Agilent 7820A
Gas Chromatograph

Advanced User Guide
Notice

Agilent Technologies, Inc. 2016

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**CAUTION**

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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.

Firmware Version

This manual is written for 7820A GCs using firmware version A.01.18.
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Software Keypad
Using the Software Keypad

The Agilent software keypad (remote controller) provides the ability to program and use the 7820A Gas Chromatograph (GC). Refer to the Operating Guide for instructions for its installation and use.

![Software Keypad](image)

**Figure 1** Software keypad (remote controller)

All instructions in this manual assume the use of the software keypad unless otherwise noted.
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Run Time Programming

Run time programming during a method allows certain setpoints to change automatically 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.

Its uses include:

- 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)
- 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)

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. See “From the run or clock time tables” on page 115.

**Using run time events**

The [Run Table] key is used to program the following timed events.

- Valves (1-2)
- Analog signal definition, zero, and range
- TCD negative polarity (on/off)
- Detector gas flow (on/off), including NPD H₂ fuel gas
Programming run time events

1. Press [Run Table].
2. Press [Mode/Type] to see the available run time events.
3. Scroll to the event to be programmed. Press [Enter].
4. Enter values for the **Time:** and the other parameter. Press [Enter] after each entry.

The run table

The programmed events are arranged in order of execution time in the Run Table. This is a brief example:

<table>
<thead>
<tr>
<th>RUN TABLE (1 of 3)</th>
<th>Event 1 rotates a valve.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time:</td>
<td>0.10</td>
</tr>
<tr>
<td>Valve #2</td>
<td>On</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>RUN TABLE (2 of 3)</th>
<th>Event 2 adjusts the signal range.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time:</td>
<td>3</td>
</tr>
<tr>
<td>Analog signal 2 range</td>
<td>2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>RUN TABLE (3 of 3)</th>
<th>Event 3 resets Valve #2 to its original position in preparation for another run. Valves do not reset automatically.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time:</td>
<td>4.20</td>
</tr>
<tr>
<td>Valve #2</td>
<td>Off</td>
</tr>
</tbody>
</table>

Adding events to the run table

1. To add new events to the run table, press [Mode/Type] while on the **Time:** line of any entry.
2. Select the event type.
3. Set appropriate **Time:** and other parameters. Some require numbers; others require [On/Yes] or [Off/No].
4. Repeat until all entries are added. Events are automatically placed in order by execution time.
Editing events in the run table

1. Press [Run Table].
2. Move the cursor to the event you want to change.
3. To edit the time for an event, move the cursor to the line labeled 'Time'. Type the desired time and press [Enter].
4. To edit a setpoint value, scroll to the setpoint line. Press [On/Yes] or [Off/No] or enter a numeric value for the setpoint. Press [Enter].

Deleting run time events

1. Press [Run Table].
2. From within this table press [Delete] to delete events from the run time table. You will be asked to confirm the deletion.
3. Press [On/Yes] to delete the current timed event; press [Off/No] to cancel this operation.
4. To delete the entire table, press [Delete][Run Table].
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. 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

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.

Programming clock time events

1. Press [Clock Table].
2. Press [Mode/Type] to see the available clock time events.
3. Scroll to the parameter to be programmed.
4. Edit Time: and the setpoints for this event.

When the clock event is executed, a confirming message appears.

Adding events to the clock table

1. Press [Clock Table].
2. Press [Mode/Type]. When entries are added, they are automatically ordered chronologically.
Select the event type.
Set appropriate parameters.
Repeat this process until all entries are added.

**Editing clock time events**

1. Press [Clock Table] to view all events programmed.
2. Scroll to the event you want to change.
3. To edit the time for an event, move the cursor to the line labelled *Time:* and type the desired time.
4. To edit a setpoint value, scroll to the setpoint item. Press [On/Yes] or [Off/No], or enter a numerical value for the setpoint.

**Deleting clock time events**

1. Press [Clock Table].
2. Use [Delete] to remove events from the clock time table. You will be asked to confirm the deletion.
3. Press [On/Yes] to delete the current timed event; press [Off/No] to cancel this operation.

To delete the entire table, press [Delete][Clock Table].
Post Run Programming

This function can be used with both isothermal and programmed methods. Post run is a period that begins at the end of the normal run. The parameters include:

- **Time**—How long is the post run period?
- **Oven Temperature**—What is the oven temperature during the post run period?

EPR (electronic pneumatics regulation) equipped GCs do not support post run parameters for Column n pres and Column n flow since column pressure and flow are set manually.

- **Column n pres**—For a column controlled in a pressure mode, enter the pressure for this column during the post run period.
- **Column n flow**—For a column controlled in a flow mode, enter the flow rate for this column during the post run period.

Post run may be used to clean out a column in preparation for the next run, backflush a column to eliminate high-boilers, and other functions.

When the Post run Time elapses, the GC returns to the initial state defined in the current method.

**To enable a post run program**

1. Press [Post Run].
2. Type a non-zero time for the post run duration and press [Enter]. The post run parameters available for the current GC configuration appear.
3. Scroll to each desired parameter, type the value for the post run period, and press [Enter].

**To disable a post run program**

1. Press [Post Run].
2. Type a 0 as the post run time and press [Enter].
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Using Hydrogen

**WARNING** When using hydrogen (H₂), as the carrier gas, be aware that hydrogen (H₂) gas can flow into the oven and create an explosion hazard. Therefore, be sure that the supply is off until all connections are made, and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen (H₂) gas is supplied to the instrument.

**WARNING** Hydrogen (H₂) is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen (H₂), leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen (H₂) supply at its source before working on the instrument.
# Inlet Overview

## Table 1  Comparing inlets

<table>
<thead>
<tr>
<th>Inlet</th>
<th>Column</th>
<th>Mode</th>
<th>Sample concentration</th>
<th>Comments</th>
<th>Sample to column</th>
</tr>
</thead>
<tbody>
<tr>
<td>Split/splitless</td>
<td>Capillary</td>
<td>Split Pulsed split*</td>
<td>High High</td>
<td>Useful with large injections</td>
<td>Very little Very little</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Splitless Pulsed splitless*</td>
<td>Low Low</td>
<td>Useful with large injections</td>
<td>All All</td>
</tr>
<tr>
<td>Purge packed column</td>
<td>Packed</td>
<td>n/a</td>
<td>Any Any</td>
<td>OK if resolution not critical</td>
<td>All All</td>
</tr>
<tr>
<td></td>
<td>Large capillary</td>
<td>n/a</td>
<td>Any Any</td>
<td></td>
<td>All All</td>
</tr>
<tr>
<td>Packed column</td>
<td>Packed</td>
<td>n/a</td>
<td>Any</td>
<td></td>
<td>All</td>
</tr>
<tr>
<td>Cool on-column</td>
<td>Capillary</td>
<td>n/a</td>
<td>Low or labile</td>
<td>Minimal discrimination and decomposition</td>
<td>All</td>
</tr>
</tbody>
</table>

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

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

### Table 2  Column size and carrier flow rate

<table>
<thead>
<tr>
<th>Column type</th>
<th>Column size</th>
<th>Hydrogen</th>
<th>Helium</th>
<th>Nitrogen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Packed</td>
<td>1/8-inch</td>
<td>30</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1/4-inch</td>
<td>60</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>Capillary</td>
<td>0.05 mm id</td>
<td>0.5</td>
<td>0.4</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>0.10 mm id</td>
<td>1.0</td>
<td>0.8</td>
<td>n/a</td>
</tr>
<tr>
<td></td>
<td>0.20 mm id</td>
<td>2.0</td>
<td>1.6</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>0.25 mm id</td>
<td>2.5</td>
<td>2.0</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>0.32 mm id</td>
<td>3.2</td>
<td>2.6</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td>0.53 mm id</td>
<td>5.3</td>
<td>4.2</td>
<td>1.5</td>
</tr>
</tbody>
</table>
About Gas Saver

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 purge and split vent flows decrease. Flows—except column flow—remain at the reduced level until you press [Prep Run].
To use gas saver

1. Press [Front Inlet] or [Back Inlet].
2. Turn gas saver On.
3. Set Gas saver flow. It must be at least 15 mL/min greater than the column flow.
4. If in split mode, set Saver time after injection time. In all other modes, set after Purge time.
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 (press [Prep Run] before each run on the GC keypad or software keypad), 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 Run light blinks on the GC, and the Pre Run indicator lights on the software keypad.
- Setpoints change to the correct values for injection.
- Inlet, detector, and oven equilibration times begin.

When all criteria for a run are met, the Not Ready light turns off. The GC is now ready for sample injection.

**The [Prep Run] key**

Press [Prep Run] before you inject a sample manually. The GC enters the Pre Run state. When the Run light stops blinking and the Not Ready light goes off, begin the analysis.

**Agilent samplers**

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

Start the sampler. It generates the [Prep Run] function, When all the setpoints are reached and the GC becomes Ready, sample injection begins.
Auto Prep Run

To set this parameter, usually for a non-Agilent integrator, workstation, or other controlling device:

1  Press [Config] to view a list of configurable parameters.
2  Scroll to Instrument and press [Enter].
3  Scroll to Auto prep run and press [On/Yes].
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.

**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.

**Septum tightening (S/SL)**

Septum retainer nuts must be tightened enough to obtain a good gas seal, but not so much as to compress the septum and make it difficult to push a syringe needle through it.

For the standard septum retainer nut, an internal spring in the septum retainer applies pressure to the septum. For inlet pressures up to 100 psi, tighten the retainer until the C-ring lifts about 1 mm above the top surface. This is adequate for most situations.

With higher inlet pressures, tighten the septum retainer until the C-ring stops turning, indicating that the retainer is in firm contact with the septum.

If using a Merlin Microseal™ septum, finger tighten the septum nut, until snug (not loose). The pressure capacity depends on the duckbill seal used.
Standard S/SL inlet

The standard split/splitless inlet is rated to 100 psi pressure at the inlet. It is appropriate for most columns.

Recommended source pressure is 120 psi.

Split/Splitless inlet split mode overview

During a split injection, a liquid sample is introduced into a hot inlet where it vaporizes rapidly. A small amount of the vapor enters the column while the major portion exits from the split/purge vent. The ratio of column flow to split vent flow is controlled by the user. Split injections are primarily used for high concentration samples when you can afford to lose most of the sample out the split/purge vent. It is also used for samples that cannot be diluted.

The split ratio is equal to the split vent flow divided by the column flow. If the column has been configured, the desired split ratio can be entered directly.

The pneumatics for this inlet in split mode operation are shown in the figure below.
**Split/Splitless inlet splitless mode overview**

In this mode, the split vent valve is closed during the injection and remains so while the sample is vaporized in the liner and transferred to the column. At a specified time after injection, the valve opens to sweep any vapors remaining in the liner out the split vent. This avoids solvent tailing due to the large inlet volume and small column flow rate. Specify the purge time and purge flow rate in the inlet parameter list.

**NOTE**

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

If you are using gas saver, the gas saver time should be *after* the purge time.
The S/SL inlet pulsed split and splitless modes

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

The pressure pulse modes increase inlet pressure just before the beginning of a run and return it to the normal value after a specified amount of time. The pressure pulse:

- reduces the solvent vapor volume
- reduces the risk of inlet overload
- tightens the sample band
- may allow use of a 2 mm liner, reducing the active glass area

If your chromatography is degraded by the pressure pulse, a retention gap may help restore peak shape.

