete inlet pressure test, it is normally performed with the column installed and configured, and provides a quick assurance that the inlet is reasonably leak-free. Agilent recommends running the check before and during inlet maintenance, so that as you tighten any fittings you can watch the inlet stop leaking. The test is appropriate for all applications, although some may require more robust leak testing.

The inlet leak check finds leaks at the:

- Inlet column fitting
- Gold seal (as applicable)
- Split vent trap housing (as applicable)
- Septum nut and septum (as applicable)
- Insert weldment nut/septum head assembly (as applicable)

To run the test:

1 Select **Diagnostics** > **Diagnostic Tests** > **Inlets** , select the desired inlet, and select **Leak & Restriction Test**.

2 Select **Start Test**.

NOTE

If you ran the test on a leak-free inlet before performing maintenance, the leak test result after maintenance should be about the same as it was before maintenance.

3 If the test still fails:

4 Replace
   - the septum
   - Reinstall the column into the inlet
   - Replace the liner and liner O-ring
   - Open the split vent trap and check the O-ring seating. Replace the split vent trap if needed.

If the test passes, but you still suspect an inlet leak, perform a pressure decay leak test. See:

- **To Perform a Pressure Decay Leak Test**
- **When Performing a Pressure Decay Leak Test for a Purged/Packed Inlet**

To Perform a Pressure Decay Leak Test

Use the Inlet Leak & Restriction Test to determine if the inlet is leaking. Open the software, select the GC, and run the test for the inlet. See **"To Perform an Inlet Leak Check"** and **"To Run the Split Vent Restriction Test"** .

If the test fails:

- Check the plugged column fitting and septum purge cap.
- Check the septum head. Tighten if needed.
- Replace the septum if necessary.
- Inspect and re-install the liner if it is leaking.
- Tighten the split vent trap if needed, or install a new cartridge and O-rings.
- Tighten the split vent line connection to the inlet.

To perform an inlet pressure decay test from the browser UI

The pressure decay test checks for leaks from the inlet flow module up to the column fitting.

After performing maintenance, first check for leaks in externally accessible areas. See **"To Check for External Leaks"**.

If a leak is known to exist, check the externally accessible inlet fittings first, especially any connection that has seen recent maintenance, such as the septum nut, column adapter, column connection, and so forth.

This test can/cannot find the following types of leaks:

The test can find leaks at the:	The test cannot find leaks at the:
septum	column fitting
septum nut	gas supply bulkhead fittings to the flow module
liner O-ring seal	tubing and connections in a transfer line connected to the inlet
gold seal/washer and reducing nut	internal leaks in an EPC or EPR module
inlet body	
flow manifold split vent valve	
split vent tubing and trap	
septum purge tubing	
seals within the tubing between the inlet flow module and the inlet body	

The below materials will be required to perform this test:

- No-hole ferrule
- 1/4-inch wrench
- Heat-resistant gloves (if the inlet is hot)
- Column nut
- New septum
- O-ring
- ECD/TCD Detector plug (part no. 5060-9055)

5 Select **Diagnostics** > **Diagnostic Tests** > **Inlets**.

6 Select **Pressure Decay Test**.

7 Select **Start Test**.

8 Follow the instructions presented on the Browser UI.

9 After the inlet passes the test, restore the GC to operating condition.

- Restore source pressures.
- Remove any caps/plugs.
- Reinstall the column.
- Restore the correct column configuration.
- Load the operating method.

To Manually Check for Leaks in a Split/Splitless Inlet

This procedure describes how to check for and fix leaks in the split/splitless inlet. Follow the procedures below based on the inlet's symptoms.

Cannot Reach Adjusted Value or Pressure Setpoint

If the split/splitless inlet does not reach its adjusted value or pressure setpoint, the GC will be "Not Ready." (EPR equipped inlets will indicate a lack or readiness via a flashing asterisk.) The status indicator above the touch screen and status tray on the touch screen will be orange. The GC will shut down in approximately 5.5 minutes if the inlet cannot pressurize and control.

If you have recently performed maintenance, first check for leaks on any fittings/parts you handled.

