Operating Manual
Volume 1. General Information

Agilent 6890 Series Gas Chromatograph

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Safety Information

The 6890 Gas Chromatograph meets the following IEC (International Electrotechnical Commission) classifications: Safety Class 1, Transient Overvoltage Category II, and Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the 6890 has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard. Disconnect the AC power cord before removing covers. The customer should not attempt to replace the battery or fuses in this instrument. The battery contained in this instrument is recyclable.

Safety Symbols

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument.
Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

WARNING

A warning calls attention to a condition or possible situation that could cause injury to the user.

CAUTION

A caution calls attention to a condition or possible situation that could damage or destroy the product or the user's work.

See accompanying instructions for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Indicates radio-active hazard.

Indicates explosion hazard.

Electromagnetic compatibility

This device complies with the requirements of CISPRII. Operation is subject to the following two conditions:

1. This device may not cause harmful interference.
2. This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1. Relocate the radio or television antenna.
2. Move the device away from the radio or television.
3. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
4. Make sure that all peripheral devices are also certified.
5. Make sure that appropriate cables are used to connect the device to peripheral equipment.
6. Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.
7. Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

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Sound Emission Certification for Federal Republic of Germany

Sound pressure Lp < 65 dB(A)
During normal operation
At the operator position
According to ISO 7779
(Type Test)

When operating the 6890 with cryo valve option, the sound pressure 74.6 dB(A) during cryo valve operation for short burst pulses.

Schallemission

Schalldruckpegel LP < 65 dB(A)
Am Arbeitsplatz
Normaler Betrieb
Nach DIN 45635 T. 19
(Typprüfung)

Bei Betrieb des 6890 mit Cryo Ventil Option treten beim Oeffnen des Ventils impulsfoermig Schalldrucke Lp bis ca. 74.6 dB(A) auf.

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1

The 6890 Series Gas Chromatograph

What's unique to this instrument.
Maintenance. Strategy.

The 6890 Series Gas Chromatograph

The 6890 Series Gas Chromatograph is referred to as “the GC” throughout this manual.

Manuals

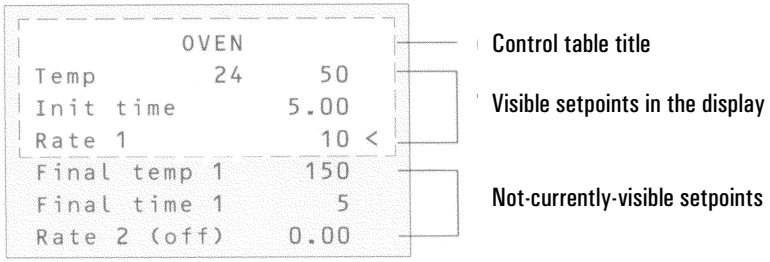
The Operating Manual is in three volumes:

- The *General Information* volume (this one) contains everything except information on Inlets and Detectors.
- The *Inlets* volume is devoted to inlet systems, including maintenance and troubleshooting procedures.
- The *Detectors* volume discusses detector systems, including maintenance and troubleshooting procedures.

Each volume has its own index.

Control tables

This GC is controlled by a lengthy list of setpoints (temperatures, times, choice of signal, and so on) that are organized into control tables. This is a typical oven control table:



- Control table title—This line identifies the table. It does not move when the rest of the table moves up or down.
- Visible setpoints in the display—The display has four lines. The title uses one line leaving three to show setpoints and, in the `Temp` line, the current actual value.
- Not-currently-visible setpoints—This table contains six setpoint lines. The lower three can be moved into the viewing window when needed.

Installed equipment

Your instrument only displays control tables for items that are physically present. There is no way to see a control table for an inlet, detector, or other device that is not installed.

Control tables that list many instrument functions, such as [Status] or [Config], only show items that are installed. Therefore, the sample displays in this manual may be somewhat different than those on your instrument.

