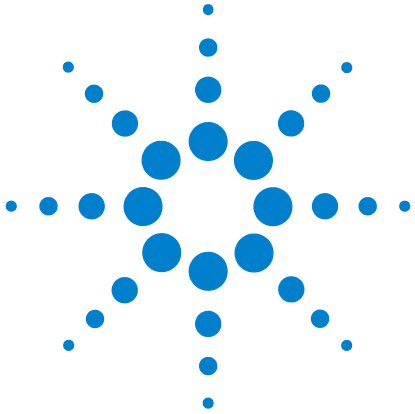
沪制01150234号



Agilent Intuvo 9000 Gas Chromatograph

Troubleshooting

Agilent Technologies

Notices

Manual Part Number

G9000-90005

Edition

Fifth edition, September 2021
Fourth edition, July 2019
Third edition, February 2018
Second edition, June 2017
First edition, September 2016

Printed in USA

Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610 USA

安捷伦科技 （上海）有限公司
上海市浦东新区外高桥保税区
英伦路 412 号
联系电话：（800）820 3278

Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

Safety Notices

CAUTION

A CAUTION notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Contents

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.

While this manual describes troubleshooting for Intuvo 9000 GCs in general, when describing GC firmware features, this manual assumes a 9000 GC using firmware 2.4.

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 touch screen and browser UI include 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**.

Self-Guided Diagnostics

The GC provides diagnostic tests. Run these tests if you suspect a problem such as a leak, clogged line, clogged jet, or if the FID isn't working correctly after maintenance.

Table 1 Self-guided diagnostics

Diagnostic test	What is checked
Gas Supply Pressure Check	Maximum pressure the gas supply can deliver in splitless mode
Jet & Restriction Test	Leaks at the inlet column connection, split vent trap, septum nut and septum, and insert weldment assembly.
Pressure Decay Test	All inlet and inlet flow module seals
Septum Purge Test	Blockages in the septum purge line.
Split Vent Restriction Test	Restrictions in the split vent line.
FID Leakage Current Test	Proper assembly and cleanliness of the upper FID parts when the flame is off (by measuring electrical connectivity).
FID Jet Restriction Test	Gas flow through the jet.

Error conditions

If a problem occurs, a status message appears. If the message indicates broken hardware, more information may be available.

Systems Configured for Enhanced Communications

When configured with other instruments that support enhanced communications, such as a 5977B MSD and 8697 and 7697 Headspace Sampler, the 9000 GC and configured instruments closely interact with each other. In addition to simply sending start run commands and general status information (ready or not ready), the instruments change their settings based on each other's status. For example:

- A GC shutdown may cause an MS to shut down or otherwise protect itself.
- Venting an MS causes changes in the GC to support the venting process.
- An MS shutdown may cause the GC to change its settings to protect itself and the MS.
- A loss of communications between the instruments may causes changes in one or both instruments.
- The GC sleep/wake cycle triggers the MS and HS sleep/wake cycle (if available).
- A configured headspace sampler determines its timings based on the loaded GC method.

Because of this interaction, always check the displays on all instruments and in the data system to learn the complete system status. In general, the GC display will present configured instrument status messages as well as its own messages.

A connected 8697 Headspace Sampler is considered an integral part of the GC, so its EMF data, troubleshooting diagnostics, and automated checks and procedures are also integrated into the GC touchscreen and browser interface.

Peak Evaluation

Peak Evaluation is a tool that lets a user track performance metrics based on a method's calibration standard. Use Peak Evaluation to analyze continuing calibration validation, and if validation fails, optionally stop processing samples. Peak Evaluation also provides a means to perform routine maintenance based on actual performance, rather than by elapsed time or an injection counter.

Using Peak Evaluation involves:

1. Creating and setting up a reference chromatogram.
2. Modifying a method to perform the peak evaluation based on the reference chromatogram.
3. Running a continuing calibration verification standard (or other appropriate sample) with the method.

Peak Evaluation normally compares an output signal from the current sample against the reference chromatogram saved in the method to determine if the GC is operating as expected. However, it can also check several absolute baseline statistics, such as baseline value and baseline noise.

Note that the Peak Evaluation can be disabled by a data system. See "Data System Feature Selection" on page 18.

For more information, refer to the Browser Interface online help topics.

Trend Analysis

The GC includes a Trend Analysis feature, which provides the ability to visualize a variety of details about the instrument's health and performance over a period of time. A Trend Analysis can be performed for:

- Diagnostic test results
- Chromatographic test results
- Early Maintenance Feedback (EMF) reset history

The Diagnostic trend plots display the results from diagnostic tests (such as inlet leak and restriction tests or FID jet restriction tests) that have been run on the instrument. The Chromatographic trend plots display results from Blank, Detector, and Peak Evaluations that have been run on the instrument. The EMF reset plots show when and at what counter value recent EMF resets were performed on the instrument.

When using trend analysis, it is good practice to save methods with a new name after making any changes. Method changes can cause shifts in the plots. It is usually easier to interpret the before and after data separately.

Diagnostic Trend Plots

The GC provides the ability to plot the long term results for certain performance metrics. Trend plotting is available for diagnostic values from existing diagnostic test results, such as from leak test results.

To search for trends:

1. Go to **Diagnostics > Diagnostic Tests**.
2. Select **Diagnostic Plot**.
3. Select the applicable device and data to plot.
   - **Device**: Select the inlet, detector, or sampler (if available) which has the available test data to be plotted.
   - **Test**: Select the desired diagnostic test for the device.
   - **Parameter**: Select the desired test parameter to plot. (Some tests have only parameter.)
   - **From**: Plot data created no earlier than this date.
   - **To**: Plot data created through this date.

4 Select **Load** to load and plot the data available for the selected date range and other selections.

- Use the legend at the bottom of the plot to show or hide limits, values, and so forth.
- See "The Trend Plot" on page 16.

After reviewing the plot data, you can choose to begin a maintenance task to correct any issue. Select Maintenance **Start** to open a list of related maintenance tasks for the device. If desired, you can select and run a task, for example, to replace the inlet septum if the inlet leak test plot shows the septum has degraded through use.

Chromatographic Trend Plots

The GC provides the ability to plot the long term results for certain performance metrics. Trend plotting is available for chromatographic values such as Blank Evaluation attributes, Detector Evaluation historical data, and Peak Evaluation attributes.

The chromatographic trend plot uses data as gathered during blank evaluations, detector evaluations, and peak evaluations. For example, a peak evaluation plot relies on the peak list and relative peak parameters set up in the Reference Chromatogram, and the use of that reference chromatogram to collect peak evaluation data.

Note that a connected data system can restrict access to peak evaluation data and trend plotting, as well as to some troubleshooting features. See "Data System Feature Selection" on page 18.

To search for trends:

1 Go to **Diagnostics**.

2 Select the desired diagnostic data to analyze:

- **Detector Evaluation Reports**: View trends in historical detector evaluation results.
- **Blank Evaluation Reports**: View trends in blank evaluation run data, such as baseline, noise, and peaks areas.
- **Peak Evaluation Reports**: View trends in peak evaluation attributes, such as retention time, peak height, or peak width.

3 Select **Chromatographic Trend Plot**.

4 Select the applicable device and data to plot, depending on the chromatographic trend plot type selected. Available choices include:

- **Method**: Select the method used for the peak evaluation data.
- **Device**: Select the detector associated with the method used to collect the diagnostic data (blank evaluation, detector evaluation, or peak evaluation).
- **Component**: For blank evaluation plots, select **Baseline** to plot baseline data (initial or final, value or noise), or select **Summary** to plot **Max Peak Height** or **Total Peak Area**.
- **Analyte**: If available, select to plot the parameters for an analyte available in the method.
- **Parameter**: Choose the desired analyte/component parameter to plot.
- **From**: Plot data created no earlier than this date.
- **To**: Plot data created through this date.

