

Agilent 6890N Gas Chromatograph



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Contents

Concepts and General Tasks Concepts How to troubleshoot using this manual 8 The [Status] key Configurable Items to Always Keep Current Column configuration **Automatic Liquid Sampler configuration** Gas configuration 10 Information to Obtain Before Calling Agilent for Service **Hardware Symptoms** 2 **Plunger Errors** 12 Vial Mishandled by ALS Syringe Needle Bends During Injection into Inlet 14 FID Does Not Ignite 15 FID Ignitor Does Not Glow During Ignition Sequence 16 Corrosion in FID Collector and Ignitor Glow Plug **FPD Does Not Ignite** NPD Adjust Offset Process Fails 19 3 **Chromatographic Symptoms** 22 **Retention Times Not Repeatable** Peak Areas Not Repeatable 23 24 **Contamination or Carryover** Isolate the source Check possible causes—all inlet and detector combinations 24 Larger Peaks Than Expected 25 Peaks Not Displayed/No Peaks 26 Table 1. Detector troubleshooting 26

Troubleshooting 3

Baseline Rise During Oven Temperature Program

27

Poor Peak Resolution 28		
Peak Tailing 29 NPD 29		
Peak Boiling Point or Molecular Weight Discrimination Poor 30 For any inlet operating in split mode with any detector 30 For any inlet operating in splitless mode with any detector 30		
Sample Decomposition in Inlet/Missing Peaks 31		
Peak Fronting 32		
Noisy Detector, Including Wander, Drift, and Baseline Spikes 33 Noisy baseline 33 Baseline wander and drift 34 Baseline spiking 34 Figure 1. Cyclic spiking 35 Figure 2. Random spiking 35		
Check for the correct jet. Low Peak Area or Height 36		
FID Flame Goes Out During a Run and Attempts to Reignite 38		
FID Baseline Output Above 20 pA 39		
FPD Flame Goes Out During a Run and Attempts to Reignite 40		
FPD Output Too High or Too Low 41		
FPD Low Peak Areas 42		
FPD Large Peak Width at Half-Height 43		
FPD Baseline Output High, > 20 pA 44		
NPD Solvent Quenching 45		
NPD Response Low 46		
NPD Baseline Output > 8 million 47		
NPD Adjust Offset Process Not Functioning Properly 48		
NPD Low Selectivity 49		
Negative Peaks Seen with TCD 50		
TCD Baseline Has Dampened Sinusoidal Noise Trailing Peaks (Ringing Baseline) 51		
TCD Peaks Have Negative Dip on Tail 52		
GC Not Ready Symptoms		
GC Never Becomes Ready 54		

Flow Never Becomes Ready 55

Troubleshooting 4

Oven Temperature Never Cools Down/Cools Very Slowly 56		
Oven Never Heats Up 57		
Temperature Never Becomes Ready 58		
Cannot Set a Flow or Pressure 59		
A Gas Does Not Reach Setpoint Pressure or Flow 60		
A Gas Exceeds Pressure Setpoint or Flow 61		
The Inlet Pressure or Flow Fluctuates 62		
Cannot Maintain a Pressure as Low as the Setpoint on a Split Inlet 63		
The Measured Column Flow Does Not Equal the Displayed Flow 64		
FID Does Not Ignite 65		
FID Ignitor Does Not Glow During Injection Sequence 66		
NPD Adjust Offset Process Fails 67		
FPD Does Not Ignite 68		
Shutdown Symptoms		
Column Shutdown 70		
Hydrogen Shutdown 71		
Thermal Shutdowns 72		
Ionfunctional GC Symptoms		
GC Does Not Turn On 74		
PC Cannot Communicate with GC 75		
Checking for Leaks		
Leak Check Tips 78		
To Check for External Leaks 79		
To Check for GC Leaks 81		
Leaks in Capillary Flow (Microfluidic) Fittings 82		
roubleshooting Tasks		
To Measure a Column Flow 84 Measuring FID, TCD, uECD, and FPD column flow 84 Measuring NPD column flow 85		
To Measure a Split Vent or Septum Purge Flow 87		
To Measure a Detector Flow 89 Measuring FID, TCD, uECD, and FPD flows 89		