You can do column pressure and flow programming when in the pressure pulse mode. However, the pressure pulse will take precedence over the column pressure or flow ramp.

Split/Splitless inlet split mode minimum operating pressures

The minimum recommended inlet total flow is 20 mL/minute. When the split/splitless inlet is operated in Split mode, there will be a minimum pressure at which the inlet can operate. Typically, low inlet pressures may be required for shorter, wide bore columns. The minimum pressure is a function of carrier gas type, total inlet flow, liner design, and possible contamination of the split vent tube or trap.

A wide bore column requires a much lower inlet pressure than a typical capillary column to maintain a given flow. Setting the split ratio (total flow) too high when using a wide bore column can create an unstable control relationship between the pressure and flow control loops.
These numbers are based on the resistance to flow of new, clean inlet systems. Sample condensation in the split vent tube or a dirty filter can make these values non-attainable.

**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 will also incorporate glass wool or some other source of surface area inside the liner that provides for complete sample vaporization across the boiling point range of the sample. Select an appropriate liner from Table 4.

<table>
<thead>
<tr>
<th>Liner Description</th>
<th>Volume (µL)</th>
<th>Mode</th>
<th>Deactivated</th>
<th>Part Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Pressure Drop – Positioning Bead</td>
<td>870</td>
<td>Split – Fast Injection</td>
<td>Yes</td>
<td>5183-4647</td>
</tr>
<tr>
<td>4mm ID, Glass Wool</td>
<td>990</td>
<td>Split – Fast Injection</td>
<td>No</td>
<td>19251-60540</td>
</tr>
<tr>
<td>Empty Pin &amp; Cup</td>
<td>800</td>
<td>Split – Manual Only</td>
<td>No</td>
<td>18740-80190</td>
</tr>
<tr>
<td>Packed Pin &amp; Cup</td>
<td>800</td>
<td>Split – Manual Only</td>
<td>No</td>
<td>18740-60840</td>
</tr>
</tbody>
</table>
Splitless liner

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 purge delay. 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 5  Splitless mode liners

<table>
<thead>
<tr>
<th>Liner</th>
<th>Description</th>
<th>Volume</th>
<th>Mode</th>
<th>Deactivated</th>
<th>Part Number</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Single Taper Glass Wool" /></td>
<td>Single Taper Glass Wool</td>
<td>900 uL</td>
<td>Splitless</td>
<td>Yes</td>
<td>5062-3587</td>
</tr>
<tr>
<td><img src="image" alt="Single Taper" /></td>
<td>Single Taper</td>
<td>900 uL</td>
<td>Splitless</td>
<td>Yes</td>
<td>5181-3316</td>
</tr>
<tr>
<td><img src="image" alt="Dual Taper" /></td>
<td>Dual Taper</td>
<td>800 uL</td>
<td>Splitless</td>
<td>Yes</td>
<td>5181-3315</td>
</tr>
<tr>
<td><img src="image" alt="2 mm Quartz" /></td>
<td>2 mm Quartz</td>
<td>250 uL</td>
<td>Splitless</td>
<td>No</td>
<td>18740-80220</td>
</tr>
<tr>
<td><img src="image" alt="2 mm Quartz" /></td>
<td>2 mm Quartz</td>
<td>250 uL</td>
<td>Splitless</td>
<td>Yes</td>
<td>5181-8818</td>
</tr>
<tr>
<td><img src="image" alt="1.5 mm" /></td>
<td>1.5 mm</td>
<td>140 uL</td>
<td>Direct</td>
<td>No</td>
<td>18740-80200</td>
</tr>
<tr>
<td><img src="image" alt="Single Taper Glass Wool" /></td>
<td>Single Taper Glass Wool</td>
<td>900 uL</td>
<td>Splitless</td>
<td>Yes</td>
<td>5062-3587</td>
</tr>
</tbody>
</table>
Agilent provides a Vapor Volume Calculator to help you determine if a liner is suitable for a method. To use the calculator install the Agilent Instrument utility provided with the GC. The calculator is also provided with the Agilent Instrument Utilities software.

### Setting parameters for the S/SL split mode

**Mode**  The current operating mode—split

**Temperature**  Actual and setpoint inlet temperatures

**Pressure**  Actual and setpoint inlet pressure

**Split ratio**  The ratio of split vent flow to column flow. Column flow is set at the Column parameter list. This line appears only if your columns in the flow path are defined.

**Split flow**  Flow, in mL/min, from the split vent. This line appears only if your columns in the flow path are defined.

**Total flow**  This is the total flow into the inlet, which is the sum of the split vent flow, column flow, and septum purge flow. When you change the total flow, the split ratio and split vent flow change while the column flow and pressure remain the same.
**Septum Purge**  Flow, in mL/min, through the septum purge line.

**If all columns in the flow path are defined**

1. Press [Front Inlet] or [Back Inlet].
2. Scroll to **Mode:** and press [Mode/Type]. Select **Split**.
3. Set the inlet temperature.

The following two steps are not applicable to EPR equipped GCs with an S/SL inlet. If you want to calculate the split ratio, adjust the total flow (by using the 2/+ and 8/- buttons on the software keypad, and then adjust the column head pressure until the desired split ratio is achieved.

4. If you want a specific split ratio, scroll to **Split ratio** and enter that number. **Split flow** will be calculated for you.
5. If you want a specific split flow, scroll to **Split flow** and enter that number. **Split ratio** will be calculated for you.

**NOTE**

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

6. If desired, turn on **Gas saver**. Set **Saver time** after the injection time. Press [Prep Run] (see “Pre Run and Prep Run” on page 25) before manually injecting the sample.

**If a column in the flow path is not defined**

1. Press [Front Inlet] or [Back Inlet].
2. Set the inlet temperature.
3. Set **Total flow** into the inlet. Measure the split vent flow using a flow meter.
4. Subtract split vent flow and septum purge flow (see “Pre Run and Prep Run” on page 25) from **Total flow** to get column flow.

**NOTE**

The following step is not supported on EPR (electronic pneumatics regulation) equipped GCs since the split ratio cannot be set.

5. Calculate the split ratio (split vent flow/column flow). Adjust as needed.
Selecting parameters for the S/SL splitless mode

A successful splitless injection consists of these steps:

1. Vaporize the sample and solvent in a heated inlet.
2. Use a low flow and low oven temperature to create a solvent-saturated zone at the head of the column.
3. Use this zone to trap and reconcentrate the sample at the head of the column.
4. Wait until all, or at least most, of the sample has transferred to the column. Then discard the remaining vapor in the inlet—which is mostly solvent—by opening a purge valve. This eliminates the long solvent tail that this vapor would otherwise cause.
5. Raise the oven temperature to release the solvent and then the sample from the head of the column.

Some experimentation is needed to refine the operating conditions. Table 6 provides starting values for the critical parameters.

Table 6  Splitless mode inlet parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Allowed setpoint range</th>
<th>Suggested starting value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oven temperature</td>
<td>24 °C to 425 °C</td>
<td>10 °C below solvent boiling point</td>
</tr>
<tr>
<td>Oven initial time</td>
<td>0 to 999.9 minutes</td>
<td>≥ Inlet purge time</td>
</tr>
<tr>
<td>Inlet purge time</td>
<td>0 to 999.9 minutes</td>
<td>2 x Liner volume Column flow</td>
</tr>
<tr>
<td>Gas saver time*</td>
<td>0 to 999.9 minutes</td>
<td>After purge time</td>
</tr>
<tr>
<td>Gas saver flow</td>
<td>15 to 1000 mL/min</td>
<td>15 mL/min greater than maximum column flow</td>
</tr>
</tbody>
</table>

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

Setting parameters for the S/SL splitless mode

Mode  The current operating mode—splitless

Oven temperature  Below solvent boiling point

Temperature  Actual and setpoint inlet temperatures
Inlets 3

There is no setpoint for the Pressure parameter in EPR (electronic pneumatics regulation) equipped GCs with an S/SL inlet.

**Pressure**  Actual and setpoint inlet pressure in psi, bar, or kPa

**Purge time**  The time, after the beginning of the run, when you want the purge valve to open. This is the time in which the vaporized sample transfers from the liner to the column.

**Purge flow**  The flow, in mL/min, from the purge vent, at Purge time. You will not be able to specify this value if any column in the flow path is not defined. For EPR equipped GCs, you can manually adjust this parameter using the 2/+ and 8/- keys on the software keypad.

There is no setpoint for the Total flow parameter in EPR (electronic pneumatics regulation) equipped GCs with an S/SL inlet.

**Total flow**  The actual flow to the inlet during a Pre-run (Pre-run light is on and not blinking) and during a run before purge time. You cannot enter a setpoint at these times. At all other times, Total flow will have both setpoint and actual values.

**If all columns in the flow path are defined**

1. Press [Front Inlet] or [Back Inlet].
3. Set the inlet temperature.

There is no setpoint for the Purge flow parameter in EPR (electronic pneumatics regulation) equipped GCs with an S/SL inlet.

4. Enter a **Purge time** and a **Purge flow**.

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

5. If desired, turn on **Gas saver**. Make certain the time is set after the **Purge time**.
6 Press [Prep Run] (see “Pre Run and Prep Run” on page 25) before manually injecting a sample (this is automatic for Agilent ALS).

If a column in the flow path is not defined

1 Press [Front Inlet] or [Back Inlet].
2 Scroll to Mode: and press [Mode/Type]. Select Splitless.
3 Set the inlet temperature.
4 Enter a Purge time.
5 Set your Total flow greater than the sum of column flow plus the septum purge flow—see “Pre Run and Prep Run” on page 25—to guarantee adequate column flow.
6 Press [Prep Run] (see “Pre Run and Prep Run” on page 25) before manually injecting a sample.

Setting parameters for the S/SL pulsed modes

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

The pulsed mode parameters are the same as the non-pulsed parameters, but with two additional values.

Pulsed pressure  The inlet pressure you want at the start of the run. The pressure rises to this value when [Prep Run] is pressed and remains constant until Pulse time elapses, when it returns to Pressure.

Pulse time  This is the time after the start of the run when the inlet pressure returns to Pressure.
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, regardless or whether or not the column is capillary and defined, or packed.
Setting parameters

The inlet operates in flow control mode.

While in flow control mode, you cannot enter pressures here.

**Temperature**  The setpoint and actual temperature values.

**Pressure**  The actual pressure (in psi, bar, or kPa) supplied to the inlet. You cannot enter a setpoint here.

**Total flow**  Enter your setpoint here, actual value is displayed. Inlet is mass flow controlled.

**Col flow**  Enter your setpoint here, actual value is displayed.

**Septum purge**  The actual flow, in mL/min, through the septum purge line.
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.
Adjusting parameters

The inlet operates in flow control mode. You cannot enter pressures here.

**Temperature**  The setpoint and actual temperature values.

**Col flow**  Adjust using the 2/+ and 8/- keys on the virtual keypad, actual value is displayed.
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. A cryogenic cooling option that uses liquid CO₂ or N₂ from the oven cryogenic system can reach sub-ambient temperatures.
**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. Use predrilled septa.
- 0.53 mm automatic on-column or retention gap
- 0.2 mm manual

To select the correct hardware for a column and injection type, refer to **Maintaining Your GC**.

**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 µL of sample injected. Information on ordering retention gaps can be found in the Agilent catalog for consumables and supplies.

If you are using a retention gap and are operating with the column defined, the length of the retention gap could affect the calculations for flow and velocity through your column. If your retention gap is the same inside diameter as your column, it is a good idea to add the retention gap and column length before entering the number on the Configure Column parameter list. If the retention gap inside diameter is larger than your column, this step may not be necessary.

