Agilent

Agilent 8860 Gas Chromatograph

# Operation Manual

# Notices



## Manual Part Number

G2790-90014

## Edition

Second edition, July 2019
First edition, January 2019

Printed in USA or China

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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 Getting Started



This document provides an overview of the Agilent 8860 Gas Chromatograph (GC) along with detailed operating instructions.

# The 8860 Gas Chromatograph


Figure 1. The 8860 GC

# Before Operating Your GC

Before operating your GC, be sure to read the safety and regulatory information included on the *Agilent GC and GC/MS User Manuals & Tools* DVD, the Browser Interface or a connected web browser. The most common safety hazards when working on the GC are:

- Burns caused by touching heated areas on or in the GC.
- Release of pressurized gas containing hazardous chemical compounds caused by opening inlets.
- Glass cuts or puncture wounds caused by sharp capillary column ends.
- Use of hydrogen as a GC carrier gas.

# Chromatography Using a GC

Chromatography is the separation of a mixture of compounds into individual components.

There are three major steps involved with separating and identifying components of a mixture using a GC. They are:

1. **Injecting** a sample into the GC. (This takes place at the inlet.)
2. **Separating** the sample into individual components. (This takes place inside the column in the oven.)
3. **Detecting** what compounds were in the sample. (This is done in the detector.)

During this process, status messages from the GC are displayed, and user changes to parameter settings can be made through the Browser Interface or the data system.

# Inlets

Inlets are where samples are injected into the GC. The Agilent 8860 GC can have a maximum of two inlets, identified as Front Inlet and Back Inlet.

The following inlet types are available:

- Split/splitless inlet (SSL)
- Purged packed inlet (PP)
- Packed column inlet (PCI)
- Cool on column inlet (COC)

The type of inlet chosen is based on the type of analysis being done, the type of sample being analyzed, and the column being used.

Samples can be injected into the inlets by hand using a syringe, or an automatic sampling device (such as an Agilent Automatic Liquid Sampler or Agilent Headspace Sampler).

## Automatic injectors

The Agilent 8860 GC can accommodate up to one autoinjector, which may be used in either the front or rear position.

## Automatic sampling valves

The optional sampling valves are simple mechanical devices that introduce a sample of fixed size into the carrier gas stream. Valves are most frequently used to sample gases in constantly flowing streams.

The Agilent 8860 GC can accommodate up to three total valves. Up to two non-heated liquid sampling valves, or up to three gas sampling valves may be used in any combination.

The valves are located inside the sampling valve box.

# GC Columns and Oven

GC columns are located inside a temperature-controlled oven. Generally, one end of the column is attached to the inlet, while the other end is attached to the detector.

Columns vary in length, diameter, and internal coating. Each column is designed for use with different compounds.

The purpose of the column and the oven is to separate the injected sample into individual compounds as it travels through the column. To aid this process, the GC oven can be programmed to speed the sample flow through the column.

The Agilent 8860 GC can accommodate up to three columns, identified as Column #1, Column #2, and Column #3.

# Detectors

Detectors identify the presence of compounds as they exit the column.

As each compound enters the detector, an electrical signal proportional to the amount of compound detected is generated. This signal is generally sent to a data analysis system—such as Agilent OpenLAB CDS ChemStation edition—where it shows up as a peak on a chromatogram.

The GC can accommodate up to three detectors (two mounted on top of the GC, and one mounted in a side carrier, or in the aux position on the top), identified as Front Detector, Back Detector, and Aux 2 Detector.

A complete selection of detectors (FID, TCD, NPD, FPD+, ECD, and MSD) are available. The type of detector chosen is based on the type of analysis required.


Figure 2. Detector locations

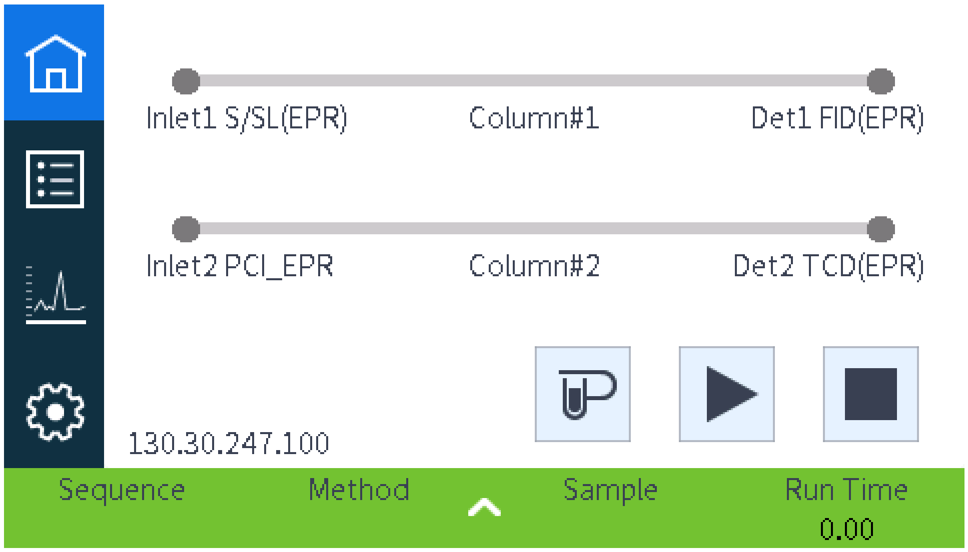
**NOTE**

**A side mounted detector may only be a TCD, ECD, or FID. Other detector types are not supported in this location.**

**An Aux Detector mounted on top may only be a FPD+ or a TCD.**

# Touchscreen

The touchscreen shows GC status and activity information, and allows you to start, stop, and prepare the GC to run a sample.




See **"Touchscreen Operation"** for a detailed description of the touchscreen functions and capabilities.

# System Operation

The GC can be controlled using the touchscreen, the Browser Interface, and an Agilent data system.

## Touchscreen

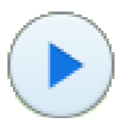
The touchscreen provides direct control of configuration settings, access to diagnostic and maintenance functions, logs, and access to help, as well as the ability to make temporary changes in setpoints. Use the touch display to:

The touchscreen provides controls for:

- Setting the GC IP address
- Selecting the touchscreen language
- Viewing status data and real-time plots.
- Monitor GC and run status
- Monitor system health
- Make system settings, for example system locale, IP address setup, and so forth

The home screen provides basic run control:

**Prep Run:** Typically required before manual injections to exit any gas saver mode and to prepare the inlet flows for injection.



**Start**

Some tasks, such as setting the IP address, can only be performed using the touchscreen.

## Browser Interface

The Browser Interface provides many of the same functions as the touchscreen.The Browser Interface provides for instrument setup and control, as well as the ability to run the instrument stand-alone (without a connected data system). The Browser Interface can be viewed using any typical web browsing device, such as a computer or tablet, provided the device is connected to the same gateway as the GC. Use the Browser Interface to:

- Create methods
- Run samples and sequences of samples
- Run diagnostic tests
- Check status and performance of the GC
- Perform maintenance procedures

- Check maintenance details and reset counters
- Change language settings
- Access on-line help

To connect to the Browser Interface:

1. On a a tablet or computer connected to the same gateway as the GC, open a browser window.
2. Navigate to **http://GC Name or IP Address**. For example, if the GC IP address is 10.1.1.100, navigate to **http://10.1.1.100**.
3. If prompted, enter the access code. (The access code is available at the GC touchscreen.)

For more information regarding the Browser Interface, see **"Browser Interface"**.

When a data system is controlling the GC, setpoint changes and other features may be disabled in the Browser Interface. If multiple users connect to a GC using the Browser Interface, only the first connection has normal control.

# Data System

The data system adapter provided with Agilent data systems, such as OpenLAB CDS, provides full control of the GC for creating methods, running samples, and so forth. Use the data system to:

- Create methods
- Run samples and sequences of samples
- Monitor GC and run status
- Monitor system health
- Track consumables usage and usable life remaining
- Make some system settings
- View system logs
- Set GC clock table and resource conservation events

In addition, the data system adapter provides access to the complete help and user information. From anywhere within the method editor for the GC, select **Help and Information Browser Interface** from the navigation tree.

Also note:

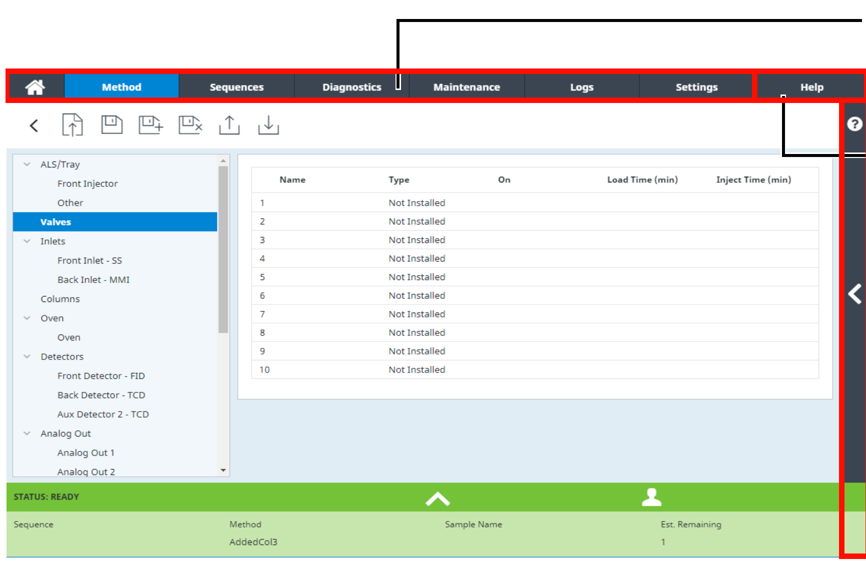
- The data system adapter does not provide direct access to all of the GC's diagnostic and maintenance features.
- The data system cannot directly use any methods or sequences created in the Browser Interface.
- When connected to the GC, the data system adapter can be set to restrict anyone from making certain changes at the touchscreen or with a Browser Interface.

# Browser Interface

You can control and monitor the GC using a web browser that is on the same gateway as the GC. An internet connection is not required. This Browser Interface can be accessed by using computer browser clients and mobile device client browsers, such as tablets. The Browser Interface provides complete control of the GC. Use it to perform tasks such as:

- Configuring GC gas types and flows
- Performing automated maintenance procedures
- Running diagnostic tests
- Creating, editing, and loading methods
- Running samples
- Creating, editing, and loading sequences of samples
- Monitoring GC performance (viewing logs, current statuses, and plots)

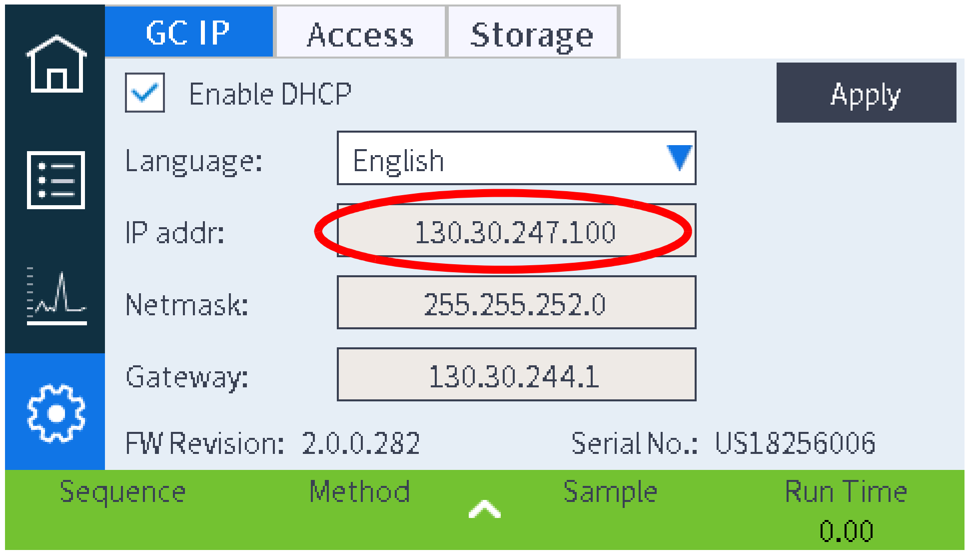
While connected to your GC with a data system, you can not edit methods, edit sequences, and start or stop runs using the Browser Interface. See **"What is a Method?"** for more details.




When using the Browser Interface, the GC stores run data internally. Access can be granted to allow a connected computer on the same gateway to access this data and copy it for analysis. Note that run data can only be deleted by using the Browser Interface directly, or by the GC according to either date or disk space settings.

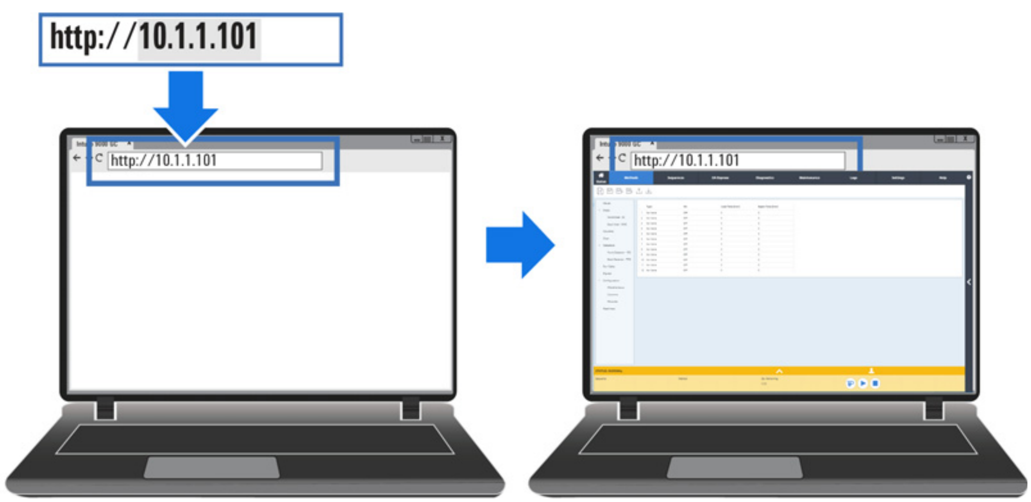
To connect to the GC using a browser:

1 If you do not know the GC's IP address or host name, use the touchscreen to find it.




2 Open a web browser. Supported browsers include Chrome, Safari (on a tablet), Internet Explorer 11, and Edge. Make sure the browser version is up to date.

3 Enter `http://xxx.xx.xx.xxx`, where `xxx.xx.xx.xxx` is the GC's IP address. (If using a host name, enter it instead.) In this example, the GC IP address is 10.1.1.101. Accessing the Browser Interface only requires that the tablet or computer be connected to the same gateway as the GC; no internet connection is required.




For more information on how to utilize the Browser Interface, click the Help tab to access the Help & Information suite, or click the **<** on the right side of the screen to access the context sensitive help. See **"Help from a Browser"** and **"Context-Sensitive Help"** for more information.

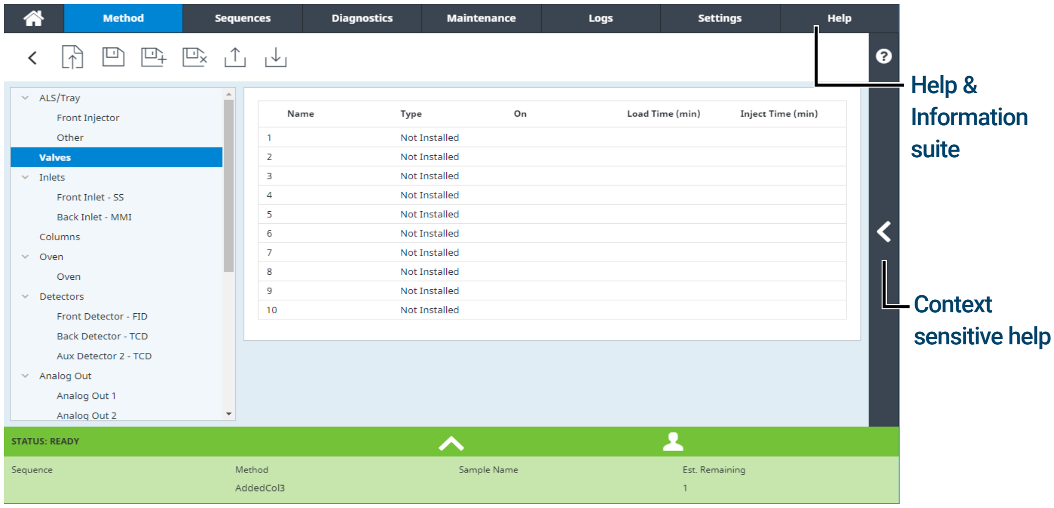



Figure 3. Accessing help from the Browser Interface

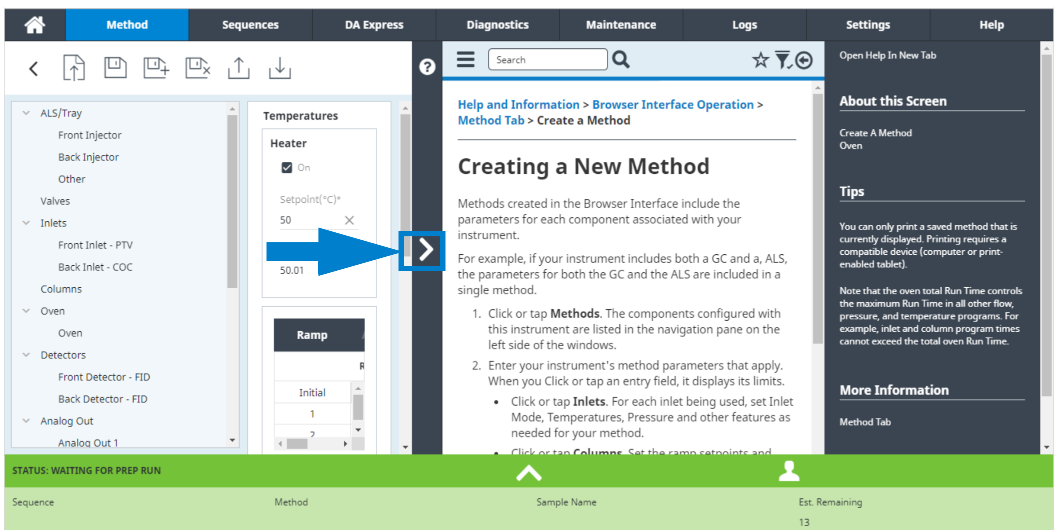



Figure 4. Context sensitive help

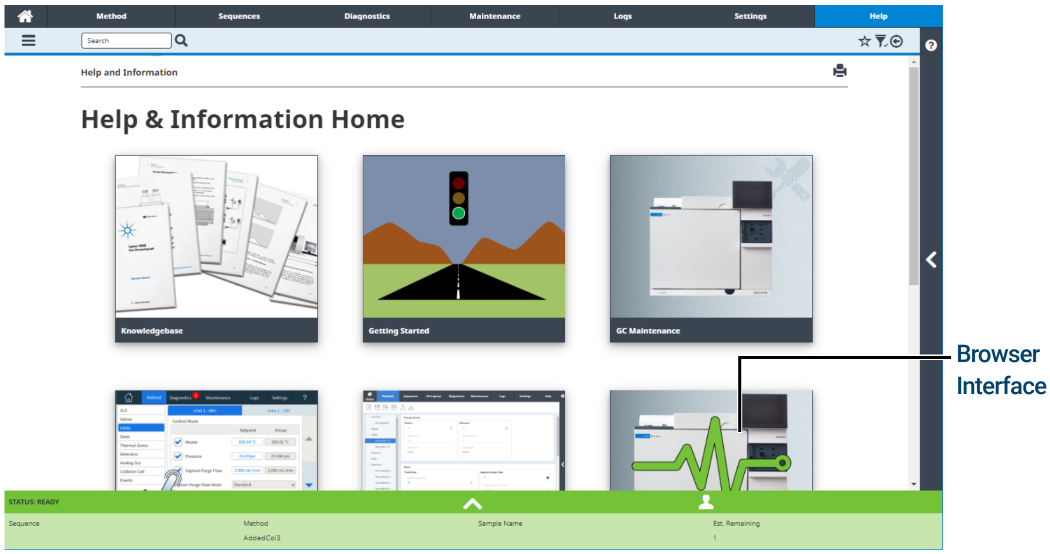



Figure 5. Complete Help and Information content

# Status Indicator

The GC includes a status indicator on the front panel to allow you to quickly determine the status and readiness of the GC. The status indicator changes color depending on the current state of the GC.

- Green: Indicates that GC is ready for operation.
- Yellow: Indicates that the GC is not ready for operation. Power is on and available, but not all parameters have reached operating setpoints. A warning or other message may exist. Check the GC touchscreen for additional information.
- Red: Indicates a fault or other serious condition. A fault or other message may exist. Check the GC touchscreen for additional information. The GC cannot be used until the fault condition is resolved.

# GC Status

When the GC is ready to begin a run, the Browser Interface shows **STATUS: READY FOR INJECTION**. Alternately, when a component of the GC is not ready to begin a run, the Browser Interface shows **STATUS: NOT READY** and the status indicator on the GC front panel is yellow. Clicking the **Diagnostics** tab displays indications of why the GC is not ready.

## Alert tones

The GC provides information via beeps.

*A series of warning beeps* sounds before a shutdown occurs. The GC starts with one beep. The longer the problem persists, the more the GC beeps. After a short time the component with the problem shuts down, the GC emits one beep, and a brief message is displayed. For example, a series of beeps sounds if the inlet gas flow cannot reach setpoint. The message **Inlet flow shutdown** is briefly displayed. The flow shuts down after 2 minutes.

**WARNING**

**Before resuming GC operations, investigate and resolve the cause of the hydrogen shutdown.**

*One beep* sounds when a problem exists, but the problem will not prevent the GC from executing the run. The GC will emit one beep and display a message. The GC can start the run and the warning will disappear when a run starts.

Fault messages indicate hardware problems that require user intervention. Depending on the type of error, the GC emits no beep or a single beep.

## Error conditions

If a problem occurs, the GC status bar changes to Not Ready, the GC status indicator turns yellow, a number appears next to the Diagnostics tab, and the status tray and Diagnostics tab list the condition that is causing the Not Ready condition. Select **Diagnostics** to view the issue and resolve it.

## Clearing a shutdown condition

When a component is shut down, the GC becomes Not Ready, the status indicator and status bar turn yellow, and the Diagnostics tab and status tray display messages about the condition that caused the shutdown.

To clear a shutdown, select **Diagnostics** > **Clear shutdown - ON** to turn on all zones, including the one that was shut down, or select **Clear shutdown - OFF** to turn on all zones except the one that was shut down. Note that if you clear the shutdown but do not resolve the problem that caused it (for example, changing a gas supply cylinder or fixing a leak), the GC will shut down again.

# Overview of Running a Sample

Operating the GC involves the following tasks:

- Setting up the GC hardware for an analytical method.
- Starting up the GC. See **"To Start Up the GC"**.
- Preparing any attached sampler. Install the method-defined syringe; configure solvent and waste bottle usage and syringe size; and prepare and load solvent, waste, and sample vials, as applicable. Refer to the documentation provided with your ALS or Headspace Sampler (HS) for details on its installation, operation, and maintenance.
- Loading the analytical method or sequence into the GC control system.
    - See the Agilent data system documentation.
    - For standalone GC operation see **"Load a Method"**.
- Running the method or sequence.
    - See the Agilent data system documentation.
    - For standalone GC operation, see **"To manually inject a sample with a syringe and start a run"** and **"To run a method to process a single ALS sample"**.
- Monitoring sample runs from the GC touchscreen, Browser Interface, or Agilent data system. See **"Home View"**, or the Agilent data system documentation.
- Shutting down the GC. See **"To Shut Down the GC for Less Than a Week"** or **"To Shut Down the GC for More Than a Week"**.

# Instrument Control

The Agilent 8860 GC is typically controlled by an attached data system such as Agilent OpenLAB CDS. Please refer to the online help included in the Agilent data system for details on how to load, run, or create methods and sequences using the data system.

# Correcting Problems

If the GC stops operation because of a fault, check the touchscreen or Browser Interface for any messages. The GC includes diagnostics functions to help you determine the cause of a fault.

1. Use the touchscreen, Browser Interface, or data system to view the alert. (See **"Home View"** and **"Diagnostics"** for details.)
2. Tap the stop button on the touchscreen, click the stop button on the Browser Interface, or turn off the offending component in the data system.
3. Diagnose the problem using the built in diagnostics tools on the GC. See **"Diagnostics"**.
4. Resolve the problem by, for example, changing gas cylinders or fixing the leak.

Once the problem is fixed, you may need to power cycle the instrument. Most shutdown errors can be cleared from the LUI, but some may require a power cycle.

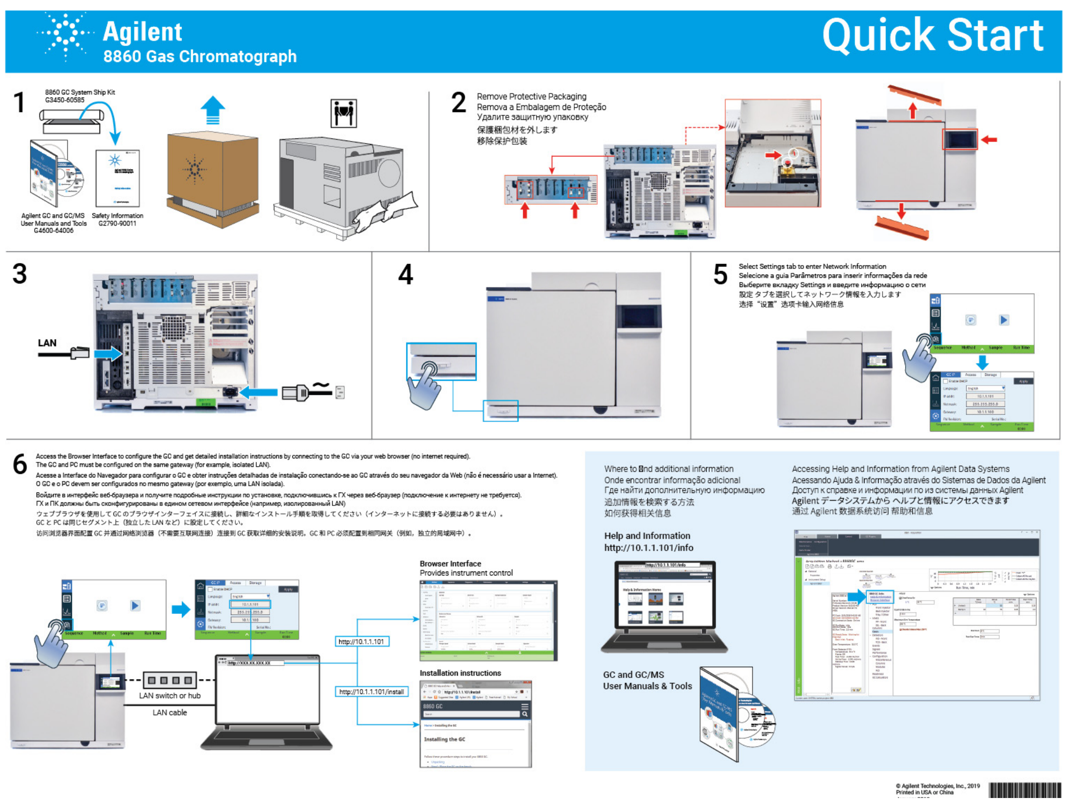
# 2 Help and Information

# Where to Find Information

Agilent provides an extensive amount of documentation for installing, operating, and maintaining the instrument directly from the 8860 GC. In addition, there are multiple ways to access the comprehensive Help and Information suite either through a browser, or from your Agilent data system.

- **"Help from a Browser"**.
  The complete set of user information is available directly from the GC by using a connected web browser.
- **"Context-Sensitive Help"**.
  In addition to the complete set of user documentation, context-sensitive information is also available from the Browser Interface.
- **"Agilent GC and GC/MS User Manuals & Tools DVD"**.
  Information about the 8860 GC, mass selective detectors, and samplers is also available on the Agilent GC and GC/MS User Manuals & Tools DVD.

When unpacking the instrument, make sure you take a look at the provided 8860 GC *Quick Start Poster* to help you quickly get familiar with your GC, as well as setup and configure your instrument. This poster can also be found in your browser help, in the familiarization section.