Measuring NPD flows 90

To Perform the GC Self-Test 92

To Adjust the FID Lit Offset 93

To Verify That the FID Flame Is Lit 94

To Verify FID Ignitor Function During Ignition Sequence 95

To Measure FID Leakage Current 96

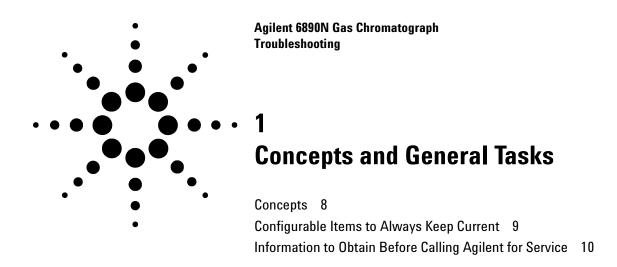
To Measure FID Baseline Output 97

To Measure NPD Leakage Current 98

To Verify That the NPD Bead Is Ignited 99

To Verify That the FPD Flame Is Lit 100

To Adjust the FPD Lit Offset 101



1 Concepts and General Tasks

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.

The [Status] key

Be sure to also use the **[Status]** and **[Info]** keys on the GC keypad while using this troubleshooting information. These keys will display additional useful information related to the status of the GC and its components.

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.

Column configuration

Reconfigure the GC 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 injector position, 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.

Information to Obtain Before Calling Agilent for Service

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 on the GC display
- · Results of any troubleshooting tests you have run
- Press the [Status] key to display previous Error, Not Ready, and Shutdown messages.

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

Agilent 6890N Gas Chromatograph Troubleshooting Agilent 6890N Gas Chromatograph Troubleshooting Hardware Symptoms Plunger Errors 12

Plunger Errors 12
Vial Mishandled by ALS 13
Syringe Needle Bends During Injection into Inlet 14
FID Does Not Ignite 15
FPD Does Not Ignite 18
NPD Adjust Offset Process Fails 19

2 Hardware Symptoms

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.

Vial Mishandled by ALS

Refer to the 7683B Automatic Liquid Sampler Installation, Operation and Maintenance manual for additional information.

When you find a mishandled sample vial, do the following:

- Check for folds or wrinkles in the crimp cap, especially near the neck of the sample vial.
- Use Agilent recommended sample vials.
- Check sample labels (if applicable).
 - Check that they are the correct size.
 - Verify that the labels do not interfere with the gripper.
- Check that the tray quadrants are clean and snapped into the tray base.

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 the 7683B Automatic Liquid Sampler Installation, Operation and Maintenance manual 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.
- 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.

FID Does Not Ignite

- Check that the FID ignitor glows during ignition sequence. (See To Verify FID Ignitor Function During Ignition Sequence.)
- Verify that the Lit Offset is ≤ 2.0 pA.
- Check for a plugged or partially plugged jet.
- Check the FID flow rates. 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.
- · Check the column flow rate.
- · Check for leaks at the FID column fitting.
- Ensure that the FID temperature is high enough for ignition (>150 $^{\circ}$ C).

FID Ignitor Does Not Glow During Ignition Sequence

WARNING

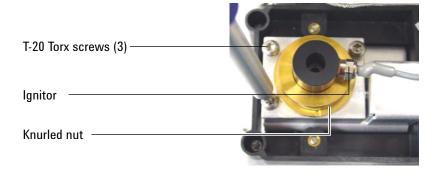
Keep body parts at a safe distance from the FID chimney while performing this task. If using hydrogen, the FID flame will not be visible.

- 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 \geq 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 100 $^{\circ}\mathrm{C}.$

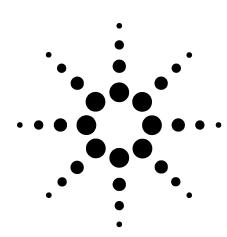
FPD Does Not Ignite

- 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.
- Measure the actual detector flows. (See To Measure a Detector Flow.)
- The column may be installed too high into the detector.
- Check that the FPD ignitor operates. (See To Verify That the FPD Flame Is Lit.)
- Check the column and makeup flow rates.
- 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.
- Check the **Lit offset** value. The typical **Lit offset** value is 2.0.
- Thoroughly leak check the whole system. (See Checking for Leaks.)

NPD Adjust Offset Process Fails

- Inspect the jet to see if it is clogged.
- Measure the actual detector flows. (See To Measure a Detector Flow.)
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- Thoroughly leak check the whole system, especially the detector column fitting. (See Checking for Leaks.)
- Set the equilibration time to 0.0.