**COC inlet temperature control**

**CryoBlast (optional)**

CryoBlast shortens the cycle time between runs. If you have a CO₂ or N₂ cryogenic valve and the CryoBlast feature, you can cool the inlet to −37 °C in either the track oven or temperature program modes.

The CryoBlast accessory uses coolant from the oven cryogenic system.
**Track oven mode**

In the **Track oven** mode, the inlet temperature stays 3 °C higher than the oven temperature throughout the oven program. You cannot enter a temperature setpoint—it is set automatically. If you have CryoBlast, the inlet will track oven temperatures to –40°C; without CryoBlast, the lower limit is set by room temperature.

**Temperature programming mode**

In this mode, you can enter up to three temperature ramps in the inlet parameter list so that the inlet and the oven operate independently. This is the recommended mode if operating below –20 °C.

At these very low oven temperatures, the inlet temperature should be at least 20 °C higher than the oven temperature. This will be more than adequate for solvent focusing.

At temperatures greater than ambient, the inlet should always be at least 3 °C warmer than the oven for proper control of the inlet temperature.

The oven temperature program controls the run. If it is longer than the inlet temperature program, the inlet will remain at its final temperature until the oven program (and the run) ends.

**Cryogenic considerations**

When using track oven mode with a cryogenic oven, all other inlets must be off or in track oven mode.

If cryo is turned on, and if the inlet is cooled during a run, cryogen is used to achieve the setpoint. This may have a negative impact on the chromatographic performance of the oven and cause distorted peaks.

The inlet uses the same cryo coolant as configured for the oven.

**Setting COC inlet flows/pressures**

1. Configure capillary column to inlet—select constant flow or pressure.
2. Set column flow, linear velocity, or inlet pressure.
3. Set Septum Purge, typically 3 to 10 mL/min.
Setting COC inlet parameters

**Track oven mode**

1. Press [Front Inlet] or [Back Inlet].
2. Press [Mode/Type] and select *Track oven*.

There is no setpoint for *Track oven* mode.

**Ramped temperature mode**

1. Press [Front Inlet] or [Back Inlet].
2. Press [Mode/Type] and select *Ramped temp*.
3. Enter a value for *Temp*. This is the starting temperature.
4. Enter an *Init time*. This is the length of time the inlet will stay at the starting temperature after a run has begun.
5. Enter *Rate 1*. This is the rate at which the inlet will be heated or cooled. A Rate of 0 halts further programming.
6. Enter *Final temp 1*. This is the inlet temperature at the end of the first ramp.
7. Enter *Final time 1*. This is the number of minutes the inlet holds *Final temp 1*.
8. To enter a second (or third) ramp, scroll to the appropriate *Rate* line and repeat steps 5 through 7.
4

Columns and Oven

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About the Oven

### Table 7  Oven capabilities

<table>
<thead>
<tr>
<th>Capability</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range</td>
<td>0 °C to the configured limit</td>
</tr>
<tr>
<td>Maximum temperature</td>
<td>425 °C</td>
</tr>
<tr>
<td></td>
<td>(100 V oven: 350°C)</td>
</tr>
<tr>
<td>Temperature programming</td>
<td>Up to five ramps</td>
</tr>
<tr>
<td>Maximum run time</td>
<td>999.99 minutes</td>
</tr>
<tr>
<td>Temperature ramp rates</td>
<td>0 to 75 °C/min, depending on instrument configuration</td>
</tr>
<tr>
<td></td>
<td>(100 V oven: 0 to 30 °C/min)</td>
</tr>
</tbody>
</table>

### Oven safety

For safety, opening the oven door turns off power to the oven heater, and fan, but maintains the setpoints in memory.

Closing the oven door returns the oven to normal operation.

If the oven cannot attain or maintain an entered setpoint temperature during normal above-ambient operation, a problem is assumed and the oven is switched off.

Possible problems include:

- The oven vent flaps not working
- The oven fan, heater, or temperature sensor not working properly
- An electronic problem

When a shutdown occurs, the Off line in the oven parameter list blinks and the oven remains off until switched on again by pressing [Oven][On] or by editing the Temperature setpoint.
Configuring the Oven

Oven configuration sets maximum temperature, equilibration time, and the cool down mode.

**Maximum temperature** Maximum allowable oven temperature setpoint. Some accessories, such as the valve box, valves and columns have specific temperature limits. When configuring **Maximum temperature**, these limits should be considered so that the accessories are not damaged. Oven setpoints are verified as they are entered; a message is displayed when an entered setpoint is inconsistent with a previously defined maximum.

**Equilibration time** The time required for the oven temperature to equilibrate after temperature is modified. Equilibration time begins when the actual oven temperature comes within 1 °C of the oven temperature setting. The **Equilibration time** setpoint can be 0 to 999.99 minutes.
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.

Programming setpoints

**Temperature**  Starting temperature of a temperature programmed run. When the program begins, this value is copied into a temporary setpoint called *Init temp*. At the end of the run, *Temperature* is reset to the value in *Init temp* and the oven returns to its starting temperature.

**Initial time**  Time in minutes that the oven will stay at the starting temperature after a programmed run has begun.

**Rate**  The rate in °C/min at which the oven will be heated or cooled.
**Final temperature**  Temperature of the oven at the end of a heating or cooling rate.

**Final time**  Time in minutes that the oven will be held at the final temperature of a temperature-programmed rate.

Total length of a run is determined by its oven temperature program. The maximum allowable time for a run is 999.99 minutes. If the program is still running at that time, the run terminates.

See also “Post Run Programming.”

**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 8 lists typical oven ramp rates.

<table>
<thead>
<tr>
<th>Temperature range (°C)</th>
<th>100 V oven ramp rate (°C/minute)</th>
<th>200/220/230/240 V oven ramp rate (°C/minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 to 70</td>
<td>30</td>
<td>75</td>
</tr>
<tr>
<td>70 to 115</td>
<td>30</td>
<td>45</td>
</tr>
<tr>
<td>115 to 175</td>
<td>30</td>
<td>40</td>
</tr>
<tr>
<td>175 to 300</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>300 to 425</td>
<td>20</td>
<td>20</td>
</tr>
</tbody>
</table>

**Setting the oven parameters for constant temperature**

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. Press [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.
Setting the oven parameters for ramped temperature

Single ramp

1. Press [Oven] to open the oven parameter list.
2. Enter a starting temperature (Temperature).
3. Enter the time (Initial time) that you want the oven to stay at Temperature.
4. Enter the rate (Rate 1) at which the oven temperature is to change.
5. Enter the final temperature (Final temperature 1).
6. Enter the time (Final time 1) the oven is to hold Final temperature 1.
7. To end the oven ramp program after Ramp 1, set Rate 2 to zero.

Multiple ramps

In a multiple-ramp program, Final time for one ramp is also Initial time for the next ramp. Thus, there is only one Initial time.

1. Set up the first oven ramp as described in “Single ramp”.
2. Enter the rate (Rate 2) at which you want the oven temperature to increase for the second oven ramp.
3. Enter the final temperature (Final temperature 2).
4. Enter the number of minutes (Final time 2) that you want the oven to hold the final temperature.
5. To end the temperature program after the second ramp, set Rate 3 to zero.
6. To add additional oven ramps, repeat the steps described.
About Columns

In all GCs, a sample—which is a mixture of several components—is vaporized in an inlet, separated in a column, and examined in a detector.

The column separates components in time because:

- When a vaporized component is presented with a gas phase and a coating phase, it divides between the two phases according to its relative attraction to the two phases.
- The “attraction” can be solubility, volatility, polarity, specific chemical interaction, or any other property that differs from one component to another.
- If one phase is stationary (the coating) and the other is moving (the carrier gas), the component will travel at a speed less than that of the moving phase. How much less depends on the strength of the attraction.
- If different components have different “attractions”, they will separate in time.

Selecting the correct packed glass column type

This topic is covered in the Maintenance manual. See To attach a packed column to the purged packed inlet for details.

About the column modes

The flow modes available are determined by the GC inlet’s control mode. When the inlet’s control mode is set to Pressure control, all of the flow modes and pressure modes below are available for the column. When the inlets control mode is set to Flow control, the column’s mode is not selectable. For an inlet’s mode of Flow control, only column flow can be entered.

The flow modes

Flow rates are corrected to NTP (normal temperature and pressure, 25 °C and 1 atmosphere.

- 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

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.

**NOTE**

Ramped pressure mode is not supported on EPR (electronic pneumatics regulation) equipped GCs.

• **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.

### Select a column mode

The column’s mode parameter is not available if the inlet’s mode parameter is set to **Flow control**.

1. Press [Col #] and enter the column number.
2. Scroll to the Mode line.
3. Press [Mode/Type] to see the column mode list.
4. Scroll to the column mode you want. Press [Enter].

**NOTE**

Ramped flow mode is not supported on EPR (electronic pneumatics regulation) equipped GCs.
This completes column mode selection. Next you must specify the inlet conditions either during the entire run (if you selected either of the constant modes) or at the beginning of the run (if you selected either of the ramped modes).

**Setting the column parameters for constant flow or constant pressure**

If the column is defined, you can enter any one of these quantities—the GC will calculate and display the other two.

For example, you may have selected **Constant pressure** as the column mode. You decide to specify, as a starting condition, the column flow. The GC will compute the pressure necessary to achieve this flow (as well as the average linear velocity) and hold this pressure constant during the run.

If you select **Constant flow** as the mode and specify column flow as the initial condition, the GC will still calculate the pressure necessary to achieve this flow, but it will adjust the pressure as necessary to maintain constant flow.

If the column is not defined, you can enter only pressure. Constant flow can still be specified, but the GC cannot know what the flow is.

1. Press **[Col #]** and enter the column number.
2. Scroll to the **Pressure** or **Flow** or **Velocity** line.
3. Type the desired initial value, followed by **[Enter]**. The GC will compute and display the other two values. Adjust them, if you choose to, by repeating steps 2 and 3 but note that changing any one changes all three.

This completes setting the initial carrier gas condition.

**Enter a flow or pressure program (optional)**

If you selected either the ramped pressure or ramped flow column mode, the column parameter list contains entries for setting up a ramp program.

You begin with an initial value, either **Initial Pressure** or **Initial Flow**, and an **Initial time**. At the end of that time, **Rate 1** begins and runs until it reaches **Final pressure** (or **Final flow**). It remains at that value for **Final time 1**. You can then add a second and third ramp, each consisting of a Rate, a Final value (pressure or flow), and a Final time.

The program ends when it reaches a **Rate** that is set to 0 (**Off**).
When a flow or pressure program is running, the **Pressure**, **Flow**, and **Velocity** lines that you used to set constant conditions show the progress of the program.

The oven program determines the length of the run. If a flow or pressure program ends before the analytical run does, the flow (or pressure) remains at the last final value.

### Programming column pressure or flow

1. Press [Col #] then enter the column number.
2. Scroll to **Initial pressure** (or **Initial flow**). Type the desired value and press [Enter].
3. Similarly, enter a value for **Initial time**. This completes the initial part of the program.
4. To begin a ramp, enter a positive value for **Rate 1**. It does not matter whether you are programming up or down—the rate is always positive.
5. If **Rate 1** is zero, the program ends here. If you enter any other value, the Final value lines for the first ramp appear and the cursor moves to the line.
6. Enter values for **Final pressure 1** (or **Final flow 1**) and **Final time 1**. This completes the first ramp.
7. To enter a second ramp, scroll to the appropriate **Rate** line and repeat steps 5 and 6. A maximum of 3 ramps can be entered.