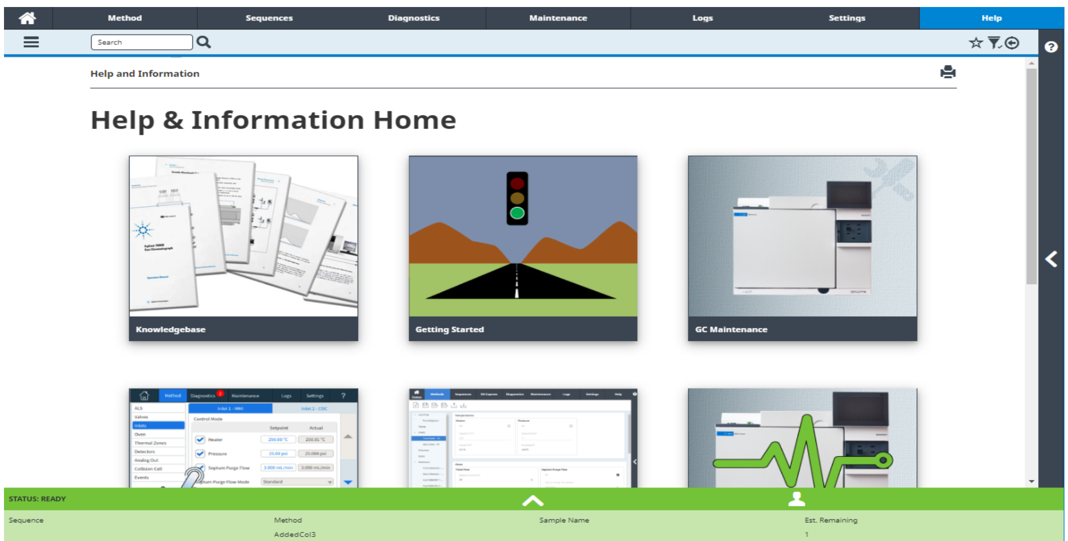


# Help from a Browser

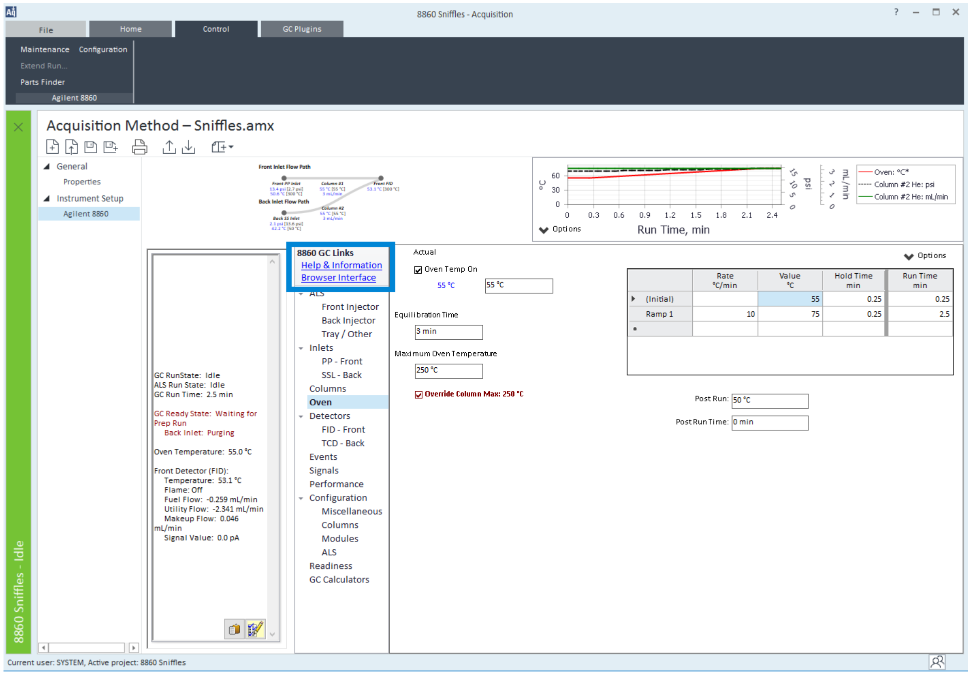
You can access an extensive amount of on-board documentation that is designed to assist with topics such as getting started, familiarization, installation, operation, maintenance, troubleshooting, and other useful information. **Access to the internet is not required to use this enhanced help package**. Accessing this help only requires that your PC or tablet be connected to the same gateway as your GC.

This enhanced version of **Help & Information** can be easily accessed through:

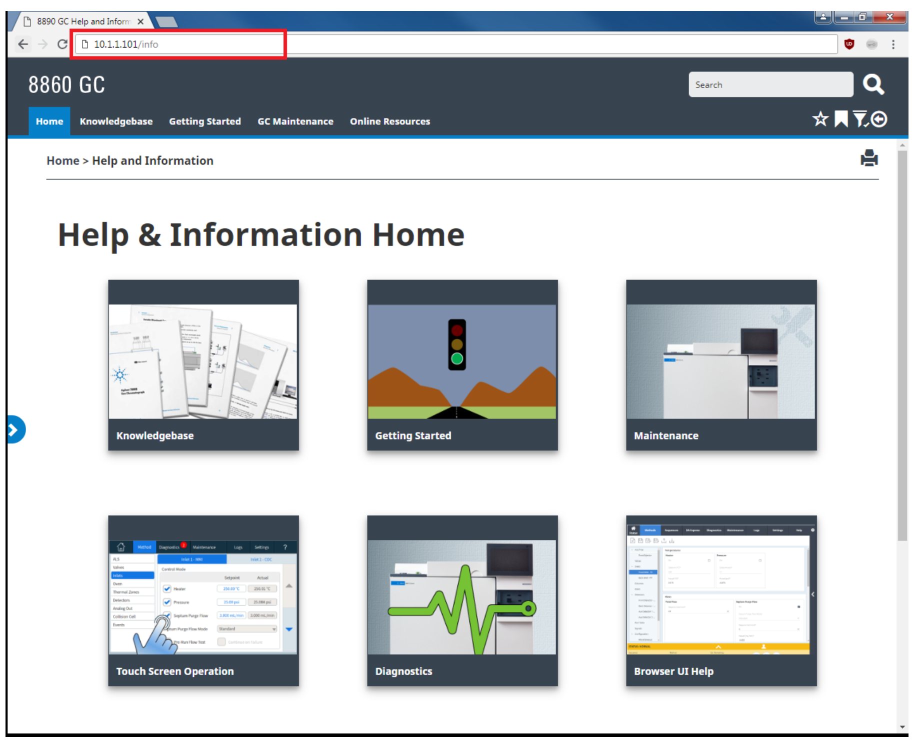
- The Browser Interface. Access the Help & Information suite by clicking the **Help** tab in the Browser Interface. See **"Browser Interface"** for instructions on connecting to the Browser Interface.




- Your Agilent data system. Access the Help & Information suite by clicking the **Help & Information Browser Interface** link at the top of the tree view.




- A web browser on any device that is on the same gateway as the GC. Access the Help & Information suite by typing `http://xxx.xx.xx.xxx/info` into your web browser of choice, where `xxx.xx.xx.xxx` is the IP address or host name of your GC.



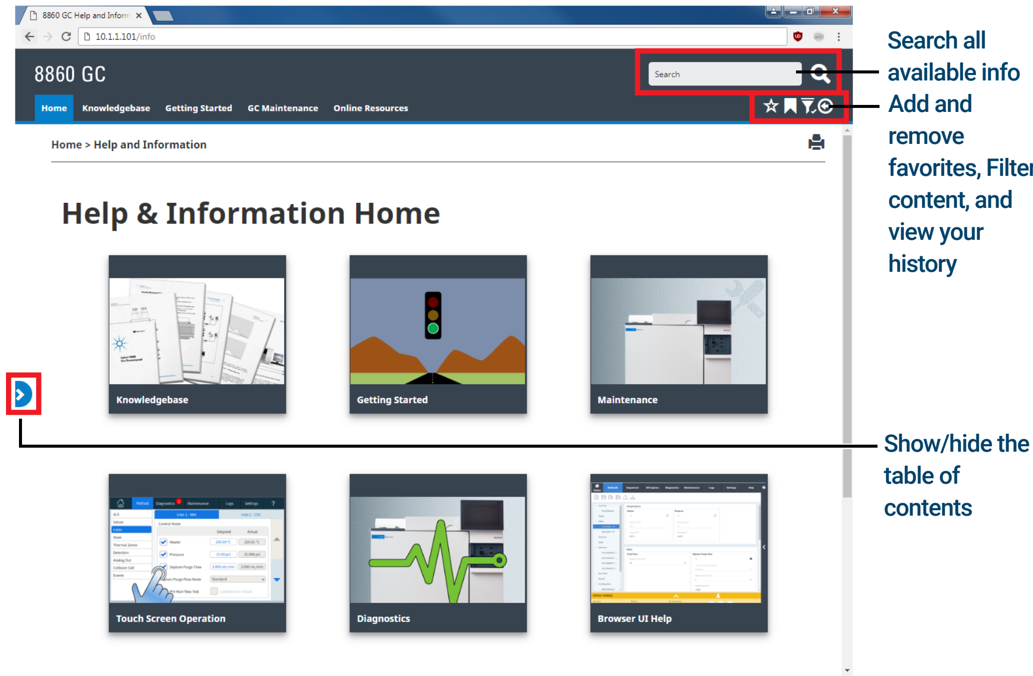

Accessing the Help & Information suite provides you with the following information:

- **Knowledgebase**: manuals, installation info, safety info, and site prep.
- **Getting Started**: BrowserUI tutorial, Quick Start Poster, eFam, a GC Feature Tour, the System Setup Wizard, and instructional videos.
- **Maintenance**: information for performing maintenance on your configuration (inlets, detectors, etc.).
- **System Operation**: information on operating your GC, such as its settings, or EMF counters.
- **Diagnostics**: information on the diagnostic tests, self-guided diagnostics, and tasks available on your GC.
- **Browser Interface Help**: help and instructions for using the Browser Interface.
- **Online Resources**: links to Agilent University, Agilent YouTube, Agilent Community, services, and more.

By default, the help is filtered to only show information pertaining to your GC's configuration. If information does not appear to be available, be sure to enable all help content. To check your active filters, click the icon in the top right corner of the Help & Information screen. These filters allow you to show or hide information pertaining to the various components of your GC, such as information about specific detectors or inlets.

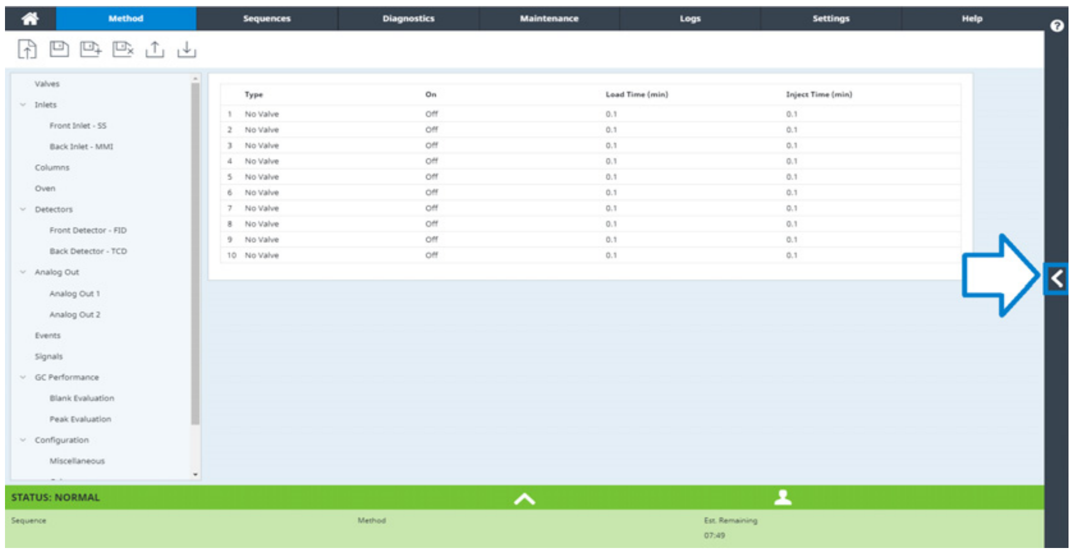
In addition, you can set frequently visited topics as favorites for easy access. To do so, navigate to the desired topic, and click the icon. Once a topic has been added to your favorites, the icon becomes filled in. Click this icon again to remove the topic from your favorites. Your favorites can be viewed at any time by clicking the icon. From here, click any of the listed topics to quickly access them, or click the to remove the topic from your favorites.

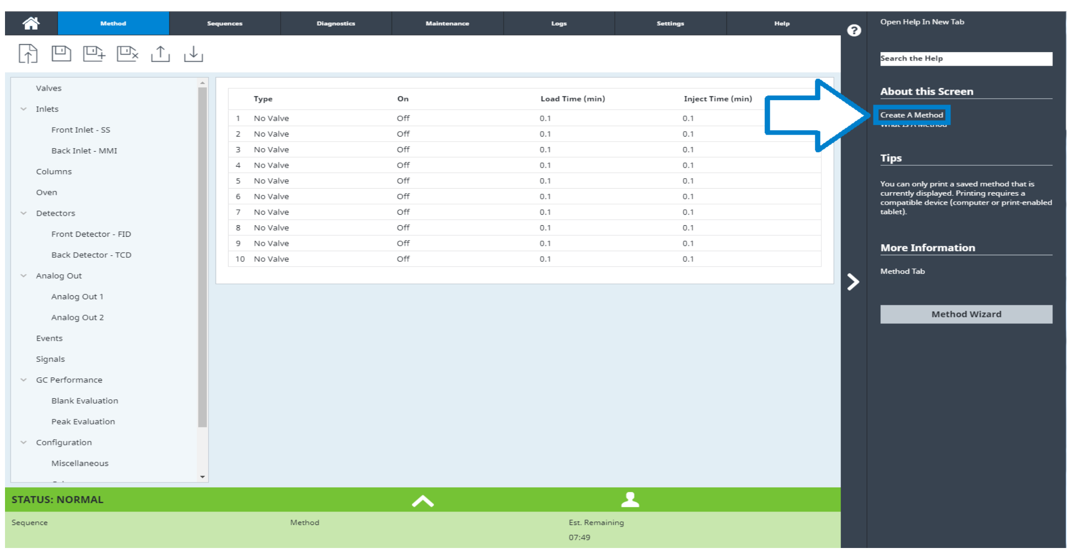
The history icon lists the recent help topics visited in the current browser session. From here, select any of the listed topics to revisit them. Select **Clear History** to remove all topics from your history.


8860 GC Help and Inform
10.1.1.101/info
8860 GC
Search
Home
Knowledgebase
Getting Started
GC Maintenance
Online Resources
Home > Help and Information
Help & Information Home
Knowledgebase
Getting Started
Maintenance
Touch Screen Operation
Diagnostics
Browser UI Help
Search all available info
Add and remove favorites, Filter content, and view your history
Show/hide the table of contents

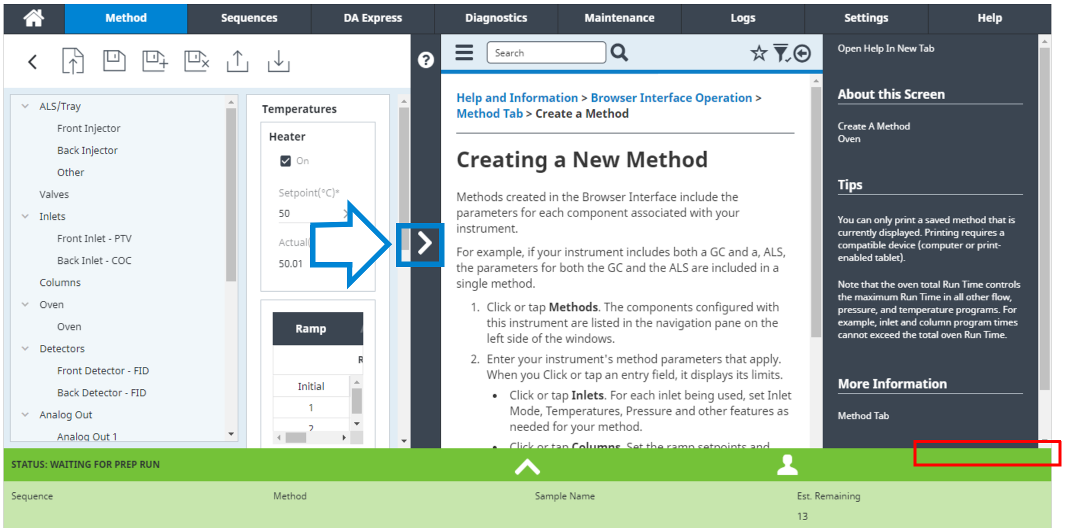
# Context-Sensitive Help

Available from each of the Browser Interface pages is the ability to access context-sensitive information or help. Select the **<** from the right side of the screen to access information and tips relevant to the page you are viewing. About this Screen provides several links to relevant help topics. When clicked, these links open their corresponding topics within the help tray. Under Tips, you will see snippets of useful information about the current page. Additionally, some pages will provide links to additional documentation, such as the link to the Method Wizard in the example below.




To minimize the topic you are viewing, click the **>** between the context sensitive help and the page you are viewing. Click the **>** again to minimize the context sensitive help tray.



Method
Sequences
DA Express
Diagnostics
Maintenance
Logs
Settings
Help
ALS/Tray
Front Injector
Back Injector
Other
Valves
Inlets
Front Inlet - PTV
Back Inlet - COC
Columns
Oven
Oven
Detectors
Front Detector - FID
Back Detector - FID
Analog Out
Analog Out 1
Temperatures
Heater
On
Setpoint(°C)*
50
Actual
50.01
Ramp
Initial
1
Search
Help and Information > Browser Interface Operation > Method Tab > Create a Method
Creating a New Method
Methods created in the Browser Interface include the parameters for each component associated with your instrument.
For example, if your instrument includes both a GC and a, ALS, the parameters for both the GC and the ALS are included in a single method.
1. Click or tap Methods. The components configured with this instrument are listed in the navigation pane on the left side of the windows.
2. Enter your instrument's method parameters that apply. When you Click or tap an entry field, it displays its limits.
• Click or tap Inlets. For each inlet being used, set Inlet Mode, Temperatures, Pressure and other features as needed for your method.
Open Help In New Tab
About this Screen
Create A Method
Oven
Tips
You can only print a saved method that is currently displayed. Printing requires a compatible device (computer or print-enabled tablet).
Note that the oven total Run Time controls the maximum Run Time in all other flow, pressure, and temperature programs. For example, inlet and column program times cannot exceed the total oven Run Time.
More Information
Method Tab
STATUS: WAITING FOR PREP RUN
Sequence
Method
Sample Name
Est. Remaining
13

# Agilent GC and GC/MS User Manuals & Tools DVD

The *Agilent GC and GC/MS User Manuals & Tools* DVD provides an extensive collection of online help and books for current Agilent gas chromatographs, mass selective detectors, and GC samplers. Included are localized versions of the information you need most, such as:

- Getting Familiar documentation
- Safety and Regulatory guide
- Site Preparation information
- Installation information
- Operation guides
- Maintenance information
- Troubleshooting details

# 3 Startup and Shutdown

# To Start Up the GC

Successful operation begins with a properly installed and maintained GC. The utility requirements for gases, power supply, venting of hazardous chemicals, and required operational clearances around the GC are detailed in the *Agilent 8860 Gas Chromatograph Site Preparation Guide*.

1 Check gas source pressures. For required pressures, see the *Agilent 8860 Gas Chromatograph Site Preparation Guide*.
2 Turn on the carrier and detector gases at their sources and open the local shutoff valves.
3 Turn on the GC power. Wait for **Power on successful** to display on the touchscreen.
4 Install the column.
5 Check that the column fittings are leak free.
6 Set a method for the GC to use.
If using a data system, download the method to the GC.
If using the Browser Interface, load a method.
7 Wait for the detector(s) to stabilize before acquiring data. The time required for the detector to reach a stable condition depends on whether the detector was turned off or its temperature was reduced while the detector remained powered.

**Table 1 Detector stabilization times**

| Detector type | Stabilization time starting from a reduced temperature (hours) | Stabilization time starting from detector off (hours) |
|---|---|---|
| FID | 2 | 4 |
| TCD | 2 | 4 |
| ECD | 4 | 18 to 24 |
| FPD+ | 2 | 12 |
| NPD | 4 | 18 to 24 |

# To Shut Down the GC for Less Than a Week

1 Wait for the current run to finish.

2 If the active method has been modified, save the changes.

**WARNING**

**Never leave flammable gas flows on if the GC will be unmonitored. If a leak develops, the gas could create a fire or explosion hazard.**

3 Reduce oven temperature to 50 °C or lower. Reduce detector and inlet temperatures to between 150 and 200 °C. If desired, the detector can be turned off. See **Table 1** to determine if it is advantageous to shut down the detector for a short time period. The time required to return the detector to a stable condition is a factor.

# To Shut Down the GC for More Than a Week

1 Put the GC into general maintenance mode by selecting **Maintenance** > **Instrument** > **Perform Maintenance** > **Start Maintenance**, and wait for the GC to become ready.

2 Turn off the main power switch.

WARNING

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

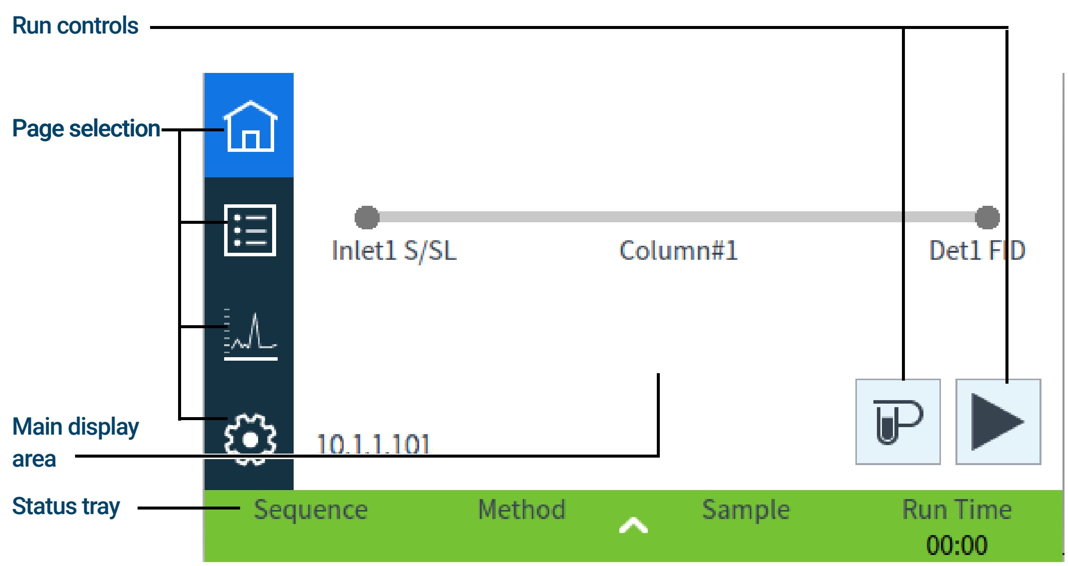
# 4 Touchscreen Operation



This section describes the basic operation of the Agilent GC touchscreen.

# Navigation

The touchscreen shows GC status and activity information (current temperatures, flows, pressures, and information about GC readiness), and allows you to start, stop, and prepare the GC to run a sample.




On the home page, selecting one of the page selection buttons loads the corresponding page.

The main display area provides information related to the selected functional area/page. This area contains status displays, controls, settable parameters, and so on.

Depending on which page is selected, additional controls may appear. This can include page selection buttons, selectable tabs, back and next buttons, scroll buttons, and so on. See **Figure 6**.

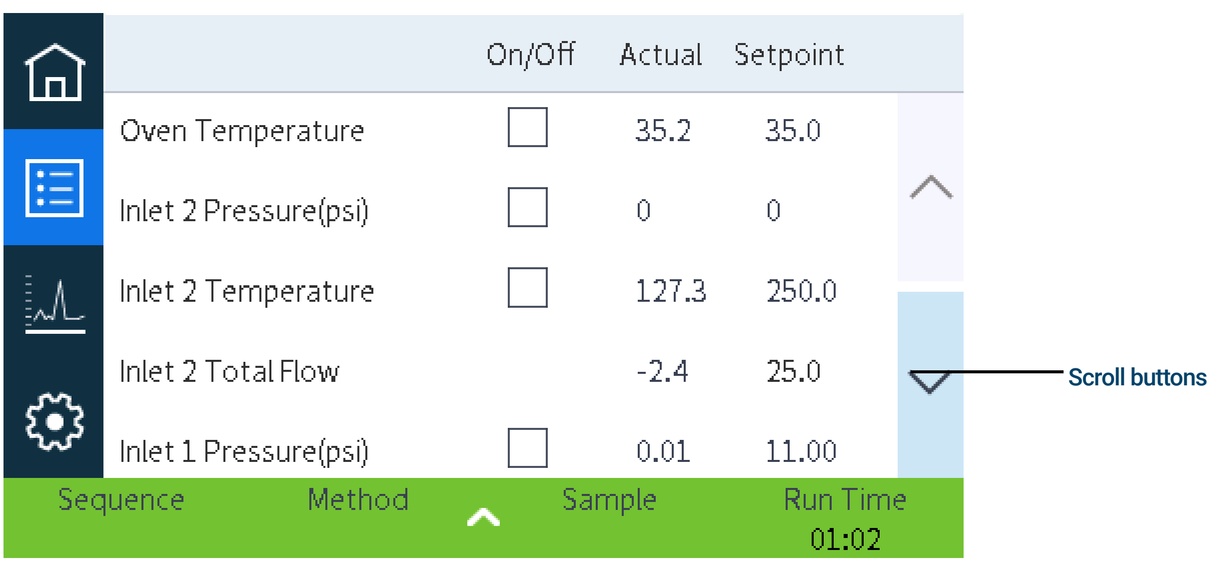



Figure 6. Additional controls

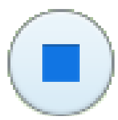
Scroll buttons are enabled if additional information or settings are available via scrolling.

# Run controls

The run controls are located on the Home page. The run controls are used to start, stop, and prepare the GC to run a sample.

The **Prep Run** control activates processes required to bring the GC to the starting condition for a run (such as turning off the inlet purge flow for a splitless injection). This is typically required before manual injections to exit any gas saver mode and to prepare the inlet flows for injection.

The **Start** control starts a run after manually injecting a sample. (When you are using an automatic liquid sampler or gas sampling valve, the run is automatically activated at the appropriate time.)



The **Stop** control immediately terminates the run. If the GC is in the middle of a run, the data from that run may be lost.

For details on running methods, see **"Running Methods"**.

# Status/control tray

The status/control tray provides details on the current status of the GC, the current sequence and method (if connected to an Agilent data system), the time remaining for the current operation being performed by the GC, run controls, and so on.

The status/control tray is color coded to reflect the run or ready status of the GC:

- Green - Ready for a run
- Yellow - Not Ready or Shutdown
- Blue - Run in Progress
- Purple - Preparing Sample
- Teal- Sleep mode
- Red- Error

Any Early Maintenance Feedback (EMF) flags are also displayed. See **"Early Maintenance Feedback (EMF)"**.

The tray can be expanded by selecting the up arrow on the tray.

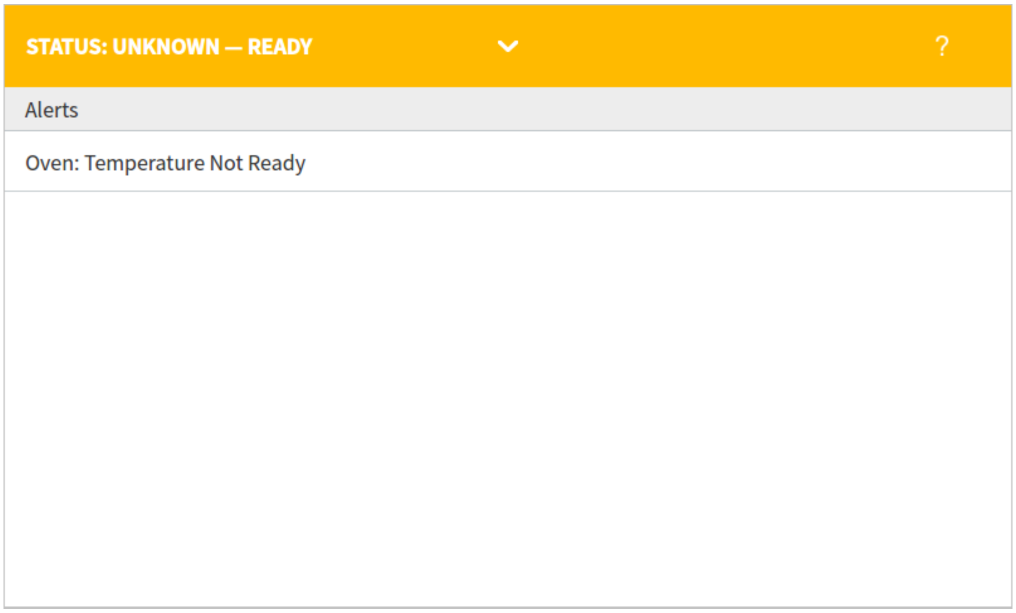



Figure 7. Status/control tray - expanded

The tray can be minimized by selecting the arrow on the tray.