1. Verify sufficient gas supply pressure delivered to the GC and verify no leaks in the gas supply (see **To Check for External Leaks**). The inlet requires 70 kPa (10 psi) above the highest pressure used in the method.
   - 120 psi maximum for 0–100 psi inlet
   - 170 psi maximum for 0–150 psi inlet
2. Check the total flow setting. Total flow must be high enough to maintain inlet pressure throughout the run. Wide bore columns require higher flow rates. Typically 50 mL/min is sufficient. To increase total flow:
   - Increase split ratio if in split mode
   - Increase the purge flow for splitless mode
3. If the pressure remains low, continue with the next step.
4. Perform the inlet run leak check. See **"To Perform an Inlet Leak Check"**. The total flow provides an indication of the magnitude of the leak. Monitor the total flow while checking/tightening fittings:
   - Septum nut
   - Column
   - Split vent trap/O-rings
   - Liner/O-rings
   - Gold seal
   - Split vent line connection to the inlet body
   - Flow block fittings on the flow manifold

   Alternately, use an electronic leak detector to check these fittings/connections.

   If these checks do not resolve the problem, contact Agilent for service.

WARNING

Be careful! The oven, inlet, and/or detector may be hot enough to cause burns. If the oven, inlet, or detector is hot, wear heat-resistant gloves to protect your hands.

Low Sensitivity or Poor Repeatability

A small leak in the split/splitless inlet can cause low sensitivity or poor repeatability. Check for and isolate a small leak as described below.

If you have recently performed maintenance, first check for leaks on any fittings/parts you handled.

1. Perform the inlet pressure decay test. See **"To Perform a Pressure Decay Leak Test"**. If the test passes, consider the inlet leak-free and check other possible causes for the low sensitivity or poor repeatability.
2. If the pressure decay test fails, perform the inlet leak check. See **"To Perform an Inlet Leak Check"**. Monitor the total flow while checking/tightening fittings:
   - Septum nut
   - Column
   - Split vent trap
   - Liner/O-ring
   - Gold seal
   - Split vent line connection to the inlet body
   - Flow block fittings on the flow manifold
3. If the inlet (prep run) leak check did not resolve the problem, the leak may be too small to detect using that test. Use an electronic leak detector to check the fittings/connections.

If these checks do not resolve the problem, contact Agilent for service.

To Correct Leaks in the Split Splitless Inlet

If the inlet fails a pressure decay test, check the following:

- Check the caps/plugs used in the test—make sure each is correctly installed and tight.
- If you performed the leak test after performing maintenance, check for proper installation of the part(s) handled during the maintenance.
- Check the tightness of the septum nut.
- Check the septum. Replace if old or damaged.
- Check the insert assembly installation.
- Check the liner and liner O-ring.
- If you changed the gold seal, verify correct installation.
- Make sure the inlet temperature remained constant during the test.

When Performing a Pressure Decay Leak Test for a Purged/Packed Inlet

Use the Inlet Pressure Decay Test to determine if a purged/packed inlet is leaking.

If the test fails:

- Tighten the septum nut/Merlin cap.
- Replace the septum or Merlin Microseal.
- Tighten the top insert weldment. Replace the liner.
- Replace the O-ring and tighten the column adapter connection. Re-install if needed.
- Check the plugged column fitting and the septum purge cap.

To Correct Leaks in the Purged Packed Inlet

1 If the inlet fails a leak test, check the following:

- Check the caps/plugs used in the test—make sure each is correctly installed and tight.
- If you performed the leak test after performing maintenance, check for proper installation of the part(s) handled during the maintenance.
- Check the tightness of the septum nut.
- Check the septum. Replace if old or damaged.
- Check that the top insert weldment is installed tightly.
- Replace the O-ring.
- Also check the glass insert.
- Replace the ferrule seal on the adapter.
- Make sure the inlet temperature remained constant during the test

2 If these items do not resolve the problem, contact Agilent for service.

To Manually Check for Leaks in a Packed Column Inlet

This method checks for leaks from the inlet flow module up to the column fitting.

After performing maintenance, first check for leaks in externally accessible areas. See **"To Check for External Leaks"**.

If a leak is known to exist, check the externally accessible inlet fittings first, especially any connection that has seen recent maintenance, such as the septum nut, column adapter, column connection, and so on.