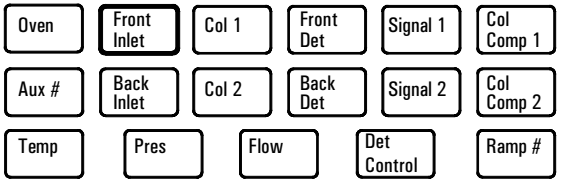
Using control tables

The general procedure for setting up the instrument is:

1. Press a key to call up a control table. It appears in the display. The first line is a title that identifies the table.
2. Inspect the setpoints in the table (you may have to scroll up or down if it is a long table).
3. Edit selected setpoints in the table.
4. Repeat this process with other tables until you have what you want.
5. Analyze the sample.

The advantage of a table is that it contains groups of related setpoints. You can inspect them and alter them easily and quickly without having to execute key sequences for each.

For example, to set up the front inlet, press [Front Inlet]



The control table for the type of inlet in the front position appears in the display. Control tables for three inlet types, all with Electronic Pneumatic Control (EPC), are on the next page.

Cool on-column

```
   FRONT INLET (COC)
Mode:    Track oven
Temp        53     53
Pressure 10.0   10.0 <
```

Purged packed

```
   FRONT INLET (PP)
Temp        250    250 <
Pressure 10.0   10.0
Total flow         5.0
```

Split/Splitless

```
Mode:  Split
Temp        250    250 <
Pressure 10.0   10.0
   FRONT INLET (S/SL)
Split ratio        100
Split flow        76.6
Tot flow 80.4   80.4
Gas saver           On
Saver flow        20.0
Saver time         2.0
```

The top of the table. Use the scroll keys (page to move it into the display).

The title line does not move.

You can view three lines of the table in the display

The bottom of the table. Use the scroll keys to move it into the display.

Figure 1 Some inlet control tables

Use the cursor keys (▲ and ▼) to move the cursor (<) to the line you wish to change, type the new value, and press [Enter]. Repeat until the table is as you want it.

Tables change depending on the modes you select and the instrument configuration. Your tables are probably somewhat different from these.

Some specifics

Gas control

The GC can control all gas streams—inlets, detectors, and three auxiliary flows—from the keyboard using Electronic Pneumatic Control (EPC). EPC allows flow and pressure setting plus a variety of program modes.

Some nonEPC inlets and detectors are also available. They function in the conventional manner using flow controllers, pressure regulators and a separate flow meter. Only on/off control is available from the keyboard.

Columns

You can control the behavior of the carrier gas in the column, specifying constant or ramped flow or constant or ramped pressure. EPC inlet systems maintain this behavior for the entire run, even with temperature programming.

The column should be set up before the inlet!

Inlets and detectors

These are covered in the separate *Inlets* and *Detectors* volumes.

Signals

Signals are the data streams that exit the GC for processing by some other device. There is a wide selection, both analog and digital.

Automation

The run time table executes commands at specified times after injection. The clock time table executes commands at specified times of day.

Methods and sequences

The active method is the set of control tables and values that is presently controlling the GC. Up to five methods can be stored in memory.

A sequence is a list of sample locations and the stored methods to be used to run them. Up to five sequences can be stored. Samples may come from either an automatic liquid sampler or a sampling valve with a stream selector valve. Sequences can be interrupted to run urgent samples.

Valves

Switching valves can be used for various column operations. Gas sampling valves can be used either manually or with a multiposition stream selection valve. If the multiposition valve is used, it can be combined with a sequence to control the sample selection and analysis.

Strategy

The GC is organized around a set of control tables, each containing a group of related setpoints. It is controlled by viewing and editing the tables to meet your analytical needs. Some suggestions for doing this are:

- The content of many tables depends on what equipment is present. While the GC can sense many of its components, some information (such as what carrier gas is in use) must be entered by you. Always configure (define) instrument elements before trying to use them.
- When setting up for analysis, configure the carrier gas first, then the column mode, and finally the inlet. Detectors can be set up at any time.
- Use the [Config] key routinely to verify that the configuration is what you believe it to be.
- Use the [Info] key for help with setpoint ranges, next action to perform, and other advice.
- Many setpoints require that you select from a list of choices. The [Mode/Type] key opens these lists. If a setpoint seems to call for an entry other than a number or [On] or [Off], try [Mode/Type] to see if there is an underlying menu.