# Entering Data

When you touch a data entry field, a touch keyboard or keypad appears, as applicable. See **Figure 8**.

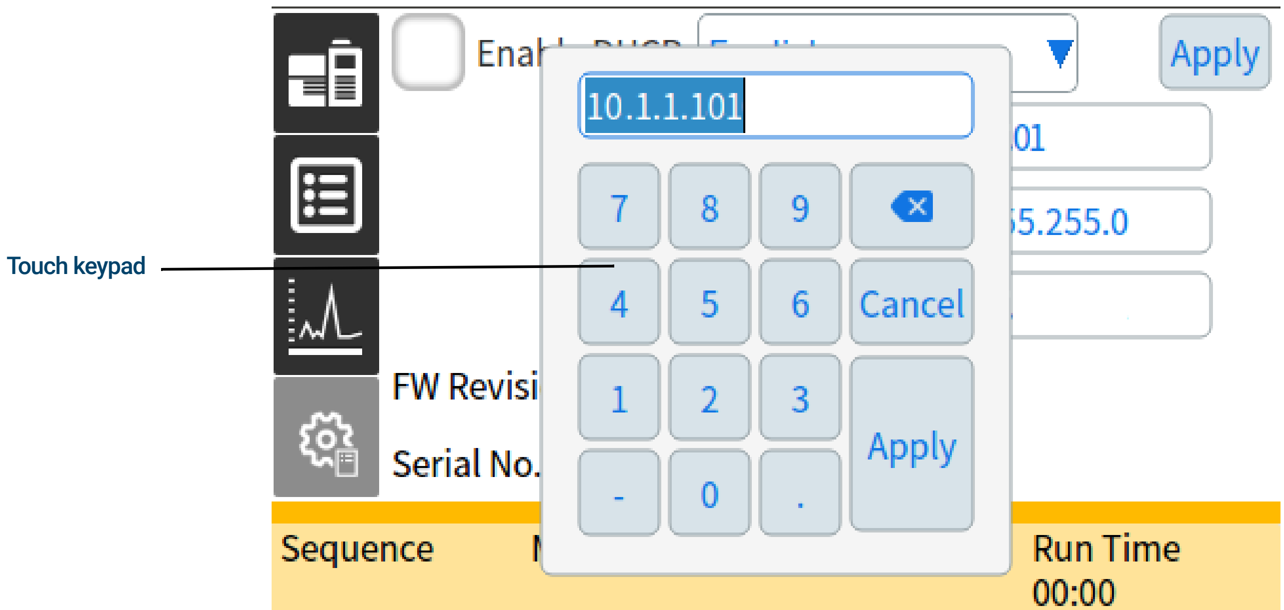



Figure 8. Touch keypad for data entry

If you enter an out of range entry, it is highlighted in a different color.

If the field is a drop-down list box (indicated by a down arrow to the right of the displayed contents of the field), select it to open the list, and then select the desired entry.

# Home View

The home view's Flow Path page shows flow path information (including current temperatures and flow rates), run status (including user selectable status items), a real-time plot of the current chromatogram, and related information. See **Figure 9**.

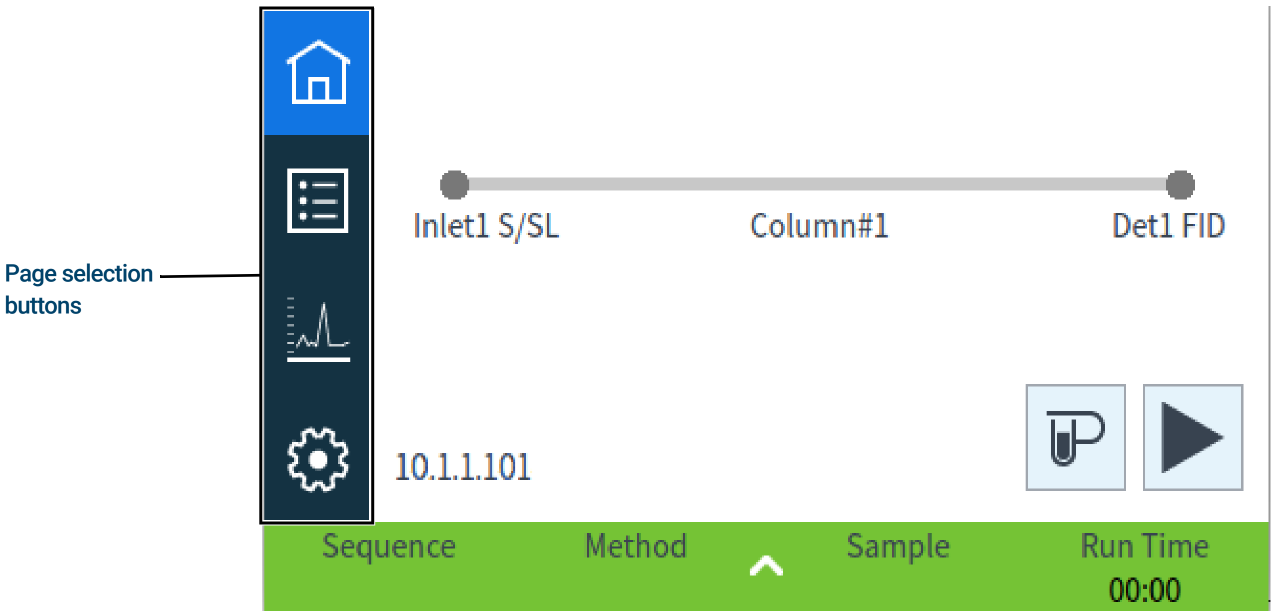



Figure 9. Home view

The other pages available from the home view are:

- Status
- Plot
- Configuration

These pages are displayed by selecting the corresponding page selection button on the left side of the home view.

The flow path page provides details on sample flow through the GC. This includes visual indications of whether an ALS is installed on the GC, the inlet type(s), column setup, and detector type(s), along with corresponding setpoints.

Each page is described below.

## Status page

The status page displays a user-selectable list of parameters, along with their setpoints and actual values. See **Figure 10**.

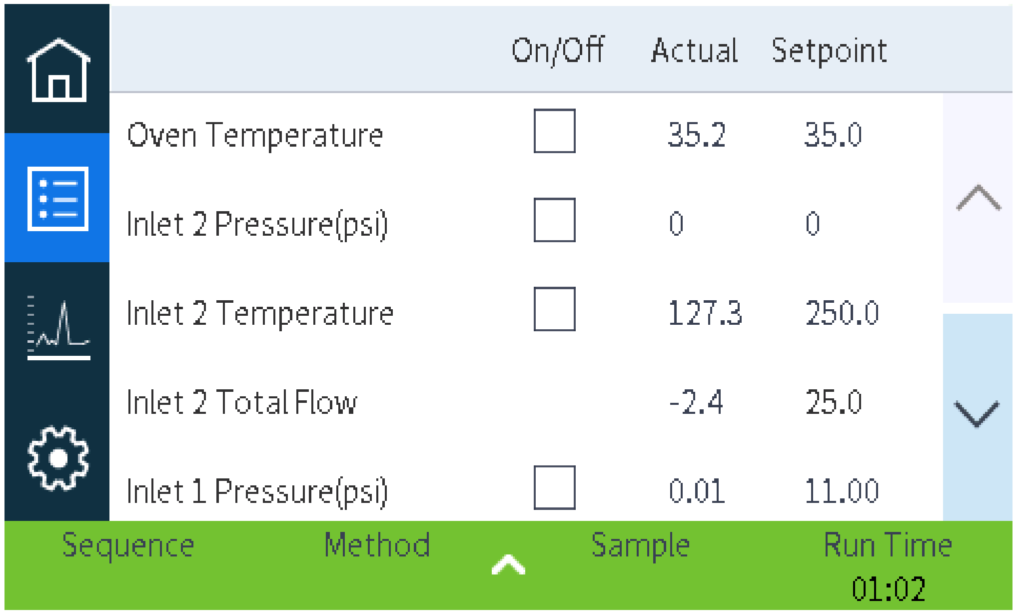



Figure 10. Home view - status page

## Plot page

The plot page displays a plot of the currently selected signal. See **Figure 11**.

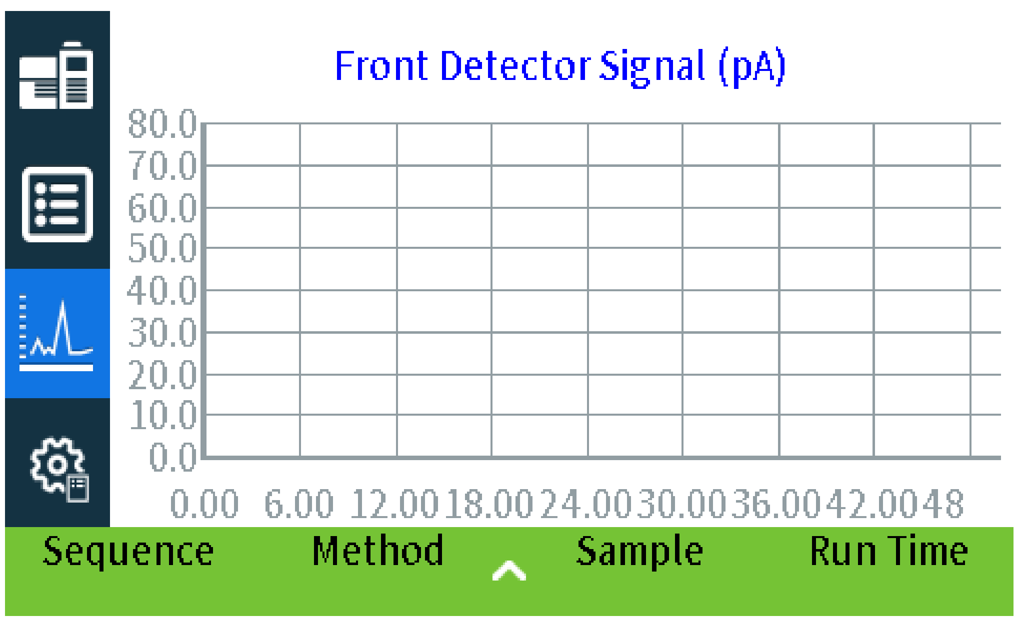



Figure 11. Home view - plot page

Selecting the displayed signal name opens a Plot Options dialog box. This allows you to select which signal to display. See **Figure 12**.

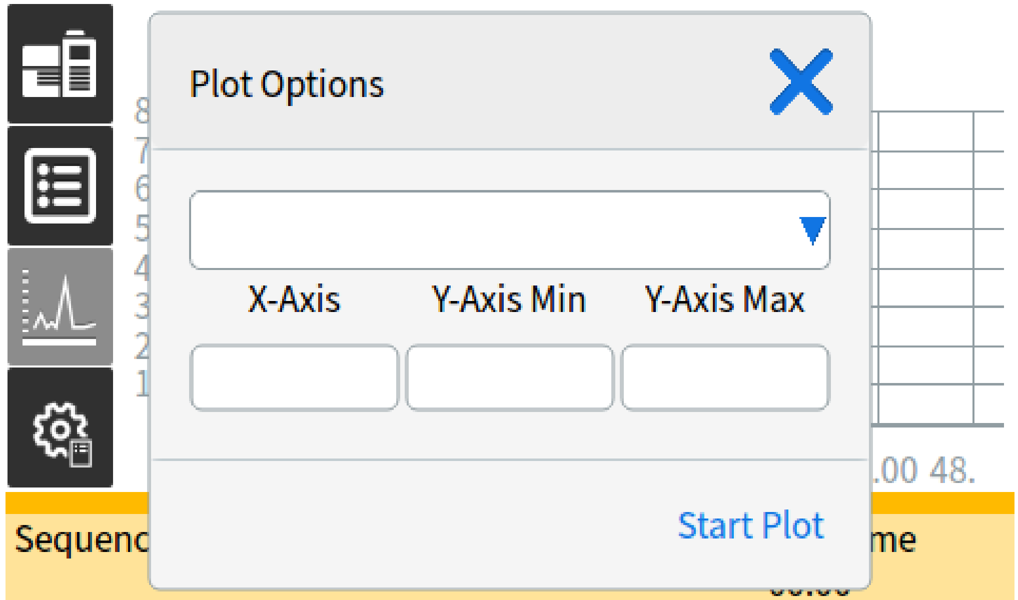



Figure 12. Plot Options dialog box

Use the drop down list box to select which parameter to display on the plot.

The displayed **X-Axis** interval is 1 to 60 minutes. The **Y-Axis Range** is negative infinity to infinity. Selecting either field brings up a keypad which allows you to set the corresponding value.

If the plot is not currently running, selecting **Start Plot** starts it. If the plot is currently running, selecting **Stop Plot** halts data collection and display. (When changing the Signal Name, it may be necessary to select **Stop Plot** and then **Start Plot** to display the signal.)

# 5 Methods

# What is a Method?

A method is the group of settings required to analyze a specific sample.

Since every type of sample reacts differently in the GC—some samples require a higher oven temperature, others require a lower gas pressure or a different detector—a unique method must be created for each specific type of analysis.

The GC also can store several specialized methods. The GC stores three methods used for resource conservation, called **SLEEP**, **CONDITION**, and **WAKE**. When configured for use with an attached MS, the GC also provides a method called **MS VENT**, used to change GC setpoints to values appropriate for a safe MS venting process. Create these methods using an Agilent data system. See **"Resource Conservation"** for more information about these specialized methods.

Methods can be created and edited using the Browser Interface. Methods can also be created, edited, and stored on a connected data system. While a data system is connected to your GC, any attempt to connect to your GC via the Browser Interface will result in the Browser Interface having limited functionality. You will not be able to edit methods, edit sequences, and start or stop runs until the data system is disconnected. Similarly, once a Browser Interface is connected to your GC, any subsequent Browser Interfaces attempting to connect will not be able to edit methods, edit sequences, and start or stop runs until the first session is disconnected.

Methods and sequences created using the Browser Interface are not directly accessible from a connected data system. Methods and sequences created using a connected data system are not directly accessible using the Browser Interface.

# What Is Saved in a Method?

Some of the settings saved in a method define how the sample will be processed when the method is used. Examples of method settings include:

- The oven temperature program
- The type of carrier gas and flows
- The type of detector and flows
- The type of inlet and flows
- The type of column
- The length of time to process a sample

NOTE

**For GCs equipped with EPR (electronic pneumatics regulation), detector, column, and inlet flows are not saved with the method.**

Data analysis and reporting parameters are also stored in a method when it is created on an Agilent data system, for example OpenLAB CDS or MassHunter software. These parameters describe how to interpret the chromatogram generated by the sample and what type of report to print.

The GC method also includes sampler setpoints. Refer to the sampler documentation for details on setpoints for the supported device:

- For the 7650A ALS, see its Installation, Operation, and Maintenance manual.
- For the 7693A ALS, see its Installation, Operation, and Maintenance manual.
- For the 7697A HS, see its Installation and First Startup manual and Operation manual.
- For the CTC PAL3 AS, see its Installation and First Startup manual and Operation manual.

Current setpoint parameters are saved when the GC is turned off, and loaded when you turn the instrument back on.

# What Happens When You Load a Method?

There are two kinds of methods:

- The active method—This is sometimes referred to as the current method. The settings defined in this method are the settings the GC is currently maintaining.
- Stored methods—User-created methods can be stored in the GC, along with one SLEEP method, one WAKE method, one CONDITION method, an MS VENT method, and a default method.

For EPC (electronic pneumatics control) equipped GCs, **when a method is loaded** from the GC or Agilent data system, the setpoints of the active method are immediately replaced with the setpoints of the method loaded.

For EPR (electronic pneumatics regulation) equipped GCs, **when a method is loaded** from the GC or Agilent data system, any manually set parameters **are not** replaced with the setpoints of the method loaded.

- The method loaded becomes the active (current) method.
- The GC front panel status indicator will be yellow (not ready) and will stay yellow until the GC reaches all of the settings specified by the method that was just loaded.

# Create a Method

Methods may be created using your Agilent data system, or via the Browser Interface. For more information on creating a method using your data system, refer to the documentation provided with your data system.

From the Browser Interface:

1 Click **Method** on the control ribbon.

2 Click the **Create New** button . You are prompted to name your method and save.

3 From the navigation tree, select each instrument device, and set its method parameters to the desired values.

4 Click **Configuration** > **Modules**. Check the gas configuration for each inlet and detector. (Gas configuration was initially set when the Setup Wizard was run at installation.) Make any changes if needed.

   Click **Configuration** > **Columns**. If the installed columns include Smart ID Keys, the column configurations are set by the Smart ID Keys. IF not, edit the column configuration as needed. Double-click a column to edit it.

5 Click **Columns**. For each column, set the control mode, select the **On** checkbox, and set the flow on the column.

6 Click **Inlets**, and then select either the front or back inlet. Set the inlet mode, temperatures, and other parameters needed for your method. Repeat for the other inlet, if present.

7 Click **Detectors**. For each detector being used, set the detector temperature and gas flows. Select all check boxes to turn the detector on.

NOTE

**Carrier Gas Flow Correction setting recommendations**

- **Column + Fuel = Constant** ($H_2$ carrier in Constant Pressure mode)
- **Column + Makeup = Constant** (He/$N_2$ carrier Constant Pressure mode)
- **Constant Makeup and Fuel Flow** (any carrier in Constant Flow mode)

8 Click **Oven**. Set the initial oven temperature, ramp temperatures, and times for your method. Select the **Heater On** checkbox. For an isothermal run, do not create any ramps.

9 Click **Signals**. Choose the signals to be included in the data file when a run is initiated. Normally this is **Front Signal** or **Back Signal** for detector output. Select the Save check box and select a data rate that meets the needs of your chromatography.

10 Click **ALS/Tray**, then select either the front or back injector. Set the **Injection Volume**, washes, and pumps.

NOTE

**It is important to do both sample and solvent washes to eliminate carryover and keep the syringe clean. Pumping the plunger multiple times eliminates air bubbles in the syringe for better reproducibility.**

11 If using a tray, click **ALS/Tray** > **Other**. Set **Sample Overlap** if desired.

12 Click the **Save** button to save your method.

# Load a Method

1. Connect to the GC using the Browser Interface. See **"Browser Interface"**.
2. Open or create the desired method. See **"Create a Method"**.
3. If needed, click the **Save** button to save the method.
4. Click the **Download** button to load the method to your GC.

# Running Methods

## Pre Run and Prep Run

With some inlets and operating modes, certain instrument setpoints are different between runs than during an analysis. To restore the setpoints for injection, you must place the GC into the Pre Run state.

You must use the Pre Run state when:

- Using gas saver with any inlet.
- Using splitless mode with any inlet.
- Using a pressure pulse mode with any inlet.

NOTE **Gas saver and pressure pulse mode are not supported on EPR (electronic pneumatics regulation) equipped GCs.**

•

There are three ways to begin Pre Run—manually (select before each run), automatically (for Agilent samplers), or using **Auto Prep Run** (for non-Agilent samplers). The three methods are discussed below.

During the Pre Run state:

- The status on the touchscreen changes to indicate the GC is preparing for the injection.
- Setpoints change to the correct values for injection.
- Inlet, detector, and oven equilibration times begin.

When all criteria for a run are met, the Status/Control tray indicates that the GC is ready for sample injection.

## Manually prepare for the run

Select before you inject a sample manually. The GC enters the Pre Run state. When the touchscreen indicates that the GC is ready, begin the analysis.

If you are using an Agilent automatic sampling system, the **Prep Run** function is automatic.

## To manually inject a sample with a syringe and start a run

1. Prepare the sample syringe for injection.
2. Load the desired method. See **"Load a Method"**.
3. Navigate to the **Home** view and select **Prep Run** . See **"Run controls"** for more information.

4 Wait for status **Ready** to display.

5 Insert the syringe needle through the septum and all the way into the inlet.

6 Simultaneously depress the syringe plunger to inject the sample and select **Start** ▶.

## To run a method to process a single ALS sample

1 Prepare the sample for injection.

2 Load the sample vial into the assigned location in the ALS tray or turret.

3 Load the desired method. (See **"Load a Method"**.)

4 Navigate to the **Home** view and select **Start** ▶ to initiate the ALS syringe cleaning, sample loading, and sample injection method. After the sample is loaded into the syringe, the sample is automatically injected when the GC reaches the ready state. See **"Run controls"** for more information.

## To abort a method

1 Select **Stop** ■.

2 When you are ready to resume running analyses, load the appropriate sequence or method. (See **"Load a Method"**.)

# Events

Run time programming during a method allows certain setpoints to be changed during a run as a function of the chromatographic run time. Thus an event that is programmed to occur at 2 minutes will occur 2 minutes after every injection.

- Controlling column switching or other valves
- Changing analog signal definition, zero, or range
- Controlling an auxiliary pressure channel
- Changing polarity of a thermal conductivity detector (TCD)
- Make sample flow bypass TCD filament
- Turning the hydrogen flow to a nitrogen-phosphorus detector (NPD) on or off
- Switching digital signal output (requires an Agilent data system)
- Pausing (“freezing”) and resuming digital signal output (requires an Agilent data system)
- Performing signal math on the front and back detectors.

The changes are entered into a run table that specifies the setpoint to be changed, the time for the change, and the new value. At the end of the chromatographic run, most setpoints changed by a run time table are returned to their original values.

Valves can be run time programmed but are not restored to their starting position at the end of the run. You must program the reset operation in the run table if this action is desired.

## Using run time events

The **Events** page in the **Method** tab is used to program the following timed events.

- Valves (1-10)
- Multiposition valve
- Signal type
- Analog signal definition, zero, and range
- Auxiliary pressures (1 through 9)
- TCD negative polarity (on/off)
- Detector gas flow (on/off), including NPD $H_2$ fuel gas
- Inlet septum purge flow
- AO signal range
- AO signal source
- AO signal zero
- Bypass TCD Filament

# Programming run time events

1 Select **Method**.

2 Select **Events**.

3 Input the time at which you want the event to occur, select the event you want to program, select the position of the hardware that is being controlled, and set the desired setpoint.

# The run table

The programmed events are arranged in order of execution time in the Run Table.

# Editing events in the run table

1 Select the event you wish to edit. If the desired event is not immediately visible, use the up and down arrows to the right to scroll up and down the table until it is.

2 Select the parameter you want to change.

3 Input the new value

# Deleting run time events

1 Select the event you wish to delete.

2 Select **Delete**.

# Inlets

The Inlets page in the Method tab is used to modify the method parameters and each inlet connected to your GC. Common parameters include the heater temperature and inlet pressure. To modify your inlets' method parameters:

1 Select **Method** > **Inlets**.

2 Select the inlet you want to modify.

3 Scroll to the desired setting, and edit as necessary.

## Carrier gas flow rates

The flow rates in **Table 2** are recommended for all column temperatures.

### Table 2 Column size and carrier flow rate

| Column type | Column size | Carrier flow rate, mL/min | | |
|---|---|---|---|---|
| | | Hydrogen | Helium | Nitrogen |
| **Packed** | 1/8-inch | | 30 | 20 |
| | 1/4-inch | | 60 | 40 |
| **Capillary** | 0.05 mm id | 0.5 | 0.4 | n/a |
| | 0.10 mm id | 1.0 | 0.8 | n/a |
| | 0.20 mm id | 2.0 | 1.6 | 0.25 |
| | 0.25 mm id | 2.5 | 2.0 | 0.5 |
| | 0.32 mm id | 3.2 | 2.6 | 0.75 |
| | 0.53 mm id | 5.3 | 4.2 | 1.5 |

## About gas saver

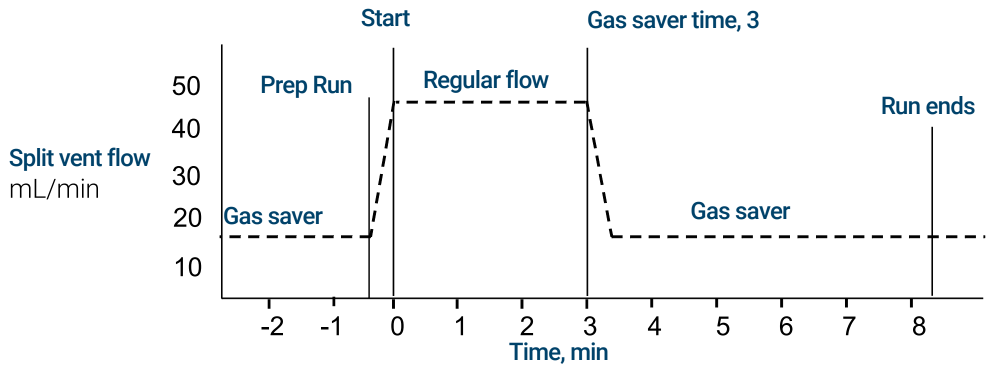
NOTE

**Gas saver is not supported on EPR (electronic pneumatics regulation) equipped GCs.**

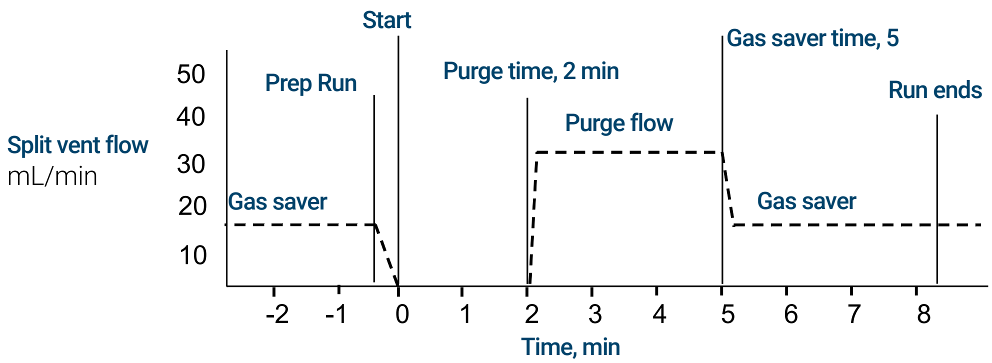
Gas saver reduces carrier flow from the split vent after the sample is on the column. It applies to the Split/Splitless inlet (all modes). It is most useful in split applications.

Column head pressure and flow rate are maintained, while split vent flows decrease. Flows—except column flow—remain at the reduced level until you press .

Split mode




Splitless mode



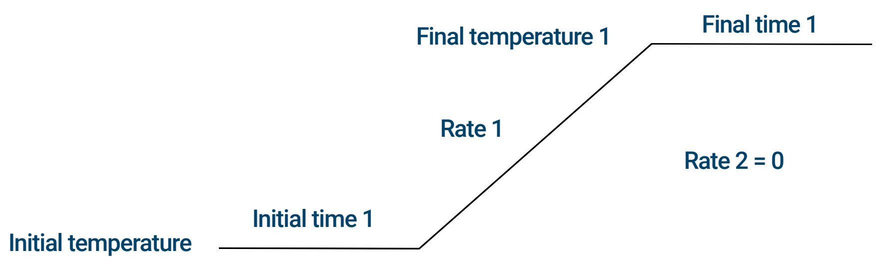

### To use gas saver

1. Select **Method** > **Inlets**.
2. Scroll down until the Gas Saver option is visible.
3. Select the checkbox next to **Gas Saver** to enable it.
4. Input the **Setpoint**. The setpoint must be at least 15 mL/min greater than the column flow.
5. Input the **Time**.