The leak test described below requires removing the column and capping the inlet column fitting. This test can/cannot find the following types of leaks:

The test can find leaks at the:	The test cannot find leaks at the:
septum	column fitting
septum nut	
adapter and ferrule	
inlet body	
top insert weldment	
seals within the tubing between the inlet flow module and the inlet body	

WARNING

This leak test is not appropriate for flammable carrier gases, such as hydrogen.

1 Gather the following:
   - 9/16-inch wrench
   - 7/16-inch wrench
   - Heat-resistant gloves (if the inlet is hot)
   - 1/8- and 1/4-inch Swagelok caps
2 Set the inlet and oven temperature to 35 °C.
3 Remove the column, if installed.
4 Remove the old septum and replace it with a new one.
5 Inspect the O-ring and replace it if it is hard and brittle or cracked.
6 If unsure of the quality of the adapter ferrule, replace it.
7 Use the 2/+ and 8/- keys on the software keypad to adjust the column flow to 5 mL/min.
8 Plug the column fitting.

- If a 1/8-inch packed column adapter is installed, use a 1/8-inch Swagelok cap (5180-4121).
- If a 1/4-inch packed column adapter is installed, use a 1/4-inch Swagelok cap (5180-4120)

9 Since there is no way for gas to escape when the column fitting is capped, the displayed column flow should drop to 0.0 mL/min or < 0.1 mL/min.

10 Double click Status to see the actual flow of the inlet.

11 If the displayed column flow is greater than 0.1 mL/min, or if it reaches the adjusted value, there is a leak. Otherwise the system has no leak.

12 After the inlet passes the test, restore the GC to operating condition.

13 Remove any caps/plugs.

14 Reinstall the column.

15 Restore the correct column configuration.

16 Load the operating method.

To Correct Leaks in the Packed Column Inlet

If the inlet fails a leak test, check the following:

- Check the caps/plugs used in the test—make sure each is correctly installed and tight.
- If you performed the leak test after performing maintenance, check for proper installation of the part(s) handled during the maintenance.
- Check the tightness of the septum nut.
- Check the septum. Replace if old or damaged.
- Check that the top insert weldment is installed tightly.
- Replace the O-ring.
- Also check the glass insert.
- Replace the ferrule seal on the adapter.
- Make sure the inlet temperature remained constant during the test.

If these items do not resolve the problem, contact Agilent for service.

To Correct Leaks in the Cool On-Column Inlet

If the inlet fails a pressure decay test, check the following:

- Check the caps/plugs used in the test—make sure each is correctly installed and tight.
- If you performed the leak test after performing maintenance, check for proper installation of the part(s) handled during the maintenance.
- Check the tightness of the septum nut or cooling tower assembly.
- Check the septum. Replace if old or damaged.
- Make sure the inlet temperature remained constant during the test.

If these items do not resolve the problem, contact Agilent for service.

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To Measure a Column Flow

Measuring FID, TCD, ECD, and FPD+ column flow

The following procedure is used to measure column flow with an FID, TCD, ECD, or FPD+.

WARNING

Hydrogen (H2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, a flow meter). Purge flowmeters with inert gas as needed. Always measure gases individually. Always turn off detectors to prevent flame/bead autoignition.

WARNING

Be careful! The detector may be hot enough to cause burns. If the detector is hot, wear heat-resistant gloves to protect your hands.

1 Gather the following:
   - Appropriate flowmeter adapter tube (included in the GC ship kit)
   - Electronic flowmeter calibrated for the gas and flow rates of concern
2 Turn off the detector.
3 Turn off the detector flows.
4 Connect the appropriate adapter to the detector exhaust.

NOTE

Flowmeter tube diameters vary by model; modify the adapter to the flowmeter tubing as needed.

A 1/8-in rubber adapter tube attaches directly to a ECD or TCD exhaust vent.



A separate adapter (19301-60660) is supplied for the FID. Insert the adapter into the detector exhaust vent as far as possible. You will feel resistance as the adapter O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure a good seal.



For the FPD+, remove the plastic tubing from the FPD+ exhaust and connect the flowmeter directly to the FPD+ vent tube. If necessary, use a 1/4-inch tube adapter between the detector exhaust and the flowmeter tubing.



5 Connect the flowmeter to the flowmeter adapter to measure flow rates.

Measuring NPD column flow

1 Gather the following:
   - NPD flowmeter adapter tool (G1534-60640)



   - Flow-measuring insert (19301-60660)
   - Electronic flowmeter calibrated for the gas and flow rates of concern

2 Turn off the bead.

3 Cool the NPD to 100 °C.

WARNING

Be careful! The detector may be hot enough to cause burns. If the detector is hot, wear heat-resistant gloves to protect your hands.