2 Hardware Symptoms



3

Chromatographic Symptoms

Retention Times Not Repeatable 22

Peak Areas Not Repeatable 23

Contamination or Carryover 24

Larger Peaks Than Expected 25

Peaks Not Displayed/No Peaks 26

Baseline Rise During Oven Temperature Program 27

Poor Peak Resolution 28

Peak Tailing 29

Peak Boiling Point or Molecular Weight Discrimination Poor 30

Sample Decomposition in Inlet/Missing Peaks 31

Peak Fronting 32

Noisy Detector, Including Wander, Drift, and Baseline Spikes 33

Check for the correct jet. Low Peak Area or Height 36

FID Flame Goes Out During a Run and Attempts to Reignite 38

FID Baseline Output Above 20 pA 39

FPD Flame Goes Out During a Run and Attempts to Reignite 40

FPD Output Too High or Too Low 41

FPD Low Peak Areas 42

FPD Large Peak Width at Half-Height 43

FPD Baseline Output High, > 20 pA 44

NPD Solvent Quenching 45

NPD Response Low 46

NPD Baseline Output > 8 million 47

NPD Adjust Offset Process Not Functioning Properly 48

NPD Low Selectivity 49

Negative Peaks Seen with TCD 50

TCD Baseline Has Dampened Sinusoidal Noise Trailing Peaks (Ringing

Baseline) 51

TCD Peaks Have Negative Dip on Tail 52



Retention Times Not Repeatable

- Replace the septum.
- Check for leaks in the inlet, liner (as applicable), and column connection. (See "Checking for Leaks" .)
- 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.
- Check for leaks at the inlet. (See "Leak Check Tips".)
- Verify that you are using the correct liner type for the sample being injected.
- Consider if this is this first run. (Has the GC stabilized?)
- If using an FID or NPD and retention times increase (drift), check the jet for contamination.

Peak Areas Not Repeatable

- Check the ALS syringe operation. (See the Troubleshooting section of the 7683B Automatic Liquid Sampler Installation, Operation and Maintenance 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 this 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

- Check the septum type and installation.
- 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.
- 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.
- Check all gas trap indicators and dates.
- Verify the gas purity. Check for supply tubing and fitting contamination.
- If you suspect that there is contamination in the inlet, column, or detector, perform the bakeout procedure.
- Verify that the oven program temperature and time are sufficient for the samples being injected.
- · Check the solvent level in the ALS wash bottles.
- Replace the ALS syringe if necessary.
- Check the sample injection volume.
- Install an Agilent column backflush system.

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.
- Check the vial caps.
- 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.
 - Verify that the turret/tray 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" .)

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.
NPD, uECD	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.
- 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.
- Check for leaks at the column fittings. (See "Checking for Leaks" .)
- Prepare and use a column compensation profile.

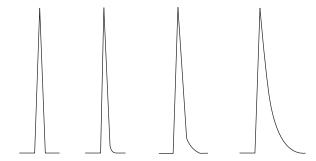
Poor Peak Resolution

- 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.
- Consider the column stationary phase (active column).
- Verify that the column was cut and installed properly.
- Consider the type of adapter, liner, and inlet seal being used. One or all of these may be contaminated or active.
- Check adapters (if installed) and liner for solid particles.
- For capillary splitless injection, consider compatibility between the solvent and column.
- Verify that the injection technique is adequate.
- Verify the inlet temperature.
- Check for dead volume in the system. Check for correct column installation at both ends.
- Inspect any transfer lines for cold spots.

NPD

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.
- Increase the inlet temperature and verify that the insulation cup is installed and contains insulation.
- Check column cut and installation into the inlet. See the topic for the SS, PTV, and VI.

For any inlet operating in splitless mode with any detector

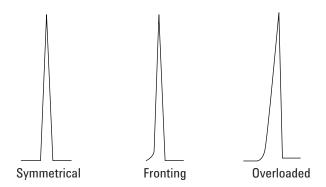
- Check the inlet for leaks. (See "Checking for Leaks".)
- Check liner type.
- Verify that the oven starting temperature is less than the solvent boiling point.
- Check column cut and installation into the inlet See the topic for the SS, PTV, and VI.
- Check that the solvent vapor volume does not exceed the liner capacity.
- Check for appropriate purge delay time.

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 (SS, PP, and PTV inlets).
- 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. See the following:
 - To Bakeout Contaminants from the Split/Splitless Inlet
 - To Bakeout Contaminants from the Purged Packed Inlet
 - To Bakeout Contaminants from the COC Inlet
 - To Bakeout Contaminants from the PTV Inlet
 - To Bakeout Contaminants from the VI Inlet

Peak Fronting

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



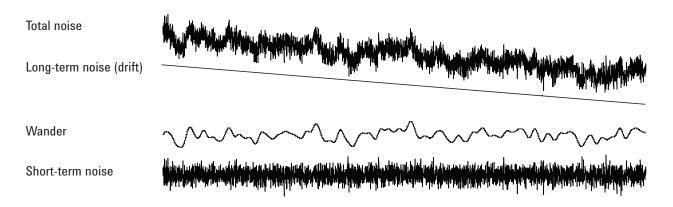
If peak fronting or overloading occurs, try the following:

- Verify that the injection volume is appropriate.
- Ensure that the column is installed properly.
- Verify that the appropriate injection technique is being used.
- 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.