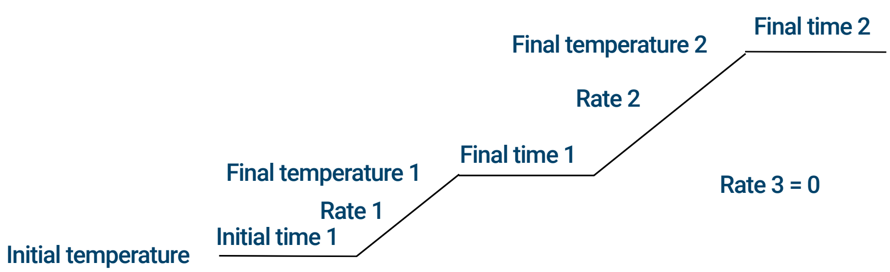
# About Oven Temperature Programming

You can program the oven temperature from an initial temperature to a final temperature using up to 5 ramps during a run.

A single ramp temperature program raises the initial oven temperature to a specified final temperature at a specified rate and holds at the final temperature for a specified period of time.




The multiple-ramp temperature program is similar. You can program the oven from an initial temperature to a final temperature, but with various rates, times, and temperatures in between. Multiple ramps can also be programmed for temperature decreases as well as increases.




## Oven ramp rates

The highest rate that you can achieve depends on many factors, including the room temperature, temperatures of the inlets and detectors, the amount of material inside the oven (columns, valves, etc.), and whether or not this is the first run of the day.

**Table 3** lists typical oven ramp rates.

**Table 3 Oven ramp rates**

| Temperature range (°C) | 100 V oven ramp rate (°C/minute) | 200/220/230/240 V oven ramp rate (°C/minute) |
|---|---|---|
| 50 to 70 | 30 | 75 |
| 70 to 115 | 30 | 45 |
| 115 to 175 | 30 | 40 |
| 175 to 300 | 30 | 30 |
| 300 to 425 | 20 | 20 |

### Isothermal runs

An isothermal run is one in which the oven is maintained at a constant temperature. For an isothermal run, set Rate 1 to zero.

1 Select **Oven** to open the oven parameter list.
2 Enter the oven temperature for the isothermal run.
3 Enter the number of minutes (Initial time) that you want the oven to stay at this temperature. This time is the duration of the run.
4 If Rate 1 is not already 0, enter zero for an isothermal run

# Columns

The column flow mode determines whether pressure or flows are available as setpoints for the GC inlets. When all columns in the flow path are defined, you can enter pressures or flows. If any column in the flow path is not defined, then the settings for the inlet may be restricted based on the inlet type and whether the column is in flow or pressure mode.

There are two different flow modes that control the mass flow rate through the column: **Constant flow** and **Ramped flow**. The flow rates are corrected to NTP (normal temperature and pressure, 25 °C and 1 atmosphere).

There are two different pressure modes that control the pressure at the head of the column; **Constant pressure** and **Ramped pressure**. These pressures are gauge pressures—the difference between the absolute pressure and the local atmospheric pressure.

**Constant flow** Maintains a constant mass flow rate of carrier gas in the column throughout the run. If the column resistance changes due to a temperature program, the column head pressure is adjusted to keep the flow rate constant. This can shorten runs significantly.

**Ramped flow** Increases the mass flow rate in the column during the run according to a program you enter. A column flow profile can have up to three ramps, each consisting of a programmed increase followed by a hold period.

The pressure modes are not available if the column is not defined or the inlet's mode is set to Flow control. Pressures are gauge pressures—the difference between the absolute pressure and the local atmospheric pressure.

Because most detectors present little resistance to the column flow, the gauge pressure at the column head is usually the same as the pressure difference between column inlet and exit. The mass selective detector and the atomic emission detector are the exceptions.

- Constant pressure— Maintains a constant gauge pressure at the head of the column throughout the run. If the column resistance and gas density changes during a run, the gauge pressure does not change but the mass flow rate does.
- Ramped pressure— Increases the column head gauge pressure during the run according to a program you enter. A column pressure profile can have up to three ramps, each consisting of a programmed increase followed by a hold period.

NOTE

**Flow modes and ramped pressure mode are not supported on SSL EPR (electronic pneumatics regulation) equipped GCs.**

**Pressure modes are not supported on PPIP EPC equipped GCs.**

**Pressure modes and ramped flow mode are not supported on PCI EPR equipped GCs.**

# Detectors

For help creating a new method, or troubleshooting detector problems, see the recommended starting conditions for each detector.

The makeup gas line of your detector parameter list changes depending on your instrument configuration. If you have an inlet with the column not defined, the makeup flow is constant.

## FID

### FID automatic reignition (Lit offset)

**Lit offset** is the expected minimum difference between the FID output with the flame lit and the output with the flame off. The GC checks this value during runs and when loading a method.

During a run, if the output falls below the **Lit offset** value, the FID will attempt to reignite three times. If after the third attempt the output does not increase by at least this value, the detector shuts down all functions except temperature and makeup gas flow.

When loading a method that includes a **Flame On** setting, the GC performs a similar check. If the detector output is less than the **Lit offset**, it will attempt reignition after reaching method setpoints.

The default setting for **Lit offset** is 2.0 picoamps. This is a good working value for all but very clean gases and systems. You may want to lower this setpoint if the detector attempts to reignite when the flame is still on, thus producing a shutdown.

To change **Lit offset**:

1. Select **Settings**.
2. Select **Configuration**.
3. Select **Detectors**.
4. Select your FID from the detectors listed at the top of the window.
5. Enter the new value.

NOTE

**The newly entered Lit offset is not applied/referenced against the detector output until the next ignition cycle.**

### Recommended starting conditions

See **Table 4** for guidelines and rules to select initial detector settings for new methods.

**Table 4 Recommended starting conditions**

| Gas type | Suggested flow rate |
|---|---|
| **Carrier gas (hydrogen, helium, nitrogen)** | |
| Packed columns | 10 to 60 mL/min |
| Capillary columns | 1 to 5 mL/min |

**Table 4 Recommended starting conditions (continued)**

| Gas type | Suggested flow rate |
|---|---|
| **Detector gases** | |
| Hydrogen | 40 mL/min* |
| Air | 450 mL/min* |
| Column plus capillary makeup ($N_2$ is recommended, or He as an alternative) | 50 mL.min* |
| **Detector Temperature** | |
| If < 150 °C, the flame will not light. Agilent recommends a temperature ≥ 300 °C to prevent condensation damage. The detector temperature should be approximately 20 °C greater than highest oven ramp temperature. | |

* The hydrogen / air ratio should be 8% to 12% to keep the flame lit

# FPD+

The sample burns in a hydrogen- rich flame, where some species are reduced and excited. The gas flow moves the excited species to a cooler emission zone above the flame where they decay and emit light. A narrow bandpass filter selects light unique to one species, while a shield prevents intense carbon emission from reaching the photomultiplier tube (PMT).

The light strikes a photosensitive surface in the PMT where a light photon knocks loose an electron. The electron is amplified inside the PMT for an overall gain of up to a million.

## Storage

The FPD+ should not be stored at temperatures above 50 °C.

## FPD+ linearity

Several mechanisms produce sulfur emission. The excited species is diatomic, so that emission intensity is approximately proportional to the square of the sulfur atom concentration.

The excited species in the phosphorus mode is monatomic, leading to a linear relationship between emission intensity and atom concentration.

## Inlet liners for use with the FPD+

Compounds containing sulfur may adsorb on an inlet liner and degrade the GC’s performance. Use deactivated, clean liners or a cool on-column inlet, which injects directly onto the column.

For best results use Agilent Ultra Inert liners:

- Splitless 5190-2293
- Split 5190-2295

## FPD+ temperature considerations

The FPD+ provides two temperatures zones, one for the transfer line (the main detector temperature) and one for the emission block. For the transfer line temperature, we recommend a temperature that is 25 °C higher than the highest column temperature.

The emission block temperature ranges from 125—175 °C. Typically, the default temperature of 150 °C is sufficient for most applications. However, when setting the emission block temperature, consider the following:

- If using the GC oven at high temperature (>325 °C) with the transfer line set to 400 °C, set the emission block temperature to 165 °C to avoid a system Not Ready if the emission block temperature cannot be maintained.
- If using the transfer line at 400 °C, set the emission block temperature to at least 150 °C to avoid a system Not Ready.
- For sulfur analyses, the highest area response will be realized with the lowest possible emission block temperature.
- For phosphorus analyses, the area response is independent of the emission block temperature.

### Recommended starting conditions

The FPD+ provides two temperatures zones, one for the transfer line (the main detector temperature) and one for the emission block. For the transfer line temperature, we recommend a temperature that is 25 °C higher than the highest column temperature. The emission block temperature ranges from 125—175 °C. Typically, the default temperature of 150 °C is sufficient for most applications.

Flows for the maximum sensitivity FPD+ flame, which is hydrogen-rich and oxygen-poor. Helium, either as carrier or makeup gas, may cool the detector gases below the ignition temperature.

Table 5 Recommended starting conditions

| Gas type | Suggested flow rate |
|---|---|
| **Carrier gas (hydrogen, helium, nitrogen)** | |
| Packed columns | 10 to 60 mL/min |
| Capillary columns | 1 to 5 mL/min |
| **Detector gases** | |
| Hydrogen | 60 mL/min |
| Air | 60 mL/min |
| Column plus capillary makeup | 60 mL.min |

As with the FID, the FPD has a Lit Offset associated with it. The default Lit OIffset for the FPD is 2.0 pA.

## NPD

### Setting NPD adjust offset on the clock table

You can use the **Clock table** feature to begin **Adjust offset** at a specified time.

## Extending the NPD bead life

These actions, together with the automated heatup and adjust procedures, can extend bead life considerably.

- Use the lowest practical **Adjust Offset** value. This will result in a lower applied bead current during operation.
- Run clean samples.
- Turn the bead off when not in use.
- Keep the detector temperature high (320 to 335 °C).
- Turn the hydrogen flow off during solvent peaks and between runs.

**Turning hydrogen off during a solvent peak** When using the NPD, the baseline shifts after a solvent peak and can take some time to stabilize, especially with chlorinated solvents. To minimize this effect, turn off the hydrogen flow during the solvent peak and turn it back on after the solvent elutes. With this technique, the baseline recovers to its original value in less than 30 seconds. This also extends the life of the bead. The hydrogen can be turned on and off automatically as part of a Run Table. See **"Events"**.

**Turning hydrogen off between runs** To extend bead life, turn off the hydrogen flow between runs. Leave all other flows and the detector temperature on. Turn on the hydrogen flow for the next run; the bead will ignite almost immediately. The process can be automated with Run Table entries.

## Recommended Starting conditions

### Table 6 Recommended starting conditions

| Gas type | Suggested flow rates |
|---|---|
| **Carrier gas** (helium, hydrogen, nitrogen*) | Capillary: choose an optimal flow rate based on column dimensions. |
| **Detector gases** | |
| Hydrogen | 1 to 3 mL/min |
| Air | 60 mL/min |
| Makeup Flow (*He*, $N_2$†) | 1 to 20 mL/min, less is recommended |
| **Bead voltage**<br>Blos bead.<br>• Use **Auto Adjust**, **Dry Bead**, and let the GC set the applied bead current for you. | |

* Flow rate must be less than 3 mL/min when using hydrogen as carrier gas.

† Helium is recommended for best peak shapes.

# TCD

### Chemically active compounds reduce TCD filament life

The tungsten-rhenium TCD filament has been chemically passivated to protect against oxygen damage. However, chemically active compounds such as acids and halogenated compounds may attack the filament. The immediate symptom is a permanent change in detector sensitivity due to a change in filament resistance.

If possible, such compounds should be avoided. If this is not possible, the TCD cell may have to be replaced frequently.

### Changing the TCD polarity during a run

**Negative polarity On** inverts the peak so the integrator or Agilent data system can measure it. **Negative Polarity** can be a run table entry; see **“Events”**.

### Detecting hydrogen with the TCD using helium carrier gas

Hydrogen is the only element with thermal conductivity greater than helium, and mixtures of small amounts of hydrogen (<20%) in helium at moderate temperatures exhibit thermal conductivities less than either component alone. If you are analyzing for hydrogen with helium carrier gas, a hydrogen peak may appear as positive, negative, or as a split peak.

There are two solutions to this problem:

- Use nitrogen or argon-methane as carrier gas. This eliminates problems inherent with using helium as carrier, but causes reduced sensitivity to components other than hydrogen.
- Operate the detector at higher temperatures—from 200 °C to 300 °C.

You can find the correct detector operating temperature by analyzing a known range of hydrogen concentrations, increasing the operating temperature until the hydrogen peak exhibits normal shape and is always in the same direction (negative relative to normal response to air or propane) regardless of concentration. This temperature also ensures high sensitivity and linear dynamic range.

Because hydrogen peaks are negative, you must turn negative polarity on at appropriate times so the peak appears positive.

### Recommended Starting conditions

**Table 7 Recommended starting conditions**

| Gas type | Suggested flowrates |
| --- | --- |
| Carrier gas<br>(hydrogen, helium, nitrogen) | Packed: 10 to 60 mL/min<br>Capillary: 1 to 5 mL/min |
| Reference<br>(same gas type as carrier) | 15 to 60 mL/min |
| Capillary makeup<br>(same gas type as carrier) | Packed: 2 to 3 mL/min<br>Capillary: 5 to 15 mL/min |
| **Detector temperature** | |

**Table 7 Recommended starting conditions (continued)**

| Gas type | Suggested flowrates |
| --- | --- |
| <135 °C, cannot turn on filament<br>If detector temperature goes below 120 °C, the filament turns off.<br>Detector temperature should be 30 °C to 50 °C greater than the highest oven ramp temperature. | |

# ECD

## ECD linearity

The ECD response factor versus concentration curve is linear for four orders of magnitude or more (linear dynamic range = $10^4$ or higher) for a broad range of compounds. You should still run a calibration curve on your samples to find the limits of the linear range for your materials.

## ECD makeup gas notes

If the carrier gas type is different from the makeup gas type, the makeup gas flow rate must be at least three times the carrier gas flow rate.

ECD sensitivity can be increased by reducing the makeup gas flow rate.

ECD chromatographic speed (for fast peaks) can be increased by increasing the makeup gas flow rate.

## ECD temperature programming

The ECD is flow sensitive. If you are using temperature programming, in which the column flow resistance changes with temperature, set up the instrument as follows:

- Set the carrier gas in the **Constant flow** mode. Set detector makeup gas to **Constant makeup**.
- If you choose to work in the constant pressure mode, the makeup gas should be set in the **Column + makeup = constant** mode.

## Recommended starting conditions for new ECD methods

Use the following information when selecting temperatures and flows. Maximum source pressure must not exceed 100 psi. Use the maximum source pressure to achieve maximum makeup flow rate.

**Table 8 Recommended starting conditions**

| Gas type | Suggested flow rates |
| --- | --- |
| **Carrier gas** | |
| Packed columns (nitrogen or argon-methane) | 30 to 60 mL/min |
| Capillary columns (hydrogen, nitrogen, or argon-methane) | 0.1 to 20 mL/min, depending on diameter |
| **Capillary makeup** (nitrogen or argon-methane) | 10 to 150 mL/min<br>(30 to 60 mL/min typical) |

### Table 8 Recommended starting conditions (continued)

| Gas type | Suggested flow rates |
| --- | --- |
| **Temperature** | |
| 250 °C to 400 °C<br>Detector temperature is typically set 25 °C greater than the highest oven ramp temperature. | |

# Valves

## The valve box

The GC holds up to three valves in a heated valve box on top of the oven.

The valve box is the preferred location for valves because it is a stable temperature zone, isolated from the column oven.

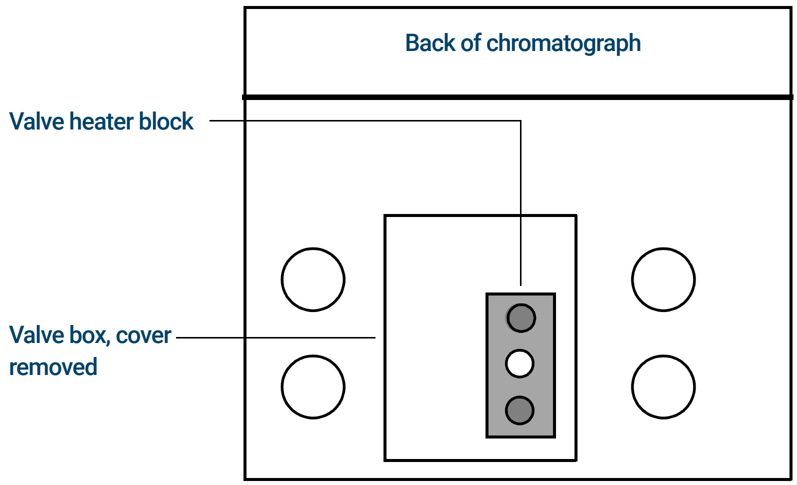



Figure 13. Diagram of valve locations on GC

## Valve control

Valves can be controlled manually from the Browser Interface or as part of a clock or run time program. Note that sampling valves automatically reset at the end of a run.

### The valve drivers

A valve driver is the software and circuitry in the GC that controls a valve or related function. There are four drivers, known as Valves 1 through 4. Each valve is independently controlled.

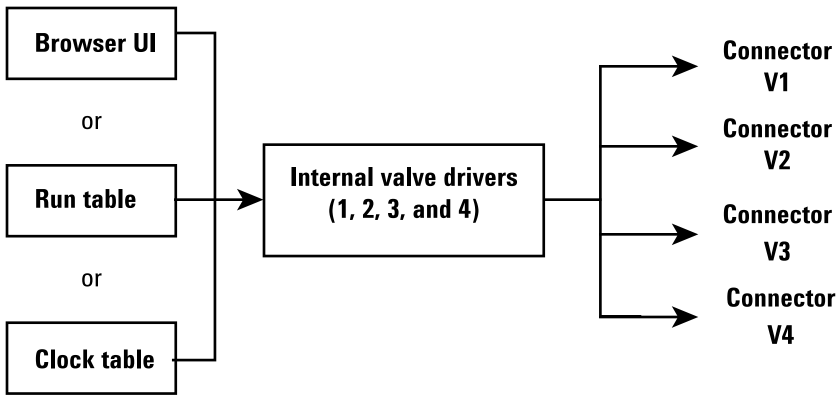
**Table 9 Valve drivers**

| Valve number | Type | Volts | Power or current | Use |
|---|---|---|---|---|
| 1, 2, 3, and 4 | Current source | 24 VDC | 13 watts | Pneumatic valve control |
| 5 and 6 | Current source | 24 VDC | 100 mA | Relays and low-power devices |
| 7 and 8 | Contact closure | 48 VDC or 48 VAC RMS | | Control an external current source |

### The internal valve drivers

Valve drivers 1, 2, 3, and 4 are usually used to control pneumatically operated valves mounted in the valve box. The wiring for these appears at a set of connectors inside the right cover of the GC.

Pneumatically driven valves are controlled by solenoids mounted near the connectors that control the flow of air to the valve actuators.




There is no direct relationship between the location of a valve in the valve box and the driver that controls it. This depends on how the solenoids are wired and the actuators are plumbed.

Manual valves must be switched by hand, and are heated or unheated.

## Valve types

The possible valve types are:

**Sampling** A two-position (load and inject) valve. In load position, an external sample stream flows through an attached (gas sampling) or internal (liquid sampling) loop and out to waste. In inject position, the filled sampling loop is inserted into the carrier gas stream. When the valve switches from **Load** to **Inject**, it starts a run if one is not already in progress. See the example on **page 77**.

**Switching** A two-position valve with four, six, or more ports. These are general-purpose valves used for such tasks as column selection, column isolation, and many others.

**Other** Could be anything.

**Not installed** There is no valve installed in this position.

## Controlling a valve

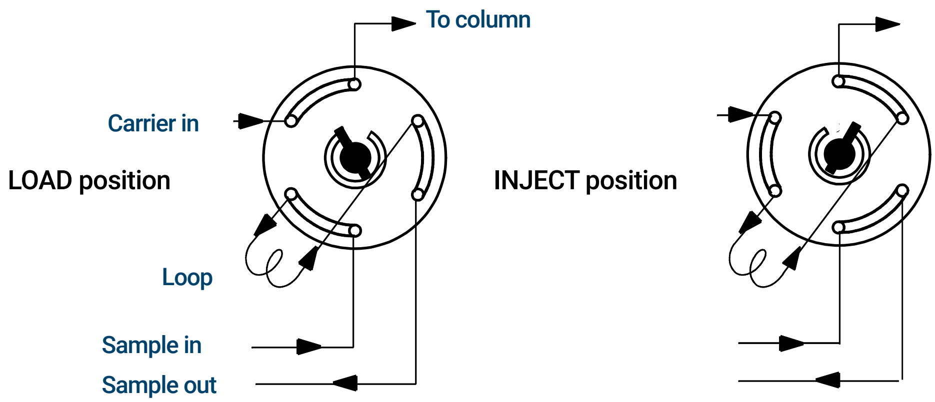
### From the run or clock time tables

The **Valve On** and **Valve Off** commands can be run time or clock time programmed. See **"Events"** and **"Clock Time Programming"**.

If a valve is rotated by a run time program, it is not automatically returned to its initial position at the end of the run. You must program this reset operation yourself.

### Sampling valve

If a valve is configured as a sampling valve, it starts a run automatically when it is switched to the Inject position. This can be done by a subsequence or clock table entry. You may have two gas sampling valves installed.




Sampling valves have two positions:

**Load position** The loop (external for gas sampling, internal for liquid sampling) is flushed with a stream of the sample gas. The column is flushed with carrier gas.

**Inject position** The filled loop is inserted into the carrier gas stream. The sample is flushed onto the column. The run starts automatically.

Carrier gas may be provided by an (optional) PCM channel. To do this, configure the column and specify the PCM channel as the inlet. The channel then becomes programmable with four operating modes.

The gas sampling valve control parameters are:

**Load time** Time in minutes that the valve remains in the Load position before becoming ready.

**Inject time** Time in minutes that the valve remains in the Inject position before returning to the Load position.

The sampling valve cycle is:

1 The sampling valve rotates to the Load position. **Load time** begins. Valve is not ready.
2 **Load time** ends. The valve becomes ready.
3 If everything else is ready, the GC becomes ready. If anything is not ready:
   - If you are using Clock Table or sequence control, the GC waits until everything is ready, then executes the valve inject command.
   - If you are not using Clock Table or sequence control, the valve injection can be made at any time from the keyboard.

4 The sampling valve rotates (keyboard command or sequence control) to the Inject position. **Inject time** begins. The run begins.

5 **Inject time** ends. Return to step **1**.

# GC Output Signals

Signal is the GC output to a data handling device, analog or digital. It can be a detector output or the output from flow, temperature, or pressure sensors. One signal output channel is provided.

Signal output can be either analog or digital, depending on your data handling device. Analog output is available at either of two speeds, suitable to peaks with minimum widths of 0.004 minutes (fast data rate) or 0.01 minutes (normal rate). Analog output ranges are 0 to 1 V, 0 to 10 V.

Digital output rates are set by your Agilent data system, such as OpenLAB CDS or MassHunter Workstation.

See **Table 10** for the conversions from units shown on the GC display to units as shown in Agilent data systems and integrators.

### Table 10 Signal conversions

| Signal type | 1 display unit is equivalent to: |
|---|---|
| **Detector:** | |
| FID, NPD | 1.0 pA ($1.0 \times 10^{-12}$ A) |
| FPD+ | 150 pA ($150 \times 10^{-12}$ A) |
| TCD | 25 uV ($2.5 \times 10^{-5}$ V) |
| ECD | 1 Hz |
| Analog input board (use to connect the GC to non-Agilent detector) | 15 µV |
| **Nondetector:**<br>Thermal | <br>1 °C |
| Pneumatic:<br>Flow<br>Pressure<br>Diagnostic | <br>1 mL/min<br>1 pressure unit (psi, bar, or kPa)<br>Mixed, some unscaled |

When outputting a column pressure signal, the GC reports the pressure in absolute units. For example, an inlet pressure of 68.9 kpa would be reported as 170.2 kpa.

## Analog signals

If you use an analog recorder, you may need to adjust the signal to make it more usable. **Zero** and **Range** in the Signal parameter list do this.

### Analog zero

**Zero** Subtracts value entered from baseline. Either select **On** to set Zero at the current signal value, or enter a number between -500,000 and +500,000 as the setpoint to subtract from the baseline.

This is used to correct baseline elevation or offsets. A common application is to correct a baseline shift that occurs as the result of a valve operation. After zeroing, the analog output signal is equal to the **Value** line of the parameter list minus the **Zero** setpoint.

**Zero** can be programmed as a run time event. For details, see **"Events"**.

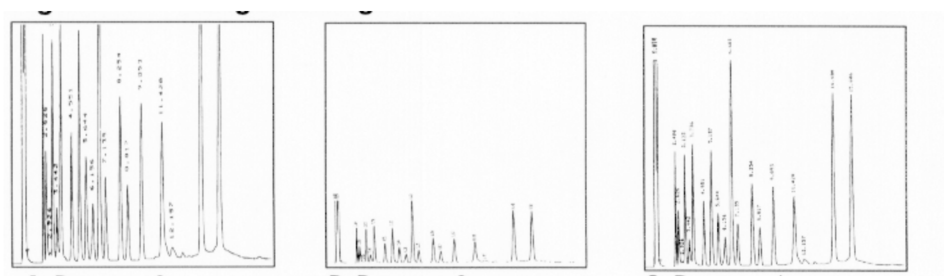
### Analog range

**Range** Scales data coming from the detector

Range is also referred to as gain, scaling, or sizing. It sizes the data coming from the detector to the analog signal circuits to avoid overloading the circuits (clamping). **Range** scales all analog signals.

If a chromatogram looks like A or B in the next figure, the data needs to be scaled (as in C) so that all peaks are visible on the paper.

Valid setpoints are from 0 to 13 and represent $2^0$ (=1) to $2^{13}$ (=8192). Changing a setpoint by 1 changes the height of the chromatogram by a factor of 2. The following chromatograms illustrate this. Use the smallest possible value to minimize integration error.



A: Range = 0 B: Range = 3 C: Range = 1

There are limits to usable range settings for some detectors. The table lists the valid range setpoints by detector.

**Table 11 Range limits**

| Detector | Usable range settings (2x) |
| --- | --- |
| FID | 0 to 13 |
| NPD | 0 to 13 |
| FPD+ | 0 to 13 |
| TCD | 0 to 6 |
| ECD | 0 to 6 |
| Analog input | 0 to 7 |

Range may be run time programmed. See **"Events"** for details.

### Analog data rates

Your integrator or recorder must be fast enough to process data coming from the GC. If it cannot keep up with the GC, the data may be damaged. This usually shows up as broadened peaks and loss of resolution.

Speed is measured in terms of bandwidth. Your recorder or integrator should have a bandwidth twice that of the signal you are measuring.

The GC allows you to operate at two speeds. The faster speed allows minimum peak widths of 0.004 minutes (8 Hz bandwidth), while the standard speed allows minimum peak widths of 0.01 minutes (1.6 Hz bandwidth).

If you use the fast peaks feature, your integrator should operate at around 15 Hz.

### Selecting fast peaks (analog output)

1. Select **Settings** > **Configuration**.
2. Select Analog Out.
3. Select the checkbox next to Fast Peaks.

Agilent does not recommend using **Fast peaks** with a thermal conductivity detector. Since the gas streams switch at 5 Hz, the gain in peak width is offset by increased noise.

## Digital signals

The GC outputs digital signals only to an Agilent data system. The following discussions describe features that impact the data sent to data systems, not the analog data available to integrators. Access these features from the data system. These features are not accessible from the GC touchscreen or Browser Interface.

### Zero signal

Available only from an Agilent data system.