4 Remove the bead and store it carefully until reinstallation.

5 Insert the NPD flowmeter adapter tool into the NPD collector.

6 Attach the flow-measuring insert to the NPD flowmeter adapter tool.



7 Place the flowmeter tubing over the flow-measuring insert to begin measuring flows.

To Measure a Split Vent or Septum Purge Flow

Note that the GC reports flows calibrated at 25 °C and 1 atmosphere. Correct flowmeter results accordingly.

WARNING

Hydrogen (H2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, a flow meter). Purge flowmeters with inert gas as needed. Always measure gases individually. Always turn off detectors to prevent flame/bead autoignition.

Septum purge and split vent flows exit through the pneumatic module at the top rear of the GC. See the figure below.

To measure split vent or septum purge flows, attach the flowmeter to the appropriate tube. Remove the GC pneumatics cover to access the back inlet exhausts.

- The split vent has a 1/8-in Swagelok threaded fitting. The vents use a 1/8-in Swagelok threaded fitting. Create and use a 1/8-in tube adapter (as shown below) to convert the 1/8-in threaded fitting into a 1/8-in tube. This prevents the rubber flowmeter tubing from leaking around the threads, which would cause an incorrect flow reading.



- The septum purge is a 1/8-in tube. Use the red rubber adapter shown to measure flows.

To Measure a Detector Flow

Detectors, especially detectors with flame, require precise flow measurements to function properly. Incorrect flows are caused by:

- Restrictions in the supply line, which will cause a **Not Ready** message on the GC display (all detectors)
- A leaking column or column adapter fitting (all detectors)
- A plugged jet (FID, NPD).
- A leak in the burner chamber, window seal, or ignitor seal (FPD+)
- A pressure sensor that needs to be zeroed.
- An EPC or EPR valve that is not operating correctly.

To isolate the problem, compare the flow of **one channel of gas** against the actual flow rate.

Measuring FID, TCD, ECD, and FPD+ flows

WARNING

Hydrogen (H2) is flammable and is an explosion hazard when mixed with air in an enclosed space (for example, a flow meter). Purge flowmeters with inert gas as needed. Always measure gases individually. Always turn off detectors to prevent flame/bead autoignition.

1 Gather the following:
   - Appropriate flowmeter adapter tube (included in the GC ship kit)
   - Electronic flowmeter calibrated for the gas and flow rates of concern

CAUTION

To avoid damaging the column, cool the oven before turning off the column flow.

2 Set the oven temperature to ambient (35 °C).

3 Turn off the column flow and pressure.

4 Shut off all detector gases.

5 Turn off the FID flame, FPD+ flame, or TCD filament (as applicable).

6 Cool the detector.

7 Connect the appropriate adapter to the detector exhaust.

NOTE

Flowmeter tube diameters vary by model; modify the adapter to the flowmeter tubing as needed.

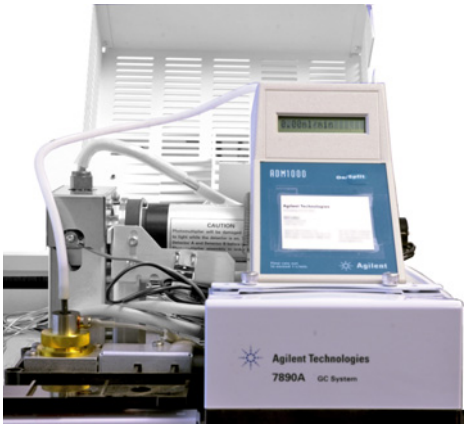
A rubber adapter tube attaches directly to a ECD, or TCD exhaust vent.



A separate adapter (19301-60660) is supplied for the FID. Insert the adapter into the detector exhaust vent as far as possible. You will feel resistance as the adapter O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure a good seal.