### Advantages of the optional Backflush Wizard

The procedures below provide simple ways to create the backflush program. However, for best results, use the optional Backflush Wizard. The wizard provides the following advantages:

- Helps you select the best technique for your setup.
- Helps you validate the hardware’s capabilities for backflushing.
- Helps you create relevant baseline data (pre-backflush).
- Helps you validate that the backflushing worked (unwanted peaks are completely removed from the columns, while wanted peaks elute normally) by performing runs at various stages and analyzing the data.
- Helps you adjust the method, if needed, until the backflushing succeeds.
• Provides the ability to create pre-column backflush methods, which require careful adjustment due to the elution times and elution order differences found on pre-columns (compared to the analytical column).

For more information, see the *GC and GC/MS User Manuals & Tools* DVDs. In addition, the Backflush Wizard contains extensive documentation.

**Procedures for manually creating a backflush method**

**To set up a post run backflush**

A main advantage of the post-run backflush method is that the GC turns off detection during this time. If using an MS, this helps prevent damage to the detector and is the recommended backflush method.

If using an Agilent data system, a Backflush Wizard provides a straightforward interface for making these settings.

1. Verify that all columns are properly configured.
2. Since the backflush will run as a post-run program, set the method so that the oven program ends after the last peak of interest returns to baseline, or after reaching the last temperature of interest.
3. Press **[Post Run]** and enter the backflush duration as the **Time**.
   - The oven temperature and column pressure/flow setpoints for installed columns appear.
   - Whether you can enter a pressure or flow for a column depends on its mode (pressure or flow).
4. Enter the oven temperature for the backflush.
5. Enter the backflush flow(s) or pressure(s).

If in flow mode:
   - Enter a negative flow for the column connected between the inlet and the CFT device. The GC will automatically establish the corresponding pressures needed in the inlet and the CFT device.
   - Increase the flow rates in the column(s) connected between the CFT device and the detector(s).

If in pressure mode:
   - Set the pressure of the column connected between the inlet and the CFT device to 0.000. This turns off flow from the inlet.
- Increase the pressure of the primary column connected between the CFT device and the detector(s). This creates the backward flow through the column and increases the flow through any connected detectors.

When developing the backflush portion of your method, consider the following:

- Ensure that the split vent flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.
- If using gas saver, ensure that the gas saver flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.

**To backflush using a ramped pressure program**

In this case, the backflush occurs as part of the run, so the detectors continue to collect data. During the backflush, you may wish to turn off data collection in the data system.

**CAUTION**

To avoid damage to an MS, Agilent strongly recommends setting up backflush as a post run event, not as part of a ramped column program. If you still choose to backflush as part of a run, be very careful that the flow into the MS does not exceed the limits of the vacuum pump.

1. Verify that all columns are properly configured.
2. Enter all method parameters for the analysis: sampler parameters, inlet parameters, oven temperature profile, detector flows and temperatures, and so forth.
3. Program the oven for the backflush.
   - Include any temperature profile needed for backflush.
   - Set the total run time to include sufficient time for backflush.
4. Program the pressure ramp for the column installed between the inlet and the CFT device. After the last analyte elutes or after reaching the last temperature of interest, program a fast ramp (for example, 30 psi/min) with a final pressure of 0.
5. Program the pressure ramp for the primary column installed between the CFT device and the detector. The pressure should increase slightly during the backflush duration so that the flow into the detectors remains relatively stable.
If you turned off data acquisition in a data system during backflush, remember to turn it on again at the end of the run.

**To backflush using a ramped flow program**

In this case, the backflush occurs as part of the run, so the detectors continue to collect data. During the backflush, you may wish to turn off data collection in the data system.

To avoid damage to an MS, Agilent strongly recommends setting up backflush as a post run event, not as part of a ramped column program. If you still choose to backflush as part of a run, be very careful that the flow into the MS does not exceed the limits of the vacuum pump.

1. Verify that all columns are properly configured.
2. Enter all method parameters for the analysis: sampler parameters, inlet parameters, oven temperature profile, detector flows and temperatures, and so forth.
3. Program the oven for the backflush.
   - Include any temperature profile needed for backflush.
   - Set the total run time to include sufficient time for backflush.
4. Program the flow ramp for the column installed between the inlet and the CFT device. After the last analyte elutes or after reaching the last temperature of interest, program a fast ramp with a final flow that is negative.
5. Program the flow ramp for the primary column installed between the CFT device and the detector. Typically hold at the method’s final value for the backflush duration.

If you turned off data acquisition in a data system during backflush, remember to turn it on again at the end of the run.

**Backflushing using a switching valve**

Backflushing is done using a column switching valve controlled by the Run Table. See “Run Time Programming” on page 12.

The valve is plumbed as follows:

- **Position 1** Carrier gas flows through the column to the detector. This is the normal flow path.
• **Position 2** Carrier gas flows through the column toward the inlet, removing components on the column through the inlet vent line.

The Run Table contains commands to perform these actions:

• After the last peak of interest appears, switch the valve to **Position 2**. Higher boiling peaks are discarded through the inlet vent.

• At the same time, turn data acquisition off.

• At the end of the backflush period (determined experimentally), switch the valve to **Position 1** and turn data acquisition on. The system is now ready for the next run.
Nickel Catalyst Tube

About the nickel catalyst tube

The Nickel Catalyst Tube accessory, G4337A, is used for trace analysis of CO and CO$_2$ with a flame ionization detector. The gas sample is separated on the column and passed over a heated catalyst in the presence of hydrogen, which converts the CO and CO$_2$ peaks to CH$_4$.

Nickel catalyst gas flows

For a standard FID installation:

<table>
<thead>
<tr>
<th>Table 9</th>
<th>Gas flows for a standard FID</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Gas</strong></td>
<td><strong>Flow rate, mL/min</strong></td>
</tr>
<tr>
<td>Carrier (helium)</td>
<td>30</td>
</tr>
<tr>
<td>FID hydrogen</td>
<td>30 (see Caution)</td>
</tr>
<tr>
<td>FID air</td>
<td>400</td>
</tr>
</tbody>
</table>
Setting temperatures for the nickel catalyst tube

The nickel catalyst tube is usually mounted in the back inlet position and controlled by the back inlet temperature setpoint. For most analyses, set these temperatures:

- Nickel catalyst tube—375 °C
- FID—400 °C

---

**Table 10**  Gas flows for a TCD/FID series installation

<table>
<thead>
<tr>
<th>Gas</th>
<th>Flow rate, mL/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carrier (helium)</td>
<td>30</td>
</tr>
<tr>
<td>TCD switching flow</td>
<td>25</td>
</tr>
<tr>
<td>FID hydrogen</td>
<td>45 (see Caution)</td>
</tr>
<tr>
<td>FID air</td>
<td>500</td>
</tr>
</tbody>
</table>

**CAUTION**

Hydrogen flow is pressure-controlled, where an FID provides a known resistance. The nickel catalyst tube increases flow resistance, so that the calibration is no longer valid. You must measure hydrogen flow with a bubble or similar meter.

The nickel catalyst can be damaged by exposure to air.
Hydrogen Sensor

The 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. See the Operation Manual for details. 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

By default, the hydrogen sensor current reading (in percent) appears in the GC status display.

If a calibration fails, the GC’s Service Due indicator lights. Press [Service Mode] on the GC keypad. The first line will indicate that the hydrogen sensor calibration failed.
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.
5 Detectors

About Makeup Gas 66
About the FID 67
About the TCD 71
About the µECD 79
About the NPD 85
About the FPD+ 96
About the FPD 102
**About Makeup Gas**

Most detectors use a makeup gas to increase the flow rate through the detector body. This sweeps peaks out of the detector quickly, avoiding mixing of components and loss of resolution. This is particularly important with capillary columns because the column flow rates are so small.

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. If you are operating with *column defined*, you have a choice of two makeup gas modes.

**Constant makeup**  This mode provides a constant flow of makeup gas to the detector.

**Column + makeup = constant**  This mode provides a variable flow of makeup gas to the detector. As column flow increases or decreases, the makeup flow changes to provide a constant combined flow to the detector. If you choose this option, enter a value under *Combined flow*. The *Combined flow* line always displays the same value, while the *Makeup* line changes as the actual makeup flow changes.

**To change the makeup gas flow mode**

1. Press [Front Det] or [Back Det].
2. Scroll to Mode. Press [Mode/Type].
3. Scroll to the correct mode and press [Enter].
About the FID

The FID passes sample and carrier gas from the column through a hydrogen-air flame. The hydrogen-air flame alone creates few ions, but burning an organic compound increases the number of ions produced. A polarizing voltage attracts these ions to a collector located near the flame. The current produced is proportional to the amount of sample being burned. This current is sensed by an electrometer, converted to digital form, and sent to an output device.

The FID uses three supply gases (hydrogen, makeup gas, and air) and two supply lines. Air flows through one supply line, while hydrogen mixed with the makeup gas flows through the other. All three supply gases use:

- A filter frit to protect the flow path and limit the flow rate
- A proportional valve to control the pressure
- A pressure sensor and restrictor to control the valve

The hydrogen and makeup mix outside the flow module and enter the detector at the base of the jet. Air enters above the jet.

\[ \text{PS = Pressure Sensor} \]
How FID units are displayed in Agilent data systems and on the GC

The GC displays the FID signal in picoamperes (pA). The following table lists how different data systems convert the display units to reporting units.

**Table 11  Unit conversions**

<table>
<thead>
<tr>
<th>Data system</th>
<th>Height units</th>
<th>LSV (height units)</th>
<th>Area units</th>
<th>Noise (ASTM)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agilent data system</td>
<td>1 pA</td>
<td>1.3 x 10^-4 pA</td>
<td>1 pA-sec</td>
<td>0.038 pA</td>
</tr>
<tr>
<td>SIGRange 0</td>
<td>1 x 10^-4 pA</td>
<td>1.3 x 10^-4 pA</td>
<td>1 x 10^-4 pA</td>
<td>0.038 pA</td>
</tr>
<tr>
<td>SIGRange 5</td>
<td>3.2 x 10^-3 pA</td>
<td>4.2 x 10^-3 pA</td>
<td>3.2 x 10^-3 pA</td>
<td>0.038 pA</td>
</tr>
<tr>
<td>Analog 1V†</td>
<td>1.25 x 10^-4 pA</td>
<td>device dependent</td>
<td>1.25 x 10^-4 pA</td>
<td>0.038 pA</td>
</tr>
</tbody>
</table>

* Noise is recommended maximum when determining MDL.
† SIGRange used with 3393 and 3396 integrators.
‡ Analog 1V is an approximate value.

To light the FID flame

Press [Front Det] or [Back Det], scroll to Flame, then press [On/Yes].

To extinguish the FID flame

Press [Front Det] or [Back Det], scroll to Flame, then press [Off/No].

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. Press [Config][Front Det] or [Config][Back Det].
2. Scroll to Lit offset.
3. Enter the new value and press [Enter].

**Recommended starting conditions for new FID methods**

See Table 12 for guidelines and rules to select initial detector settings for new methods.