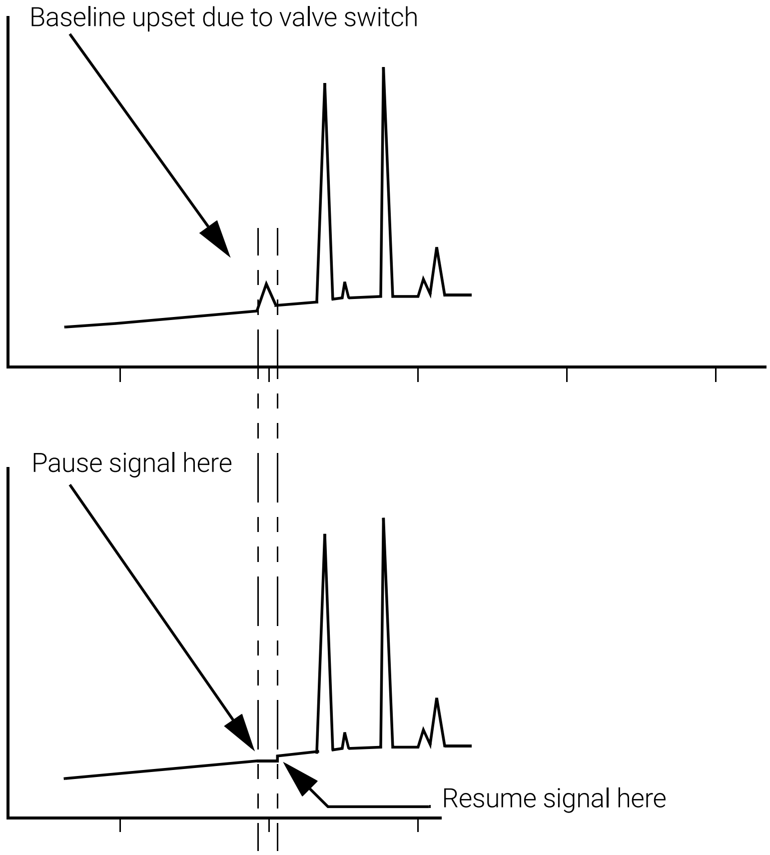
Digital signal outputs respond to a zero command by subtracting the signal level at the time of the command from all future values.

### Signal Freeze and Resume

Available only from an Agilent data system.

Some run time operations, such as changing signal assignments or switching a valve, can cause baseline upsets. Other factors can cause baseline upsets also. The GC can compensate for this by pausing (freezing) the signal at a particular value, using that signal value for a specified duration, and then resuming normal signal output.

Consider a system that uses a switching valve. When the valve switches, an anomaly occurs in the baseline. By freezing and resuming the signal, the anomaly can be removed so that the peak identification and integration software operates more smoothly.




### Data rates with Agilent data systems

The GC can process data at various data rates, each corresponding to a minimum peak width. The table shows the effect of data rate selection.

#### Table 12 Agilent data system data processing

| Data rate, Hz | Minimum peak width, minutes | Relative noise | Detector | Column type |
|---|---|---|---|---|
| 1000 | 0.0002 | 6.96 | NPD | Narrow-bore, 0.05 mm |
| 500 | 0.0004 | 5 | FID/NPD | Narrow-bore, 0.05 mm |
| 200 | 0.001 | 3.1 | FID/FPD+/NPD | Narrow-bore, 0.05 mm |
| 100 | 0.002 | 2.2 | FID/FPD+/NPD | Capillary |
| 50 | 0.004 | 1.6 | ECD/FID/FPD+/NPD | |
| 20 | 0.01 | 1 | ECD/FID/FPD+/NPD | |
| 10 | 0.02 | 0.7 | ECD/FID/FPD+/NPD | |
| 5 | 0.04 | 0.5 | ECD/FID/FPD+/NPD /TCD | to |

### Table 12 Agilent data system data processing (continued)

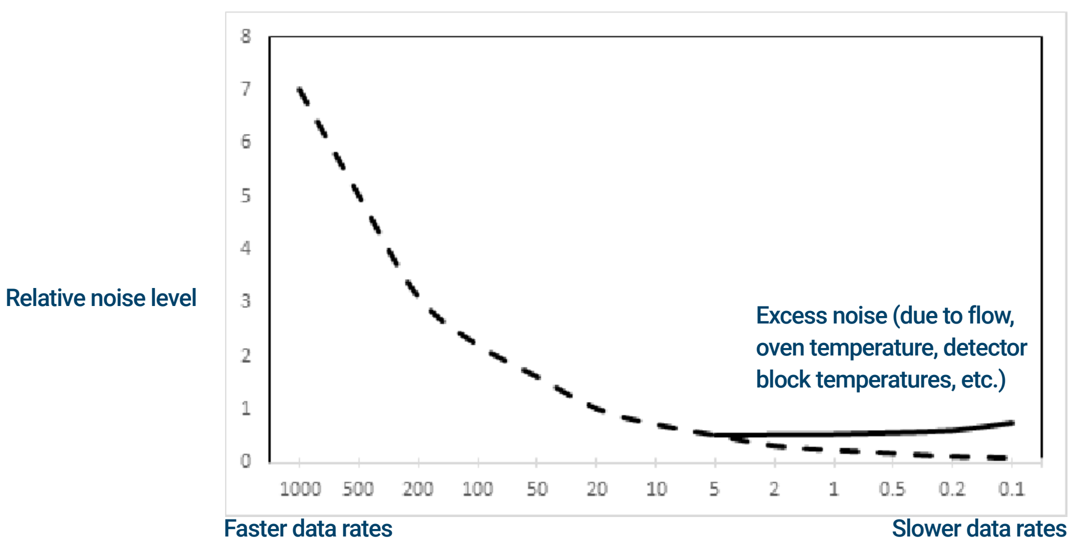
| Data rate, Hz | Minimum peak width, minutes | Relative noise | Detector | Column type |
|---|---|---|---|---|
| 2 | 0.1 | 0.3 | ECD | |
| 1 | 0.2 | 0.22 | ECD | |
| 0.5 | 0.4 | 0.16 | ECD | |
| 0.2 | 1.0 | 0.10 | ECD | |
| 0.1 | 2.0 | 0.07 | ECD | Slow packed |

You cannot change the data rate during a run.

You will see higher relative noise at the faster sampling rates. Doubling the data rate can double peak height while the relative noise increases by 40%. Although noise increases, the signal-to-noise ratio is better at the faster rates.

This benefit only occurs if the original rate was too low, leading to peak broadening and reduced resolution. We suggest that rates be chosen so that the product of data rate and peak width in seconds is about 10 to 20.

The figure shows the relationship between relative noise and data rates. Noise decreases as the data rate decreases until you get to data rates of around 5 Hz. As the sampling rate slows, other factors such as thermal noise increase noise levels.



# 6 Sequences

# What is a Sequence?

A sequence is a list of samples to be analyzed along with the method to be used for each analysis. Sequences can be set up from the Browser Interface, or from the connected Agilent data system. Refer to the help provided in the Browser Interface or data system for details.

# Recoverable Errors

For details on how this feature works in your data system, refer to its help and documentation.

Some types of errors, such as an ALS missing vial error or a headspace sampler vial size mismatch, may not always justify stopping an entire sequence. These errors are called *recoverable errors*, since you may be able to recover from them and continue running a sequence, if desired. Agilent data systems now provide features to allow you to control how the system will react to these types of errors. When using an Agilent data system, the data system will now control whether or not the sequence pauses, aborts completely, continues with the next sample, and so on, for each type of recoverable error.

Note that the data system only controls what happens to the *next* run in the sequence, not the *current* run, except when set to immediately abort. (In that case, the data system typically aborts the current run and the sequence.)

For example, selecting the **Stop** control on the GC always halts the current run. However, the data systems can allow you to choose whether to continue with the next run or to pause or to abort the whole sequence.

# 7 Diagnostics

# About Diagnostics

The GC provides diagnostics features for inlets, detectors, and other installed components. This includes tests that are performed by the operator and automated testing that is performed by the GC without operator intervention.

The Diagnostics view provides access to the system health report and operator initiated diagnostic tests.

Additionally, the view provides a list of all current alerts. When a condition arises, the GC lists the problem in Diagnostics. Select a condition to see a description of the issue and recommended diagnostic tasks that might help resolve it. If a listed diagnostic task is automated, select it to run it.

## The system health report

To access the system health report, select **System Health Report**. The system health report appears.

The system health report includes the following types of information:

- System information
- System Configuration details
- Active instrument conditions
- Column details
- Early Maintenance Feedback details
- Diagnostic test results
- Network information
- Status snapshot information

## Automated testing

The GC performs continuous, automated testing of the following items. If a failure occurs, an alert appears on the diagnostic tab. In addition, an entry is made in the appropriate log.

See below for a list of the continuous monitoring dialogs:

Detector:

- Supply voltage
- ADC (analog to digital converter) references
- FID flameout
- NPD bead open/short
- Igniter open/short
- Collector short
- Electrometer not plugged in
- TCD filament open or short

- TCD switching valve open or short
- FPD flameout

EPC (Electronic Pneumatic Control) - Inlets, detectors, and other modules.

ADC (analog to digital converter) references

Actuator movements

Thermal:

- Sensor open/short
- Missing heater
- Wrong heater
- Heater current:
  - Quiescent
  - Leakage

Configuration Mismatch

# Self-guided diagnostics

The GC offers several useful diagnostic checks to use when troubleshooting issues with the GC or method. See below for a list of self-guided diagnostic tests available:

Inlet tests:

- Leak & Restriction test
- Gas Supply Pressure test
- Split Vent Restriction test
- Pressure Decay test
- Septum Purge test

Detector tests:

- FID Jet Restriction test
- FID Leakage Current test

# Using the Diagnostics View

To use the Diagnostics view:

1. Select **Diagnostics**. The Diagnostics view appears. The view provides a list of all current alerts.
2. Select **Diagnostic Tests**. The Diagnostic Tests page appears. .
3. Select **Inlets, Detectors**, or **Other**, as desired. The corresponding page will appear. For example, selecting **Inlets** brings up the Inlet Diagnostic Tests page.

# Performing Diagnostic Tests

To perform a diagnostic test:

1 Access the desired test from the Diagnostics view. See **"Using the Diagnostics View"**.

2 Select the desired test. The corresponding test page appears. The test page includes a test description and an indication of the parameter being tested.

3 Select **Start Test**. Testing is initiated. Test details are displayed along with test results.

The currently running test can be aborted by selecting **Cancel**. This brings up a dialog box which allows you to confirm that you want to cancel the test.

# 8 Maintenance



Selecting the maintenance navigation tab shows buttons for counters by component, lets you view the maintenance log, and lets you start automated, step-by-step maintenance procedures for various components. These procedures also include automated tests as needed to help-be sure the GC is ready when maintenance is complete. See the *8860 Maintaining Your GC manual* for more information.

# Early Maintenance Feedback (EMF)

The 8860 provides injection- and time-based counters for various consumable and maintenance parts. Use these counters to track usage and replace or recondition these items before potential degradation impacts the chromatographic results.

If using an Agilent data system, these counters can be set and reset from within the data system.

## Counter types

Counters are provided for injections, runs, and time. Each type is described below.

**Injection** counters increment whenever an injection occurs on the GC via an ALS injector, headspace sampler, or sampling valve. Manual injections do not increment counters. The GC differentiates between front and back injections, and only increments counters associated with the configured injection flow path.

For example, consider the following GC:

**Table 13**

| Configured front flow path | Configured back flow path |
| --- | --- |
| Front injector | Back injector |
| Front inlet | Back inlet |
| Column 1 (GC oven) | Column 2 (GC oven) |
| Purged Union / Aux EPC 1 | Back detector |
| Column 3 (GC oven) | |
| Front detector | |

In this example, for a front ALS injection, the GC will increment counters for the front injector, front inlet, and the front detector, but it would not increment the back injector, back inlet, or back detector counters. For the columns, the GC would increment the injection counters for columns 1 and 3, and the oven cycles counter for all 3 columns.

**Run** counters increment against the number of runs performed on the GC.

**Time** counters increment against the GC clock. Changing the GC clock changes the age of tracked consumables.

## Thresholds

The EMF feature provides two warning thresholds: **Service Due** and **Service Warning**. When either threshold is passed, an indication appears on the **Maintenance** tab on the GC touchscreen ribbon.

Selecting the **Maintenance** tab brings up the Maintenance view.

Selections are available for any installed component.

Two thresholds are settable for any given item:

- **Service Due**: When the counter exceeds this number of injections, runs, or days, a red warning icon appears on the corresponding button, and an entry is made in the **Maintenance Log**.
- **Service Warning**: When the counter exceeds this number of injections or days, an orange warning icon appears on the corresponding button, indicating that the component needs maintenance soon.

Both thresholds are set independently for each counter. You can enable one or both, as desired. The **Service Due** limit must be larger than the **Service Warning** limit.

## Default Thresholds

Selected counters have default thresholds to use as a starting point.

If you want to change a default limit, enter a conservative limit based on your experience. Use the warning feature to alert you when service is approaching, then track performance to determine if the **Service Due** threshold is too high or too low.

For any EMF counter, you may need to adjust the threshold values based on the demands of your applications.

# Perform Maintenance

For many common maintenance procedures, your GC contains a detailed walkthrough of each procedure. These procedures may be accessed by selecting **Maintenance** > your desired component > **Perform Maintenance**. Once here, select your desired maintenance procedure, and select **Start Maintenance** to begin the walkthrough.

Many maintenance procedures require you to put the GC in maintenance mode prior to performing any maintenance. To do so, select **Maintenance** > **Instrument** > **Perform Maintenance**, select the checkbox next to Maintenance Mode, and then select **Start Maintenance**. Putting the GC into maintenance mode can additionally include:

- Setting low temperatures to avoid burns and other injuries
- Setting reduced flows to avoid safety hazards and to prevent damage to the instrument
- Venting a mass selective detector (MSD)
- Making other settings to prevent damage to the instrument (electronics, columns, and so forth) or to connected instruments (MSD)

For example, to change the gas clean filter, select **Maintenance** > **Instrument** > **Perform Maintenance**, select the checkbox next to Gas Clean Filter Maintenance, and then select **Start Maintenance**. This cools down the required components of the GC, and then walks you through the procedure for replacing the gas clean filter.

# Available Counters

**Table 14** lists the most common counters available. The available counters will vary based on the installed GC options, consumables, and future updates.

### Table 14 Common EMF counters

| GC Component | Parts with a counter | Type | Default value |
|---|---|---|---|
| **Detectors** | | | |
| FID | Collector | Number of injections | |
| | Jet | Number of injections | |
| | Ignitor | Number of ignition attempts | |
| TCD | Switching solenoid | On time | |
| | Filament on time | On time | |
| ECD | Insert liner | Number of injections | |
| | Time since wipe test | On time | 6 months |
| NPD | Bead | Number of injections | |
| | Collector | Number of injections | |
| | Bead baseline offset | pA Value | |
| | Bead baseline voltage | Voltage value | Blos bead: 1.045 |
| | Bead current integral | pA-sec Value | |
| | Bead on time | On time | Blos bead: 2400 h |
| FPD+ | Ignitor | Number of ignition attempts | |
| | PMT | Number of injections | |
| | PMT | On time | 6 months |
| **Inlets** | | | |
| SSL | Gold seal | Number of injections | 5000 |
| | Gold seal | Time | 90 days |
| | Liner | Number of injections | 200 |
| | Liner | Time | 30 days |
| | Liner O-ring | Number of injections | 1000 |
| | Liner O-ring | Time | 60 days |
| | Septum | Number of injections | 200 |
| | Split vent trap | Number of injections | 10,000 |
| | Split vent trap | Time | 6 months |
| PP | Liner | Number of injections | 200 |
| | Liner | Time | 30 days |

### Table 14 Common EMF counters (continued)

| GC Component | Parts with a counter | Type | Default value |
|---|---|---|---|
| | Septum | Number of injections | 200 |
| | Top weldment O-ring | Number of injections | 10,000 |
| | Top weldment O-ring | Time | 1 year |
| COC | Septum | Number of injections | 200 |
| **Columns** | | | |
| Column | Injections onto column | Number of injections | |
| | Oven cycles | Number of injections | |
| | Length | Value | |
| | Run count | Number of runs | |
| **Valves** | | | |
| Valve | Rotor | Activations (number of injections) | |
| | Maximum temperature | Value | |
| **Instrument** | | | |
| Instrument | On time | Time | |
| | Run count | Number of runs | |
| | Filters | Time | |
| **ALS Injectors** | | | |
| ALS | Syringe | Number of injections | 800 |
| | Syringe | Time | 2 months |
| | Needle | Number of injections | 800 |
| | Plunger moves | Value | 6000 |
| **Mass spectrometers** | | | |
| Mass spectrometer | Pump | Time (days) | 1 year |
| | Filament 1 | Time (days) | 1 year |
| | Filament 2 | Time (days) | 1 year |
| | Source (time since last cleaning) | Time (days) | 1 year |
| | EMV at last tune | V | 2600 |

# Viewing Maintenance Counters

To view the maintenance counters:

1 Select the **Maintenance** tab.

2 Select the desired component type. The Status column lists the counter for the corresponding component.

3 Scroll to view additional components, as applicable.

# To Enable, Reset, or Change a Limit for an EMF Counter

When using the GC without a data system, enable or change the limit for a counter as follows:

1 Locate the counter you wish to change. See **"Viewing Maintenance Counters"**.

2 Select the Component listing for the counter you want to change.

3 To modify a threshold:

  a Select the threshold entry. A data entry dialog box appears.

  b Enter the desired value. See **"Default Thresholds"**.

4 To enable or disable a warning, select or deselect **Enable** for the corresponding counter.

5 Select **Apply**. The dialog box closes. The entered value is displayed in the corresponding field.

6 To reset the counter:

  a Select **Reset Counter**. A confirmation dialog box appears.

  b Select **Yes**. The confirmation dialog box closes.

7 Select **Apply**.

# EMF Counters for Autosamplers

The GC provides access to the counters for the autosampler. The functionality for ALS counters depends on the ALS model and firmware version. In all cases, the GC shows EMF counter status and allows you to enable, disable, and reset the counters using the Browser Interface.

## Counters for 7693A and 7650 ALS with EMF-enabled firmware

If using an Agilent 7693 injector with firmware version G4513A.10.8 (or higher), or a 7650 injector with firmware version G4567A.10.2 (or higher), each injector independently tracks its EMF counters.

- The injector counters will increment as long as the injector is used on an 8860 Series GC. You can change positions on the same GC or install the injector on a different GC without losing the current ALS counter data.
- The ALS will report an exceeded limit only when mounted on an 8860 GC.

## Counters for ALS with earlier firmware

If using a 7693 or 7650 injector with earlier firmware, or if using another injector model, the GC tracks the counters for that injector. The GC uses the injector serial number to distinguish between installed injectors, but only maintains up to two sets of counters—one for the front injector, and one for the back injector.

- The GC will track injector counters regardless of installed position (front or back inlet). Because the GC tracks the injector serial number, you can change the injector position without losing the counters as long as the injector remains installed on the GC.
- Each time the GC detects a new injector (different model or different serial number), the GC resets the ALS counters in the new injector’s position.

# EMF Counters for MS Instruments

For GC-MS, GC-HS, and GC-MS-HS systems, all EMF counters are available at the Browser Interface. In addition, most counters can be reset at the Browser Interface. Some counter types, for example, a counter which requires a calibration to be performed on a headspace sampler, cannot be reset at the Browser Interface, but can still be viewed.

# 9 Logs



This section describes the log features available on the Agilent 8860 GC.

# Logs View

The Logs view provides listings of GC events including maintenance events, run events, and system events, sorted by date/time.

Selecting one of the buttons on the Logs view brings up the corresponding log page.

For Maintenance and System log items, items are sorted by date and time. For Run log items, relative time (from the start of the run) is used.

Use the scroll buttons to scroll through the log entries.

Select **Cancel** to return to the Logs view.

## Maintenance logs

The maintenance log contains entries made by the system when:

- A system event occurs (for example, a detector shutdown)
- Any component counter reaches a monitored limit

The log entry contains a description of the maintenance event and the date/time that the event occurred. In addition, each user task related to the counter is recorded in the log, including resetting, enabling, or disabling monitoring, and changing limits or units (cycles or duration).

## Run log

The run log is cleared at the start of each new run. During the run, any deviations from the planned method (including touchscreen or Browser Interface intervention) are listed in the run log table.

## System log

The system log records significant events that occur during GC operation. Some of the events also appear in the run log if they occur during a run.

# 10 Settings

# About Settings

The Settings view provides access to configuration and system settings for the GC.

Selecting **Settings** on the touchscreen control ribbon brings up the Settings view.

- Select **Scheduler** to access the GC Instrument Schedule settings. See **"Resource Conservation"**.
- Select **Service Mode** to access the service mode settings for the GC. See **"Service Mode"**.
- Select **About** to get information about this GC. See **"About"**.
- Select **Calibration** to access the calibration functions. See **"Calibration"**.
- Select **System Settings** to access the system settings for the GC, including setting the network address, system date and time, touchscreen settings, system setup information, and so on. See **"System Settings"**.
- Select **Tools** to access the Tools page. See **"Tools"**.

# Service Mode

The Service Mode feature allows you to view the installed GC system component details. This includes serial numbers, firmware versions, voltages, currents, temperatures, and so on.

To view the specifications for the various components of your instrument:

1. Select the desired component type. The Service Mode page for the selected component appears.
2. Use the page selection buttons on the left side of the page to display related functional information.

# About

The About feature allows you to view details about the GC.

The About screen lists the GC manufacturing date, serial number, firmware revision, and help and information revision.

Select **Close** on the About page to return to the Settings view.

# Calibration

Calibration allows you to adjust the following items (when available):

- ALS
- Inlets
- Oven
- Detectors
- EPC modules

To change the calibration settings:

1. Use the page selection buttons on the left side of the page to display related functional information.
2. Make changes to calibration settings as desired. See **"Maintaining EPC calibration—inlets, detectors, PCM, and AUX"**, **"To zero a specific flow or pressure sensor"** for more information.
3. Select **Apply**. The entered changes are saved to the GC.

## Maintaining EPC calibration—inlets, detectors, PCM, and AUX

The EPC gas control modules contain flow and/or pressure sensors that are calibrated at the factory. Sensitivity (slope of the curve) is quite stable, but zero offset requires periodic updating.

### Flow sensors

All inlet modules use flow sensors, as does channel 1 of a PCM. If the **Auto flow zero** feature is selected, they are zeroed automatically after each run. This is the recommended setting. They can also be zeroed manually—see **"To zero a specific flow or pressure sensor"**."

### Pressure sensors

All EPC control modules use pressure sensors. They must be zeroed individually. There is no automatic zero for pressure sensors.

### Auto Zero flow

A useful calibration option is **Auto Zero flow**. When selected, after the end of a run, the GC shuts down the flow of gases to an inlet, waits for the flow to drop to zero, measures and stores the flow sensor output, and turns the gas back on. This takes about two seconds. The zero offset is used to correct future flow measurements.

### Auto zero septum purge

This is similar to **Auto zero flow**, but is for the septum purge flow.

### Zero conditions

Flow sensors are zeroed with the carrier gas connected and flowing.

Pressure sensors are zeroed with the supply gas line disconnected from the gas control module.

### Zero intervals

**Table 15 Flow and Pressure Sensor Zero Intervals**

| Sensor type | Module type | Zero interval |
|---|---|---|
| Flow | All | Use Auto flow zero and/or Auto zero septum purge |
| Pressure | Inlets | |
| | Small capillary columns (id ≤ 0.32 mm) | Every 12 months |
| | Large capillary columns (id > 0.32 mm) | At 3 months, at 6 months, then every 12 months |
| | Auxiliary channels | Every 12 months |
| | Detector gases | Every 12 months |

# To zero a specific flow or pressure sensor

1 Select **Settings** > **Calibration** > **Detectors**, and select the desired detector.

2 Select **On** next to the desired sensor to zero it.

3 For **Flow sensors**. Verify that the gas is connected and flowing (turned on).

4 For **Pressure sensors**. Disconnect the gas supply line at the back of the GC. Turning it off is not adequate; the valve may leak.

5 Reconnect any gas line disconnected in the previous step and restore operating flows.

# System Settings

System settings includes setting the network address, system date and time, touchscreen theme, disk space and data settings, locale setting, system setup information, and status parameter settings.

Use the page selection buttons on the left side of the page to display related functional information.

Select **Save** to apply any changes made to the GC.

## Configuring the IP address for the GC

For network (LAN) operation, the GC needs an IP address. It can get this from a DHCP server, or it can be entered directly from the touchscreen. In either case, see your LAN administrator for appropriate settings.

### To use a DHCP server

1 From the System Settings page, select the **Network** page selection button. The Network Configuration page appears.

2 Select **Enable DHCP**.

3 Select **Apply**.

4 If prompted, restart the GC.

### To set the LAN address at the touchscreen

1 Select the System Settings page.

2 If **Enable DHCP** is selected:

   a Deselect **Enable DHCP**.

   b When prompted, restart the GC.

   c Return to the System Settings page

3 Enter the **Host Name** or the **IP Address** in its corresponding field

4 Enter the **Gateway** number in the corresponding field.

5 Enter the subnet mask in the **Net Mask** field.

6 Select **Apply**.

7 If prompted, restart the GC.

## To change the system locale

1 From the System Settings page, select the **Locale** page selection button. The Locale Settings page appears.

2 Choose the desired **Language** from the corresponding drop-down list box.

3 Select **Apply**. The GC saves the change made. The system is changed to the selected locale. This may take a few moments.

**NOTE**

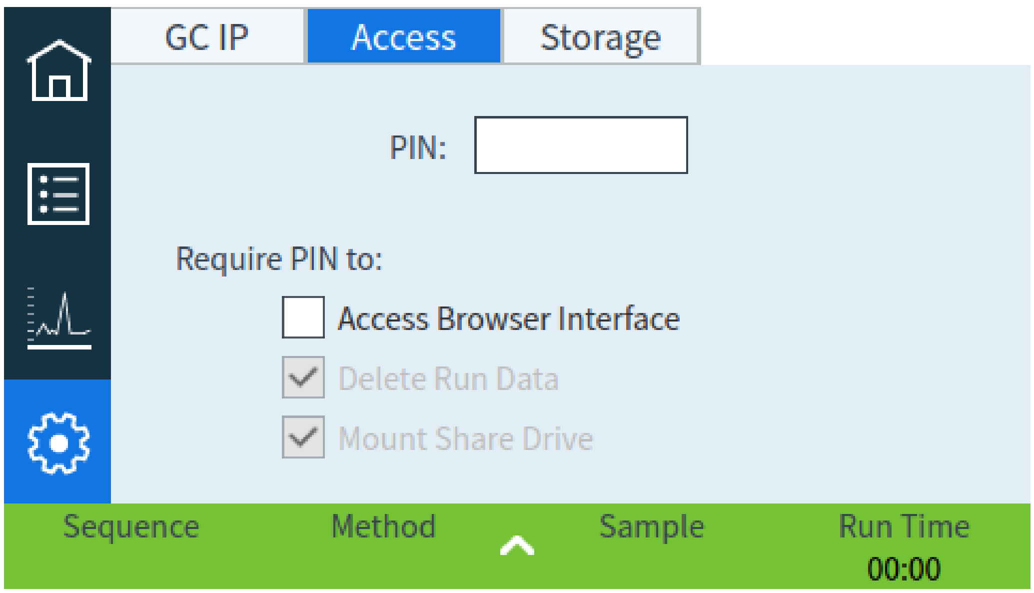
**The dim display value must be less than the display brightness value.**

**The display brightness value must be greater than 0.**

## To access stored run data

If using the Browser Interface to perform runs and collect data, the GC stores the result data internally. To access that data:

1 From the **Settings** page, select the **Access** tab. Note the displayed PIN.




2 Select the **Storage** tab. Note the path to the GC share.

3 On your PC, map a network drive to the GC share. When prompted, connect using the credentials:

user: results

password: the PIN (default: 0000).

## To control Browser Interface access

The GC is set so that a four-digit PIN must be used to perform the following actions for your GC:

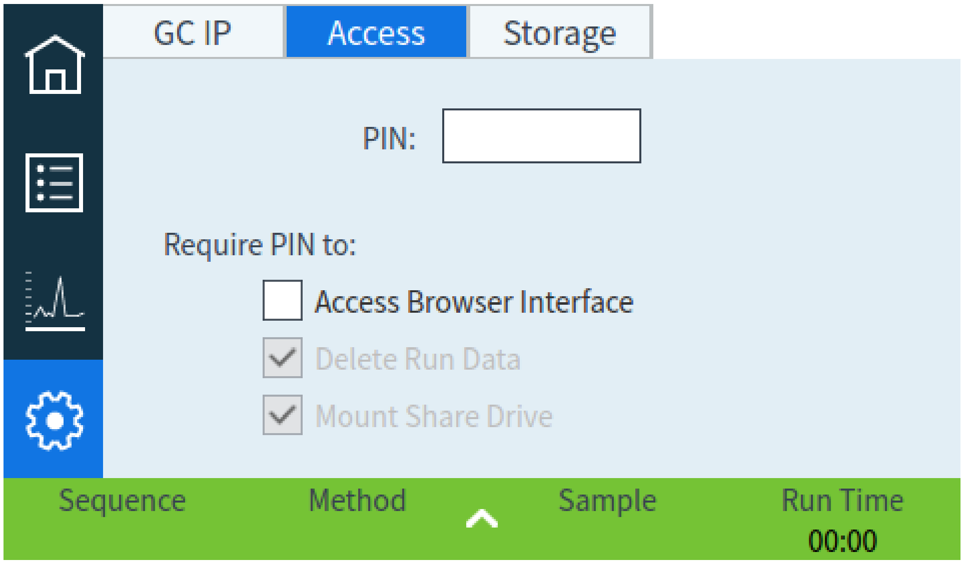
- Delete run data.
- Mount a share drive.