Noisy Detector, Including Wander, Drift, and Baseline Spikes

Noise should be measured under "normal" operating conditions, with a column connected and carrier gas on. 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.



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 Measure NPD Leakage Current.
- 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. See the following:
 - To Bakeout Contaminants from the Split/Splitless Inlet
 - To Bakeout Contaminants from the Purged Packed Inlet
 - To Bakeout Contaminants from the COC Inlet

3 Chromatographic Symptoms

- To Bakeout Contaminants from the PTV Inlet
- To Bakeout Contaminants from the VI Inlet
- Verify the purity of carrier and detector gases.
- Verify proper reassembly after recent maintenance.
- Inspect the detector for contamination.

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

- Inspect the detector for contamination.
- Inspect the column and inlet for contamination.
- Inspect the FID or NPD jet for contamination.
- Verify that the FPD photomultiplier tube (PMT) is properly installed. If it is not, light leaks and ultimately noise will result.

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. If experiencing baseline wander, check for leaks, especially at the septum and at the column. (See "Checking for Leaks" .) 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.

Baseline spiking

There are two types of spiking on the baseline output: cyclic and random.



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

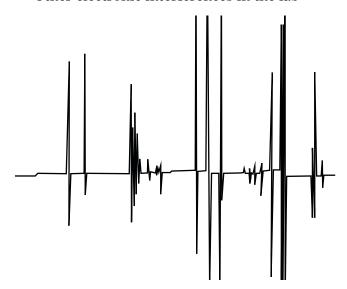


Figure 2 Random spiking

- 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.
- For a packed column, check that the packed column exit is properly sealed with glass wool.
- · Check packed column installation.

3 Chromatographic Symptoms

Check for the correct jet. Low Peak Area or Height

- If using an inlet in split mode, check the split ratio.
- Check for leaks. (See "Checking for Leaks".)
- 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.
- Check the supply gas purity.
- Check all trap indicators and dates.
- Verify that the method parameters are correct.
- 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 using an FID:

- Verify that the correct jet is installed.
- Check for a dirty jet.

If using a uECD:

- Replace the fused silica indented mixing liner.
- · Replace and reinstall column.
- Clean the makeup gas adapter.

If using an NPD:

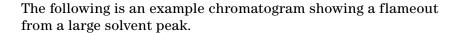
- Check the detector for contamination.
- Replace ceramic insulators.
- Replace the bead.

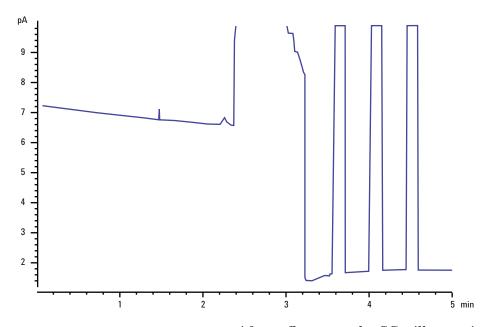
If using an FPD:

• Verify correct column installation.

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

FID Flame Goes Out During a Run and Attempts to Reignite





After a flameout, the GC will try to ignite the flame three times.

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

- See if an aromatic 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" .)
- 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.
- 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.
- Check that the FID leakage current is < 2.0 pA. (See "To Measure FID Leakage Current" .)

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" .)
- Verify the detector 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" .)

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.

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.

NPD Solvent Quenching

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

- Turn hydrogren off/on around the solvent peak.
- Use nitrogen as the makeup gas.
- Set the total column flow and makeup gas to less than $10~\mathrm{mL/min}$.
- Increase the air flow by 10 mL/min.
- Increase the detector temperature to 325 °C.
- Implement an Agilent Dean's switch solvent vent solution.

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. Increase the bead voltage.
- Measure the actual gas flow at the detector. (See "To Measure a Detector Flow" .)
- Check for a partially plugged jet.
- Verify that the bead is activated. Look through the vent hole on the detector lid to see if the bead is glowing orange. Replace the insulators/collector.

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.
- Measure the actual detector flows. (See To Measure a Detector Flow.)
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- Thoroughly leak check the whole system, especially the detector column fitting. (See Checking for Leaks.)
- Set the equilibration time to 0.0.