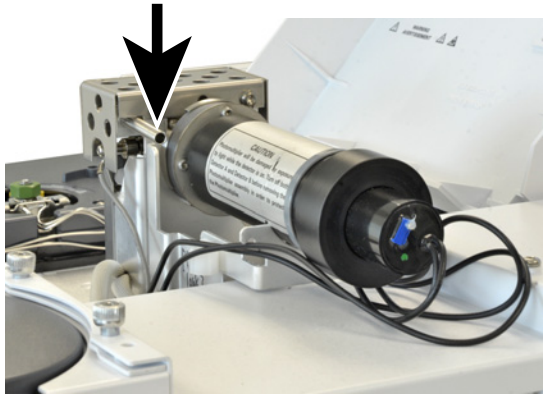
For the FPD+, remove the plastic tubing from the FPD+ exhaust and connect the flowmeter directly to the FPD+ vent tube. If necessary, use a 1/4-inch tube adapter between the detector exhaust and the flowmeter tubing.











8 Connect the flowmeter to the flowmeter adapter.

9 Measure the actual flow rate of each gas one at a time.

Measuring NPD flows

1 Gather the following:

- NPD flowmeter adapter tool (G1534-60640)



- Flow-measuring insert (19301-60660)
- Electronic flowmeter calibrated for the gas and flow rates of concern

2 Turn off the bead.

3 Cool the NPD to 100 °C.

WARNING

Be careful! The detector may be hot enough to cause burns. If the detector is hot, wear heat-resistant gloves to protect your hands.

4 Remove the bead and store it carefully until reinstallation.

5 Insert the NPD flowmeter adapter tool into the NPD collector.

6 Attach the flow-measuring insert to the NPD flowmeter adapter tool.



7 Place the flowmeter tubing over the flow-measuring insert to begin measuring flows.

To Perform the GC Self-Test

1. Turn the GC off.
2. Wait one minute, then turn the GC back on. If the main GC status screen appears, the GC has passed the self-test.

To Check or Monitor Split Vent Line Backpressure

Agilent provides a built-in test that measures backpressure in the split vent trap and line for the split/splitless inlet. The test measures the pressure developed in the split vent flow path at a given, user-selected flow rate. This flow rate can be your method **Split flow** setpoint, or the default 400 mL/min used by Agilent to compare "typical" values.

By running the test on a clean system, you can establish a baseline for expected backpressure in the split vent line. Then, you can re-run the test periodically to determine whether or not the trap needs to be replaced before it impacts your chromatography.

The pressure measured by the test depends on:

- The liner installed
- The flow rate used

Therefore, the actual measured value will vary between different setups and from GC to GC.

The test checks for:

- Liner restriction
- Contamination on the gold seal
- Restrictions in the split vent line, such as condensed sample contamination in the split vent line and trap

The test can also provide a measure of suitability for the installed hardware. Run the test using your method's setpoints and hardware. If the measured test pressure is close to the desired column head pressure, this means that even a minor amount of restriction in the split vent line can cause the GC to become not ready. You may wish to install a different liner or adjust the method. (For splitless liners, first try reinstalling the liner. Splitless liners create more backpressure than split liners, and so minor variations in orientation can make a difference at low head pressures.)

To Run the Split Vent Restriction Test

The following materials may be required to perform this test:

- No-hole ferrule (5020-8294) and column nut (5181-8130), or capillary column blanking nut (5020-8294)
- 1/4-inch wrench

Heat-resistant gloves (if the inlet is hot)

From the browser UI:

1. Select **Diagnostics** > **Diagnostic Tests** > **Inlets**.
2. Select **Split Vent Restriction Test**.
3. Follow the instructions that appear on the Browser UI.

To Adjust the FID Lit Offset

To adjust the FID **Lit offset**:

1. Select **Settings > Configuration > Detector**.
2. Tap the **Lit offset** value to show a keypad. Use the keypad to input the desired value.
3. Lit offset should be < 2.0 pA or lower than the normal FID output when lit.
4. Select **Apply** to save your changes.

To Verify That the FID Flame Is Lit

To verify that the FID flame is lit, hold a mirror or other reflective surface over the collector exhaust. Steady condensation indicates that the flame is lit.

Typically the FID output will be between 5.0 and 20.0 pA when lit and < 2.0 pA when not lit.

If the flame does not light, do the following:

- Verify that the detector temperature is above 150 °C. Agilent recommends operating the FID ≥300 °C.
- Verify correct detector flows.
- Inspect the jet for contamination.
- Verify that the jet is installed correctly.
- Check the column connections for leaks.