**Table 12  Recommended starting conditions**

<table>
<thead>
<tr>
<th>Combustible gas mix</th>
<th>Make sure that the final hydrogen-to-air ratio is between 8% and 12%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detector temperature</td>
<td>Set to 20 °C above the highest oven temperature, depending on the column type.</td>
</tr>
<tr>
<td></td>
<td>A temperature of 300 °C provides a good starting point and easier ignition, and minimizes water condensation.</td>
</tr>
<tr>
<td></td>
<td>The GC will not attempt to ignite the flame at a temperature &lt;150 °C.</td>
</tr>
<tr>
<td>Carrier gas flow (hydrogen, helium, nitrogen)</td>
<td></td>
</tr>
<tr>
<td>Packed columns</td>
<td>Suggest 10 to 60 mL/min</td>
</tr>
<tr>
<td>Capillary columns</td>
<td>Suggest 1 to 5 mL/min</td>
</tr>
<tr>
<td>Detector gases</td>
<td>Flow range mL/min</td>
</tr>
<tr>
<td>Standard installation</td>
<td></td>
</tr>
<tr>
<td>Column plus capillary makeup</td>
<td>10 to 60</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>24 to 60</td>
</tr>
<tr>
<td>Air</td>
<td>200 to 600</td>
</tr>
<tr>
<td>With Nickel Catalyst Accessory: Standard installation</td>
<td></td>
</tr>
<tr>
<td>Column plus capillary makeup</td>
<td>10 to 60</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>24 to 60</td>
</tr>
<tr>
<td>Air</td>
<td>200 to 600</td>
</tr>
</tbody>
</table>
Verifying the installation of the FID column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

To set the FID parameters:

1. Verify:
   - Makeup gas is configured
   - Installed jet type is correct for column type
3. Set the detector temperature. The temperature must be greater than 150 °C for the flame to light.
4. Set the hydrogen flow rate, if desired, and press [Off/No].
5. Change the air flow rate, if desired, and press [Off/No].
6. If using a packed column, set the FID makeup gas to 0.0/Off.
7. If using a defined capillary column, set the makeup gas flow or combined column plus makeup gas flow.
8. Scroll to Flame and press [On/Yes]. This turns on the air and hydrogen and initiates the ignition sequence. The signal typically increases to 5 to 20 pA after ignition.

Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the collector exit. Steady condensation indicates that the flame is lit.
About the TCD

The TCD compares the thermal conductivities of two gas flows—pure carrier gas (the reference gas) and carrier gas plus sample components (the column effluent).

This detector contains a filament that is heated electrically so that it is hotter than the detector body. The filament temperature is held constant while alternate streams of reference gas and column effluent pass over it. When a sample component appears in the effluent, the power required to keep the filament temperature constant changes. The two gas streams are switched over the filament five times per second (hence the ticking sound) and the power differences are measured and recorded.

When helium (or hydrogen) is used as carrier gas, the sample causes the thermal conductivity to fall. If nitrogen is used, the thermal conductivity usually goes up because most things are more conductive than nitrogen.

Because the TCD does not destroy the sample during the detection process, this detector can be connected in series to a flame ionization detector or other detector.
Column effluent is forced away from the filament. TCD measures reference gas.

Column effluent is forced toward the filament. TCD measures peaks (if present).
TCD pneumatics

The figure below shows the pneumatics design of the TCD.

![TCD pneumatics diagram]

PS = Pressure Sensor

TCD carrier, reference, and makeup gas

Reference and makeup gas must be the same as the carrier gas, and the gas type must be specified in both the inlet and detector parameter lists.

When using packed columns, we recommend a small makeup gas flow (2 to 3 mL/min) to get the best peak shapes.

Use the next figure to select a value for reference gas flow for either capillary or packed columns. Any ratio within ±0.25 of that in the figure is suitable.
TCD gas pressures

Choose a flow, find a pressure, set source pressure 10 psi (70 kPa) higher.
Selecting reference and makeup flows for the TCD

Table 13  Recommended flow rates and temperatures

<table>
<thead>
<tr>
<th>Gas type</th>
<th>Flow range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carrier gas (hydrogen, helium, nitrogen)</td>
<td>Packed, 10 to 60 mL/min</td>
</tr>
<tr>
<td></td>
<td>Capillary, 1 to 5 mL/min</td>
</tr>
<tr>
<td>Reference (same gas type as carrier)</td>
<td>15 to 60 mL/min</td>
</tr>
<tr>
<td></td>
<td>See the figures to select a value.</td>
</tr>
<tr>
<td>Capillary makeup (same gas type as carrier)</td>
<td>5 to 15 mL/min—capillary columns</td>
</tr>
<tr>
<td></td>
<td>2 to 3 mL/min—packed columns</td>
</tr>
</tbody>
</table>

Detector temperature

<150 °C, cannot turn on filament
Detector temperature should be 30 °C to 50 °C greater than highest oven ramp temperature.

Sample components with higher thermal conductivities than the carrier gas produce negative peaks. For example, helium or hydrogen form a negative peak with nitrogen or argon-methane as the carrier gas.

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 filament 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 “Run Time Programming” on page 12.

**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.

**Setting parameters for the TCD**

1. Press [Front Det] or [Back Det].
2. Set the detector temperature. Do not set higher than the maximum temperature allowed for the column because part of the column passes through the heated block and into the cell.
3 Verify that makeup gas type is the same as that plumbed to your instrument (next to Makeup line in the parameter list). Change the gas type, if necessary.

4 Set the reference gas flow rate.

5 If you are using packed columns, turn off the makeup gas (or proceed to step 6 and enter 2 to 3 mL/min, see “TCD carrier, reference, and makeup gas” on page 73) and proceed to step 7

6 If you are using capillary columns: choose a flow mode and set the makeup gas flow or combined flow.

7 Turn on the filament. Allow about 30 minutes for thermal stabilization. A longer period may be needed for the highest sensitivity.

8 If necessary, turn **Negative polarity [On/Yes]** to invert negative-going peaks. When a sample contains components giving both positive- and negative-going peaks, **Negative polarity** can be switched on and off during a run as a timetable event.

**Example: Packed mode (packed and large capillary columns)**

Column flow is 15 to 60 mL/min. Set the reference flow to 1.5 times the sum of column flow + makeup flow.

Makeup gas is recommended with all capillary columns. It allows the column to be inserted all the way into the detector and withdrawn 1 mm. If makeup is not used, the column must be no more than 3 mm above the ferrule. Minimum makeup flow is 1 mL/min.

**1/8-inch stainless steel column** If column flow is 30 mL/min, set the reference flow to $30 \times 1.5 = 45$ mL/min. Total detector flow is $30 + 45 = 75$ mL/min.

**10 m × 0.53 mm column** If column flow is 15 mL/min and makeup flow = 2 mL/min, set the reference flow to $1.5 \times (15 + 2) = 25.5$ mL/min. Total detector flow is $17 + 25.5 = 42.5$ mL/min.

**Example: Capillary mode (small capillary columns)**

If combined column plus makeup flow is between 5 and 10 mL/min, set the reference flow at 3× the combined flow. For a combined flow between 10 and 15 mL/min, use a multiplier of 2. This will bring the TCD within 25% of the maximum response. For further optimization, adjust the reference flow.
**2 m × 0.2 mm capillary column**  If column flow is 0.75 mL/min, the makeup must be at least 4.25 mL/min. Set it = 5. Reference flow will then be $3 \times 5.75 = 17.25$ mL/min. Total detector flow = $5.75 + 17.25 = 22.5$ mL/min.

**25 m × 0.32 mm capillary column**  If column flow = 10 mL/min, set makeup low to minimize sample dilution. Set it = 2 mL/min. Reference flow will then be $12 \times 2 = 24$ mL/min. Total detector flow = $12 + 24 = 36$ mL/min.
About the µECD

The micro-cell detector (µECD) contains a cell plated with $^{63}\text{Ni}$, a radioactive isotope. The $^{63}\text{Ni}$ releases $\beta$ particles that collide with carrier gas molecules to produce low-energy electrons—each $\beta$ particle produces approximately 100 electrons. The free electrons produce a small current—called the reference or standing current—that is collected and measured in a pulsed circuit.

When a sample component molecule comes into contact with the free electrons, the electrons may be captured by the sample molecules to create negatively charged ions. The voltage across the cell electrodes is pulsed to collect the remaining free electrons while the heavier ions are relatively unaffected and swept out the vent with the carrier gas flow.

Cell current is measured and compared to a reference current. The pulse rate is adjusted to maintain a constant cell current. The more uncaptured electrons, the lower the pulse frequency required to match the reference current. When a component that captures electrons passes through the cell, the pulse rate rises. This pulse rate is converted to a voltage and recorded.

µECD safety and regulatory information

The $^{63}\text{Ni}$ isotope

The radioactive isotope used in the cell is $^{63}\text{Ni}$. It is plated onto the inner surface of the cell body and is solid at temperatures used in chromatography. Some other properties are listed below.

<table>
<thead>
<tr>
<th>Table 14</th>
<th>Properties of $^{63}\text{Ni}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Property</td>
<td>Value</td>
</tr>
<tr>
<td>Half–life:</td>
<td>101.1 years</td>
</tr>
<tr>
<td>Emission:</td>
<td>65.87 keV max., beta radiation</td>
</tr>
<tr>
<td>Melting point:</td>
<td>1453 °C</td>
</tr>
<tr>
<td>Dimensions of the active part of the µECD:</td>
<td>Inside diameter: 6 mm</td>
</tr>
<tr>
<td></td>
<td>Height: 4.2 mm</td>
</tr>
<tr>
<td>Total activity (µECD cell):</td>
<td>555 MBq (15 millicuries) maximum</td>
</tr>
</tbody>
</table>

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µECD licenses

Customers in the United states can purchase an exempt model µECD. Customers outside the United States should contact their local Agilent sales office for information.

µECD warnings

Although beta particles at this energy level have little penetrating power—the surface layer of the skin or a few sheets of paper will stop most of them—they may be hazardous if the isotope is ingested or inhaled. For this reason the cell must be handled with care: Radioactive leak tests must be performed at the required intervals (not applicable to exempt models), the inlet and outlet fittings must be capped when the detector is not in use, corrosive chemicals must not be introduced into the detector, and the effluent from the detector must be vented outside the laboratory environment.

**WARNING**

Materials that may react with the $^{63}$Ni source, either to form volatile products or to cause physical degradation of the plated film, must be avoided. These materials include oxidizing compounds, acids, wet halogens, wet nitric acid, ammonium hydroxide, hydrogen sulfide, PCBs, and carbon monoxide. This list is not exhaustive but indicates the kinds of compounds that may cause damage to $^{63}$Ni detectors.

**WARNING**

In the extremely unlikely event that both the oven and the detector heated zone should go into thermal runaway (maximum, uncontrolled heating in excess of 400 °C) at the same time, and that the detector remains exposed to this condition for more than 12 hours, take the following steps:

- After turning off the main power and allowing the instrument to cool, cap the detector inlet and exhaust vent openings. Wear disposable plastic gloves and observe normal laboratory safety precautions.
- Return the cell for exchange, following directions included with the License Verification Form (part no. 19233-90750).
- Include a letter stating the condition of abuse.
It is unlikely, even in this very unusual situation, that radioactive material will escape the cell. However, permanent damage to the $^{63}$Ni plating within the cell is possible, and therefore, the cell must be returned for exchange.

**WARNING**

Do not use solvents to clean the µECD.

**WARNING**

You may not open the µECD cell unless authorized to do so by your local nuclear regulatory agency. Do not disturb the four socket-head bolts. These hold the cell halves together. United States customers removing or disturbing them is a violation of the terms of the exemption and could create a safety hazard.