5 Select **Load** to load and plot the data available for the selected date range and other selections.

6 Use the legend at the bottom of the plot to show or hide limits, values, and so forth.

7 See "The Trend Plot" on page 16.

After reviewing the plot data, you can choose to begin a troubleshooting task to correct any issue found. Select Troubleshooting **Start** to begin automated chromatographic troubleshooting. If the GC finds at least seven (7) data points, it can suggest troubleshooting tasks most related to the plotted attributes. If there are fewer than 7 data points, select troubleshooting tasks manually.

The Trend Plot

The plot shows the selected results by date. Where applicable, the plot also includes the pass/fail and warning criteria.

Trend plot legend and controls:

- **Troubleshooting**: Select to toggle the pass/fail criteria. For peak evaluation, this is the limit where Peak Evaluation will fail.
- **Warning**: For Peak Evaluation, select to toggle the warning limits set in the reference chromatogram (whether strict or lenient).
- **Value**: Select to toggle the test result value.

- **Method/Config**: Indicates the date when the method or configuration changed (if applicable), for example, when a column was installed. Any method or configuration change is plotted, regardless of whether it was associated with the flowpath or method being plotted.
- **Maintenance**: Select to toggle markers for dates maintenance was performed for the relevant hardware.

Scaling the plot:

- Click and drag on the plot to zoom in on that selected plot region.
- Double-click on the plot to zoom out to the previous magnification level.

Annotate the plot:

- To annotate the plot, click/tap a data point on the Value plot line.

EMF Trend Plots

The GC automatically plots EMF reset data to make it easier to see trends in performance related to routine maintenance. The GC logs every counter reset, including the counter value, date, and time. When using EMF counters and the automated maintenance tasks, this occurs automatically. If not using the automated maintenance tasks, the GC logs all manual counter resets.

To search for trends, view the counter details for the EMF item. Go to **Maintenance**, select the component type (inlet, detector, and for forth), then select the counter and show its details.

The EMF trend plot includes the following controls:

- **User Reset**: Select to toggle manual counter resets.
- **Maintenance Reset**: Select to toggle counter resets performed automatically when using an automated maintenance procedure.
- **Service Due**: Select to toggle the Service Due limit on the plot.
- **Service Warning**: Select to toggle the Service Warning limit on the plot.

Note that any change to a Service Due or Service Warning limit does not appear until the next counter reset.

Data System Feature Selection

When controlled by a data system, the data system can restrict certain advanced GC features so that these features are no longer available at the GC touchscreen or in the browser interface. These changes apply whether or not the data system is currently controlling the GC.

When a feature has been disabled by a data system, the browser interface will indicate this by making the tab or control gray, and by displaying a small lock icon on it ().

Table 2 summarizes the possible changes to the browser interface. Each feature can be enabled/disabled independently of the others.

Table 2 Possible feature changes

Feature	Changes in browser interface if feature is enabled	Changes in GC touchscreen if feature is enabled
Start	The browser interface cannot start a sequence.	The Start button only works for manual injections.
Real-time chromatograms	Real-time plots are not available. The Home tab Plot button will be gray, inactive, and will display a small lock icon.	Real-time plots are not available. The Home tab Plot button will be gray, inactive, and will display a small lock icon.
Date and time changes	Running system setup will not show a dialog to edit the GC date and time. Set the GC date and time from the data system.	The UI for setting the data and time is unavailable. Set the GC date and time from the data system.
GC Troubleshooting verification	When running a troubleshooting, maintenance, or diagnostic automated task, the GC will skip any verification run step. For example, the GC will not prompt to perform a blank run or checkout run.	When running a troubleshooting, maintenance, or diagnostic automated task, the GC will skip any verification run step. For example, the GC will not prompt to perform a blank run or checkout run.
Peak Evaluation	Peak evaluation is completely disabled. Existing data is retained, but methods which use peak evaluation will not generate or analyze any new peak evaluation data. Existing peak evaluation data will not be available for viewing or analysis. (Re-enabling peak evaluation restores access to the existing data.)	Not applicable.

Table 2 Possible feature changes (continued)

Feature	Changes in browser interface if feature is enabled	Changes in GC touchscreen if feature is enabled
Sequence menu	The browser sequence tab is gray and unavailable. Existing browser interface sequences are retained, but cannot be loaded, edited, viewed, or run.	The Sequence tab is disabled and the browser interface cannot enable it.
Deletion of all on-instrument chromatographic data	No changes. (On-instrument chromatographic data can only be deleted from the touchscreen.)	The control is disabled.

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

Be sure to configure the GC and all related components to reflect an inlet or detector change. Below are some examples of components to keep current after making changes to the inlet or detector:

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. Configure the flow according to the FPD$^+$ filter installed (phosphorus versus sulfur).

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 View the Run Log, Maintenance Log, and Event Log

Use these logs to troubleshoot problems, especially when a message no longer appears on the display.

Run Log

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

Maintenance Log

The maintenance log contains an entry for each time an Early Maintenance Feedback limit is reached, reset, or changed. The log records details such as the counter item, the counter value, the new counter value, and whether or not the counter was reset (indicating a part replacement).

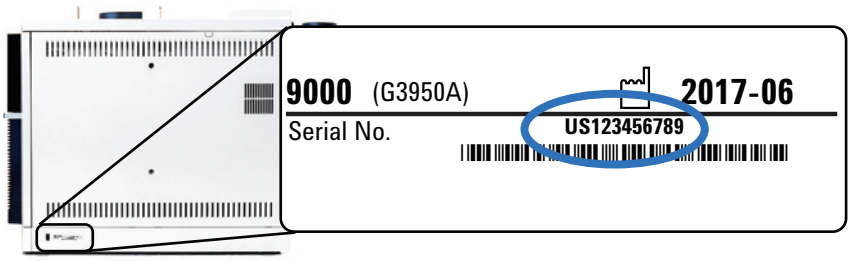
Event Log

The event log records events such as shutdowns, warnings, faults, and GC state changes (start run, stop run, and so forth) that occur during GC operation.

Information to Obtain Before Calling Agilent for Service

Gather the following information before contacting Agilent for service:

- System health report (obtain using a web browser) contains this information. You can view the system help report from a web browser and print or save the report from the browser.
- 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
- Instrument details. Obtain the following information:



  - GC firmware revision

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

2
ALS and Detector 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.
- 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.

Vial Mishandled by ALS (7693A)

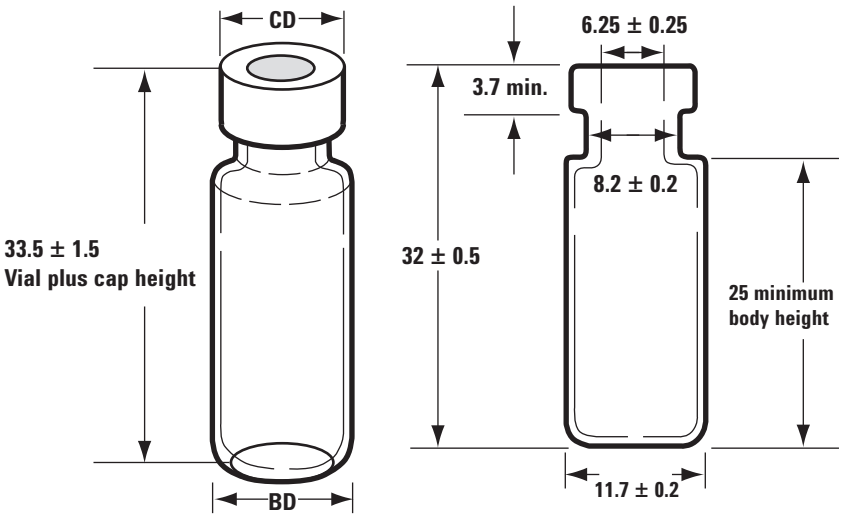
Refer to your sampler operating documentation for additional information.