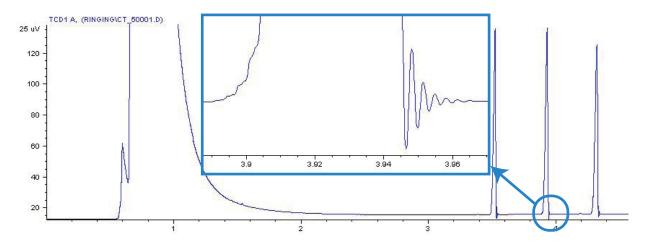
NPD Low Selectivity

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

Negative Peaks Seen with TCD

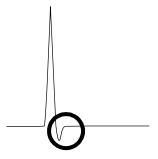
- Verify that the correct gas type is being used.
- Check for a leak in the system, especially at the detector column fitting. (See "Checking for Leaks" .)
- Consider sensitivity to analytes.
- Check the flow settings, then measure the actual detector flows. (See "To Measure a Detector Flow" .)

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

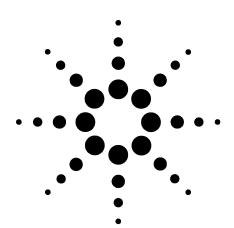


Wrong data rate is selected in the data system. For TCD, the data rate should be \leq 5 Hz.

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.



GC Never Becomes Ready 54
Flow Never Becomes Ready 55
Oven Temperature Never Cools Down/Cools Very Slowly 56
Oven Never Heats Up 57
Temperature Never Becomes Ready 58
Cannot Set a Flow or Pressure 59
A Gas Does Not Reach Setpoint Pressure or Flow 60
A Gas Exceeds Pressure Setpoint or Flow 61
The Inlet Pressure or Flow Fluctuates 62
Cannot Maintain a Pressure as Low as the Setpoint on a Split Inlet 63
The Measured Column Flow Does Not Equal the Displayed Flow 64
FID Does Not Ignite 65
FID Ignitor Does Not Glow During Injection Sequence 66
NPD Adjust Offset Process Fails 67
FPD Does Not Ignite 68

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:

- Press [Status] or a component key (for example, [Front inlet]) 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 press [**Prep Run**] 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.

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 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

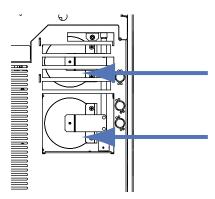
If the oven does not cool down or cools down very slowly:

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.



If using cryo cooling:

- Check for sufficient cryo coolant.
- Check if operating limits are exceeded.

Oven Never Heats Up

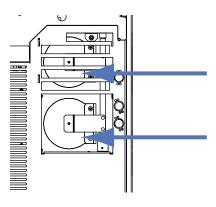
• Press [Status] to check for errors to report to Agilent.

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.



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.
- If using a cool on-column with CryoBlast or a PTV inlet:
 - Check cryo coolant level.
 - Check if operating limits have been exceeded.

Cannot Set a Flow or Pressure

If you cannot set a flow or pressure using the split/splitless, PTV, VI, or cool on-column inlets, 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.

If you cannot set a flow or pressure using the purged packed inlet, do the following:

- Check the column mode. The packed inlet uses *flow* control for undefined columns.
- Check that the flow is turned on.

A Gas Does Not Reach Setpoint Pressure or Flow

If an inlet does not reach its pressure setpoint, it will shut down in an amount of time determined by the type of inlet. 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".)
- 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.

If you are using a split/splitless inlet, PTV inlet, or volatiles interface:

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

A Gas Exceeds Pressure Setpoint or Flow

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

If using a split/splitless inlet, PTV inlet, or volatiles interface:

- Decrease the split ratio.
- Replace the split vent filter.
- Verify that the correct liner is selected (for split/splitless and PTV inlets).
- Check the gold seal for contamination (for split/splitless inlet).

If using an FID or NPD:

• Check for a plugged jet.

Valves:

• Check for a misaligned 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.
- Check for leaks. (See "Checking for Leaks".)
- Check for large restrictions in the inlet liner or split vent trap.
- Verify that the correct liner is installed.
- Check for a restriction in headspace, purge and trap, and any other external sampling devices.

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.
- Check for a plugged liner.
- Check for contamination in the split vent line. Contact Agilent service to replace, if necessary.
- 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, do the following:

- Verify that the measured flows are corrected to 25 $^{\circ}\mathrm{C}$ and 1 atmosphere.
- Verify that the correct column dimensions are configured accurately, including the actual (trimmed) column length.
- The split vent line or trap may be partly plugged, creating an actual inlet pressure higher than the setpoint pressure.