Maintenance information

Fuses and batteries

The GC requires fuses and batteries for proper operation. These should only be accessed by Agilent service personnel.

Table 1 Mainboard Fuses and Batteries

Fuse designation	Fuse rating and type
F1, F2, F3, F4	8A, 250 Vac, IEC 127 type f (non-time delay), glass body
Battery designation	**Battery rating and type**
BT1	3-volt lithium battery, Panasonic BR3032

Table 2 AC Board Fuses

Fuse designation	Line voltage	Fuse rating and type
F1, F2	120 V	20 A, 250 Vac, IEC 127 type f (non-time delay), ceramic body
F1, F2	200 V - 240 V	15 A, 250 Vac, IEC type f (non-time delay), ceramic body
F3, F4	All	8 A, 250 Vac, IEC type f (non-time delay), glass body

Maintenance schedule

The frequency of maintenance depends upon:

- The level of usage of the GC
- The type of samples injected
- Whether injections are manual or automatic
- Whether the instrument is used for multiple applications or dedicated to one
- Other environmental factors, such as dirt, ambient temperature, etc.

Table 3 Maintenance Schedule

Maintenance frequency	Items
Daily	Change septa, run a calibration sample, check the tightness of liner and column nuts[1]
Weekly	Change glass liners and O-rings, if applicable
Monthly	Clean the split/splitless inlet vent line trap Perform a leak check for hydrogen. Check all the connections from the initial supply. At the GC, leak check the inlet and the column connections to the inlet and detector.
Quarterly	Renew gas cylinders[2]
Semiannually	Clean detectors, perform wipe test on ECD
Annually	Recondition or replace internal and external traps and chemical filters

[1] Very important for temperature programming using Vespel or Vespel/graphite ferrules

[2] With typical usage, A-size cylinders will supply two dual-channel chromatographs for about three months. Replace the cylinder when its pressure drops below 500 psig.

General warnings

Many internal parts of the GC carry dangerous voltages

If the GC is connected to power sources, even if the power switch is off, potentially dangerous voltages exist on:

- The wiring between the detector power cord and power switch
- The wiring between the GC power cord and the AC power supply, the AC power supply itself, and the wiring from the AC power supply to the power switch.

With the power switches on, potentially dangerous voltages also exist on:

- All electronics boards in the instrument
- The internal wires and cables connected to these boards.

WARNING All these parts are shielded by covers. With the covers in place, it should be difficult to accidentally make contact with dangerous voltages. Unless specifically instructed to, never remove a cover unless the detector, inlet, or oven are turned off.

If the power cord insulation is frayed or worn, the cord must be replaced. Contact your Agilent service representative.

Electrostatic discharge is a threat to GC electronics

The printed circuit (PC) boards in the GC can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions. Wear a grounded wrist strap any time you must remove the electronics side panel.

Many parts are dangerously hot

Many parts of the GC operate at temperatures high enough to cause serious burns. These parts include but are not limited to:

- The inlets
- The oven
- The detectors
- The column nuts attaching the column to an injection port or detector

You should always cool these areas of the GC to room temperature before working on them. They will cool faster if you first set the temperature of the heated zone to room temperature. Turn the zone off after it has reached the setpoint. If you must perform maintenance on hot parts, use a wrench and wear gloves. Whenever possible, cool the part of the instrument that you will be maintaining before you begin working on it.

WARNING Be careful when working behind the instrument. During cooldown cycles, the GC emits hot exhaust which can cause burns.

The insulation around the inlets, detectors, valve box, and the insulation cups is made of refractory ceramic fibers. To avoid inhaling fiber particles, we recommend the following safety procedures: ventilate your work area; wear long sleeves, gloves, safety glasses, and a disposable dust/mist respirator; dispose of insulation in a sealed plastic bag; wash your hands with mild soap and cold water after handling the insulation.