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.

  The figure below shows the critical dimensions for sample vials and microvial inserts to be used with the 7693A ALS system. These dimensions do not make up a complete set of specifications.

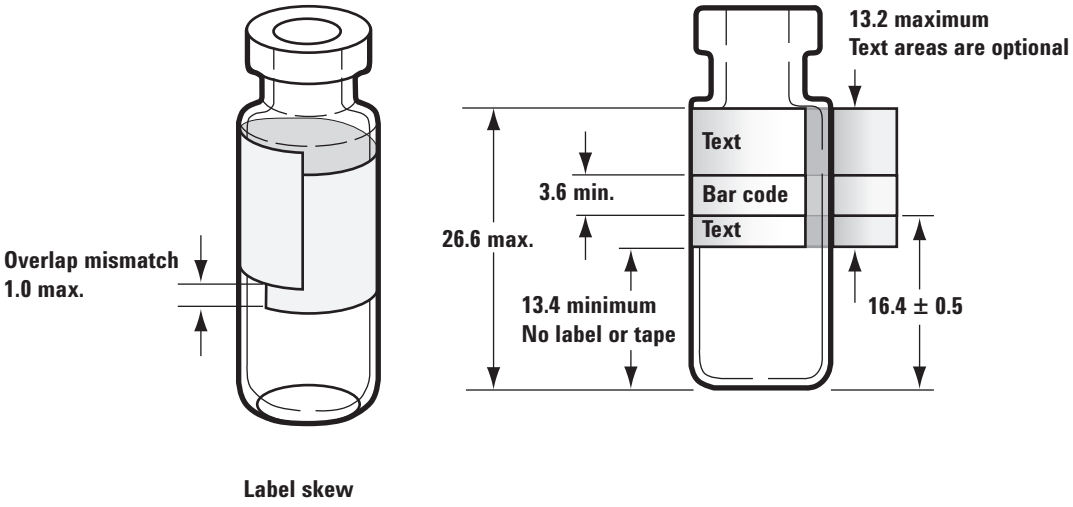
Body Diameter (BD) = 11.7 ± 0.2
Cap Diameter (CD) = BD × 1.03 maximum
All dimensions in millimeters



- Verify the transfer turret is installed if using a G4514A tray.
- Check sample labels (if applicable).
  - Check that they are the correct size.
  - Verify that the labels do not interfere with the gripper.

  Vials are available with a write-on spot for easy marking. If you choose to make and apply your own labels, Agilent Technologies recommends the positioning and maximum label thickness shown in the figure below.

All dimensions in millimeters



- Check that the tray vial racks are clean and snapped into the tray base.
- Calibrate the system.

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, Operation 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 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.
- 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. See “Vial Mishandled by ALS (7693A)”.
- Check that the crimp cap is properly installed. Refer to your sampler documentation.

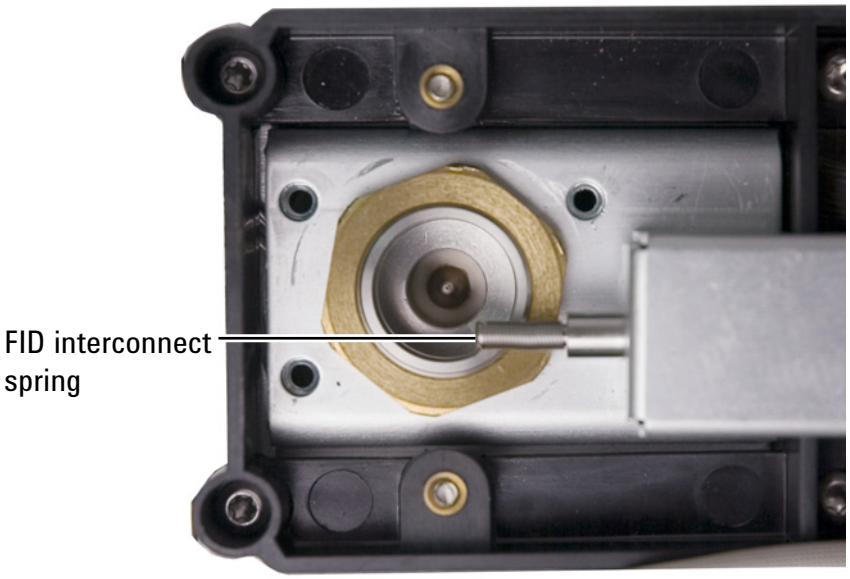
FID Fails Leakage Current Test

Possible causes

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

Procedure

1. If you have just performed maintenance on the FID, first verify that the detector was reassembled properly before troubleshooting detector problems.
2. Replace the PTFE (FID) for contamination.
3. Make sure that the interconnect spring is not damaged, bent, or dirty. The interconnect spring should be touching the bottom of the collector. If the interconnect spring is damaged, bent, or dirty, call Agilent for service.

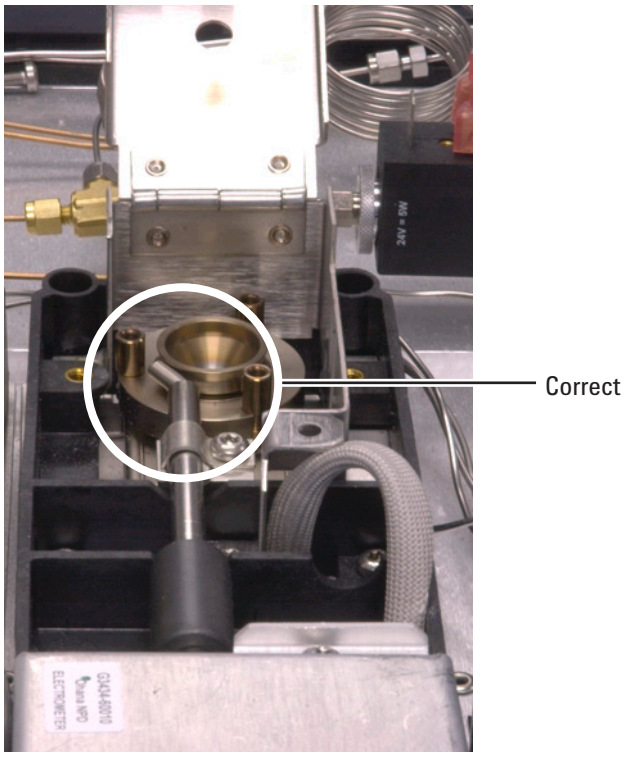


NPD Fails Leakage Current Test

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 adjust offset. 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. If the spring is damaged, bent, or dirty, call Agilent for service.



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 ≤ 1.0 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.)
- Check that the air and hydrogen pressures meet Agilent's recommendations (hydrogen > 35 psi [210 kPa] and air > 55 psi [380 kPa]). See the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
- 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 or a weak ignitor.
- 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.)
- 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.
- 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.