FID Does Not Ignite

- Check that the FID ignitor glows during ignition sequence. (See To Verify FID Ignitor Function During Ignition Sequence.)
- Verify that the Lit Offset is ≤ 2.0 pA.
- Check for a plugged or partially plugged jet.
- Check the FID flow rates. 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.
- · Check the column flow rate.
- · Check for leaks at the FID column fitting.
- Ensure that the FID temperature is high enough for ignition (>150 $^{\circ}$ C).

FID Ignitor Does Not Glow During Injection Sequence

WARNING

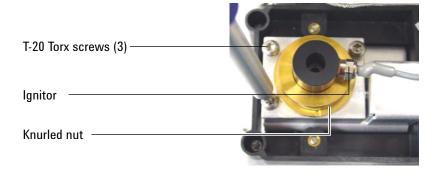
Keep body parts at a safe distance from the FID chimney while performing this task. If using hydrogen, the FID flame will not be visible.

- 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 \geq 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.

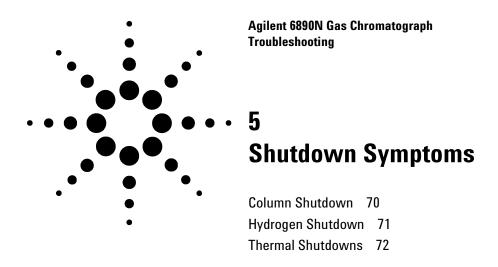


NPD Adjust Offset Process Fails

- Inspect the jet to see if it is clogged.
- Measure the actual detector flows. (See To Measure a Detector Flow.)
- Check the condition of the bead. Replace if necessary.
- Verify that the flow settings are correct.
- Thoroughly leak check the whole system, especially the detector column fitting. (See Checking for Leaks.)
- Set the equilibration time to 0.0.

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.
- Measure the actual detector flows. (See To Measure a Detector Flow.)
- The column may be installed too high into the detector.
- Check that the FPD ignitor operates. (See To Verify That the FPD Flame Is Lit.)
- Check the column and makeup flow rates.
- 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.
- Check the **Lit offset** value. The typical **Lit offset** value is 2.0.
- Thoroughly leak check the whole system. (See Checking for Leaks.)



Column Shutdown

If a carrier gas (which can include an auxiliary flow module or pneumatics control module) shuts down, the following happens:

- The oven turns off to avoid column damage.
- The oven flaps in the back of the oven open halfway.
- The oven temperature setpoint flashes **Off**.
- All flows for the column turn off. When viewed, their parameters flash **Off**. For example, the septum purge and column flows for a split/splitless inlet would turn off.
- All other heaters turn off. When viewed, their temperature parameters flash **Off**.
- Attempts to turn on a shut-down zone fail with an error message.
- The instrument beeps.

To recover from this state.

- 1 Fix the cause of the shutdown.
 - · Check for a broken column.
 - Replace the inlet septum.
 - Replace the inlet O-ring.
 - Check the supply pressure.
 - · Check for leaks.
- 2 Press the key for the device that initiated the shutdown. Scroll to the pneumatic parameter that is flashing Off, then press [On] or [Off].

For example, if the front inlet ran out of carrier gas, press [Front Inlet], scroll to the pressure or flow parameter, then press [On].

Hydrogen Shutdown

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

The GC monitors inlet and auxiliary gas streams. If a stream shuts down because it is unable to reach its flow or pressure setpoint and if that stream is configured to use hydrogen, the GC assumes that a leak has occurred and shuts down components to create a safe state. The GC:

- Closes the carrier supply valve to the inlet and closes and turns off both pressure and flow controls. When viewed, these parameters will flash **Off**.
- Opens the split vent valves in the split/splitless and PTV inlets.
- 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). When viewed, these parameters will flash Off.
- · Sounds an alarm.

To recover from this state:

- **1** Fix the cause of the shutdown:
 - Replace the inlet septum.
 - Replace the inlet O-ring.
 - · Check for broken column.
 - Check the supply pressure.
 - Check the system for leaks. See Checking for Leaks.
- 2 Press the key for the device that initiated the shutdown. Scroll to the pneumatic parameter that is flashing Off, then press [On] or [Off]. For example, if the front inlet ran out of carrier gas, press [Front Inlet], scroll to the pressure or flow parameter, then press [On].

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.

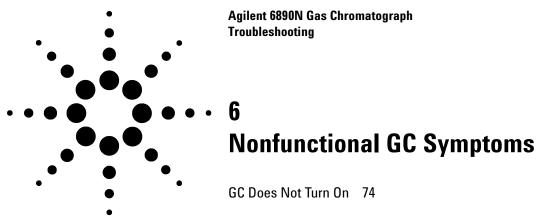
5 Shutdown Symptoms

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.