By default, the PIN is set to 0000. Additionally, you may choose to require the PIN to access the Browser Interface. To set the PIN:

1 From the **Settings** page, select the **Access** tab.

2 Select the four-digit PIN to input a new PIN.

3 If desired, select the checkbox next to Access Browser Interface to require the PIN for Browser Interface access.



# Tools

The Tools page allows you to perform column compensation runs for the installed columns on the GC.

In temperature programmed analysis, bleed from the column increases as the oven temperature rises. This causes a rising baseline which makes peak detection and integration more difficult. Column compensation corrects for this baseline rise.

A column compensation run is made with no sample injected. The GC collects an array of data points from any installed detectors. If a detector is not installed or is turned off, that part of the array is filled with zeros.

Each array defines a set of curves, one for each detector, that can be subtracted from the real run to produce a flat baseline.

When a connected data system is used, the raw signal, and the column compensation data, is output to the data system so that a compensated, and uncompensated, signal are available for analysis.

## Performing a column compensation run

All conditions must be identical in the column compensation run and the real run. The same detector and column must be used, operating under the same temperature and gas flow conditions.

Up to four column compensation runs can be made. The GC retains the results of these runs for later use.

Any column compensation run can then be used to compensate a rising baseline during a run.

1 With the Tools page displayed, select the desired **Column Compensation** in the **Start Specified Run** column. The GC performs the column compensation run. No injection occurs as a part of this run.

2 Use the connected data system to edit the method. Set the detector to **Subtract from Signal: Column compensation Curve #x** (where **x** is the number of the column compensation run).

3 Run the method. The results use the column compensation run data to compensate for baseline changes in the column.

# 11 Configuration

# About Configuration

A device's configuration properties are constant for an instrument hardware setup, unlike method settings which can change from sample run to sample run. Two example configuration settings are the gas type flowing through a pneumatic device and the operating temperature limit of a device. The GC will try to set any configuration properties necessary for any device that it finds installed. For example, if the GC knows it is connected to an MSD, it will automatically configure the MSD heated transfer line. The only settings that need to be configured manually are items that can be changed depending on your analysis, such as installed columns and carrier gas type, or an item which may be installed in a non-standard location. For example, an auxiliary EPC module installed in an inlet EPC module position may require manual configuration.

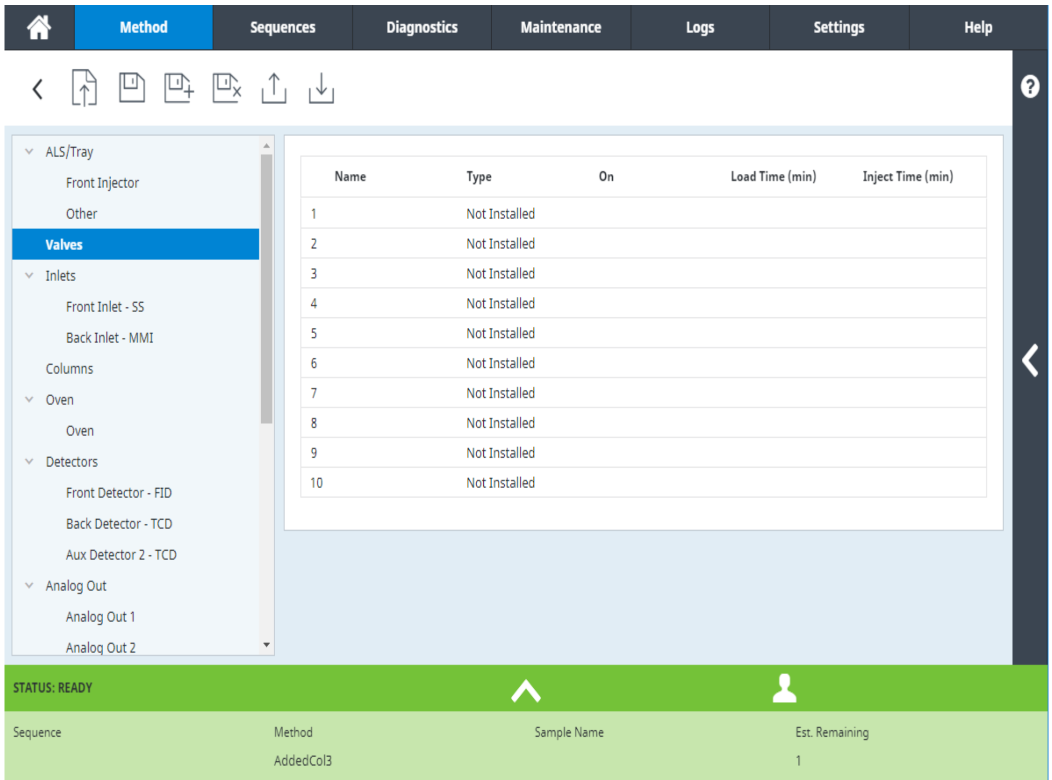
Ensure all devices have been configured correctly prior to running any samples.

# Configuration Changes

4

To change the configuration properties for a device:

Select **Method**.




5 Select the desired device type from the list on the left side of the screen. The properties for the selected device type appear on the right side of the screen.

6 Scroll to the device setting and change the property. This can involve making a selection from a list, or entering a numeric value.

7 When all required changes have been made, select the Save icon .

# Valve Configuration

Valve configuration provides the ability to specify valve types, loop volumes, step times, and BCD inversion settings. BCD inversion allows for changing the BCD input (1's become 0's and 0's become 1's). This feature accommodates coding convention differences between valve manufacturers.

Note that the Valves page appears regardless of whether any valves are currently installed in the GC.

## To configure valves

1. Select **Method** > **Valves**.
2. Use the **Valve Type** drop-down list box to select the corresponding valve type. The possible valve types are:
   - **Gas Sampling** Two-position (load and inject) valve. In load position, an external sample stream flows through an attached (gas sampling) or internal (liquid sampling) loop and out to waste. In inject position, the filled sampling loop is inserted into the carrier gas stream. When the valve switches from Load to Inject, a run starts if one is not already in progress.
   - **Switching** Two-position valve with four, six, or more ports. These are general-purpose valves used for such tasks as column selection, column isolation, and many others.
   - **Other** Something else.
   - **Not installed** Self-explanatory.
3. To enter the loop volume and step time, select **Method** > **Configuration** > **Miscellaneous**, and set the values as desired.
4. When all required changes have been made, select the Save icon .

# Inlet Configuration

1. Select **Method** > **Inlets** > your desired inlet. The properties for the selected device type appear on the right side of the screen.
2. Select the desired gas type from the **Carrier Gas Type** drop-down list box.
3. Select Readiness from the list of devices on the left, and then click the checkbox next to your inlet to enable its readiness. If Readiness is disabled, the GC becomes ready even if this device has not reached or cannot reach its setpoints.
4. Adjust the settings for the second inlet as necessary.
5. When all required changes have been made, select the Save icon.

# Columns

**Length** The length, in meters, of a capillary column. Enter **0** for a packed column or if the length is not known.

**Diameter** The inside diameter, in millimeters, of a capillary column. Enter **0** for a packed column.

**Film thickness** The thickness, in microns, of the stationary phase for capillary columns.

**Inlet** Identifies the source of gas for the column.

**Outlet** Identifies the device into which the column effluent flows.

**Thermal zone** Identifies the device that controls the temperature of the column.

## To configure a single column

You define a capillary column by entering its length, diameter, and film thickness. You then enter the device controlling the pressure at the Inlet (end of the column), the device controlling the pressure at the column Outlet, and the Thermal zone that controls its temperature.

With this information, the instrument can calculate the flow through the column. This has great advantages when using capillary columns because it becomes possible to:

- Enter split ratios directly and have the instrument calculate and set the appropriate flow rates.
- Enter flow rate or head pressure or average linear velocity. The instrument calculates the pressure needed to achieve the flow rate or velocity, sets that, and reports all three values. (Split/splitless inlet only.)
- Perform splitless injections with no need to measure gas flows.
- Choose any column mode. Except for the simplest configurations, such as a column connected to a specific inlet and detector, we recommend that you begin by making a sketch of how the column will be connected.

To configure a capillary column:

1. Select **Method** > **Configuration** > **Columns**.
2. Double click the column you want to configure.
3. Under Column Type, select **Capillary**.
4. Input the Length, Diameter, and Film Thickness into the appropriate fields.

   If you do not know the column dimensions—they are usually supplied with the column—or if you do not wish to use the GC calculating features, enter **0** for either **Length** or **Diameter**. The column will be not defined.

NOTE

**Agilent recommends always defining capillary columns.**

5 Under Inlet connection, select the desired inlet from the dropdown menu. Selections include the installed GC inlets and installed PCM channels.

6 Under Outlet connection, select the desired outlet from the dropdown menu.

- When a detector is selected, the outlet end of the column is controlled at 0 psig for the FID, TCD, FPD+, NPD, and ECD or vacuum for the MSD.
- If the column exhausts into a nonstandard detector or environment (neither ambient pressure nor complete vacuum), select **Other** and enter the outlet pressure.

7 Under Heated By, select the desired thermal zone from the dropdown menu.

8 Set the Min Temp, Max Temp, and Max Program Temp for your column.

9 Set the column's

- Manufacturer
- Serial Number

10 Click OK to confirm your changes.

11

12 When all required changes have been made, select the Save icon .

# To configure a packed column

To configure a packed column:

1 Select **Method** > **Configuration** > **Columns**.

2 Double click the column you want to configure.

3 Under Column Type, select **Packed**.

4 Set the Min Temp, Max Temp, and Max Program Temp for your column.

5 Set the column's

- Manufacturer
- Serial Number

6 Under Inlet connection, select the desired inlet from the dropdown menu. Selections include the installed GC inlets and installed PCM channels.

7 Under Outlet connection, select the desired outlet from the dropdown menu.

- When a detector is selected, the outlet end of the column is controlled at 0 psig for the FID, TCD, FPD+, NPD, and ECD or vacuum for the MSD.
- If the column exhausts into a nonstandard detector or environment (neither ambient pressure nor complete vacuum), select **Other** and enter the outlet pressure.

8 Under Heated By, select the desired thermal zone from the dropdown menu.

9 Select **Apply**. This saves the changes to the GC.

# Additional notes on column configuration

You should check configurations for all columns to verify that they specify the correct pressure control device at each end. The GC uses this information to determine the flow path of the carrier gas. Only configure columns that are in current use in your GC's carrier gas flow path. Unused columns configured with the same pressure control device as a column in the current flow path cause incorrect flow results.

It is possible, and sometimes appropriate, to configure both installed columns to the same inlet.

Some pneumatic setpoints change with oven temperature because of changes in gas viscosity. This may confuse users who observe pneumatics setpoints changing when their oven temperature changes. However, the flow condition in the column remains as specified by the column mode (constant flow or pressure, ramped flow or pressure) and the initial setpoint values.

# Oven

**Oven Standby** When enabled, the oven temperature changes to the specified Temperature after the GC has been idle for the entered Time. A usage example would be to set the standby temperature to a relatively high value to keep the system clean between runs (reduce carryover) when the GC may sit idle for long periods.

**Maximum temperature** Sets an upper limit to the oven temperature. Used to prevent accidental damage to columns. The range is 70 to 425 °C. See the column manufacturer's recommendations.

## To configure the oven

1 Select **Method** > **Oven**.

2 Use the oven configuration window to set the Maximum Oven Temperature, Equilibration Time, and oven Temperatures.

3 When all required changes have been made, select the Save icon .

# Detector Configuration

If you are operating with column defined, you have a choice of two makeup gas modes. To set the makeup gas for a detector:

1. Select **Method** > **Detectors**, then select the detector you wish to configure.
2. If available, select the makeup gas type that is plumbed to the detector. Depending on your detector type and the carrier gas selection, the gas type may be already set for you.
3. Make or enter settings for the detector, as applicable:
   - **Lit Offset**. FID and FPD+. See **"Detectors"**.
   - Readiness. Enable if using this detector. When disabled, the GC becomes ready even if this device has not reached or cannot reach its setpoints.
   - Target Offset. NPD only. Enter the default target offset. The GC will slowly try to reach this target value after the detector flows are read, the temperature is stable, and the dry bead time has elapsed.
4. When all required changes have been made, select the Save icon.

# Analog Output Settings

When connecting the GC to an integrator or recorder, the device must be fast enough to process data coming from the GC. If it cannot keep up with the GC, the data may be incorrect or corrupted. This usually shows up as broadened peaks and loss of resolution.

Speed is measured in terms of bandwidth. The recorder or integrator should have a bandwidth twice that of the signal being measured.

The GC can operate at two speeds. The faster speed allows minimum peak widths of 0.004 minutes (8 Hz bandwidth), while the standard speed allows minimum peak widths of 0.01 minutes (1.6 Hz bandwidth).

If using fast peaks feature, the integrator should operate at approximately 15 Hz.

NOTE

**Agilent does not recommend using Fast peaks with a TCD detector. Since the gas streams switch at 5 Hz, the gain in peak width is offset by increased noise.**

To change the analog output settings:

1 Select **Method** > **Analog Out**, and then select either **Analog Out 1** or **Analog Out 2**.

2 To use fast peaks for either analog output channel, select the corresponding **Fast Peaks** check box.

# MSD Configuration

## MSD configuration

The method for configuring a connected MSD varies based on the model of MSD being used.

### 5977B GC/MSD

The 5977B connects to the GC via an LVDS cable to one of the ELVDS communication ports on the rear of the GC. Because of this, the GC treats the MSD as a detector. No communication configuration is necessary.

To change the MSD settings:

1 From the touchscreen, select the settings page, and then the MSD config tab. See **Figure 14**.

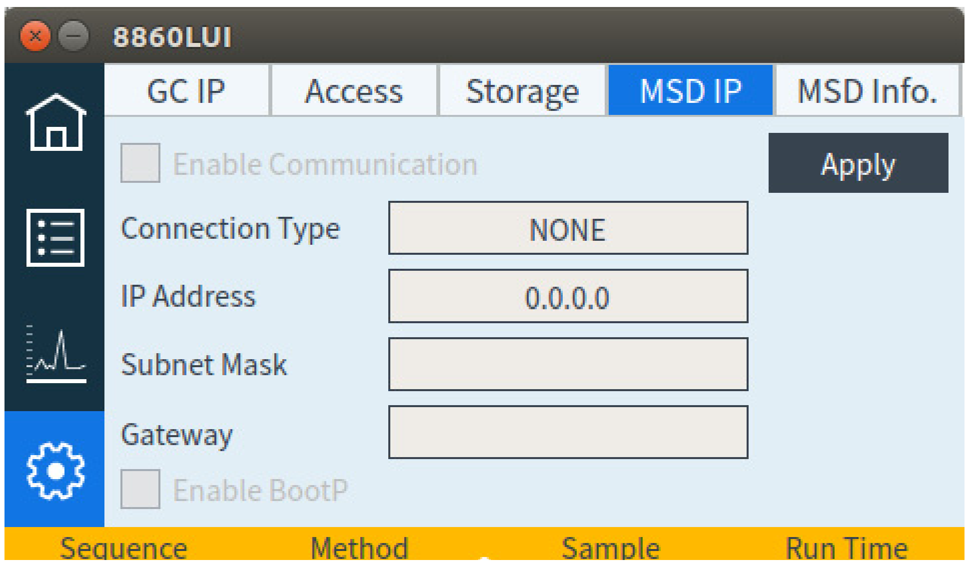



Figure 14. Detector MSD settings page

2 Enter details for, and control, the MSD.

## To enable or disable MS communications

To temporarily disable GC-MS communications:

1 From the Browser Interface, select **Method** > **Detectors**, and select your MS. Alternatively, from the touchscreen, select the settings page, and then the **MSD IP** tab.

2 Scroll until the Enable Communication checkbox is visible.

3 Select or deselect the checkbox next to **Enable Communication** to enable or disable communication with the MS.

# To use the GC when the MS is shut down

To use the GC while an MS is being repaired or maintained, do the following:

Be careful to avoid settings that send carrier gas into the MS, or which raise the temperature of parts which might cause burns when working on the MS.

1 Disable MS communications. See **"To enable or disable MS communications"**.

2 If needed, completely uninstall the MS from the GC.

# Miscellaneous Settings

The GC provides the option to change the pressure units displayed by the GC.

To change the displayed pressure units:

1 Select **Method** > **Configuration** > **Miscellaneous**.
2 Select the desired units type from the **Pressure Units** list.
   - **psi**—pounds per square inch, $lb/in^2$
   - **bar**—absolute cgs unit of pressure, $dyne/cm^2$
   - **kPa**—mks unit of pressure, 103 $N/m^2$
3 Enable or disable Slow Fan for the oven.
4 Set the Thermal Aux type.
5 Set the Loop volume, Step Time, and BCD Inverted for any installed valves.
6 When all required changes have been made, select the Save icon .

# Readiness

The states of the various hardware components are among the factors that determine whether the GC is Ready for analysis.

Under some circumstances, you may not wish to have a specific component's readiness considered in the GC readiness determination. This parameter lets you make that choice. The following components allow readiness to be ignored: inlets, detectors, the oven, and PCM.

For example, suppose an inlet heater is defective but you don't plan to use that inlet today. By deselecting the **Enable** checkbox under **Readiness** for that inlet, you can use the rest of the GC. After the heater is repaired, reselect the checkbox or the run could start before that inlet's conditions are ready.

To ignore a component's readiness, click **Method > Readiness**. Click the checkbox next to the desired component to remove its checkmark.

To re-enable a component's readiness, click **Method > Readiness**. Click the checkbox next to the desired component to restore its checkmark.

# Valve Box

The valve box mounts on top of the column oven. It may contain up to three valves mounted on heated blocks.

Valve positions on the blocks are numbered. We suggest that valves be installed in the blocks in numeric order.

All heated valves in a valve box are controlled by the same temperature setpoint.

To Configure a Valve:

1. Select **Method** > **Configuration** > **Miscellaneous**.
2. For each valve installed, select the valve type, then set the loop volume, step time, and BCD inversion as applicable to the valve type.
3. When all required changes have been made, select the Save icon.

# PCMs

To configure a PCM:

1 Select **Settings** > **Configuration** > **PCMs**.

2 Select the PCMs Aux Mode:

- Forward Pressure: Pressure is being sensed downstream from the flow proportional valve.
- Back pressure: Pressure is being sensed upstream from the flow proportional valve.

3 Select the Gas Type for channel 1, the forward-pressure control channel.

4 Select the Gas Type for the auxiliary channel (channel 2).

Channel 1 of the PCM provides forward pressure or forward flow control. It can be used to provide column flow or pressure for valved systems, sample preparation devices, or advanced flow devices like splitters or switches.

Channel 2, or the auxiliary channel, can only provide forward pressure regulation when plumbed normally, or back pressure regulation when reversed. Thus, channel 2 (reversed) can be used as a controlled leak. If the input pressure drops below setpoint, the regulator can close down to restore it. If the input pressure rises above setpoint, the regulator can open to restore it.

# Aux EPCs

An auxiliary pressure controller provides three channels of forward-pressure regulation. Three modules can be installed for a total of nine channels.

The numbering of the channels depends on where the controller is installed. Within a single module, channels are numbered and labeled.

To configure an Aux EPC:

1 Select **Settings** > **Configuration** > **Aux EPCs**.

2 For each channel, select the Gas Type.

# 12 Resource Conservation



This section describes the resource-saving features of the GC.

# Resource Conservation

The GC provides an instrument schedule to conserve resources such as electricity and gases. Using the instrument schedule, you can assign sleep, wake, and conditioning methods that allow you to program resource usage. A Sleep method sets low flows and temperatures. A Wake method sets new flows and temperatures, typically to restore operating conditions. A Condition method sets flows and temperatures for a specific run time, typically high enough to clean out contamination if present.

Load the sleep method at a specified time during the day to reduce flows and temperatures. Load the wake or condition method to restore analytical settings before operating the GC again. For example, load the sleep method at the end of each day or work week, then load the wake or condition method an hour or so before arriving to work the next day.

## Sleep methods

Use a connected data system to create a sleep method to reduce gas and power usage during times of reduced activity.

When creating a sleep method, consider the following:

- The detector. While you can reduce temperatures and gas usage, consider the stabilization time required to prepare the detector for use. The power savings is minimal.
- Connected devices. If connected to an external device such as a mass spectrometer, set compatible flows and temperatures.
- The inlets. Maintain sufficient flow to prevent contamination.

See **Table 16** for general recommendations.

**Table 16 Sleep method recommendations**

| GC Component | Comment |
| --- | --- |
| **Columns** | • Maintain some carrier gas flow to protect the columns. |
| **Oven** | • Reduce temperature to save power.<br>• Turn off to save the most power. |
| **Inlets** | For all inlets:<br>• Reduce temperatures. Reduce temperatures to 40 °C or Off to save the most power. |
| Split/splitless | • Use split mode to prevent diffusion of contamination from the vent line. Use reduced split ratio.<br>• Reduce pressure. Consider using current Gas Saver levels, if used. |
| PPIP/PCI | • Use split mode to prevent diffusion of contamination from the vent line. Use reduced split ratio.<br>• Reduce pressure. Consider using current Gas Saver levels, if used.<br>• Reduce temperature. |
| **Detectors** | |
| FID | • Turn off the flame. (This turns off hydrogen and air flows.)<br>• Reduce temperatures. (Keep at or above 150 °C to reduce contamination)<br>• Turn off makeup flow. |

Table 16 Sleep method recommendations (continued)

| GC Component | Comment |
| --- | --- |
| FPD+ | • Turn off the flame. (This turns off hydrogen and air flows.)<br>• Reduce temperatures.<br>  • Leave emission block at 125-175 °C.<br>  • Reduce transfer line to 150 °C.<br>• Turn off makeup flow. |
| ECD | • Reduce makeup flow. Try using 15–20 mL/min and test results.<br>• Maintain temperature to avoid long recovery/stabilization times. |
| NPD | • Maintain flows and temperatures. Sleep not recommended due to recovery times and also thermal cycling can reduce bead life. |
| TCD | • Leave filament on.<br>• Leave block temperature on.<br>• Reduce reference and makeup flows. |
| **Other devices** | |
| Valve box | • Reduce temperature. (Keep valve box temperature high enough to prevent sample condensation, if applicable.) |
| Aux thermal zones | • Reduce or turn off. Also refer to the manuals for any connected device (for example, a connected MSD). |
| Aux pressures or flows | • Reduce or turn off as appropriate for connected columns, transfer lines, and so forth. Always refer to the manual for any connected device or instrument (for example, a connected MSD), to maintain at least the minimum recommended flows or pressures. |

# Wake and condition methods

The GC can be programmed to wake in one of several ways:

- By loading the last Active Method used before going to sleep
- By loading the **Wake** method
- By running a method called **Condition**, then loading the last Active Method
- By running a method called **Condition**, then loading the **Wake** method

NOTE

**The GC can also store wake, sleep, and condition methods which have been created by a connected data system. Although these methods are not visually displayed on the GC, once downloaded to the GC from the data system, they can be used by the GC scheduler functionality.**

These choices provide flexibility in how you prepare the GC after a sleep cycle.

**A Wake method** sets a temperatures and flows. The oven temperature program is isothermal since the GC does not start a run. When the GC loads a **Wake** method, it remains at those settings until you load another method using the touchscreen, data system, or by starting a sequence.

A **Wake** method can include any settings, however it typically will do the following:

- Restore inlet, detector, column, and transfer line flows.
- Restore temperatures.

- Ignite the FID or FPD+ flame.
- Restore inlet modes.

**A Condition method** sets flows and temperatures for the duration of the method's oven program. When the program ends, the GC loads either the **Wake** method or the last Active Method before sleep, as specified in the instrument schedule (or when manually exiting the sleep state).

One possible use for a condition method is to set higher than normal temperatures and flows to bake out any possible contamination that may have collected in the GC during sleep.

# To Set the GC to Conserve Resources

To set the GC to conserve resources by creating and using an Instrument Schedule:

1. Select the **Settings > Scheduler**.
2. If needed, select the **Sleep/Wake** tab.
3. Create the **Instrument Schedule**. You do not have to program events for every day. For example, you can program the GC to sleep on Friday evening, then wake on Monday morning, keeping it continuously at operating conditions during weekdays.
4. Choose **Set Wake Method** for each desired day, as applicable. This will cause the Wake method to be run when the GC wakes on the selected days.
5. Enter a **Wake Time** for each desired day, based on a 24-hour clock (for example, 9:00 AM is 09:00, 9:00 PM is 21:00).
6. Choose **Set Sleep Method** for each desired day, as applicable. This will cause the Sleep method to be run prior to the GC going to sleep on the selected days.
7. Enter a **Sleep Time** for each desired day, based on a 24-hour clock.
8. If desired, edit the sleep, wake or conditioning methods. Select **Edit Wake Method**, **Edit Conditioning Method**, or **Edit Sleep Method** as needed.
9. Select **Scheduler Options**.
10. Decide how to restore flows. Choose the desired options:
    - **Wake to last active method before sleep**: At the specified time, the GC will restore the last active method used before it went to sleep.
    - **Perform a conditioning run before waking**: At the specified time, the GC will load the conditioning method. This method runs once.
11. Select **Apply**. The settings are saved to the GC.

# 13 Programming

# Clock Time Programming

Clock time programming allows certain setpoints to change automatically at a specified time during a 24-hour day. Thus, an event programmed to occur at 14:35 hours will occur at 2:35 in the afternoon. A running analysis or sequence has precedence over any clock table events occurring during this time. When this happens, such events are not executed.

Possible clock time events include:

- Valve control
- Method and sequence loading
- Starting sequences
- Initiating blank and prep runs
- Column compensation changes
- Adjustments of the detector offset
- Initiating blank and prep runs

## Using clock time events

The Clock Table function allows you to program events to occur during a day based on the 24-hour clock. Clock table events that would occur during a run or sequence are ignored.

For example, the clock table could be used to make a blank run before you even get to work in the morning.

## Adding events to the clock table

1 Select **Settings > Scheduler > Clock Table**.
2 Select **Add**.
3 Choose your **Clock Type** and **Frequency** from their respective dropdown menus.
4 Set the Time you want this event to occur.
5 Select **Add** to add this entry to the clock table.
6 Select **Apply**.
7 Repeat this process until all entries are added.

## Deleting clock time events

1 Select **Settings > Scheduler > Clock Table**.
2 Touch or click **Select**.
3 Touch or click the event to delete, then select **Delete**.
4 Select **Done** to accept the change.
5 Select **Apply**.

# 14 Flow and Pressure Modules

# About Flow and Pressure Control

The GC uses four types of electronic flow or pressure controllers; inlet modules, detector modules, pressure control modules (PCMs), and auxiliary pressure controllers (Aux EPCs).

All of these modules mount in the slots at the top rear of the GC. The slots are identified by numbers, as shown here.

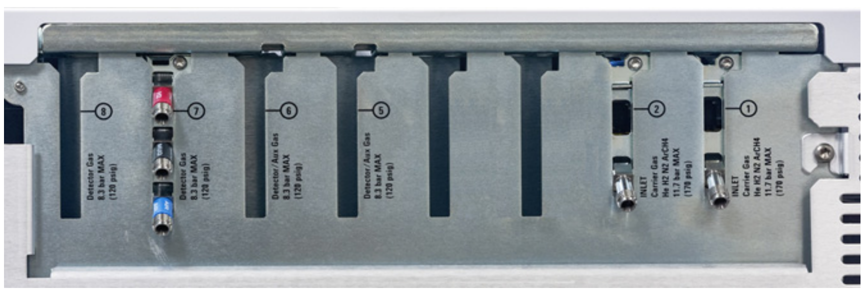



Figure 15. EPC module slots

### Table 17 EPC Modules

| Number | Usage |
|---|---|
| 1 | Inlet module |
| 2 | Inlet module |
| 3 | blank |
| 4 | blank |
| 5 | Aux EPC |
| 6 | Detector module/AuxEPC |
| 7 | Detector module |
| 8 | Detector module |

# Maximum Operating Pressure

We recommend a maximum continuous operating pressure of 170 psi to avoid excessive wear and leaks.