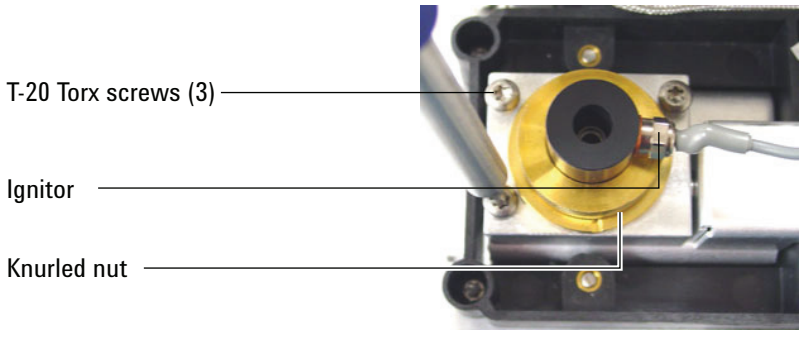
2 Turn the FID flame **On**.

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.

Table 3 FPD+ recommended flows

	Sulfur mode flows, mL/min	Phosphorus mode flows, mL/min
Carrier (hydrogen, helium, nitrogen, argon)		
Capillary columns	1 to 5	1 to 5
Detector gases		
Hydrogen	60	60
Air	60	60
Carrier + makeup	60	60

- Measure the actual detector flows. (See To Measure a Detector Flow.)
- Check that the FPD+ ignitor operates. (See To Verify That the FPD+ Flame Is Lit.)
- Check the column and makeup flow rates.
- Check the **Lit offset** value. The typical **Lit offset** value is 1.0. 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.
- Try increasing the supply pressures to the FPD+ flow module. This makes the flame easier to light without changing the setpoints.
- Under some operating conditions, the flame will light more easily with the vent tube removed. After lighting the flame, reinstall the vent tube.

- Try changing to the phosphorus mode flows, lighting the flame, and gradually alter the flows to the sulfur values.
- Check cable connections to coupling, coupling connection to glow plug, tight glow plug.

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.

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.
- Check for fault messages. You can also read the bead voltage.
- Check the condition of the bead. Replace if necessary.
- 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.

Blinking Not Ready Light: Detector Hardware Fault/TCD Filament Voltage

If the TCD filament is open, the Not Ready LED blinks and the GC will not become Ready.

3
Chromatographic Symptoms

Automated Chromatographic Troubleshooting

The GC provides automated troubleshooting procedures that are designed to help resolve the following common chromatographic symptoms:

Failed Peak Evaluation

No Peaks

Low response

High Response

Retention Time Shift

Area Repeatability

Peak Tailing

Peak Fronting

Resolution Loss

Each of these procedures provide a step-by-step process to try to determine the cause for any of the chromatographic symptoms listed. When applicable, the procedure will allow the user to load a specific method. The procedures may prompt for information such as the sample type, amount, and so forth, and will also prompt to perform automated leak checks and similar when applicable. These automated checks can include validation runs, for example, to verify that a fix resolved the problem. These procedures can be started manually, or may be suggested following a failed Blank Evaluation, Detector Evaluation, or Peak Evaluation.

If using the GC's Trend Analysis features, the trend plots for Blank Evaluation, Detector Evaluation, and Peak Evaluation also provide a link to begin automated troubleshooting.

Note that when the GC has been controlled by a data system, the data system users can choose to prevent the GC from performing runs locally. In this case, the automated troubleshooting procedures will go through all steps and automated checks except that the GC will not prompt to perform any type of validation run, whether with a blank or a sample. To validate the results of any troubleshooting fixes with a validation run, perform the validation run using the data system.

Also, the troubleshooting procedures can only access methods stored on the GC (methods created by the browser interface, or direct changes to the GC setpoints using the touchscreen). If using a data system, you may need to apply the correct method to the GC, so that the method is the active method on the GC.

Start a manual troubleshooting session

To manually start a troubleshooting session:

1 Load the desired method onto the GC.

2 Go to Diagnostics > Troubleshoot and select Troubleshoot.

Select the most applicable symptom and start the troubleshooting session.

The troubleshooting session

Once started, each troubleshooting routine will step through a detailed series of questions and tests designed to identify and resolve common causes for the selected symptom. You may need to confirm specific hardware details, depending on the GC model and specific configuration. For example, you may need to confirm a gas type, column type and dimensions, or (for GC configurations with more than one possible flow path) which flow path to troubleshoot. The troubleshooter can analyze only one flow path at a time.

- During troubleshooting, the GC will not be available to run samples (other than a verification run associated with the specific troubleshooting procedure, as applicable).
- During the troubleshooting, you may be prompted to perform tasks or verification runs. The troubleshooting routine may allow you to pause and resume the routine to allow for time to perform any tasks required.
- Troubleshooting evaluates the currently-loaded method.

Once complete, the troubleshooting report becomes available, and the GC updates the System Log to indicate that a troubleshooting session occurred.

Note that verification runs associated with troubleshooting tasks may be skipped if disabled by a data system. See Feature Selection.

Resolution

If the method used for troubleshooting (the active method on the GC) was set up to include any type of evaluation, then that method includes a reference chromatogram. The GC compares the verification run against the stored reference chromatogram. If acceptable, the troubleshooting is considered resolved, the GC logs it, and the troubleshooting report becomes available. You will also have the option to update the method's current reference chromatogram with the new one.

If the method used for troubleshooting was not set up to include any type of evaluation, then the GC presents the resulting chromatogram for review and acceptance (or rejection).

If the troubleshooter made changes to the loaded method, save the changes if desired. Navigate to the Method tab. Upload the method from the GC and select Save or Save As. If using a data system, upload the modified method into the data system.

Retention Times Not Repeatable

- Replace the septum.
- Check for leaks in the inlet, liner (as applicable), and column connection.
- 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.
  - To Check for a Plugged FID Jet
  - To Check for a Plugged NPD Jet

Peak Areas Not Repeatable

Check the ALS syringe operation.

- Replace the syringe.
- Check for leaks in the inlet, liner (as applicable), and column connection.
- 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?)

For a multimode or split/splitless inlet in split mode, also check for:

- An abnormal split vent restriction.

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 SS and MMI: Set the purge flow to at least 3 mL/min to keep the septum clean. 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, and replace the guard chip. If using a jumper chip, consider installing a guard chip instead.
- 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.
- Install an Agilent column backflush system.
- Install a short column without a stationery phase, for example the OQ/PV column, 19019-60620E. Make a blank run. If the problem goes away, the column is the problem. perform column maintenance or replace the column. Alternately, use any known clean column.

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 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.
  - 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".)
- Check for leaks. (See "To Correct a Leak at a Click and Run Fitting".)
- 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.
  - For 7693A ALS, use the vial heater (accessory G4514A Bar Code Reader/Mixer/Heater) to warm the sample vial.

If the problem is with the detector, see Table 4.

Table 4 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.)

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 .“To Correct a Leak at a Click and Run Fitting”.)
- Prepare and use a column compensation profile.

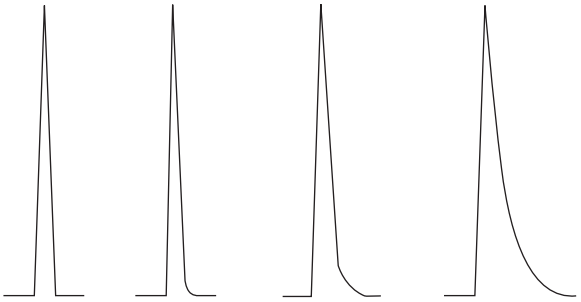
Poor Peak Resolution

- 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, and replace the guard chip. If using a jumper chip, consider installing a guard chip instead.
- 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.
  - 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 Intuvo 9000 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.
  - Replace the column.
- Verify that the column was installed properly.
  - Confirm the installation is leak free.
- 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.
  - If using a jumper chip, consider installing a guard chip instead.

- 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.
- 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:

- 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.