---

- Never eat, drink, or smoke when handling µECDs.
- Always wear safety glasses when working with or near open µECDs.
- Wear protective clothing such as laboratory jackets, safety glasses, and gloves, and follow good laboratory practices. Wash hands thoroughly with a mild non-abrasive cleaner after handling µECDs.
- Cap the inlet and outlet fittings when the µECD is not in use.
- Connect the µECD exhaust vent to a fume hood or vent it to the outside. See the latest revision of title 10, Code of Federal Regulations, part 20, (including appendix B) or the applicable State regulation. For other countries, consult with the appropriate agency for equivalent requirements.

Agilent Technologies recommends a vent line inside diameter of 6 mm (1/4 inch) or greater. With a line of this diameter, the length is not critical.
The µECD 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.

**µECD detector gas**

The µECD operates with either nitrogen or argon/methane as the makeup and anode gas. Purity is critical; gases must exceed 99.9995% purity.

Because of the high detector sensitivity, carrier and makeup gas must be dry and oxygen-free. Moisture, chemical, and oxygen traps in good condition should be installed in carrier and makeup gas supply lines. Do not use plastic (including PTFE) tubing, plastic-bodied traps, or O-ring seals.
**µECD temperature**

To prevent peak tailing and to keep the cell clean, the detector temperature should be set higher than the highest oven temperature used—the setpoint should be based on the elution temperature of the last compound. If you operate at excessively high temperatures, your results will not necessarily improve and you may increase sample and column decomposition.

**µECD analog output**

If you intend to use the analog output from the µECD, you must set the output Range to 10.

1. Press [Analog Out 1] or [Analog Out 2].
2. Scroll to Range.
3. Type 10 and press [Enter].

**Recommended starting conditions for new µECD 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 15**  
Starting values

<table>
<thead>
<tr>
<th>Gas</th>
<th>Recommended flow range</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Carrier gas</strong></td>
<td></td>
</tr>
<tr>
<td>Packed columns</td>
<td>30 to 60 mL/min</td>
</tr>
<tr>
<td>(nitrogen or argon-methane)</td>
<td></td>
</tr>
<tr>
<td>Capillary columns</td>
<td>0.1 to 20 mL/min, depending on diameter</td>
</tr>
<tr>
<td>(hydrogen, nitrogen, or argon-methane)</td>
<td></td>
</tr>
<tr>
<td><strong>Capillary makeup</strong></td>
<td>10 to 150 mL/min</td>
</tr>
<tr>
<td>(nitrogen or argon-methane)</td>
<td></td>
</tr>
<tr>
<td><strong>Temperature</strong></td>
<td></td>
</tr>
<tr>
<td>250 °C to 400 °C</td>
<td>Detector temperature is typically set 25 °C greater than the highest oven ramp temperature.</td>
</tr>
</tbody>
</table>

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

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

**µECD temperature programming**

The µECD 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.

**Setting parameters for the µECD**

Verify that your detector gases are connected, a column is properly installed, and the system is free of leaks. Set the oven temperature and the inlet temperature and flow. Make sure your carrier gas type is the same as that plumbed to your GC.

1. Press [Front Det] or [Back Det].
2. Set the detector temperature. To keep the µECD cell clean, this temperature must be higher than the oven temperature.
3. Verify that the makeup gas type is the same as that plumbed to your instrument. The gas type is in parentheses next to the **Makeup** line on the parameter list. Change the gas type, if necessary.
4. Enter a value for the makeup gas flow.
   - If you are using **packed columns**, turn off the makeup gas.
   - If your **capillary column** is defined, choose a flow mode and set the makeup or combined gas flow.
   - If your **capillary column** is not defined, only constant makeup flow is available. Enter a makeup gas flow.
About the NPD

We strongly recommend that you allow the firmware to perform Auto Adjust and set the Bead Voltage.

NPD flows and general information

The NPD passes sample and carrier through a hydrogen/air plasma. A heated ceramic or glass source, called the bead, is just above the jet. The low hydrogen/air ratio cannot sustain a flame, minimizing hydrocarbon ionization, while the alkali ions on the bead surface facilitate ionization of nitrogen- or phosphorus-organic compounds. The output current is proportional to the number of ions collected. It is sensed by an electrometer, converted to digital form, and sent to an output device.
NPD flow, temperature, and bead recommendations

Table 16  General operating values

<table>
<thead>
<tr>
<th>Gas or Setting</th>
<th>Recommendation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Carrier gas</strong> (helium, hydrogen, nitrogen)</td>
<td>Capillary, choose optimum flow based on column dimensions.</td>
</tr>
<tr>
<td><strong>Detector gases</strong></td>
<td></td>
</tr>
<tr>
<td>Hydrogen</td>
<td>Ceramic bead</td>
</tr>
<tr>
<td></td>
<td>2 to 5 mL/min</td>
</tr>
<tr>
<td>Air</td>
<td>60 mL/min</td>
</tr>
<tr>
<td>Capillary makeup (helium, nitrogen)</td>
<td>Ceramic bead</td>
</tr>
<tr>
<td></td>
<td>Nitrogen: 5 to 10 mL/min</td>
</tr>
<tr>
<td></td>
<td>Helium: less than 5 mL/min</td>
</tr>
</tbody>
</table>

**Temperature**
Default is 250 °C; operating range is 150 °C to 400 °C.
- <150 °C, the Adjust offset process will not start.
- 325 to 335 °C is recommended.
- Detector temperature should be greater than the highest oven temperature. With higher detector temperatures, less bead heating voltage is required.

**Adjust offset**
Default is 30 pA, suggested operating range is 20 to 40 pA, and allowable range is 0 to 99.9 pA.
- ≥ 50 pA increases sensitivity but reduces bead life.
- Lower settings reduce sensitivity and increase bead life, but settings too low will result in solvent quenching.
- The time required for Adjust offset depends on the bead type and condition.

**Bead voltage**
Ceramic bead. Range is 0 to 4.095 V.
- Use Auto Adjust On, Dry Bead, and let the GC set the Bead Voltage for you.

**Source gas pressures**
Choose a flow, find a pressure, and set source pressure 10 psi (70 kPa) higher.
Temperature programming

The NPD 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.

NPD required gas purity

Because of its high sensitivity, the NPD requires very pure (at least 99.9995%) gases. We strongly recommend that moisture and organics traps be used on the carrier gas and all detector
gases, including the detector hydrogen, air, and makeup gases. Do not use plastic (including PTFE) tubing, plastic-bodied traps, or O-ring seals.

Setting parameters for the NPD

Before operating the NPD, make sure that detector gases are connected, a column is installed, and the system is free of leaks. Set the oven temperature, inlet temperature, and column flow.

WARNING Make sure that a column is installed or the NPD column fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

1. Select the bead (white ceramic, black ceramic).
2. Select the jet.
3. Install bead and jet as required. (See the Maintenance Manual for details.)
4. Press [Config][Front Det] or [Config][Back Det].
5. If you are using makeup gas, verify that the configured makeup gas type is the same as that plumbed to your instrument. Change the gas type, if necessary. Nitrogen is recommended.
6. If the displayed Bead Type is incorrect, set the Bead Type using the [Mode/Type] key.
7. Set Auto Adjust (On recommended).
8. Set Dry Bead (On recommended).
10. Set the detector temperature. The recommended range is 325 to 335 °C.
11. Enter a hydrogen flow (3.0 mL/min is recommended). Turn the flow On.
12. Enter an air flow (60 is recommended for ceramic beads). Turn the flow On.
   a. If you are using packed columns, turn off makeup gas and proceed to step 13.
   b. If your capillary column is defined, choose a flow mode and set the makeup gas flow. For a column in the constant
flow mode, choose **Constant makeup**. For a column in the constant pressure mode, choose **Column + makeup = constant**.

c  If your column is *not defined*, enter a makeup gas flow. Only constant flow is available.

13  Monitor the offset adjustment process.

  a  If **Auto Adjust** is **On**, the adjust offset process starts automatically when the detector reaches setpoint. If **Auto Adjust** is **Off**, the Bead Voltage will gradually go to the last setpoint after the bead reaches setpoint temperature and the Dry Bead time has elapsed.

  b  If you need to set a new target offset, enter an **Adjust offset** value. Adjust offset starts when the detector reaches setpoint.

  c  If **Auto Adjust** is **Off**, you can manually start the Adjust offset process by scrolling to **Adjust offset**, then pressing **[On/Yes]**.

  d  If your standard operating procedures require that you set the bead voltage directly, see “Setting NPD bead voltage manually (optional)” on page 94.

**Selecting an NPD bead type**

Two beads are available:

<table>
<thead>
<tr>
<th>Table 17</th>
<th>NPD beads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bead type</td>
<td>Part number</td>
</tr>
<tr>
<td>White ceramic</td>
<td>G1534-60570</td>
</tr>
<tr>
<td>Black ceramic</td>
<td>5183-2007</td>
</tr>
</tbody>
</table>

![Ceramic beads](image)
Selecting an NPD jet

Open the oven door and locate the column connection fitting at the base of the detector. It will look like either a capillary optimized fitting or an adaptable fitting.

- If you have an application that tends to clog the jet, select a jet with a wider tip id.
- When using packed columns in high column-bleed applications, the jet tends to clog with silicon dioxide.

For capillary optimized fittings, select one of the following from Table 18.

### Table 18  Jets for capillary optimized fittings

<table>
<thead>
<tr>
<th>Figure 3 ID</th>
<th>Jet type</th>
<th>Part number</th>
<th>Jet tip id</th>
<th>Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Capillary with extended jet (recommended)</td>
<td>G1534-80580</td>
<td>0.29 mm (0.011 inch)</td>
<td>51.5 mm</td>
</tr>
<tr>
<td>2</td>
<td>Capillary</td>
<td>G1531-80560</td>
<td>0.29 mm (0.011 inch)</td>
<td>43 mm</td>
</tr>
<tr>
<td>3</td>
<td>High-temperature</td>
<td>G1531-80620</td>
<td>0.47 mm (0.018 inch)</td>
<td>43 mm</td>
</tr>
</tbody>
</table>

For the adjustable NPD, select one of the following from Table 19.
To configure the NPD

In addition to the Ignore Ready and Makeup gas type, the NPD requires the following configuration settings. Scroll to each and enable/disable using [On/Yes] or [Off/No].

**Auto Adjust Bead**  Recommended On. When On, the automatic adjust offset process starts when the bead reaches the temperature setpoint after having been turned off or cooled below 150 °C. Auto adjust starts after Dry Bead hold time, if enabled. Auto Adjust Bead uses the adjust offset feature to protect the bead—especially new beads—by making sure that the desired offset is obtained with the lowest possible bead voltage. When Off, the bead voltage will rise as soon as the Dry Bead time elapses, or as soon as the temperature setpoint is reached if Dry Bead is off.

**Dry Bead**  Recommended On. When On, the bead temperature holds at 150 °C for 5 minutes before continuing to the setpoint. This allows any condensation to evaporate and be swept out of the detector.
**Maximum Bead Voltage**  Display only. Shows the current maximum bead voltage for the configured bead type (4.095 V for ceramic beads).

**Automatically adjusting NPD bead voltage**

Agilent recommends using the Adjust offset feature to automatically determine the lowest bead voltage needed to give the desired response.

When the detector is turned on, the temperature rises at a controlled rate.

- If Dry Bead is On, temperature holds at 150 °C for 5 minutes to drive off moisture, then continues to the setpoint.
- If Dry Bead is Off, the temperature rises directly to the setpoint.