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.



PC Cannot Communicate with GC 75

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:

```
Microsoft Windows XP [Version 5.1.2600]
(C) Copyright 1985-2001 Microsoft Corp.

C:\>ping 10.1.1.101

Pinging 10.1.1.101: bytes=32 time<ims ITL=128
Reply from 10.1.1.101: bytes=32 time</ims ITL=128
Reply from 10.1.1.101: bytes=32 time<ims ITL=128

Ping statistics for 10.1.1.101:

Packets: Sent = 4, Received = 4, Lost = 0 (0% loss),
Approximate round trip times in milli-seconds:

Minimum = 0ms, Maximum = 0ms, Average = 0ms

C:\>
```

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.
- Check that a crossover cable for single GC to computer direct connection is installed.

6 Nonfunctional GC Symptoms

Agilent 6890N Gas Chromatograph Troubleshooting 7 Checking for Leaks Leak Check Tips 78 To Checking for September 70

Leak Check Tips 78

To Check for External Leaks 79

To Check for GC Leaks 81

Leaks in Capillary Flow (Microfluidic) Fittings 82

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 (H_2) 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.

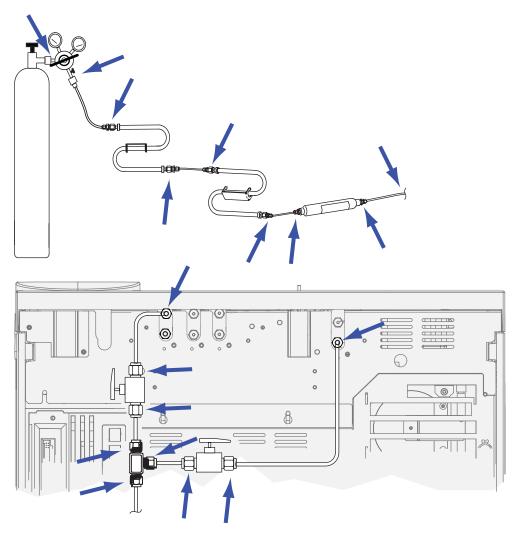
WARNING

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).

7 Checking for Leaks

- **3** Fully turn the regulator knob counterclockwise to shut the valve.
- **4** Wait 5 min. If there is a measurable drop in pressure, there is a leak in the external connections. No drop in pressure indicates that the external connections are not leaking.

To Check for GC Leaks

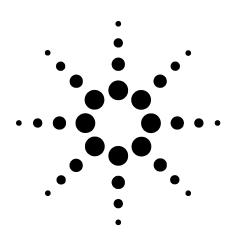
Check for leaks at these connections:

- 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
- Agilent capillary flow fittings

Leaks in Capillary Flow (Microfluidic) 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. (See To Attach a Capillary Column Using SilTite Metal Fittings.)

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



Troubleshooting Tasks

To Measure a Column Flow 84

To Measure a Split Vent or Septum Purge Flow 87

To Measure a Detector Flow 89

To Perform the GC Self-Test 92

To Adjust the FID Lit Offset 93

To Verify That the FID Flame Is Lit 94

To Verify FID Ignitor Function During Ignition Sequence 95

To Measure FID Leakage Current 96

To Measure FID Baseline Output 97

To Measure NPD Leakage Current 98

To Verify That the NPD Bead Is Ignited 99

To Verify That the FPD Flame Is Lit 100

To Adjust the FPD Lit Offset 101

To Measure a Column Flow

Measuring FID, TCD, uECD, and FPD column flow

The following procedure can be used to measure column flow with an FID, TCD, uECD, and FPD.

WARNING

Hydrogen (H_2) 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 (can be found 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.

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

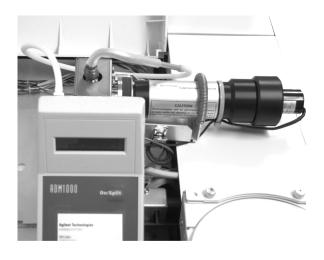


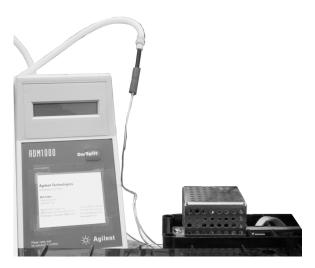
A separate adapter (19301-60660) is supplied for the FID. Insert the adapter into the detector exhaust vent as far as possible. You will feel resistance as the adapter O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure a good seal.