Shutting down the GC

For less than one week

In general you can always leave the GC power on when not in use. If you will not use the GC for up to approximately one week, conserve gases and energy as follows:

- Reduce detector, inlet, and column temperatures to 150–200°C to save energy.
- Turn off corrosive or potentially hazardous gas flows, such as oxygen and hydrogen.
- Reduce flows of carrier and makeup gases.
- Turn off coolant supplies at their sources.

WARNING Never leave flammable gas flows on if the GC will be unmonitored for long periods of time. If a leak develops, the gas could create a fire or explosion hazard.

Maintaining the instrument with a lowered temperature and reduced carrier and makeup gas flows keeps impurities from building in your column, inlet, and detector.

For more than one week

1. Set all heated zones to ambient temperature and turn off the detector support gas flows. Leave the carrier gas flow on.
2. When the GC is cool, turn it off.
3. Turn off all gas and coolant supplies at their sources.
4. Remove the column and cap its ends to prevent contamination. Store the column in a cool, dry place.
5. To prevent contamination, cap the inlet and detector column fittings.
6. If gas connections are removed from the GC, cap the intake fittings on the back panel of the GC and on the inlet manifold.
7. If desired, replace the split vent trap filter cartridge (if present).

2

The Keyboard and Display

Keyboard, control tables, and the display.

The Keyboard and Display

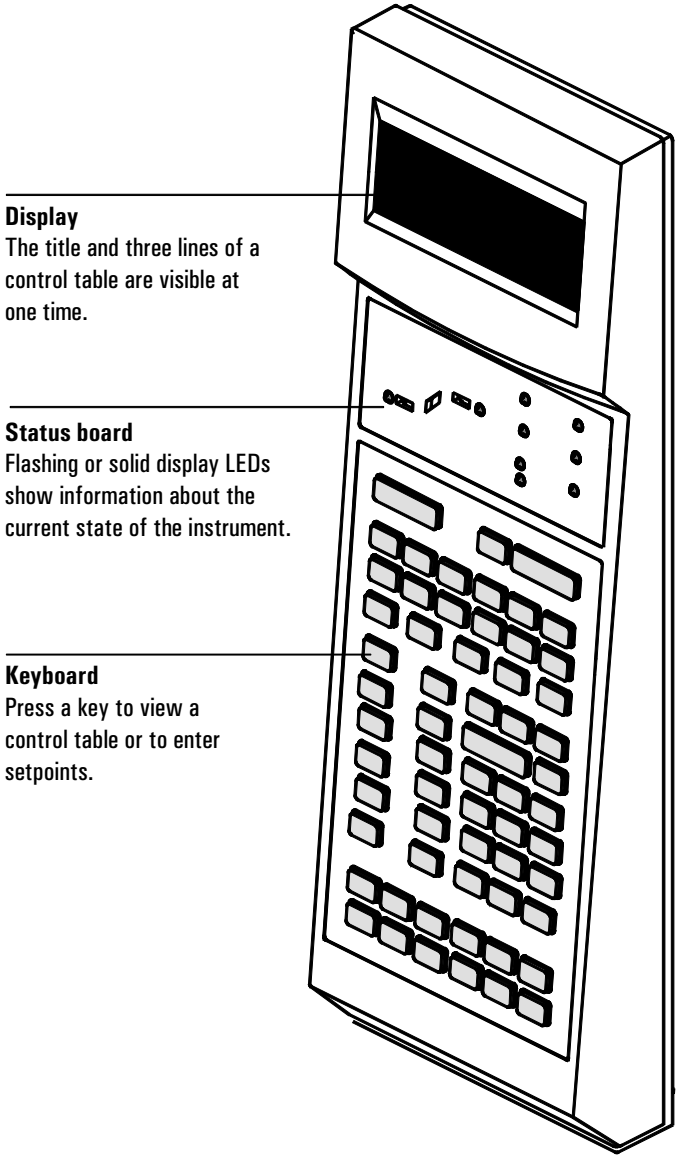


Figure 2 The GC controls

The display

Think of this as a window through which you view the control tables. The top line is a title—the other lines show the table content. If the table is more than three lines long, use the scroll keys to see the additional lines.