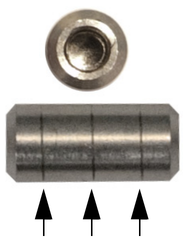
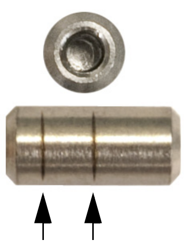
# Auxiliary Pressure Controllers

The Auxiliary Pressure Controller (Aux epc) is also a general purpose device. It has three independent forward-pressure regulated channels. Channels are designated by numbers 1 through 3.

# Restrictors

The Aux EPC and aux PCM channel use frit-type restrictors to allow accurate flow control.To work properly, there must be adequate flow resistance downstream of the pressure sensor. Each channel provides a frit-type restrictor. Four frits are available.

**Table 18 Auxiliary channel frits**

| Frit marking | Flow resistance | Flow characteristic | Often used with |
|---|---|---|---|
| Three rings<br>Blue  | High | 3.33 ± 0.3 SCCM @ 15 PSIG | NPD Hydrogen |
| Two rings<br>Red  | Medium | 30 ± 1.5 SCCM $H_2$ @ 15 PSIG | FID Hydrogen |
| One ring<br>Brown  | Low | 400 ± 30 SCCM AIR @ 40 PSIG | FID Air, FPD+ Air, QuickSwap, Splitter, Deans Switch |
| None (brass tube)  | Zero | No restriction | Headspace vial pressurization |

The one ring frit (low resistance, high flow) is installed in all channels in the AUX EPC when the instrument (or accessory) is shipped. No frit ships in the PCM Aux channel.

When installing or replacing a frit, always use a new O-ring (5180-4181, 12/pk).

# Selecting a frit

The frits change the control range of the channels. The objective is to find a frit that allows the required range of flows at reasonable source pressures.

- For an auxiliary channel ordered as an option (part of the GC order), use the frit supplied by the factory.
- For an auxiliary channel ordered as an accessory (separate from the GC order), see the instruction information supplied with the accessory.
- For a non-Agilent instrument, you must experiment to find the appropriate frit.

When you change a frit, you change the physical characteristics of the channel. It may be desirable (or necessary) to change the PID constants for that channel. See **"PIDs"**.

# Example: Using the PCM Channels

The two channels in a PCM are different. Channel 1 is used to *supply* a pressure. Channel 2 may be used in the same way, but can also be used to *maintain* a pressure by reversing the input and output connections.

### Channel 1: Forward-pressure

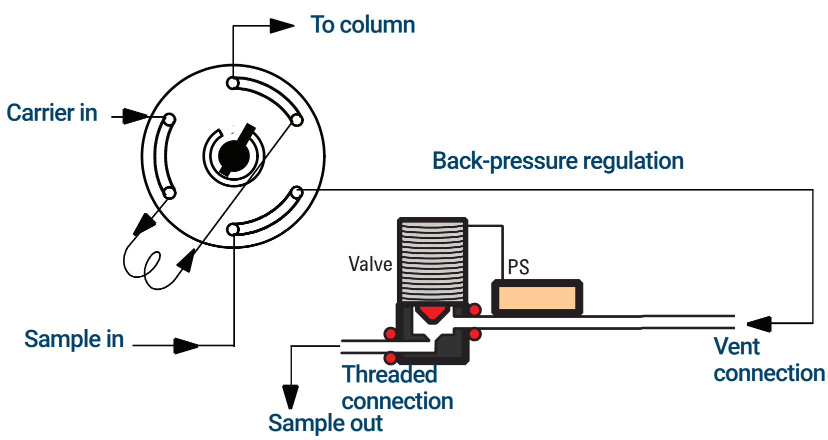
This is identical to the carrier gas channel for the packed column inlet.

### Channel 2: Two-way channel

If gas is supplied at the threaded connection and delivered by the tubing, this operates as a forward pressure regulator. But the connections can be reversed—requiring some fittings—so that it will maintain the gas supplied to it at a fixed pressure. In this mode it behaves as a controlled leak.

This can be used to maintain the gas in a gas sampling valve at a fixed pressure, even if the supply varies somewhat. The result is improved reproducibility in sample amount.

The figure shows the connections with the valve in the Load position.



# PIDs

The behavior of a pressure control module is governed by a set of three constants, called **P** (proportional), **I** (integral), and **D** (differential).

Certain gases or special applications (such as headspace vial pressurization, flow splitter, and backflush applications) require different PIDs than those provided at the factory.

If you need to update or change the pneumatic PID values for an application, use the utility program on the *GC and GC/MS User Manuals & Tools* DVD provided with the GC.

The table summarizes custom PID values required for selected applications. Note that if you are updating an AUX EPC module, you will need to change the frit for the channel used. See also "**Restrictors**."

### Table 19 PIDs and frits

| Application | Module | AUX frit | Select Available PID Values |
|---|---|---|---|
| Headspace vial pressurization | AUX EPC | No color or rings | AUX_EPC_Headspace |
| Headspace sampling loop | PCM in backpressure control | | PCM_Headspace |

# 15 Inlets

# Inlet Overview

### Table 20 Comparing inlets

| Inlet | Column | Mode | Sample concentration | Comments | Sample to column |
|---|---|---|---|---|---|
| Split/splitless | Capillary | Split<br>Pulsed split*<br><br>Splitless<br>Pulsed splitless* | High<br>High<br><br>Low<br>Low | | Very little<br>Very little<br><br>All<br>All |
| Purge packed column | Packed<br>Large capillary | n/a<br>n/a | Any<br>Any | <br>OK if resolution not critical | All<br>All |
| Packed column | Packed | n/a | Any | | All |
| Cool on-column | Capillary | n/a | Low or labile | Minimal discrimination and decomposition | All |

* Pulsed split and pulsed splitless modes are not available for split/splitless inlets on EPR (electronic pneumatics regulation) equipped GCs.

# About the Split/Splitless Inlet

This inlet is used for split, splitless, pulsed splitless, or pulsed split analyses. You can choose the operating mode from the inlet parameter list. The split mode is generally used for major component analyses, while the splitless mode is used for trace analyses. The pulsed splitless and pulsed split modes are used for the same type of analyses as split or splitless, but allow you to inject larger samples. Sleep mode is used for creating sleep methods, or if all gas needs to flow onto the column.

NOTE

**Pulsed split and pulsed splitless modes are not supported on EPR (electronic pneumatics regulation) equipped GCs.**

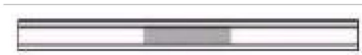
This inlet is available in both EPC (electronic pneumatics control) and EPR (electronic pneumatics regulation) equipped GCs.

## Selecting the correct S/SL inlet liner

### Split liner

A good liner for split mode operation will offer very little restriction to the split flow path between the bottom of the liner and the inlet gold seal and between the outside of the liner and the inside of the injection port body. The preferred Agilent split liner, part number 5183-4647, incorporates a glass positioning bead on the bottom to facilitate this. It also has glass wool inside the liner that provides for complete sample vaporization across the boiling point range of the sample. Select an appropriate liner from **Table 21**.

**Table 21 Split mode liners**

| Liner | Description | Volume | Mode | Deactivated | Part Number |
|---|---|---|---|---|---|
|  | Low Pressure Drop – Positioning Bead | 870 µL | Split – Fast Injection | Yes | 5183-4647 |
|  | 4mm ID, Glass Wool | 990 µL | Split – Fast Injection | No | 19251-60540 |
| | Low Pressure Drop, Glass Wool | 870 µL | Split | Yes | 5190-2295 |

### Splitless liner

NOTE

**Pulsed split and pulsed splitless modes are not supported on EPR (electronic pneumatics regulation) equipped GCs.**

The liner volume must contain the solvent vapor. The liner should be deactivated to minimize sample breakdown during the splitless time. Solvent vapor volume can be reduced by using Pulsed Splitless mode. Use the Vapor Volume Calculator to determine vapor volume requirements.

**Vapor volume < 300 µL** Use 2 mm liner (250 µL volume), 5181-8818 or similar.

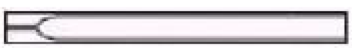
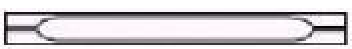
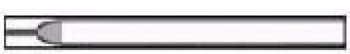
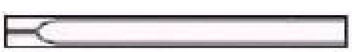
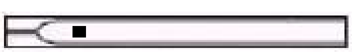
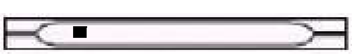
**Vapor volume 225 – 300 µL** Consider pulsed splitless mode to reduce vapor volume.

**Vapor volume > 300 µL** Use 4 mm liner, 5062-3587 or similar.

**Vapor volume > 800 µL** Consider pulsed splitless mode to reduce vapor volume.

For thermally labile or reactive samples, use G1544-80700 (open top) or G1544-80730 (top taper) liners.

Table 22 Splitless mode liners

| Liner | Description | Volume | Mode | Deactivated | Part Number |
|---|---|---|---|---|---|
|  | Single Taper Glass Wool | 900 uL | Splitless | Yes | 5062-3587 |
|  | Single Taper | 900 uL | Splitless | Yes | 5181-3316 |
|  | Dual Taper | 800 uL | Splitless | Yes | 5181-3315 |
|  | 2 mm Quartz | 250 uL | Splitless | No | 18740-80220 |
|  | 2 mm Quartz | 250 uL | Splitless | Yes | 5181-8818 |
|  | 1.5 mm | 140 uL | Direct Inject, Purge and Trap, Headspace | No | 18740-80200 |
|  | Single Taper Glass Wool | 900 uL | Splitless | Yes | 5062-3587 |
|  | Single Taper | 900 uL | Splitless | Yes | 5181-3316 |
|  | 4 mm Single Taper | Direct column connect | | Yes | G1544-80730 |
|  | 4 mm Dual Taper | Direct column connect | | Yes | G1544-80700 |

# About the Purged Packed Column Inlet

This inlet is used with packed columns when high-efficiency separations are not required. It can also be used with wide-bore capillary columns, if flows greater than 10 mL/min are acceptable.

The inlet is flow controlled when using an undefined column (packed columns), an flow or pressure controlled when using capillary columns.

# About the Packed Column Inlet

This inlet is used with packed columns when high-efficiency separations are not required.

The inlet is flow controlled regardless of whether the column is defined or not.

This inlet is available in EPR (electronic pneumatics regulation) equipped GCs only.

# About the Cool On-Column Inlet

This inlet introduces liquid sample directly onto a capillary column. To do this, both the inlet and the oven must be cool at injection, either at or below the boiling point of the solvent.

Because the sample does not vaporize immediately in the inlet, problems with sample discrimination and sample alteration are minimized. If done properly, cool-on column injection also provides accurate and precise results.

You can operate the inlet in track oven mode, where the inlet temperature follows the column oven, or you can program up to three temperature ramps.

## Setup modes of the COC inlet

The COC inlet hardware must be set up for one of three usages, depending on the type of injection and column size.

- 0.25 mm or 0.32 mm automated on-column.
- 0.53 mm automatic on-column or retention gap
- 0.2 mm manual

## Retention gaps

Because the sample is injected directly onto the column, it is strongly suggested that a retention gap—or guard column—be used to protect your column. A retention gap is a deactivated column that is connected between the inlet and the analytical column. If you choose to use one, it is suggested that at least 1 m of retention gap be installed per 1 mL of sample injected. Information on ordering retention gaps can be found in the Agilent catalog for consumables and supplies.

# 16 Columns and Oven

# Hydrogen Sensor

The optional Hydrogen Sensor module checks for uncombusted free hydrogen in the GC column oven. During normal operation with hydrogen as a carrier gas, leaks from the inlets or detectors could possibly put hydrogen gas directly into the oven. Hydrogen–air mixtures are potentially explosive in concentrations of 4–74.2% hydrogen by volume. The sensor monitors the free hydrogen level in the oven and will trigger a shutdown of all hydrogen gas flows if the hydrogen level in the oven is > 1%.

In the event of a hydrogen safety shutdown, the GC records the event in its Event log.

See the *Troubleshooting* manual for more information about GC shutdown events and how to clear them.

The GC can only shut down hydrogen gas flows that have been configured properly. Always configure the gas types used for the inlets, detectors, and so forth.

## Instrument logs

The GC will log the following hydrogen sensor events in its event logs:

- Hydrogen safety shutdowns initiated by the hydrogen sensor
- Calibrations
- Hydrogen sensor tests

## Calibration

The hydrogen sensor requires periodic calibration for optimal performance. If the sensor is not calibrated on schedule or if a calibration fails for any reason (for example, a lack of calibration gas), the sensor continues to use its existing calibration data.

## Status information

If a calibration fails, a notification will appear on the diagnostics tab.

## Operation with an Agilent data system

Using the hydrogen sensor with an Agilent data system provides additional features. Use the data system to:

- Print calibration reports. The report includes a plot of all calibration data stored in the GC,
- Access the automated calibration schedule control (on/off).
- Store lot number and expiration date information for the calibration gas cylinder.
- View the hydrogen sensor status information in the GC status user interface. The status shows the current percent hydrogen level and any messages related to the hydrogen sensor.
- Plot the measured hydrogen level as a diagnostic signal, if desired.
- View and print all logged entries for calibrations, cylinder information, and shutdowns.

# 17 Chromatographic Checkout



This section describes the general procedure for verifying performance against the original factory standards. The checkout procedures described here assume a GC that has been in use for some period of time. Therefore the procedures ask that you perform bakeouts, replace consumable hardware, install the checkout column, and so forth.

# About Chromatographic Checkout

The tests described in this section provide basic confirmation that the GC and detector can perform comparably to factory condition. However, as detectors and the other parts of the GC age, detector performance can change. The results presented here represent typical outputs for typical operating conditions and are not specifications.

The tests assume the following:

- Use of an automatic liquid sampler. If not available, use a suitable manual syringe instead of the syringe listed.
- Use of a 10 μL syringe in most cases. However, a 5 μL syringe is an acceptable substitute.
- Use of the septa and other hardware (liners, jets, adapters, and so forth) described. If you substitute other hardware, performance can vary.

# To Prepare for Chromatographic Checkout

Because of the differences in chromatographic performance associated with different consumables, Agilent strongly recommends using the parts listed here for all checkout tests. Agilent also recommends installing new consumable parts whenever the quality of the installed ones is not known. For example, installing a new liner and septum ensures that they will not contribute any contamination to the results.

When the GC is delivered from the factory, these consumable parts are new and do not need replacement.

NOTE

**For a new GC, check the installed inlet liner. The liner shipped in the inlet may not be the liner recommended for checkout.**

1. Check the indicators/dates on any gas supply traps. Replace/recondition expended traps.
2. Install new consumable parts for the inlet and prepare the correct injector syringe (and needle, as needed).

**Table 23 Recommended parts for checkout by inlet type**

| Recommended part for checkout | Part number |
|---|---|
| **Split splitless inlet** | |
| Syringe, 10-µL | 5181-1267 |
| O-ring | 5188-5365 |
| Septum | 5183-4757 |
| Liner | 5190-2295 |
| **Purged packed column inlet** | |
| Syringe, 10-µL | 5181-1267 |
| O-ring | 5080-8898 |
| Septum | 5183-4757 |
| **Packed column inlet** | |
| Syringe, 10-µL | 5181-1267 |
| O-ring | 5080-8898 |
| Septum | 5183-4757 |

### Table 23 Recommended parts for checkout by inlet type

| Recommended part for checkout | Part number |
|---|---|
| **Cool on-column inlet** | |
| Septum | 5183-4758 |
| Septum nut | 19245-80521 |
| Syringe, 5-µL on-column | 5182-0836 |
| 0.32-mm needle for 5-µL syringe | 5182-0831 |
| 7693A ALS: Needle support insert, COC | G4513-40529 |
| Insert, fused silica, 0.32-mm id | 19245-20525 |

### Table 24 Checkout standards

| Standard | Part number | Number of ampoules |
|---|---|---|
| FID checkout | 5188-5372 | 3 |
| TCD checkout | 18710-60170 | 3 |
| ECD checkout | 18713-60040 | 3 |
| NPD checkout | 18789-60060 | 3 |
| FPD+ checkout (methyl parathion) | 5188-5953 | 3 |
| OQ/PV Headspace checkout | 5182-9733 | 1 |

# To Check FID Performance

FID performance is checked differently depending on the inlet being used. For GCs equipped with a packed column inlet (PCI), use **"To check FID performance with a packed column inlet (PCI)"**. For all other inlet types, use **"To check FID performance with a purged packed, split splitless or cool-on column inlet"**.

## To check FID performance with a packed column inlet (PCI)

**1** Gather the following:

- Evaluation column, 10% OV-101, 5 ft, OD 1/8, ID 2 mm (G3591-81093)
- FID MDL sample (5188-5372)
- Chromatographic-grade isooctane
- 4-mL solvent and waste bottles or equivalent for autoinjector
- 2-mL sample vials or equivalent for sample
- Inlet and injector hardware (See "**To Prepare for Chromatographic Checkout**.")

**2** Verify the following:

- Packed column jet installed. If not, select and install a packed column jet.
- Packed column adapter installed. If not, install it.
- Chromatographic-grade gases plumbed and configured: helium as carrier gas, nitrogen, hydrogen, and air.
- Empty waste vials loaded in sample turret.
- 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.

**3** Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**"To Prepare for Chromatographic Checkout"**."

**4** Install the evaluation column. (See the procedure for the PCI in the Maintenance manual.)

- Bake out the evaluation column for at least 30 min at 180 °C. (See the procedure for the PCI in the Maintenance manual.)
- Be sure to configure the column.

**5** Check the FID baseline output. The output should be between 5 pA and 20 pA and relatively stable. (If using a gas generator or ultra pure gas, the signal may stabilize below 5 pA.) If the output is outside this range or unstable, resolve this problem before continuing.

**6** If the output is too low:

- Check that the electrometer is on.
- Check that the flame is lit.
- Check that the signal is set to the correct detector.

**7** Create or load a method with the parameter values listed in **Table 25**.

### Table 25 FID Checkout Conditions - Packed Column Inlet

| | |
|---|---|
| **Column and sample** | |
| Type | 10% OV-101, 5 ft, OD 1/8, ID 2 mm (G3591-81093) |
| Sample | FID MDL sample (5188-5732) |
| Column flow | 20 mL/min |
| Column mode | Flow mode |
| **Packed column inlet** | |
| Temperature | 250 °C |
| **Detector** | |
| Temperature | 300 °C |
| H2 flow | 30 mL/min |
| Air flow | 400 mL/min |
| Makeup flow ($N_2$) | OFF |
| Mode | Constant makeup flow OFF |
| Flame | On |
| Lit offset | Typically 2 pA |
| **Oven** | |
| Constant temperature | 180 °C |
| Time | 15 min |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 µL |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 µL |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |

**Table 25 FID Checkout Conditions (continued)- Packed Column Inlet**

| **Manual injection** | |
|---|---|
| Injection volume | 1 µL |
| **Data system** | |
| Data rate | 5 Hz |

8 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

If not using a data system, create a one sample sequence using the Browser Interface.

9 Start the run.

If performing an injection using an autosampler, start the run using the data system or press ▶ on the Browser Interface.

If performing a manual injection (with or without a data system):

a Press [Prep Run] to prepare the inlet for splitless injection.

b When the GC becomes ready, inject 1 µL of the checkout sample and press ▶ on the Browser Interface.

c The following chromatogram shows typical results for a new detector with new consumable parts installed and nitrogen makeup gas.

# To check FID performance with a purged packed, split splitless or cool-on column inlet

1 Gather the following:

- Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (19091J-413)
- FID performance evaluation (checkout) sample (5188-5372)
- Chromatographic-grade isooctane
- 4-mL solvent and waste bottles or equivalent for autoinjector
- 2-mL sample vials or equivalent for sample
- Inlet and injector hardware (See "**To Prepare for Chromatographic Checkout**.")

2 Verify the following:

- Capillary column jet installed. If not, select and install a capillary column jet.
- Capillary column adapter installed. If not, install it.
- Chromatographic-grade gases plumbed and configured: helium as carrier gas, nitrogen, hydrogen, and air.
- Empty waste vials loaded in sample turret.
- 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.

3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."

4 Install the evaluation column.

- Bake out the evaluation column for at least 30 min at 180 °C.
- Be sure to configure the column.

5 Check the FID baseline output. The output should be between 5 pA and 20 pA and relatively stable. (If using a gas generator or ultra pure gas, the signal may stabilize below 5 pA.) If the output is outside this range or unstable, resolve this problem before continuing.

6 If the output is too low:
- Check that the electrometer is on.
- Check that the flame is lit.
- Check that the signal is set to the correct detector.

7 Create or load a method with the parameter values listed in **Table 26**.

### Table 26 FID Checkout Conditions

| **Column and sample** | |
|---|---|
| Type | HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413) |
| Sample | FID checkout 5188-5372 |
| Column flow | 6.5 mL/min |
| Column mode | Constant flow for EPC equipped GCs. Constant pressure mode (30 psi) for EPR equipped GCs. |
| **Split/splitless inlet** | |
| Temperature | 250 °C |
| Mode | Splitless |
| Purge flow | 40 mL/min |
| Purge time | 0.5 min |
| Gas saver | Off |
| **Purged packed column inlet** | |
| Temperature | 250 °C |
| **Cool on-column inlet** | |
| Temperature | Track oven |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 300 °C |
| H2 flow | 30 mL/min |
| Air flow | 400 mL/min |
| Makeup flow (N2) | 25 mL/min |
| Lit offset | Typically 2 pA |

### Table 26 FID Checkout Conditions

| | |
|---|---|
| **Oven** | |
| Initial temp | 75 °C |
| Initial time | 0.5 min |
| Rate 1 | 20 °C/min |
| Final temp | 190 °C |
| Final time | 0 min |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 µL |
| **Data system** | |
| Data rate | 5 Hz |

8 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

If not using a data system, create a one sample sequence using the GC Browser Interface.

9 Start the run.

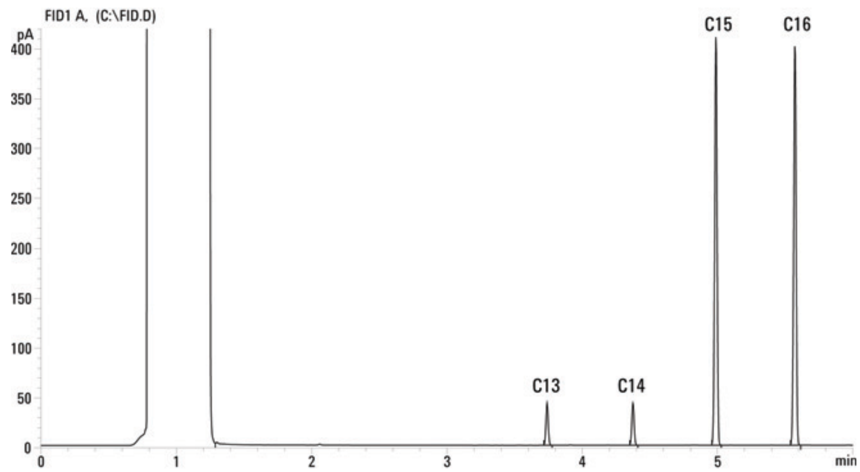
If performing an injection using an autosampler, start the run using the data system, or press ▶.

If performing a manual injection (with or without a data system):

**a** Select [icon] to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and select ▶ on the GC.

**c** The following chromatogram shows typical results for a new detector with new consumable parts installed and nitrogen makeup gas.



# To Check TCD Performance

TCD performance is checked differently depending on the inlet being used. For GCs equipped with a packed column inlet (PCI), use **"To check TCD performance with a packed column inlet (PCI)"**. For all other inlet types, use **"To check TCD performance with a purged packed, split splitless or cool-on column inlet"**.

## To check TCD performance with a packed column inlet (PCI)

1 Gather the following:
   - Evaluation column, 10% OV-101, 5 ft, OD 1/8, ID 2 mm (G3591-81093)
   - FID/TCD performance evaluation (checkout) sample (18710-60170)
   - 4-mL solvent and waste bottles or equivalent for autoinjector
   - Chromatographic-grade hexane
   - 2-mL sample vials or equivalent for sample
   - Chromatographic-grade helium as carrier, makeup, and reference gas
   - Inlet and injector hardware (See "**To Prepare for Chromatographic Checkout**.")
2 Verify the following:
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas and reference gas.
   - Empty waste vials loaded in sample turret.
   - 4-mL solvent vial with diffusion cap filled with hexane and inserted in Solvent A injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."
4 Install the evaluation column. (See the procedure for the PCI in the Maintenance manual.)
   - Bake out the evaluation column for at least 30 min at 180 °C. (See the procedure for the **PCI** in the Maintenance manual.)
   - Configure the column
5 Create or load a method with the parameter values listed in **Table 27**.

**Table 27 TCD Checkout Conditions - Packed Column Inlet**

| **Column and sample** | |
|---|---|
| Type | 10% OV-101, 5 ft, OD 1/8, ID 2 mm (G3591-81093) |
| Sample | FID/TCD checkout 18710-60170 |
| Column flow | 20 mL/min |
| Column mode | Flow mode |

### Table 27 TCD Checkout Conditions (continued)- Packed Column Inlet

| | |
|---|---|
| **Packed column inlet** | |
| Temperature | 250 °C |
| **Detector** | |
| Temperature | 300 °C |
| Reference flow (He) | 20 mL/min |
| Makeup flow (He) | OFF |
| Baseline output | < 30 display counts on Agilent OpenLAB CDS ChemStation Edition (< 750 µV) |
| **Oven** | |
| Constant temp | 180 °C |
| Time | 15 min |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 µL |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 µL |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 2 µL |
| **Data system** | |
| Data rate | 5 Hz |

6 Display the signal output. A stable output at any value between 12.5 and 750 µV (inclusive) is acceptable.

- If the baseline output is < 0.5 display units (< 12.5 µV), verify that the detector filament is on. If the offset is still < 0.5 display units (< 12.5 µV), your detector requires service.

- If baseline output is > 30 display units (> 750 µV), there may be chemical contamination contributing to the signal. Bakeout the TCD. If repeated cleanings do not give an acceptable signal, check gas purity. Use higher purity gases and/or install traps.

7 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

8 Start the run.

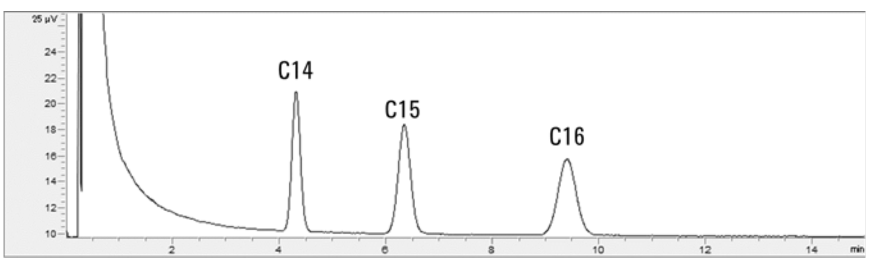
If performing an injection using an autosampler, start the run using the data system or press ▶ on the GC.

If performing a manual injection (with or without a data system):

**a** Click to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and press ▶ on the GC.

**c** The following chromatogram shows typical results for a new detector with new consumable parts installed.

- 


# To check TCD performance with a purged packed, split splitless or cool-on column inlet

1 Gather the following:

- Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (19091J-413)
- FID/TCD performance evaluation (checkout) sample (18710-60170)
- 4-mL solvent and waste bottles or equivalent for autoinjector
- Chromatographic-grade hexane
- 2-mL sample vials or equivalent for sample
- Chromatographic-grade helium as carrier, makeup, and reference gas
- Inlet and injector hardware See "**To Prepare for Chromatographic Checkout**."

2 Verify the following:

- Chromatographic-grade gases plumbed and configured: helium as carrier gas and reference gas.
- Empty waste vials loaded in sample turret.
- 4-mL solvent vial with diffusion cap filled with hexane and inserted in Solvent A injector position.

3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."