For the FPD, remove the vent tube assembly and use a 1/4-in 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)

8 Troubleshooting Tasks



- Flow-measuring insert (19301-60660)
- Electronic flowmeter calibrated for the gas and flow rates of concern
- 2 Set the bead voltage to 0.0 V.
- 3 Cool the NPD to $100 \, ^{\circ}$ C.
- **4** Remove the bead and store it carefully until re-installation.
- **5** Insert the NPD flowmeter adapter tool into the NPD collector.
- **6** Attach the flow-measuring insert to the NPD flowmeter adapter tool.



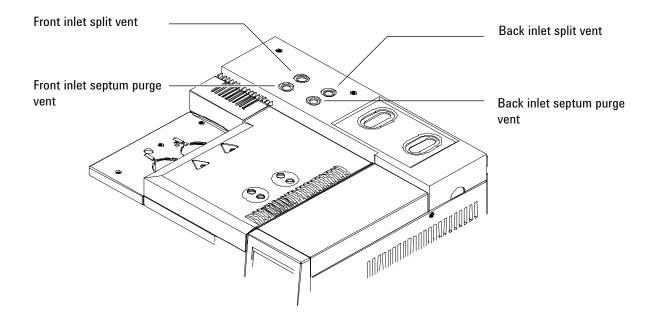
7 Place the flowmeter tubing over the flow-measuring insert to begin measuring flows.

To Measure a Split Vent or Septum Purge Flow

WARNING

Hydrogen (H_2) 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 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 will result in leakage and thus an incorrect flow reading.

8 Troubleshooting Tasks



To Measure a Detector Flow

Measuring FID, TCD, uECD, and FPD flows

WARNING

Hydrogen (H_2) 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 (can be found 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** Turn off (where applicable): the FID flame, FPD flame, and TCD filament.
- **5** Cool the detector.
- **6** Connect the appropriate adapter to the detector exhaust.

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



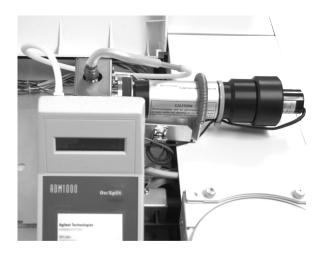
A separate adapter is supplied for the FID. Insert the adapter into the detector exhaust vent as far as possible. You will feel resistance as the adapter O-ring is forced into the detector exhaust vent. Twist and push the adapter during insertion to ensure a good seal.

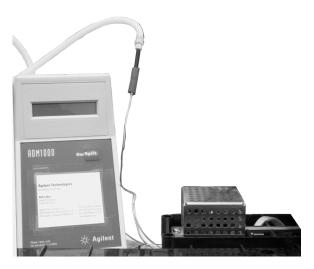


For FPD, remove the vent tube assembly and use a 1/4-inch tube adapter between the detector exhaust and the flowmeter tubing.

8 Troubleshooting Tasks









7 Connect the flowmeter to the flowmeter adapter and measure flows.

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 Set the bead voltage to 0.0 V.
- 3 Cool the NPD to $100 \, ^{\circ}$ C.
- **4** Remove the bead and store it carefully until re-installation.
- **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 1 min, then turn the GC back on. If the main GC status screen appears, the GC has passed the self-test.

Agilent 6890N GC Vesion N.05.05

Power on successful

To Adjust the FID Lit Offset

To adjust the FID Lit offset:

- 1 Press [Config].
- 2 Scroll to Front detector or Back detector (wherever the detector is installed) and press [Enter].
- 3 Scroll to **Lit offset**. With the **Lit offset** line highlighted, enter the new parameter for the detector and press [**Enter**].
- 4 Lit offset should be ≤ 2.0 pA or lower than the normal FID output when lit.

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.

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 $^{\circ}$ C.
- **2** Turn off the FID flame.
- **3** Verify that the FID electrometer is on.
- 4 Press [Front Detector] or [Back Detector], then scroll to Output.
- **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 Press [Front Detector] or [Back Detector], then scroll to Output.
- **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 Measure NPD Leakage Current

- 1 Load the analytical method.
- 2 Set the NPD Adjust Offset to Off and the Bead Voltage to 0.00 V.
 - Leave the NPD at operating temperature.
 - Leave flows on or off.
- 3 Press [Front Detector] or [Back Detector], then scroll to Output.
- 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 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 Press [Config].
- 2 Scroll to Front detector or Back detector (wherever the detector is installed) and press [Enter].
- 3 Scroll to Lit offset. With the Lit offset line highlighted, enter the new parameter for the FPD (typical value is 2.0 pA), and press [Enter].

8 Troubleshooting Tasks