When the temperature reaches the setpoint, the Bead Voltage gradually rises until it produces the desired output.

**Adjust offset**

When you enter a value here, or press [On/Yes] to use the stored value, detector gas flows turn on, the bead heats, and the bead voltage adjusts until Output is stable and equal to the entered value. There are five stages of Adjust offset.

**Detector off**  When the detector is off, Adjust offset and Bead voltage are Off and initial Output is displayed.

**Detector on—detector temperature less than 150 °C.**  When you enter an Adjust offset value or press [On], detector gases turn on and the display blinks the Temp not ready message.

**Detector on—waiting for oven and/or detector to reach temperature setpoint and equilibrium.**  If the oven or detector is not at setpoint, the display continues to blink the Temp not ready message.

**Detector on—Dry Bead On**  If Dry Bead is On, the temperature rise holds at 150 °C for 5 minutes to remove moisture, then continues to the setpoint.

**Detector on—during adjust offset.**  When the detector and oven temperatures reach setpoint and equilibrate, the Adjust offset process begins. The bead voltage is slowly increased until the output is close to the Adjust offset value. The display blinks Detector Slewing.
Detector on and ready. When the Adjust offset value is reached, the Adjust offset line reads Done and displays the offset target setpoint. Your detector is on and ready. The display shows the actual Bead voltage.

Setting NPD adjust offset on the clock table

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

Aborting NPD adjust offset

Press [Delete] with the cursor on the Adjust offset line. This cancels the adjustment without turning off the detector gases and bead voltage.

Extending the NPD bead life

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

- Use the lowest practical Adjust offset value. This will result in a lower Bead Voltage 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 “Run Time Programming” on page 12.

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.
Turning off the detector

**CAUTION** If you turn **Adjust offset** [Off] at any time, the bead voltage, hydrogen, and air flows all turn off.

Setting the initial bead voltage for new beads

Before you turn on the bead for the first time, manually set its voltage to a safe value so that the new bead is not destroyed.

1. Make sure **Adjust Offset** is turned **Off**.
2. After the temperature stabilizes at setpoint, set the initial Bead Voltage, depending on bead type:
   - Ceramic bead (white or black): 0.0 V to 2.0 V

Setting NPD bead voltage manually (optional)

**Bead voltage** shows the voltage used to heat the bead. It can be a value derived from the **Adjust offset** value, or can be entered as a setpoint. Entering a setpoint causes the voltage to change at 13 mV/second until it reaches the setpoint provided that
- the detector is at the temperature setpoint
- temperature is at least 150 °C
- gas flows are on
- **Dry Bead** time, if **On**, has elapsed

**Bead voltage** is also useful for small adjustments between runs. If you observe a baseline drift, you can enter a small, one-time change to compensate for the drift.

If you are not using the recommended Adjust offset process, note that large voltage jumps reduce bead life. Use increments no greater than 0.05 V, spaced 10 seconds apart, until you reach the desired offset.

**New beads**

After a new bead reaches the initial voltage, begin to increase the voltage value in 0.05 V increments until the bead ignites. Wait about 10 seconds between each voltage adjustment. Monitor the detector output. When the bead ignites, the output will rise suddenly, then decrease towards a more stable value. It is best to allow the NPD to remain in this state without further adjustment for about 24 hours. Then you may adjust the bead voltage in small increments (0.05 to 0.1 V) until reaching the
desired offset. With a clean environment, clean gas supplies, and low bleed column, a typical offset may decrease 6-12 pA during a 24 hour period.

Typical voltages for new ceramic beads range from 2.5 to 3.7 volts. Higher values reduce bead life.
About the 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.

Figure 4  FPD$^+$ gas flows

The current from the PMT is amplified and digitized by the FPD$^+$ electronics board. The signal is available either as a digital signal on the communications output or as a voltage signal on the analog output.

The FPD$^+$ should not be stored at temperatures above 50 °C, based on the original manufacturer’s specifications for the PMT.
**FPD+ gas flows**

The hydrogen fuel gas and makeup gas mix at the EPC module, and enter the bottom of the detector where the column enters the transfer line. See Figure 4. This gas mixture eliminates any dead volume near the column entrance before mixing with air in the emission block for combustion.

A second flow of makeup gas is used to sweep the volume between the thin-walled tube (that carries the column sample stream) and the transfer line walls. This gas flow also enters the emission block but is separate from the sample stream. The purge flow rate is not measured directly, but is controlled by a fixed restrictor and the makeup flow setting. A makeup flow setting of 60 mL/min will result in about 12 mL/min of purge flow.

The total flow from the detector is:

\[
\text{column flow} + \text{makeup flow} + \text{purge flow} = \text{total flow}
\]

However, the total flow that carries the sample is equal to just the column flow plus the makeup flow.

**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.

**FPD+ Lit Offset**

The default **Lit Offset** is 2.0 pA.

**FPD+ photomultiplier protection**

The PMT is extremely sensitive to light. Always turn the **PMT voltage** off (which turns off the high voltage to the PMT) before removing the PMT housing or opening the emissions chamber. Failing to do this can destroy the PMT.

Even with the **PMT voltage** off, protect the PMT from room light. Cap the housing when removed, place it end down to exclude light, reduce room light level before exposing the PMT, and so
on. A brief exposure (always with the **PMT voltage** turned off) will not damage it but prolonged exposure will cause a gradual loss of sensitivity.

**FPD⁺ optical filters**

The filters are marked on the edge with the transmission wavelength. Each filter has a small arrow or triangle on its side which must point toward the PMT when installed.

The sulfur filter is silvery on both sides and transmits at 393 nanometers.

The phosphorus filter is yellow/green and transmits at 525 nanometers.

**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 with splitless injection, use liner 5181-3316.

**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.
FPD$^+$ gas purity

High-purity gases have a lower sulfur content. Standard purity gases have a higher sulfur content which impairs sulfur detection in the compound being studied. Instrument or Chromatographic grades work well.

Agilent recommends using helium carrier, nitrogen makeup gas, and air with 99.9995% purity or better. Use carbon, oxygen, and moisture traps. Select traps to remove sulfur compounds from detector air and nitrogen gases. A helium getter is also recommended. See the GC, GC/MS, and ALS Site Preparation Guide and the Agilent web site at http://www.agilent.com for more information.

FPD$^+$ gas flows

Table 20 gives the flows for the maximum sensitivity FPD$^+$ flame, which is hydrogen-rich and oxygen-poor.

<table>
<thead>
<tr>
<th>Table 20</th>
<th>Recommended flows</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>mL/min</strong></td>
<td>Carrier (hydrogen, helium, nitrogen, argon)</td>
</tr>
<tr>
<td>Packed columns</td>
<td>10 to 60</td>
</tr>
<tr>
<td>Capillary columns</td>
<td>1 to 5</td>
</tr>
<tr>
<td><strong>mL/min</strong></td>
<td>Detector gases</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>60</td>
</tr>
<tr>
<td>Air</td>
<td>60</td>
</tr>
<tr>
<td>Carrier + makeup</td>
<td>60</td>
</tr>
</tbody>
</table>

Helium, either as carrier or makeup gas, may cool the detector gases below the ignition temperature. We recommend using nitrogen rather than helium.

Lighting the FPD$^+$ flame

Before trying to light the flame, heat the detector to operating temperature and let it stabilize.
During the ignition sequence, the detector does the following:

1. Until the detector temperature zones reach setpoint, the GC purges the detector with all gas flows.
2. Once the detector stabilizes at its thermal setpoints, the GC turns off hydrogen and makeup gas flows. Carrier and air flows remain on.
3. Sets air flow to 500 mL/min.
4. Turns the glow plug ignitor on.
5. Ramps the hydrogen flow from 10 to 70 mL/min.
6. Resets the air flow to the air flow setpoint.
7. Resets the hydrogen flow to the hydrogen flow setpoint.
8. Turns the makeup gas on.
9. Compares the signal change with the Lit offset value. If the change is greater than Lit offset, declares the flame on (lit). If it is less, declares the flame off (not lit).

For this process to work, there must be enough air pressure to the pneumatics module to provide up to 500 mL/min flow. We recommend a supply pressure of at least 60 psi. See the Installation and First Startup manual.

**Manual ignition**

1. Press [Front Det] or [Back Det].

**Automatic ignition**

If the FPD⁺ output with the flame on falls below the flame-off output plus the Lit offset value, this is interpreted as a flame-out condition. The FPD⁺ runs the flame ignition sequence to relight the flame. If this fails, it runs the sequence again. If the second attempt also fails, the detector shuts down all functions except temperature and makeup gas flow.

**Setting parameters for the FPD⁺**

---

**WARNING** Verify that a column is installed or the FPD⁺ fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.
1  Press [Front det] or [Back det].

2  Set the detector temperature. Scroll to Temperature and input the desired value, and press [Enter]. It must be greater than 120 °C for the flame to light.

3  Scroll to H2 flow and change the hydrogen flow rate, if desired. Press [Enter]. If the flame is not lit, press [Off/No] to turn off the flow. (It is not good practice to pass uncombusted hydrogen into the air. The hydrogen flow will be turned on automatically when igniting the flame.)

4  Scroll to Air flow and change the air flow rate, if desired. Press [Off/No] to turn off the flow for now.

5  If you are using packed columns, turn off the makeup gas and proceed to step 9.

6  If you are using capillary columns, scroll to the Makeup parameter:

   a  Verify that makeup gas type is the same as that plumbed to your instrument. The configured gas type is shown next to Makeup in the parameter list. Change the gas type, if necessary.

   b  If your capillary column is defined, choose a flow mode and set the makeup gas flow or combined flow.

   c  If your capillary column is not defined, enter a makeup gas flow. Only constant flow is available.

7  Scroll to PMT voltage and make sure it is turned on.

8  Optionally, set the emission block temperature. (Normally, the default temperature of 150 °C is sufficient for most applications.) Scroll to Emission Block, input the desired temperature, and press [Enter]. See “FPD+ temperature considerations” on page 98 for more information.

9  Scroll to Flame and press [On/Yes]. This starts the automatic ignition sequence (see “Lighting the FPD+ flame” on page 99).

   On successful ignition, the signal increases. Typical signal output increases are 4 to 40 pA in sulfur mode, and 10 to 70 pA in phosphorus mode. You can also verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the vent exit. Steady condensation indicates that the flame is lit.
About the 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.

The current from the PMT is amplified and digitized by the FPD electronics board. The signal is available either as a digital signal on the communications output or as a voltage signal on the analog output.

The FPD should not be stored at temperatures above 50 °C, based on the original manufacturer’s specifications for the PMT.
**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.

**FPD Lit Offset**

The default Lit Offset is 2.0 pA.

**Starting Up and Shutting Down the FPD**

The FPD creates a great deal of water vapor when the flame is on. This could condense in the vent tube on top of the detector and drop onto the flame, possibly extinguishing it. To avoid this, turn the heaters on, wait 20 minutes for the vent to heat up, and then ignite the flame. Water vapor will now make it over the top of the vent tube before condensing.

For similar reasons, extinguish the flame before turning the heaters off.

**FPD photomultiplier protection**

The PMT is extremely sensitive to light. Always turn the PMT voltage off (which turns off the high voltage to the PMT) before removing the PMT housing or opening the emissions chamber. Failing to do this can destroy the PMT.

Even with the PMT voltage off, protect the PMT from room light. Cap the housing when removed, place it end down to exclude light, reduce room light level before exposing the PMT, and so on. A brief exposure (always with the PMT voltage turned off) will not damage it but prolonged exposure will cause a gradual loss of sensitivity.