4 Install the evaluation column.
   - Bake out the evaluation column for at least 30 min at 180 °C.
   - Configure the column

5 Create or load a method with the parameter values listed in **Table 28**.

### Table 28 TCD Checkout Conditions

| | |
|---|---|
| **Column and sample** | |
| Type | HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413) |
| Sample | FID/TCD checkout 18710-60170 |
| Column flow | 6.5 mL/min |
| Column mode | Constant flow for EPC equipped GCs. Constant pressure mode (30 psi) for EPR equipped GCs. |
| **Split/splitless inlet** | |
| Temperature | 250 °C |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **Purged packed column inlet** | |
| Temperature | 250 °C |
| **Cool on-column inlet** | |
| Temperature | Oven track |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 300 °C |
| Reference flow (He) | 30 mL/min |
| Makeup flow (He) | 2 mL/min |
| Baseline output | < 30 display counts on Agilent OpenLAB CDS ChemStation Edition (< 750 µV) |
| **Oven** | |
| Initial temp | 75 °C |

**Table 28 TCD Checkout Conditions (continued)**

| | |
|---|---|
| **Initial time** | 0.5 min |
| Rate 1 | 20 °C/min |
| Final temp | 190 °C |
| Final time | 6.25 min |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 µL |
| **Data system** | |
| Data rate | 5 Hz |

6 Display the signal output. A stable output at any value between 12.5 and 750 µV (inclusive) is acceptable.
   - If the baseline output is < 0.5 display units (< 12.5 µV), verify that the detector filament is on. If the offset is still < 0.5 display units (< 12.5 µV), your detector requires service.
   - If baseline output is > 30 display units (> 750 µV), there may be chemical contamination contributing to the signal. Bakeout the TCD. If repeated cleanings do not give an acceptable signal, check gas purity. Use higher purity gases and/or install traps.

7 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

8 Start the run.

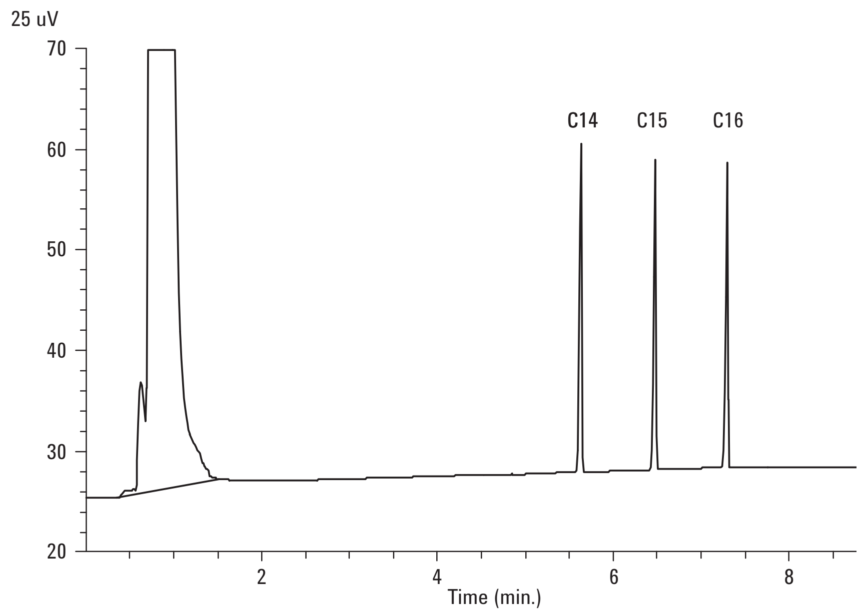
If performing an injection using an autosampler, start the run using the data system or press ▶.

If performing a manual injection (with or without a data system):

**a** Select [prep run icon] to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and select ▶ on the touchscreen.

**c** The following chromatogram shows typical results for a new detector with new consumable parts installed.



# To Check NPD Performance

1 Gather the following:
   - Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (19091J-413)
   - NPD performance evaluation (checkout) sample (18789-60060)
   - 4-mL solvent and waste bottles or equivalent for autoinjector.
   - Chromatographic-grade isooctane
   - 2-mL sample vials or equivalent for sample.
   - Inlet and injector hardware See "**To Prepare for Chromatographic Checkout**."
2 Verify the following:
   - Capillary column jet installed. If not, select and install a capillary column jet.
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas, nitrogen, hydrogen, and air.
   - Empty waste vials loaded in sample turret.
   - 4-mL vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."
4 If present, remove any protective caps from the inlet manifold vents.
5 Install the evaluation column.
   - Bake out the evaluation column for at least 30 min at 180 °C.
   - Be sure to configure the column
6 Create or load a method with the parameter values listed in **Table 29**.

**Table 29 NPD Checkout Conditions**

| Column and sample | |
|---|---|
| Type | HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413) |
| Sample | NPD checkout 18789-60060 |
| Column mode | Constant flow |
| Column flow | 6.5 mL/min (helium) |

### Table 29 NPD Checkout Conditions (continued)

| | |
|---|---|
| **Split/splitless inlet** | |
| Temperature | 200 °C |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **Purged packed column inlet** | |
| Temperature | 200 °C |
| **Cool on-column inlet** | |
| Temperature | Track oven |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 300 °C |
| H2 flow | 3 mL/min |
| Air flow | 60 mL/min |
| Makeup flow (N2) | Makeup + column = 3 mL/min |
| Output | 20 display units (20 pA) |
| **Oven** | |
| Initial temp | 60 °C |
| Initial time | 0 min |
| Rate 1 | 20 °C/min |
| Final temp | 200 °C |
| Final time | 3 min |

**Table 29 NPD Checkout Conditions (continued)**

| **ALS settings (if installed)** | |
|---|---|
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 µL |
| **Data system** | |
| Data rate | 5 Hz |

**7** If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

**8** Start the run.

If performing an injection using an autosampler, start the run using the data system, or creating a one sample sequence and pressing ▶ on the Browser Interface.

If performing a manual injection (with or without a data system):

**a** Select [icon] to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and press ▶.

**c** The following chromatogram shows typical results for a new detector with new consumable parts installed.

# To Check ECD Performance

1 Gather the following:
   - Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (19091J-413)
   - ECD performance evaluation (checkout) sample (18713–60040, Japan: 5183-0379)
   - 4-mL solvent and waste bottles or equivalent for autoinjector.
   - Chromatographic-grade isooctane
   - 2-mL sample vials or equivalent for sample.
   - Inlet and injector hardware See "**To Prepare for Chromatographic Checkout**."
2 Verify the following:
   - Clean fused silica indented mixing liner installed. If not, install it.
   - Chromatographic-grade gases plumbed and configured: helium for carrier gas, nitrogen for makeup.
   - Empty waste vials loaded in sample turret.
   - 4-mL vial with diffusion cap filled with hexane and inserted in Solvent A injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."
4 Install the evaluation column.
   - Bake out the evaluation column for at least 30 minutes at 180 °C.
   - Be sure to configure the column.
5 Display the signal output to determine baseline output. A stable baseline output at any value between 0.5 and 1000 Hz (OpenLAB CDS ChemStation Edition display units) (inclusive) is acceptable.
   - If the baseline output is < 0.5 Hz, verify that the electrometer is on. If the offset is still < 0.5 Hz, your detector requires service.
   - If the baseline output is > 1000 Hz, there may be chemical contamination contributing to the signal. Bakeout the ECD. If repeated cleanings do not give an acceptable signal, check gas purity. Use higher purity gases and/or install traps.
6 Create or load a method with the parameter values listed in **Table 30**.

**Table 30 ECD Checkout Conditions**

| **Column and sample** | |
|---|---|
| Type | HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413) |
| Sample | ECD checkout (18713-60040 or Japan: 5183-0379) |
| Column mode | Constant flow |
| Column flow | 6.5 mL/min (helium) |

### Table 30 ECD Checkout Conditions (continued)

| | |
|---|---|
| **Split/splitless inlet** | |
| Temperature | 250 °C |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **Purged packed column inlet** | |
| Temperature | 250 °C |
| **Cool on-column inlet** | |
| Temperature | Track oven |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 300 °C |
| Makeup flow (N2) | 25 mL/min (constant + makeup) |
| Baseline output | Should be < 1000 display counts. In Agilent OpenLAB CDS ChemStation Edition (< 1000 Hz) |
| **Oven** | |
| Initial temp | 80 °C |
| Initial time | 0 min |
| Rate 1 | 15 °C/min |
| Final temp | 180 °C |
| Final time | 10 min |

### Table 30 ECD Checkout Conditions (continued)

| **ALS settings (if installed)** | |
|---|---|
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 μL |
| Syringe size | 10 μL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 μL |
| **Data system** | |
| Data rate | 5 Hz |

7 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

8 Start the run.

   If performing an injection using an autosampler, start the run using the data system or press .

   If performing a manual injection (with or without a data system):

   a Select to prepare the inlet for splitless injection.

   b When the GC becomes ready, inject 1 μL of the checkout sample and press .

9 The following chromatogram shows typical results for a new detector with new consumable parts installed. The Aldrin peak will be missing when using the Japanese sample 5183-0379.

# To Check FPD+ Performance (Sample 5188-5953)

To check FPD$^+$ performance, first check the phosphorus performance, then the sulfur performance.

## Preparation

1 Gather the following:
   - Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (19091J-413)
   - FPD+ performance evaluation (checkout) sample (5188-5953), 2.5 mg/L (± 0.5%) methylparathion in isooctane
   - Phosphorus filter
   - Sulfur filter and filter spacer
   - 4-mL solvent and waste bottles or equivalent for autoinjector.
   - 2-mL sample vials or equivalent for sample.
   - Chromatographic-grade isooctane for syringe wash solvent.
   - Inlet and injector hardware See "**To Prepare for Chromatographic Checkout**."
2 Verify the following:
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas, nitrogen, hydrogen, and air.
   - Empty waste vials loaded in sample turret.
   - 4-mL vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."
4 Verify that the **Lit Offset** is set appropriately. Typically, it should be about 2.0 pA for the checkout method.
5 Install the evaluation column.
6 Set the oven, inlet, and detector to 250 °C and bake out for at least 15 minutes.
7 Be sure to configure the column.

## Phosphorus performance

1 If it is not already installed, install the phosphorus filter.
2 Create or load a method with the parameter values listed in **Table 31**.

### Table 31 FPD$^+$ Checkout Conditions (P)

| | |
|---|---|
| **Column and sample** | |
| Type | HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413) |
| Sample | FPD checkout (**5188-5953**) |
| Column mode | Constant flow |
| Column flow | 6.5 mL/min |
| **Split/splitless inlet** | |
| Temperature | 180 °C Split/splitless |
| Mode | Splitless |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **Purged packed column inlet** | |
| Temperature | 180 °C |
| **Cool on-column inlet** | |
| Temperature | Track oven |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 200 °C (On) |
| Hydrogen flow | 60 mL/min (On) |
| Air (Oxidizer) flow | 60 mL/min (On) |
| Mode | Constant makeup flow OFF |
| Makeup flow | 60 mL/min (On) |
| Makeup gas type | Nitrogen |
| Flame | On |
| Lit offset | Typically 2 pA |
| PMT voltage | On |
| Emission Block | 125 °C |

### Table 31 FPD+ Checkout Conditions (continued)(P)

| | |
|---|---|
| **Oven** | |
| Initial temp | 70 °C |
| Initial time | 0 min |
| Rate 1 | 25 °C/min |
| Final temp 1 | 150 °C |
| Final time 1 | 0 min |
| Rate 2 | 5 °C/min |
| Final temp 2 | 190 °C |
| Final time 2 | 7 min |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 µL |
| **Data system** | |
| Data rate | 5 Hz |

3 Ignite the FPD+ flame, if not lit.

4 Display the signal output and monitor. This output typically runs around 10. Wait for the output to stabilize. This takes approximately 1 hour.

If the baseline output is too high:

- Check column installation. If installed too high, the stationary phase burns in the flame and increases measured output.
- Check for leaks.
- Bake out the detector and column at 250 °C.
- Wrong flows set for installed filter.

If the baseline output is zero, verify the electrometer is on and the flame is lit.

5 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

6 Start the run.

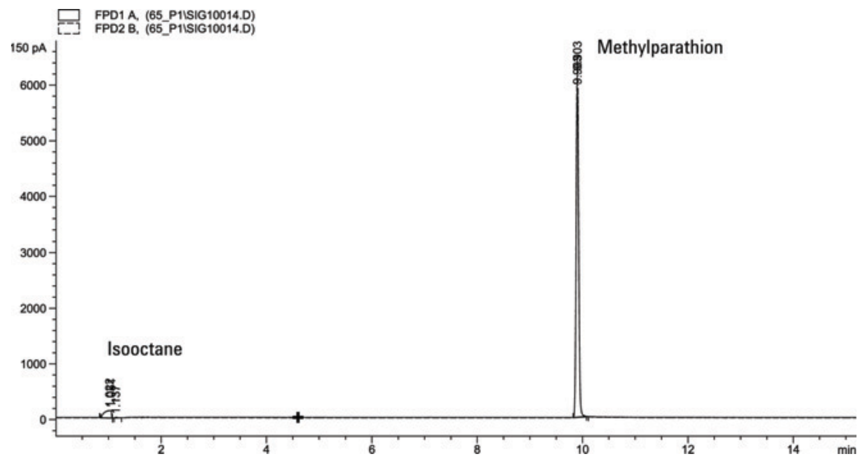
If performing an injection using an autosampler, start the run using the data system or press ▶.

If performing a manual injection (with or without a data system):

a Select the prep run key to prepare the inlet for splitless injection.

b When the GC becomes ready, inject 1 µL of the checkout sample and press ▶.

c The following chromatogram shows typical results for a new detector with new consumable parts installed.




# Sulfur performance

1 Install the sulfur filter and filter spacer.

2 Ignite the FPD+ flame if not lit.

3 Display the signal output and monitor. This output typically runs between 50 and 60 but can be as high as 70. Wait for the output to stabilize. This takes approximately 1 hour.

If the baseline output is too high:

- Check column installation. If installed too high, the stationery phase burns in the flame and increases measured output.
- Check for leaks.
- Bake out the detector and column at 250 °C.
- Wrong flows set for installed filter.

If the baseline output is zero, verify the electrometer is on and the flame is lit.

4 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

5 Start the run.

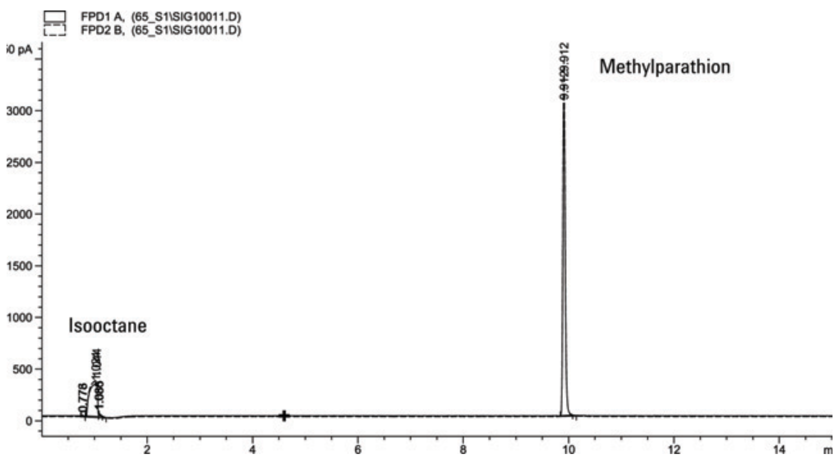
If performing an injection using an autosampler, start the run using the data system or press ▶.

If performing a manual injection (with or without a data system):

a Select the prep run button to prepare the inlet for splitless injection.

b When the GC becomes ready, inject 1 µL of the checkout sample and press ▶.

6 The following chromatogram shows typical results for a new detector with new consumable parts installed.



# To Check FPD+ Performance (Sample 5188-5245, Japan)

To verify FPD$^+$ performance, first check the phosphorus performance, then the sulfur performance.

## Preparation

1 Gather the following:
   - Evaluation column, DB5 15 m × 0.32 mm × 1.0 µm (123-5513)
   - FPD performance evaluation (checkout) sample (5188-5245, Japan), composition: n-Dodecane 7499 mg/L (± 5%), Dodecanethiol 2.0 mg/L (± 5%), Tributyl Phosphate 2.0 mg/L (± 5%), tert-Butyldisulfide 1.0 mg/L (± 5%), in isooctane as solvent
   - Phosphorus filter
   - Sulfur filter and filter spacer
   - 4-mL solvent and waste bottles or equivalent for autoinjector.
   - 2-mL sample vials or equivalent for sample.
   - Chromatographic-grade isooctane for syringe wash solvent.
   - Inlet and injector hardware See "**To Prepare for Chromatographic Checkout**."
2 Verify the following:
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas, nitrogen, hydrogen, and air.
   - Empty waste vials loaded in sample turret.
   - 4-mL vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "**To Prepare for Chromatographic Checkout**."
4 Verify the lit offset is set appropriately. Typically, it should be about 2.0 pA for the checkout method.
5 Install the evaluation column.
   - Set the oven, inlet, and detector to 250 °C and bake out for at least 15 minutes.
6 Configure the column.

## Phosphorus performance

1 If it is not already installed, install the phosphorus filter.
2 Create or load a method with the parameter values listed in **Table 32**.

### Table 32 FPD+ Phosphorus Checkout Conditions

| | |
|---|---|
| **Column and sample** | |
| Type | DB-5MS, 15 m × 0.32 mm × 1.0 µm (123-5513) |
| Sample | FPD checkout (**5188-5245**) |
| Column mode | Constant flow |
| Column flow | 7.5 mL/min |
| **Split/splitless inlet** | |
| Temperature | 250 °C |
| Mode | Splitless |
| Total purge flow | 69.5 mL/min |
| Purge flow | 60 mL/min |
| Purge time | 0.75 min |
| **Purged packed column inlet** | |
| Temperature | 250 °C |
| **Cool on-column inlet** | |
| Temperature | Oven track |
| Septum purge | 15 mL/min |
| **Detector** | |
| Temperature | 200 °C (On) |
| Hydrogen flow | 60.0 mL/min (On) |
| Air (oxidizer) flow | 60.0 mL/min (On) |
| Mode | Constant makeup flow Off |
| Makeup flow | 60.0 mL/min (On) |
| Makeup gas type | Nitrogen |
| Flame | On |
| Lit offset | Typically 2 pA |
| PMT voltage | On |
| Emission Block | 125 °C |

Table 32 FPD+ Phosphorus Checkout Conditions (continued)

| | |
|---|---|
| **Oven** | |
| Initial temp | 70 °C |
| Initial time | 0 min |
| Rate 1 | 10 °C/min |
| Final temp | 105 °C |
| Final time | 0 min |
| Rate 2 | 20 °C/min |
| Final temp 2 | 190 °C |
| Final time 2 | 7.25 min for sulfur<br>12.25 min for phosphorus |
| **ALS settings (if installed)** | |
| Sample washes | 2 |
| Sample pumps | 6 |
| Sample wash volume | 8 (maximum) |
| Injection volume | 1 µL |
| Syringe size | 10 µL |
| Solvent A pre washes | 2 |
| Solvent A post washes | 2 |
| Solvent A wash volume | 8 |
| Solvent B pre washes | 0 |
| Solvent B post washes | 0 |
| Solvent B wash volume | 0 |
| Injection mode (7693A) | Normal |
| Airgap Volume (7693A) | 0.20 |
| Viscosity delay | 0 |
| Inject Dispense Speed (7693A) | 6000 |
| PreInjection dwell | 0 |
| PostInjection dwell | 0 |
| **Manual injection** | |
| Injection volume | 1 µL |
| **Data System** | |
| Data rate | 5 Hz |

3 Ignite the FPD+ flame, if not lit.

4 Display the signal output and monitor. This output typically runs around 10. Wait for the output to stabilize. This takes approximately 1 hour.

If the baseline output is too high:

- Check column installation. If installed too high, the stationery phase burns in the flame and increases measured output.
- Check for leaks.
- Bake out the detector and column at 250 °C.
- Wrong flows set for installed filter.

If the baseline output is zero, verify the electrometer is on and the flame is lit.

**5** If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram.

**6** Start the run.

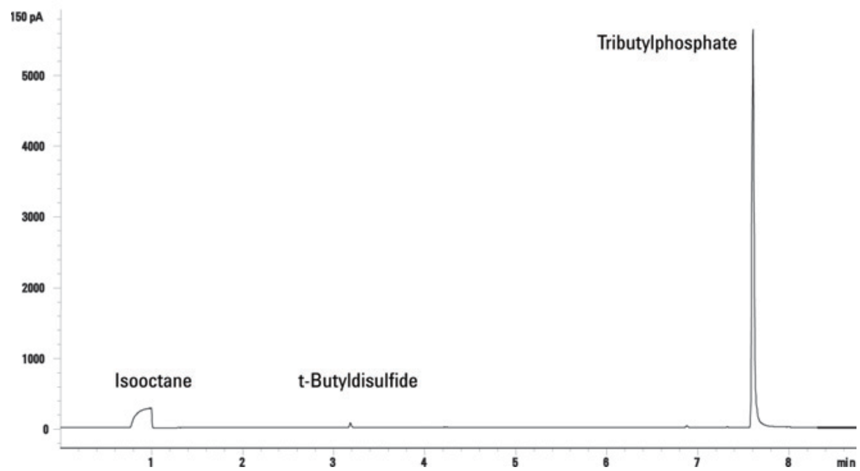
If performing an injection using an autosampler, start the run using the data system or press [Start].

If performing a manual injection (with or without a data system):

**a** Select [Prep Run] to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and press [Start].

**7** The following chromatogram shows typical results for a new detector with new consumable parts installed.




# Sulfur performance

**1** Install the sulfur filter.

**2** Ignite the FPD+ flame, if not lit.

**3** Display the signal output and monitor. This output typically runs between 50 and 60 but can be as high as 70. Wait for the output to stabilize. This takes approximately 2 hours.

If the baseline output is too high:

- Check column installation. If installed too high, the stationery phase burns in the flame and increases measured output.
- Check for leaks.
- Bake out the detector and column at 250 °C.

- Wrong flows set for installed filter

If the baseline output is zero, verify the electrometer is on and the flame is lit.

4 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure the data system will output a chromatogram.

5 Start the run.

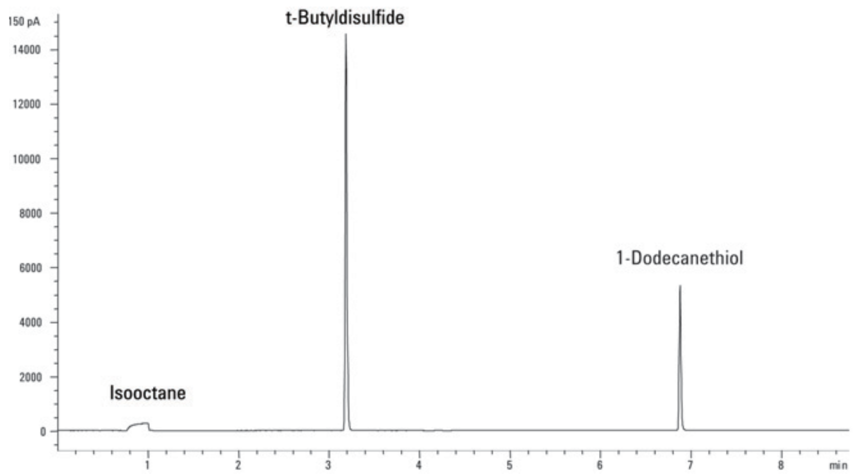
If performing an injection using an autosampler, start the run using the data system or press ▶.

If performing a manual injection (with or without a data system):

**a** Select [P] to prepare the inlet for splitless injection.

**b** When the GC becomes ready, inject 1 µL of the checkout sample and press ▶.

6 The following chromatogram shows typical results for a new detector with new consumable parts installed.



# 18 Chinese Metrology Testing



The 8860 GC complies with the following company standard: Q31/0115000033C005-2016-02.

China Metrology testing of the 8860 GC is performed in accordance with company standard Q31/0115000033C005-2016-02. This chapter provides information and techniques to correctly identify noise and drift when testing an FPD+ or ECD.

# FPD+ and ECD Unit Conversion Factors

At the time of publication, China Metrology testing requires noise and drift metrics as shown below:

| Detector | Reporting units |
| --- | --- |
| FID | A |
| TCD | mV |
| NPD | A |
| FPD+ | A |
| ECD | mV |

However, data collection is required to come through the digital output available through the GC and data system. For the FID, NPD, and TCD, the data system provides data in the desired reporting units. However, for the ECD and FPD+, Agilent reports output to its data systems in "display units," (DU). This section describes how to accurately convert/scale the FPD+ and ECD digital results to make them consistent with Chinese Metrology requirements.

The conversion factors for the FPD+ and ECD take the display unit output from the Agilent data system digital path to an absolute value for current or voltage. Agilent developed the conversion factors empirically, based on measurements from a single system that simultaneously output both the digital and analog data. The conversion factors also incorporate:

- The scaling applied to analog versus digital signals
- An analog signal range setting of 5 (2^5) at the GC
- The unique filtering applied by the 35900 ADC
- The differences in bandwidth (BW) associated with the GC digital channel (5 Hz) and the 35900 ADC analog path (3 Hz)

The differences in channel bandwidth between the analog and digital signal paths can be taken into consideration as follows:

BW = 35900 ADC path / GC digital path = $\sqrt{(3 \text{ Hz} / 5 \text{ Hz})} = 0.7$

## Conversion factors for the FPD+

For the FPD+, the conversion factor is the same whether the phosphorus or sulfur filter is used:

FPD+ (phosphorus): 1 DU = $1 \times 10^{-12}$ A

FPD+ (sulfur): 1 DU = $1 \times 10^{-12}$ A

## Conversion factor for the ECD

For the ECD, the China Metrology standard was established based on an earlier model ECD. Agilent relates display units and Hz (the base unit of measure for the ECD) at a different ratio for the ECD compared to the ECD used to develop the standard. The ECD correlates a DU to 1 Hz, whereas the older ECD correlates 1 DU to 5 Hz. Therefore the conversion also includes the difference in the digital signal reporting between the ECD and the ECD. To convert the ECD noise output into a value comparable to the CMC specification, use the following formula:

ECD: 1 DU = 0.2 mV

The ECD conversion factor shows that the comparable conversion factor for the ECD would be 1 mV/DU = 1 mV/1 Hz.

# Using the Conversion Factors

To use the conversion factors, multiply the ASTM noise reported from the Agilent data system for the GC digital signal path by the appropriate conversion factor.

For example, consider applying the FPD+ and ECD conversion factors to a statistical sampling of digital noise performances measured for both detectors at Agilent:

Average FPD+ ASTM noise, DU[1,2]: 1.54

Average ECD ASTM noise, DU[3]: 0.16

Applying the conversion factors:

FPD+: 1.54 DU × (1 × $10^{-12}$ A/1 DU) = 1.54 × $10^{-12}$ A

ECD: 0.16 DU × (0.2 mV/1 DU) = 0.032 mV

1 Agilent data for FPD+ noise in this example represents sulfur mode only.

2 Data collected for purposes of comparison should be acquired with a nominal FPD+ offset of < 100 DU in sulfur mode and < 20 DU in phosphorus mode and at a data rate of 5 Hz.

3 Data collected for purposes of comparison should be acquired with a nominal ECD baseline at or below 150 DU and at a data rate of 5 Hz.

# References

"Calculation of Performance Factors for the HP 6890 Gas Chromatograph Using Different Data Handling Devices" Agilent Technologies publication 5964-0282E.

"Calculation of Performance Factors for the HP 6890 Gas Chromatograph Using Different Data Handling Devices" Agilent Technologies publication 5091-9207E.

"Calculation of Performance Factors for the HP 6890 Gas Chromatograph Using Different Data Handling Devices" Agilent Technologies publication 5965 8901E.

# 19 Glossary

The terms in **Table 33** are used in discussing this product. They are collected here for your convenience.

**Table 33 Terms**

| Term | Definition |
|---|---|
| ADC | Analog to digital converter |
| ALS | Automatic liquid sampler |
| AS | Autosampler |
| BCD | Binary coded decimal |
| COC | Cool on column inlet |
| DHCP | Dynamic host creation protocol |
| ECD | Electron capture detector |
| ELVDS | Port for external communications with Agilent MSDs |
| EMF | Early maintenance feedback |
| EPC | Electronic pneumatics control |
| EPR | Electronic pneumatics regulation |
| FID | Flame ionization detector |
| FPD+ | Flame photometric detector plus |
| GC | Gas chromatograph |
| HS | Headspace sampler |
| LAN | Local area network |
| LUI | Local User Interface |
| LVDS | Low-voltage differential signaling |
| MS | Mass spectrometer |
| MSD | Mass selective detector |
| NPD | Nitrogen-phosphorus detector |
| NTP | Normal temperature and pressure (25 °C and 1 atmosphere) |
| PCI | Packed column inlet |
| PCM | Pressure Control Module |
| PID | Proportional integral and differential |
| PP | Purged packed inlet |
| PTFE | Polytetraflouroethylene |
| SSL | Split/splitless inlet |
| TCD | Thermal conductivity detector |

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Second Edition, July 2019

G2790-90014

Agilent