To Verify FID Ignitor Function During Ignition Sequence

WARNING **Keep body parts at a safe distance from the FID chimney while performing this task. If using hydrogen, the FID flame will not be visible.**

1. Remove the detector top cover.
2. Turn the FID flame **On.**
3. Observe the ignitor plug though the FID chimney. The small hole should glow during ignition sequence.

To Measure FID Leakage Current

1. Load the analytical method.
   - Make sure flows are acceptable for ignition.
   - Heat the detector to operating temperature or to 300 °C.
2. Turn off the FID flame.
3. Verify that the FID electrometer is on.
4. Select **Method** > **Detectors**, then scroll until **Detector Specifics** is visible.
5. Verify that the output is stable and < 1.0 pA.

   If the output is unstable or > 1.0 pA, turn off the GC and check for proper assembly of the upper FID parts and for contamination. If the contamination is confined to the detector, bakeout the FID.
6. Turn on the flame.

To Measure FID Baseline Output

1. With the column installed, load your checkout method.
2. Set the oven temperature to 35 °C.
3. Select **Method** > **Detectors**, then scroll until **Detector Specifics** is visible.
4. When the flame is lit and the GC is ready, verify that the output is stable and < 20 pA (this may take some time).
5. If the output is not stable or > 20 pA, the system or gas may be contaminated. If this contamination is isolated to the detector, bakeout the FID.

To Isolate the Cause of FID Noise

FID noise is the result of mechanical, electrical and chemical factors. FID noise can be a subjective parameter. Often FID baseline noise is perceived based on history of a given detector or comparison with another detector in the lab. For proper diagnosis of noise it is important to evaluate the detector noise under documented conditions against a known standard. Find more detailed information about noise in **“Noisy Detector, Including Wander, Drift, and Baseline Spikes”** .

Before troubleshooting the detector, perform a noise test using your Agilent data system. If the detector fails the noise test, then troubleshoot the cause as described below.

To isolate the cause of FID noise:

1. If the noise test fails, remove the column and re-evaluate detector noise with the FID capped and re-ignited, using only H2/air and makeup detector gases. If it passes, suspect contaminated column/carrier gas.
2. If the noise failed with no column installed, repeat the noise test with only H2 and air - set makeup flow to "Off". If it passes, suspect contaminated makeup gas.
3. If the leakage test fails, replace or clean the collector and PTFE insulators, the interconnect with spring, and/or the entire FID electrometer assembly.
4. If the leakage current test is OK, suspect a contaminated jet or contaminated H2 or Air detector gas supplies (gases, tubing, traps), especially if the background of the detector when lit is >20 pA.

To Measure NPD Leakage Current

1. Load the analytical method.
2. Set the **NPD Adjust Offset** to **Off** and turn off the bead.
   - Leave the NPD at operating temperature.
   - Leave flows on or off.
3. Select **Method** > **Detectors**, then scroll until **Detector Specifics** is visible.
4. Verify that the output (leakage current) is stable and < 1.0 pA.
5. The output should slowly drop toward 0.0 pA, and should stabilize in the tenths of a picoamp. Current > 2.0pA indicates a problem.

To Check for a Plugged FID Jet

The most common cause of FID ignition problems is a plugged or partially plugged jet. If the jet is not completely plugged and the flame still ignites, a secondary symptom will be lengthening peak retention times. Jet plugging is most common with thick-film/high bleed or packed columns and high temperature applications. It is best to operate the column oven within the temperature limits of the column and also to operate the FID at least 20 °C hotter than the maximum oven temperature in the GC method. If the FID jet is becoming plugged, actual H2, Makeup and Capillary carrier flows will be lower than the values indicated by the GC.

To check for a plugged FID jet:

1. Leave the column installed in the FID. If already removed, cap off the detector column fitting in the oven. (Leaving the column installed will determine if the column is installed too high in the jet, plugging the orifice.)
2. Set the makeup flow to “Off”. Confirm a reading on the GC display of 0.0 mL/min for actual makeup flow.
3. Set the hydrogen flow to 75 mL/minute (increase the H2 supply pressure as needed to achieve this flow rate setting.)
4. Monitor the makeup flow “Actual” reading

   If the makeup flow is indicating a value in excess of 1.0 mL/min, this indicates that the jet is plugged or partially plugged; pressure is backing up from the H2 channel into the makeup channel of the EPC or EPR module, resulting in a false flow indication on the makeup channel.