**FPD optical filters**

The filters are marked on the edge with the transmission wavelength. Each filter has a small arrow on its side which must point toward the PMT when installed.

The sulfur filter is silvery on both sides and transmits at 393 nanometers.
The phosphorus filter is yellow/green and transmits at 525 nanometers.

**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 with splitless injection, use liner 5181-3316.

**FPD temperature considerations**

The minimum detector temperature to prevent water condensation is 120 °C. We recommend a temperature that is 25 °C higher than the highest column temperature, but no higher than 250 °C.

**FPD gas purity**

High-purity gases have a lower sulfur content. Standard purity gases have a higher sulfur content which impairs sulfur detection in the compound being studied. Instrument or Chromatographic grades work well.

Agilent recommends using helium carrier, nitrogen makeup gas, and air with 99.9995% purity or better. Use carbon, oxygen, and moisture traps. Select traps to remove sulfur compounds from detector air and nitrogen gases. A helium getter is also recommended. See the GC, GC/MS, and ALS Site Preparation Guide for and the Agilent web site at http://www.agilent.com for more information.

**FPD gas flows**

*Table 20* gives the flows for the maximum sensitivity FPD flame, which is hydrogen-rich and oxygen-poor.

**Table 21**  
Recommended flows

<table>
<thead>
<tr>
<th>Carrier (hydrogen, helium, nitrogen, argon)</th>
<th>Sulfur mode flows, mL/min</th>
<th>Phosphorus mode flows, mL/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Packed columns</td>
<td>10 to 60</td>
<td>10 to 60</td>
</tr>
<tr>
<td>Capillary columns</td>
<td>1 to 5</td>
<td>1 to 5</td>
</tr>
</tbody>
</table>
Helium, either as carrier or makeup gas, may cool the detector gases below the ignition temperature. We recommend using nitrogen rather than helium.

**Lighting the FPD flame**

Before trying to light the flame, have the detector at operating temperature. Removing the condensate tubing may help, but be sure to replace it before making runs.

It is difficult to light the flame with the flows shown in Table 20, particularly in the sulfur mode. If the flame will not light with the sulfur mode flows shown, change to the phosphorus mode flows. After ignition, gradually reduce the flows to the sulfur values. Some experimentation will be needed.

When either of the flame ignition methods in this section is used, the FPD automatically performs this sequence:

1. Turns all detector gases—air, hydrogen, makeup—off. Carrier remains on.
2. Sets air flow to 200 mL/min.
3. Turns the glow plug ignitor on.
4. Ramps the hydrogen flow from 10 to 70 mL/min.
5. Resets the air flow to the air flow setpoint.
6. Resets the hydrogen flow to the hydrogen flow setpoint.
7. Turns the makeup gas on.
8. Compares the signal change with the Lit offset value. If the change is greater than Lit offset, declares the flame on (lit). If it is less, declares the flame off (not lit).

For this process to work, there must be enough air pressure to the pneumatics module to provide 200 mL/min flow. We recommend a supply pressure of 90 psi.

---

**Table 21** Recommended flows (continued)

<table>
<thead>
<tr>
<th>Detector gases</th>
<th>Sulfur mode flows, mL/min</th>
<th>Phosphorus mode flows, mL/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>50</td>
<td>75</td>
</tr>
<tr>
<td>Air</td>
<td>60</td>
<td>100</td>
</tr>
<tr>
<td>Carrier + makeup</td>
<td>60</td>
<td>60</td>
</tr>
</tbody>
</table>

---
Manual ignition

1  Press [Front Det] or [Back Det].
2  Scroll to Flame. Press [On/Yes]. The flame ignition sequence begins.

Automatic ignition

If the FPD output with the flame on falls below the flame-off output plus the Lit offset value, this is interpreted as a flame-out condition. The FPD runs the flame ignition sequence to relight the flame. If this fails, it runs the sequence again. If the second attempt also fails, the detector shuts down all functions except temperature and makeup gas flow.

Setting parameters for the FPD

**WARNING** Verify that a column is installed or the FPD fitting is plugged before turning on the air or hydrogen. An explosion may occur if air and hydrogen are allowed to leak into the oven.

1  Press [Front det] or [Back det].
2  Set the detector temperature. It must be greater than 120 °C for the flame to light.
3  Change the hydrogen flow rate, if desired. Press [Off/No].
4  Change the air flow rate, if desired. Press [Off/No].
5  If you are using packed columns, turn off the makeup gas and proceed to step 9.
6  If you are using capillary columns:
   a  Verify that makeup gas type is the same as that plumbed to your instrument (next to Makeup in the parameter list). Change the gas type, if necessary.
   b  If your capillary column is defined, choose a flow mode and set the makeup gas flow or combined flow.
   c  If your capillary column is not defined, enter a makeup gas flow. Only constant flow is available.
7  Scroll to Flame and press [On/Yes]. This turns on the air and hydrogen and initiates the ignition sequence.
On ignition, the signal increases. Typical levels are 4 to 40 pA in sulfur mode, 10 to 70 pA in phosphorus mode. Verify that the flame is lit by holding a cold, shiny surface, such as a mirror or chrome-plated wrench, over the vent exit. Steady condensation indicates that the flame is lit.
6 Valves

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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.

![Diagram of valve locations on GC](Figure 5)

Heating the valves

The valve box contains a heated block with two valve mounting locations (shaded in Figure 5). The middle hole on each block is used to pass tubing into the column oven.

If two valves are used, they share the same temperature setpoint.

Valve temperature programming

Most valve applications are isothermal; however, you can define three temperature ramps if desired. Program this ramp the same as an oven ramp. Refer to "Setting the oven parameters for ramped temperature" on page 52 for more information.
Valve Control

Valves can be controlled manually from the software keyboard 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 two drivers, known as Valve 1 and Valve 2. If a third valve is installed, Valve 2 controls itself and Valve 3 simultaneously.

Table 22 Valve drivers

<table>
<thead>
<tr>
<th>Valve number</th>
<th>Type</th>
<th>Volts</th>
<th>Power or current</th>
<th>Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 and 2</td>
<td>Current source</td>
<td>24 VDC</td>
<td>13 watts</td>
<td>Pneumatic valve control</td>
</tr>
</tbody>
</table>

The internal valve drivers

Valve drivers 1 and 2 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 115.

**Not installed**  Self-explanatory.
Configuring a Valve

1. Press [Config]. Scroll to Valve #.
2. Enter the valve number and press [Enter]. The current valve type is displayed.
3. To change the valve type, press [Mode/Type], select the new valve type, and press [Enter].
Controlling a Valve

From the keyboard

Valves have two positions controlled by the [On] and [Off] keys. The keyboard commands for two-position valves are:

[Valve #] <scroll to the valve> [On]  Rotates valve to one stop and
[Valve #] <scroll to the valve> [Off]  Rotates valve to the other stop

From the run or clock time tables

The Valve On and Valve Off commands can be run time or clock time programmed. See “Run Time Programming” on page 12 and “Clock Time Programming” on page 15.

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.

Gas sampling valve

If a valve is configured as a gas sampling valve, it starts a run automatically when it is switched to the Inject position. This can be done with a keyboard command or 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 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.
7

GC Output Signals

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**About 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, and 0 to 1 mV.

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

See Table 23 for the conversions from units shown on the GC display to units as shown in Agilent data systems and integrators.

### Table 23  Signal conversions

<table>
<thead>
<tr>
<th>Signal type</th>
<th>1 display unit is equivalent to:</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Detector:</strong></td>
<td></td>
</tr>
<tr>
<td>FID, NPD</td>
<td>1.0 pA ($1.0 \times 10^{-12}$ A)</td>
</tr>
<tr>
<td>TCD</td>
<td>25 µV ($2.5 \times 10^{-5}$ V)</td>
</tr>
<tr>
<td>μECD</td>
<td>1 Hz</td>
</tr>
<tr>
<td>Analog input board (use to connect the GC to non-Agilent detector)</td>
<td>15 µV</td>
</tr>
<tr>
<td><strong>Nondetector:</strong></td>
<td></td>
</tr>
<tr>
<td>Thermal</td>
<td>1 °C</td>
</tr>
<tr>
<td><strong>Pneumatic:</strong></td>
<td></td>
</tr>
<tr>
<td>Flow</td>
<td>1 mL/min</td>
</tr>
<tr>
<td>Pressure</td>
<td>1 pressure unit (psi, bar, or kPa)</td>
</tr>
<tr>
<td>Diagnostic</td>
<td>Mixed, some unscaled</td>
</tr>
</tbody>
</table>
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. Press [On/Yes] to set to current Value or [Off/No] to cancel.

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 “Run Time Programming” on page 12.

1  Verify that the detector is on and in a ready state.
2  Press [Analog Out].
3  Scroll to Zero.
4  Press [On/Yes] to set Zero at the current signal value, or Enter a number between -500000 and +500000. A value smaller than the current Zero shifts baseline up.

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

### Table 24  Range limits

<table>
<thead>
<tr>
<th>Detector</th>
<th>Usable range settings (2^x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FID</td>
<td>0 to 13</td>
</tr>
<tr>
<td>NPD</td>
<td>0 to 13</td>
</tr>
<tr>
<td>FPD</td>
<td>0 to 13</td>
</tr>
<tr>
<td>TCD</td>
<td>0 to 6</td>
</tr>
<tr>
<td>µECD</td>
<td>0 to 6</td>
</tr>
<tr>
<td>Analog input</td>
<td>0 to 7</td>
</tr>
</tbody>
</table>

Range may be run time programmed. See “Run Time Programming” on page 12 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. Press [Config][Analog Out].
2. Scroll to Fast peaks and press [On].

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 keypad.

Digital zero

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>
<thead>
<tr>
<th>Data rate, Hz</th>
<th>Minimum peak width, minutes</th>
<th>Relative noise</th>
<th>Detector</th>
<th>Column type</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>0.0001</td>
<td>5</td>
<td>FID</td>
<td>Narrow-bore, 0.05 mm</td>
</tr>
<tr>
<td>200</td>
<td>0.001</td>
<td>3.1</td>
<td>FID</td>
<td>Narrow-bore, 0.05 mm</td>
</tr>
<tr>
<td>100</td>
<td>0.002</td>
<td>2.2</td>
<td>FID/NPD only</td>
<td>Capillary</td>
</tr>
<tr>
<td>50</td>
<td>0.004</td>
<td>1.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>0.01</td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>0.02</td>
<td>0.7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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.
Zero Init Data Files

This feature applies to digital output only, and is mainly intended for non-Agilent data systems. It may help systems that have trouble with non-zero baseline output.

When you turn it **On**, the GC immediately begins to subtract the current detector output value(s) from any future values. For example, if you turn it on when the output is 20 pA, the GC subtracts 20 pA from the digital output until you turn Zero Init Data Files **Off**.

You will not see any change in the GC display, but you will see the change in the online plot available in the data system.

To change this setting, press [Config], scroll to Instrument, then scroll to Zero Init Data Files.
8 Auxiliary Devices

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About Pressure Control

Pressure units

There are two common ways of expressing gas pressures:

**psia**  Absolute pressure, measured relative to vacuum.

**psig**  Gauge pressure, measured relative to atmospheric pressure. This name is used because most pressure gauges have one side of the sensing element exposed to the atmosphere.

The two measurements are related by:

\[ psia = psig + \text{atmospheric pressure} \]