For any inlet operating in splitless mode with any detector

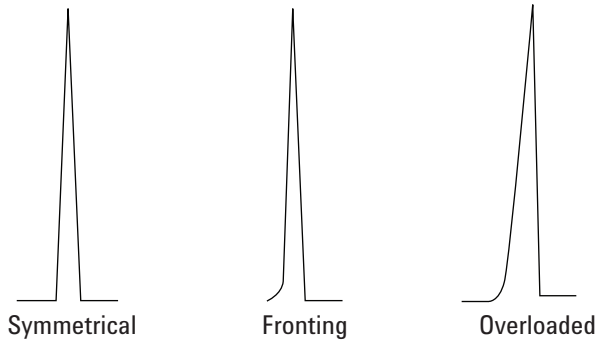
- Check the inlet for leaks.
- Check liner type. Use a liner optimized for splitless analysis (deactivated, large volume).
- Verify that the oven starting temperature is less than the solvent boiling point.
- Check that the solvent vapor volume does not exceed the liner capacity.
- Check for appropriate 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 and liner.
- 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.



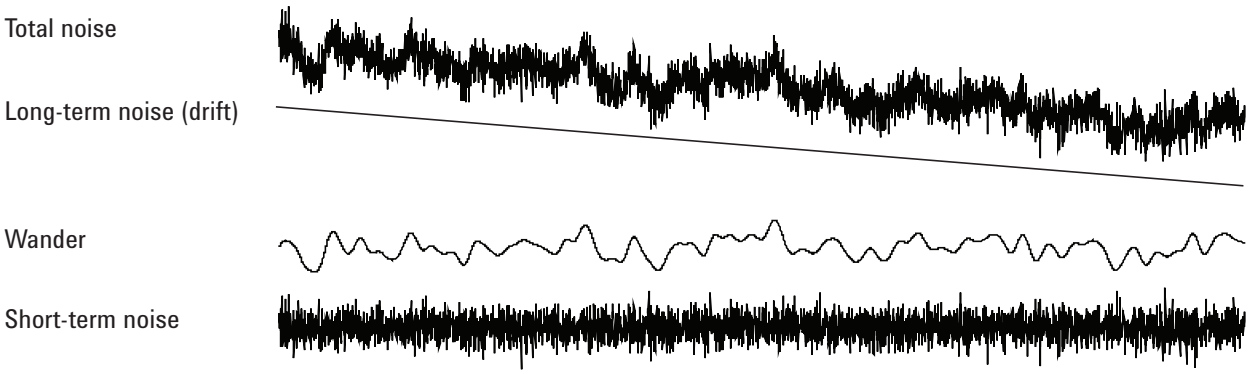
If peak fronting or overloading occurs, try the following:

- 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.
- 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 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. (See the 9000 Series Maintenance manual.)
- 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:

- 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.

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.

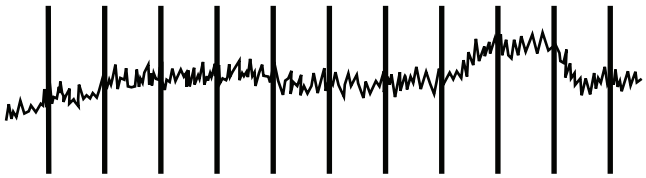


Figure 1 Cyclic spiking

Cyclic spiking can be caused by the following:

- An electric motor
- Building heating/cooling system
- Other electronic interferences in the lab

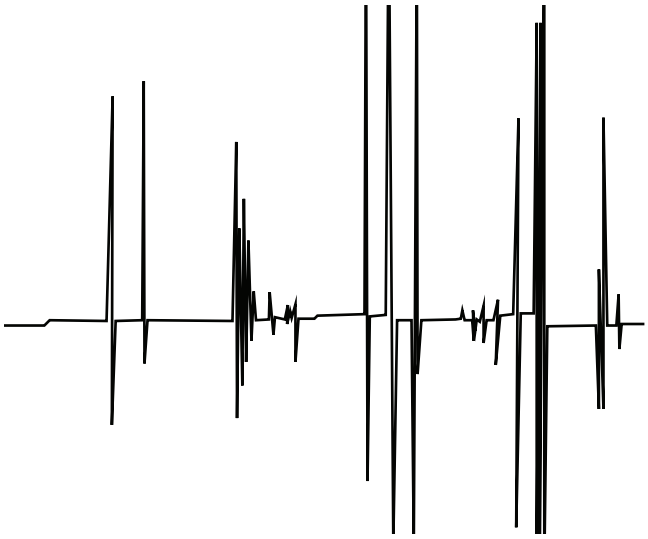


Figure 2 Random spiking

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.
- Check that the detector temperature is not too low.

Low Peak Area or Height (Low Sensitivity)

- If using an inlet in split mode, check the split ratio.
- Check for leaks.
- Check the inlet for contamination. (See "Contamination or Carryover".)
- 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:

- Check for a dirty jet.
- Check for contaminated detector parts.
  - To Check for a Plugged FID Jet

If using an NPD:

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

If using an FPD^{+}:

- 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.

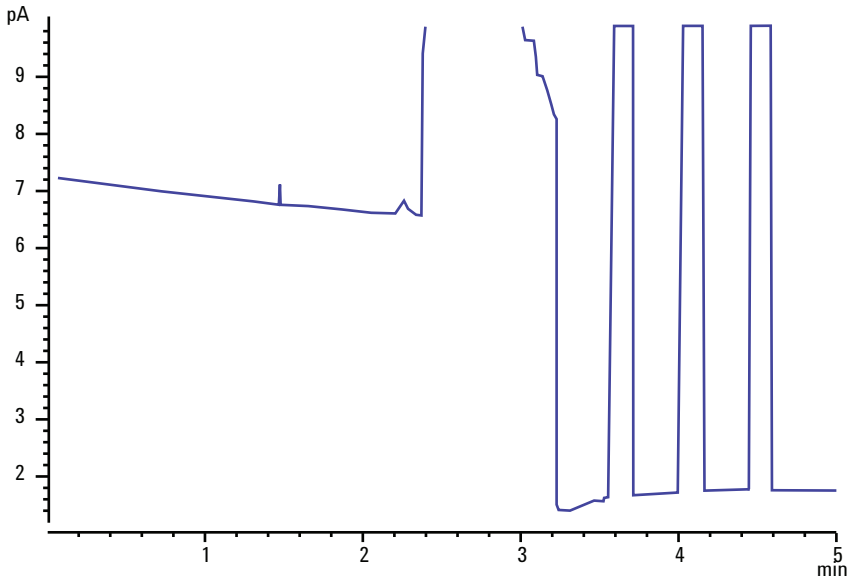
3 Check inlet parameters that control venting, such as split ratio and splitless purge delay time. Make sure the sample is not being inadvertently vented.

4 Perform inlet maintenance (replace all consumable parts) and pressure test the inlet when completed.

5 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.

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 < 1.0 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 that column is properly installed.
- Check for leaks.

FID Baseline Output Above 20 pA

- Verify the purity of the carrier and detector gas supply. See the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
- 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.
- FPD$^+$: Verify the transfer line temperature is set ≥ 200 °C.

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.
- 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 1.0.

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.