Alternately, remove the jet from the housing and hold it up to a light source. Check the holes in the jet for contamination. If plugged, replace the jet.

To Check for a Plugged NPD Jet

The detector EPC module controls flow by maintaining a calibrated gas pressure against a fixed restriction. A plugged jet will cause inaccurate flow readings.

To check for a plugged NPD jet, measure the actual hydrogen and makeup flows. (See **Measuring NPD flows**.) If the flows are lower than the displayed values, replace the jet.

To Verify That the NPD Bead Is Ignited

WARNING

Hot exhaust! Detector exhaust is hot and can cause burns.

To verify that the bead is ignited, look through the vent hole on the detector lid to see if the bead is glowing orange.

The NPD output is selected by the operator as part of the adjust offset process and generally is between 5.0 and 50.0 pA.

To Verify That the FPD+ Flame Is Lit

To verify that the FPD+ flame is lit:

1. Remove the rubber drip tube from the detector vent.
2. Hold a mirror or shiny surface near the aluminum exhaust tube. Steady condensation means that the flame is lit.

To Adjust the FPD+ Lit Offset

To adjust the FPD+ **Lit offset**:

1. Select **Settings > Configuration > Detector**.
2. Tap the **Lit offset** value to show a keypad. Use the keypad to input the desired value.
3. Lit offset should be < 2.0 pA or lower than the normal FPD+ output when lit.
4. Select **Apply** to save your changes.

When to Change Gas Purifiers

Agilent highly recommends using purifying traps in the gas lines to prevent impurities from entering and contaminating the GC system or damaging the column. Some traps are single-purpose for the removal of oxygen, moisture, or hydrocarbons, while combination traps remove all those contaminants.

The best way to know when it is time to change a trap is to use an indicating trap, which should be placed after a high capacity trap. Agilent recommends using glass indicating traps such the Gas Clean Filter system, whose clear tubes display a distinct color change in response to contamination. This color change tells the analyst it is time to change traps.

If no indicating traps are used, it is best to follow the manufacturer's recommendation for replacement frequency. Typically, the manufacturer will state how many gas cylinders can be purified with a given trap. If desired, it is possible to estimate when to replace the trap by performing a rough calculation. For example: you have a standard "K" size cylinder with He at 99.995% purity that contains 7,800 L of He. Assuming a worst case scenario in which the remaining 0.005% is all oxygen, you should have 39 mL, or about 56 mg of O2 in the tank. Agilent's OT3 oxygen trap, for example, has a capacity of 600 mg O2. Therefore, you need to replace the OT3 trap every 10 cylinders. This is just a rough estimate, and it is prudent to change traps too early rather than too late.

To Ignore a Device's Readiness State

By default, the GC monitors the status of all configured devices (inlets, detectors, valve box heaters, valves, oven heater, EPC or EPR modules, and so forth) and becomes ready when all of them reach setpoint. If the GC senses a problem in one of these devices, the GC will either never become ready, or may go into a shutdown state to protect itself or prevent a safety hazard. However, there may be a time when you do not wish to have the readiness state of a device prevent a run from starting. An important example is when an inlet or detector heater is defective. Normally, this fault prevents the GC from becoming ready and starting a run. However, you can set the GC to ignore this problem so that you can use the other inlet or detector until the device is repaired.

Not all devices can be ignored. You can ignore the readiness state of inlets, detectors, EPC or EPR modules, or the oven. The readiness state of other devices and components can never be ignored, for example, injection devices such as a switching valve or automatic liquid sampler.

To ignore the status of a device:

1. Turn off the device's heater and gas flows as applicable. (Make sure that it cannot create a safety hazard.)
2. Select **Settings > Congifuration**, then select the element.
3. Deselect the **Enable** checkbox in the **Readiness** field.

You can now use the GC until the device is repaired.

CAUTION

Do not ignore the readiness state of a device that is being used unless you do not care if it reaches setpoint.

Be sure to return a broken device to Ignore Ready = False after it is repaired. Otherwise, its state (temperature, flow, pressure, and so forth) will continue to be ignored, even if using this device in the analysis.

To consider a device's readiness, set **Ignore Ready** to **False**.