	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.
- Consider the filter type (sulfur or phosphorus).
- Check the system 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

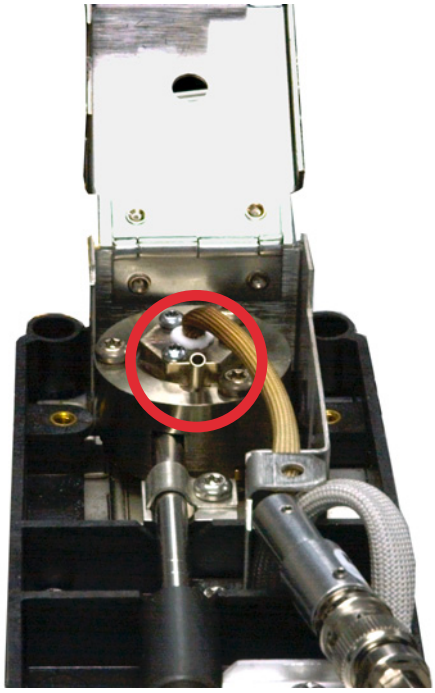
If the baseline does not recover after a solvent peak, try the following:

- Turn 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.
- A large concentration of solvent has extinguished the hydrogen/air plasma. Increase the bead voltage. 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.
- If the upper ceramic insulator is contaminated, a high offset (2 to 15 pA or more) will occur when the bead is off. This directly affects sensitivity. Replace the ceramic insulator.

- Verify that the bead is activated. 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. Reduce the bead voltage to zero to establish a reference level, and then look for a sudden sharp increase in output as the bead voltage increases, which indicates that ignition occurred. If 4 V are being supplied to the bead but it is not igniting, the bead is probably burned out. Replace the bead.



- 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.

NPD Low Selectivity

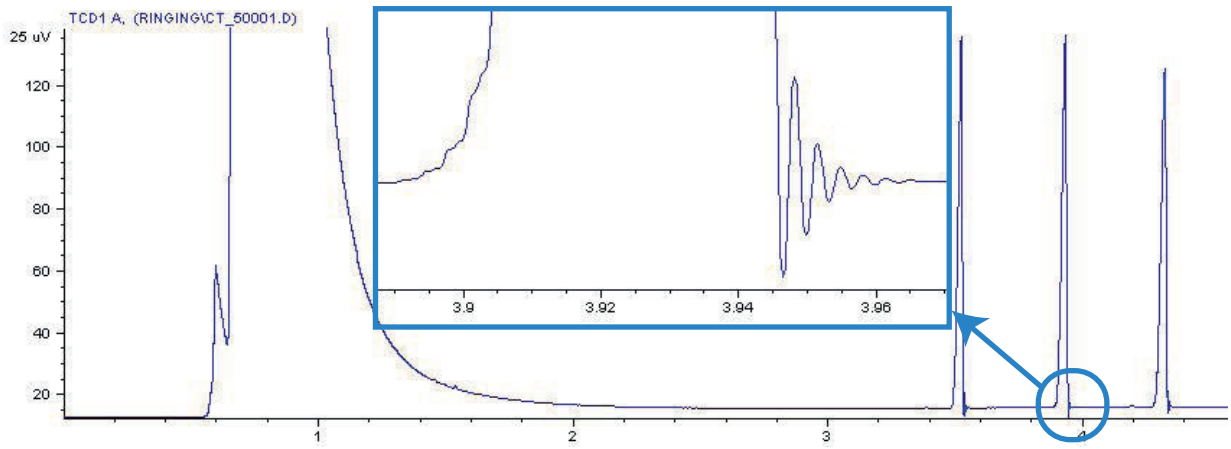
(High hydrocarbon response relative to nitrogen or phosphorus.)

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

Negative Peaks Seen with TCD

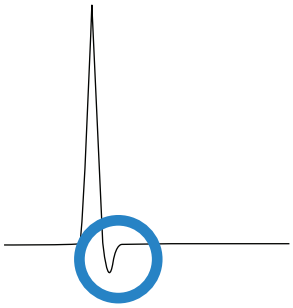
- Verify that the correct gas type is being used.
- Check for a leak in the system.
- Consider thermal conductivity of analytes relative to carrier.
- Check the flow settings, then measure the actual detector flows. (See “To Measure a Detector Flow”.)

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.
- Upgrade the detector to a passivated filament.

4
GC Not Ready Symptoms

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:

- 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 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.

Cannot Set a Flow or Pressure

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

- Check the column mode.
- Check that a capillary column is configured to the correct inlet.
- 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. 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 a defective inlet or detector pressure sensor.

If you are using a split/splitless inlet, MMI inlet:

- 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 or MMI inlet:

- Decrease the split ratio.
- Replace the split vent filter.
- Check the split vent trap line for contamination or an abnormal restriction. Run the split vent restriction test. See:
- Verify that the correct liner is selected.
- Verify the method pressure settings for the SS inlet are above the minimum viable settings available on the GC. See Table 5.

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.

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. 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 or restriction in the split vent line.
- Replace the guard chip or jumper chip.

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.
- 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 ≤ 1.0 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.)
- Check that the air and hydrogen pressures meet Agilent's recommendations (hydrogen > 35 psi [210 kPa] and air > 55 psi [380 kPa]). See the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
- 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 or a weak ignitor.
- 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.)
- 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.
- 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.

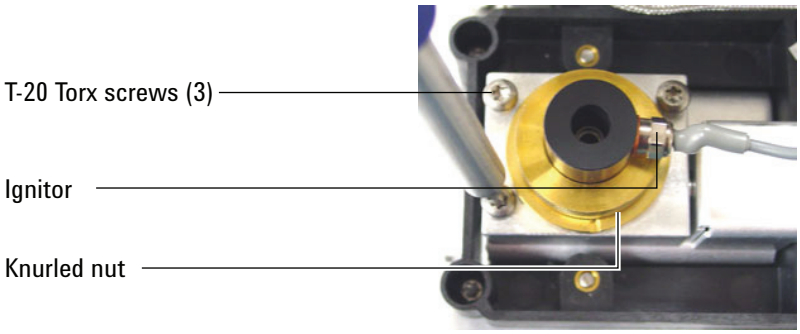
2 Turn the FID flame **On**.

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"

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.

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.

Table 5 FPD+ recommended flows

	Sulfur mode flows, mL/min	Phosphorus mode flows, mL/min
Carrier (hydrogen, helium, nitrogen, argon)		
Capillary columns	1 to 5	1 to 5
Detector gases		
Hydrogen	60	60
Air	60	60
Carrier + makeup	60	60

- Measure the actual detector flows. (See To Measure a Detector Flow.)
- Check that the FPD+ ignitor operates. (See To Verify FID Ignitor Function During Ignition Sequence.)
- 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 column and makeup flow rates.
- Check the **Lit offset** value. The typical **Lit offset** value is 1.0. 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.
- Try increasing the supply pressures to the FPD+ flow module. This makes the flame easier to light without changing the setpoints.

- Under some operating conditions, the flame will light more easily with the vent tube removed. After lighting the flame, reinstall the vent tube.
- Try changing to the phosphorus mode flows, lighting the flame, and gradually alter the flows to the sulfur values.
- Check cable connections to coupling, coupling connection to glow plug, tight glow plug.

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.

Blinking Not Ready Light: Detector Hardware Fault/TCD Filament Voltage

If the TCD filament is open, the Not Ready LED blinks and the GC will not become Ready.

5
Shutdown Symptoms

Column Shutdowns

The GC monitors inlet and auxiliary gas streams. If a carrier gas (which can include an auxiliary flow module or pneumatics control 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.
- Flashes oven temperature setpoint **Off**.
- Turns off all flows for the column. When viewed, their parameters flash **Off**. For example, the septum purge and 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.
- Communicates with the configured MS, if present, so the MS can react to the shutdown event.

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. See the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
   - Check for a broken column.
   - Check for leaks.
   - Replace the inlet O-ring.
   - Check the supply pressure.
2. Press the key for the device that initiated the shutdown.

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. When viewed, these parameters will flash **Off**.
- Opens the split vent valves in the split/splitless inlets.
- Turns off the oven heater and fan.
- 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**.
- 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.
- Communicates with a configured MS, if present, so the MS can react to the shutdown event.

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 O-ring. See the Maintenance manual.
   - Check for broken column.
   - Check the supply pressure. Make sure the gas supply meets the pressure recommendations listed in the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
   - Check the system for leaks.
2 Power cycle the GC.
3 Turn the flow back on.

Detector Air Shutdowns

When air is used to combust a fuel gas (hydrogen) in a detector, an insufficient air supply will cause a shutdown for the air channel. In addition, the **Air** and **Hydrogen** flows for the associated detector will be turned **Off**.

To recover from this state.

1. Fix the cause of the shutdown. Verify the air supply. The FID and NPD require a delivered gas pressure of 380–690 kPa (55–100 psi). The FPD^+ requires a delivered gas pressure of 690–827 kPa (100–120 psi). See the *Agilent Intuvo 9000 GC, GC/MS, and ALS Site Preparation Guide*.
2. Turn the flow back on.

9000 MS Shutdown

If the 9000 receives a shutdown event from, or loses communications with, a configured MS, the GC reacts with changes such as:

- Turning off the column oven.
- Setting low pressures/flows for the MS flow path.
- Turning off hydrogen carrier gas flow, if used.
- Aborting a current run.
- Turning off the MS transfer line heater.
- Blocking all setpoint changes.

The exact changes depend on the event that triggered the shutdown. For example, the GC may react differently to a communications loss than to a failure in the MS high vacuum pump.

When troubleshooting an MSD Shutdown:

1 Check all GC, MS and data system events and logs. The GC will enter MS Shutdown if:
   - The GC cannot maintain carrier flow into the MS.
   - The MS reports a shutdown or fault.
   - The GC and MS lose communications with each other.

2 Check the current communications status.

   7000D, 7010B:

   a The **MS Communication** setting should read **Connected**. If not, check the entered GC and MS IP addresses in the GC, the MS, and the PC. All GC and MS IP addresses must match in all three places.

   b Check the GC and MS LAN connections. Is the LAN switch or hub operating?

   5977B:

   a The **Lvds communication** setting should read **On**.

   b Check the GC and MS LAN connections. Is the LAN switch or hub operating?

Clearing an MS Shutdown

Unlike other shutdowns, you cannot clear this state by turning off a setpoint (because setpoint changes are blocked). Instead, clear this state by disabling GC to MS communications.

If the GC started the shutdown, you can now troubleshoot the problem and resolve the GC fault.

After resolving an MS Shutdown

After resolving the GC or MS problem, always restore GC to MS communications.

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
GC Power On and Communication Symptoms

GC Does Not Turn On

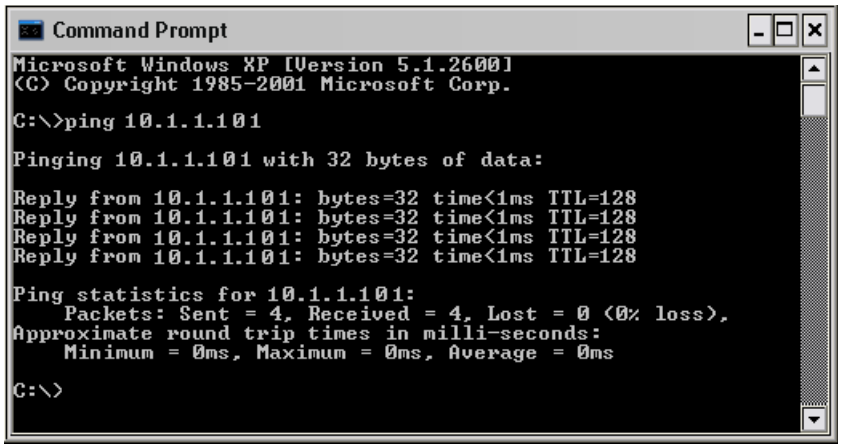
If the GC does not turn on:

- 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. (If using a client/server data system, run the ping commands from the controller PC, since the controller PC blocks normal LAN access to the GC.) For example:



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

If the problem is that a data system cannot connect to the GC, check if another PC is controlling the GC.

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.
- If using a direct PC to GC setup, make sure you are using a crossover cable. If using a setup with a hub or switch (that is, connecting to a building or site LAN), make sure you are NOT using a crossover cable.

GC Cannot Communicate with a Configured MS or HS

1. First, check that GC to MS (or GC to HS) communications are enabled.
2. **Ping** each instrument from a computer on the same local LAN. (See "PC Cannot Communicate with GC" for more information about the PC **ping** command.)
3. If using an Agilent data system, check the GC, HS, and MS IP addresses entered into the data system software. Again, these must exactly match the values entered into each instrument.

GC Does Not Recover After Firmware Update

If the GC starts but does not display the “Power on successful” message, look for any error messages. Record any messages. Then resolve the problem as follows:

1 Try power cycling the GC.

2 If the update fails again, contact Agilent for service.

GC Turns On, Then Stops During Startup (During Self-Test)

If the GC turns on but the normal display does not appear:

1. Turn the GC power switch **Off**. Wait one minute, then turn the GC power **On**.
2. If the GC does not return to normal, record any messages that appear on the display.

GC Features Missing or Disabled

The GC provides features for the currently-configured components. Sometimes, if a component is broken, it will no longer be considered a part of the GC and therefore its UI components will not appear. Help and information is still available from the GC for this component: see the browser interface help topics on filtering information, and see the PDF versions of the manuals available through the browser interface.

Also, when controlled by a data system, the data system can disable certain GC features. See "Data System Feature Selection" on page 18 for more information.

7
Troubleshooting Tasks

To Measure a Column Flow

Measuring FID, TCD, and FPD+ column flow

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

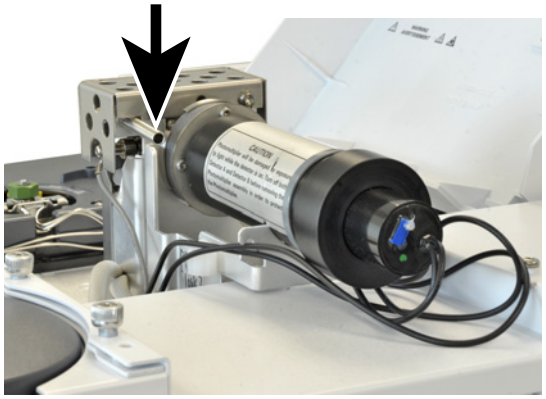
1 Gather the following:
   - Appropriate flowmeter adapter tube (can be found in the GC ship kit)
   - Electronic flowmeter calibrated for the gas and flow rates of concern
2 Turn off the detector.
3 Turn off the detector flows.
4 Connect the appropriate adapter to the detector exhaust.

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 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 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.**

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

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

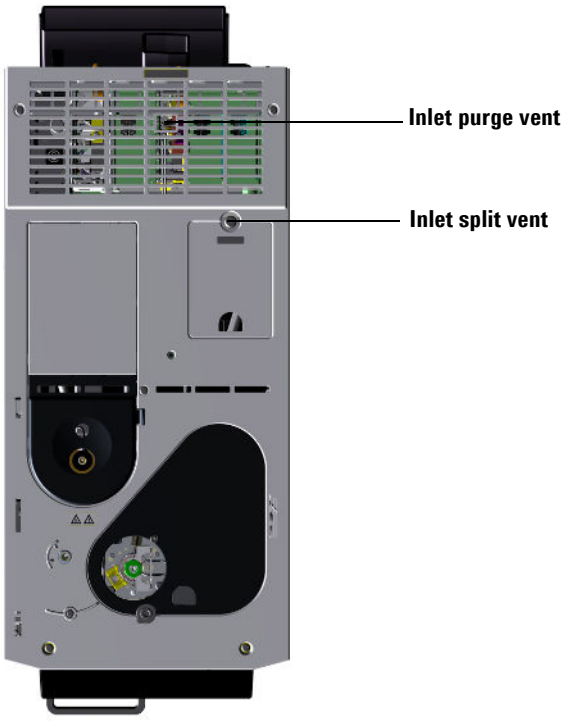
5 Place the flowmeter tubing over the flow-measuring insert to begin measuring flows.

To Measure a Split Vent or Septum Purge Flow

Note that the GC reports flows calibrated to 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.**

See the figure below.



To measure split vent or septum purge flows, attach the flowmeter to the appropriate tube.

- 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.

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 (all detectors)
- A plugged jet.
- A leak in the burner chamber, window seal, or ignitor seal (FPD+)
- A pressure sensor that needs to be zeroed.
- An EPC 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, 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 (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 Shut off all detector gases.

5 Turn off (where applicable): the FID flame, FPD+ flame, and TCD filament.

6 Cool the detector.

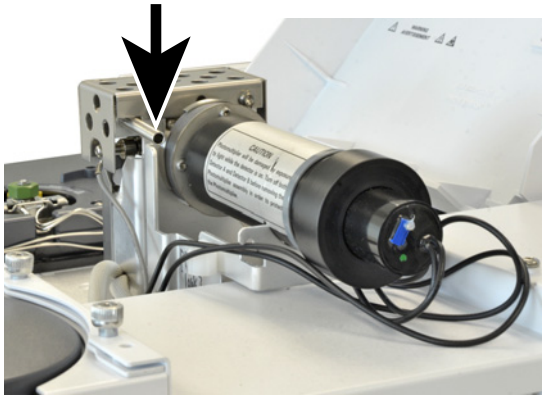
7 Connect the appropriate adapter to the detector exhaust.

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

A rubber adapter tube attaches directly to a 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 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.**

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

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

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

To Perform the GC Self-Test

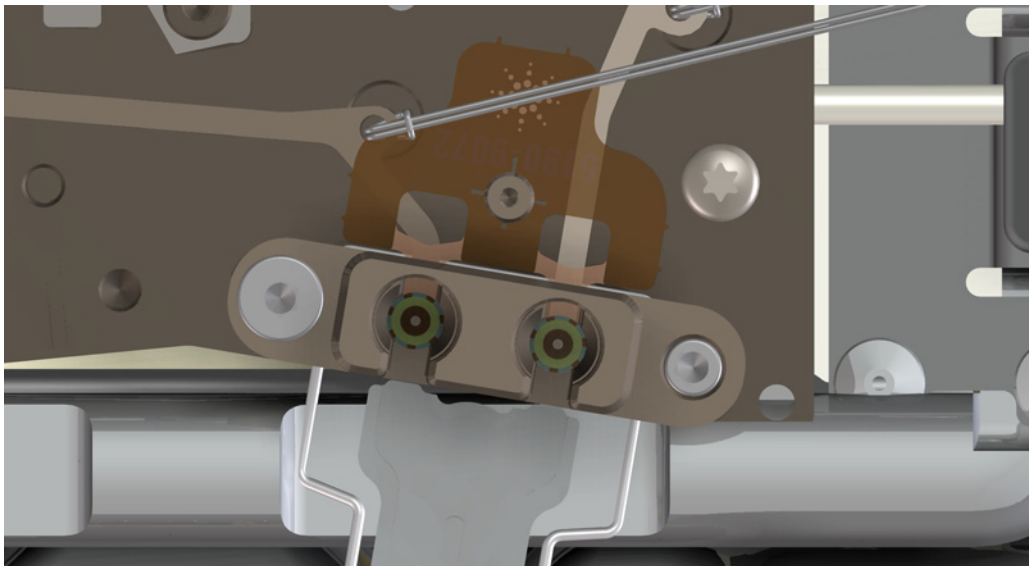
1 Turn the GC off.

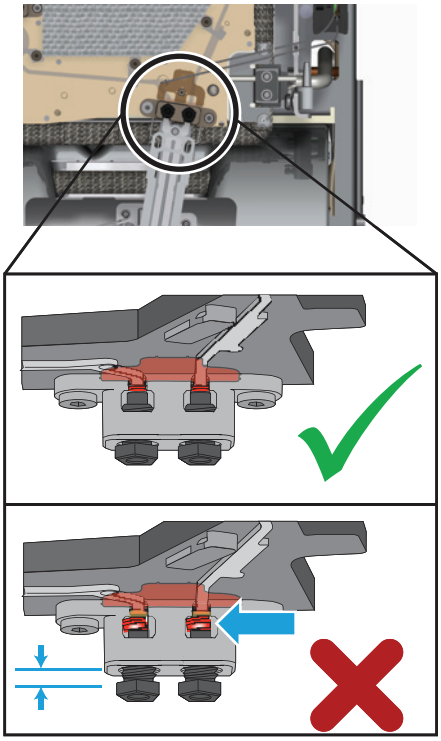
2 Wait 1 min, then turn the GC back on. If the main GC screen appears, the GC has passed the self-test.

To Correct a Leak at a Click and Run Fitting

If a leak is found at a click-and run fitting on a bus:

- Remove the compression bolts. Check that the mating surfaces of the bottom flow chip, gasket, and column or top flow chip are concentrically aligned and laying flat against each other. See the figure below. The stacked parts should rest flat against the bus.





- Replace the gasket.
- Replace the compression bolts and the gasket.
- Check the flow chip sealing surfaces.

For a guard chip, replace the guard chip. When installing the new chip, be sure that the guard chip is flat and level between the inlet base and the little bus. See the maintenance documentation for details.

Agilent recommends replacement of all Intuvo flow path parts (flow chips, columns, and detector tail) when changing between polyimide gaskets and nickel gaskets.

To Adjust the FID Lit Offset

On the GC touch screen, go to **Settings > Configuration > Detector** and check the lit offset.

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 < 1.0 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 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.
4. 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. When the flame is lit and the GC is ready, verify that the output is stable and < 20 pA (this may take some time).
4. If the output is not stable or > 20 pA, the system or gas may be contaminated.

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 noise test still fails, see To Measure FID Leakage Current. 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**.

- Leave the NPD at operating temperature.
- Leave flows on or off.

3 Verify that the output (leakage current) is stable and < 1.0 pA.

4 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. Set the makeup flow to “Off”. Confirm a reading on the GC display of 0.0 mL/min for actual makeup flow.
2. Set the hydrogen flow to 75 mL/minute (increase the H2 supply pressure as needed to achieve this flow rate setting.)
3. 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 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.

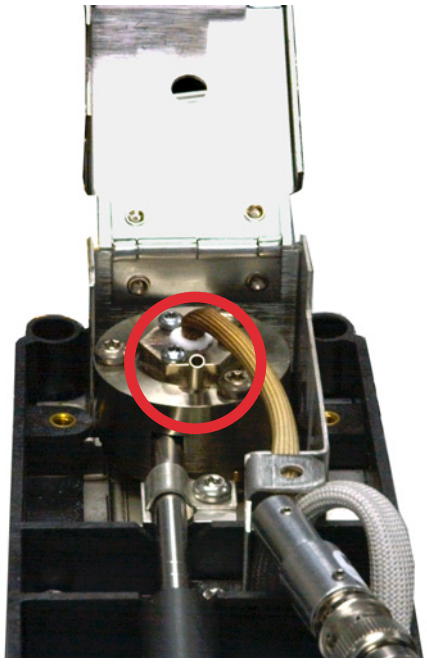
To Check for a Plugged NPD Jet

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 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.

Agilent Technologies

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.

Printed in USA, September 2021