Scroll keys (▲,▼)

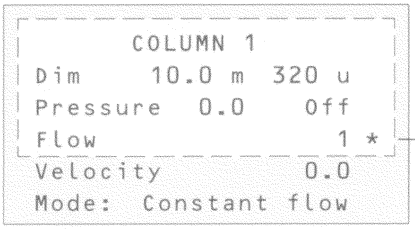
Move the control table up or down in the display window.

The cursor (<)

Points to the line that is in editing position. Changes that you make using the keyboard apply to the "cursored" line.

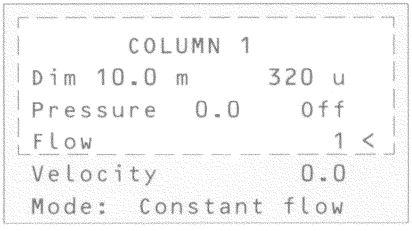
Asterisk (*)

A blinking asterisk prompts you to press [Enter] to store a setpoint or [Clear] to abort the entry. You cannot perform any other task until this is done.

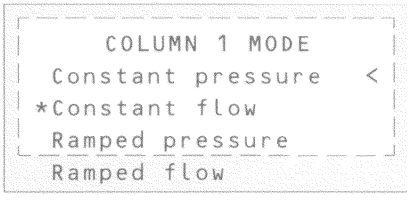


If this asterisk blinks, you cannot perform any other function until you press [Enter] or [Clear].

The asterisk on the left of a Mode/Type table indicates the current selection.



Press [Mode/Type] twice.



Beeping instrument

If a gas flow cannot reach setpoint, you hear a series of beeps. The flow shuts down after 1 or 2 minutes.

If a hydrogen flow is shut down or a thermal shutdown occurs, a continuous beep sounds. Cancel the beep by pressing [Clear].

Any other type of fault, warning, or shutdown is accompanied by one beep.

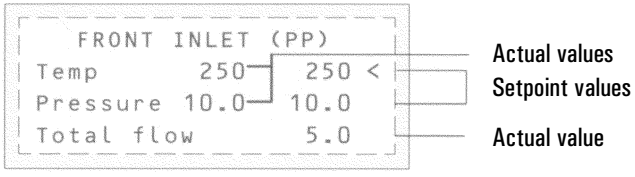
Blinking setpoint

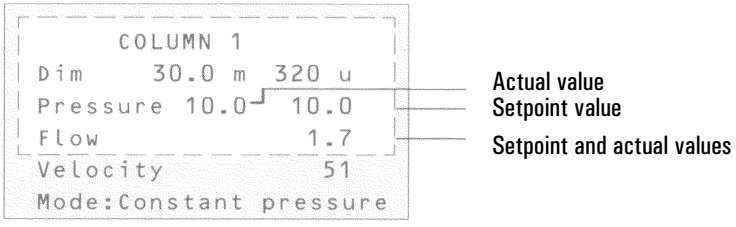
If a gas flow, multiposition valve, or the oven is shut down by the system, `Off` will blink at the appropriate line of the control table. This helps you identify where the problem occurred.

The detector On/Off line blinks if there is a pneumatics shutdown or a failure in another part of the detector, such as a TCD filament.

Actual and setpoint values

When there are two values in one line of a control table, the left value is always actual and the right value is always a setpoint. When there is only one value, it is either an actual or setpoint, depending on the table. On some control tables—such as those controlling columns—the far right number is both actual and setpoint.



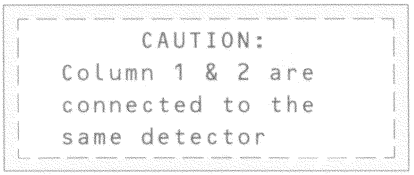


Messages

Cautions are reminders that your instrument may be configured incorrectly. You see this message when:

- [Column 1] and [Column 2] are configured to one inlet or one detector.
- An auxiliary flow channel is used as an inlet, and the auxiliary carrier gas type is configured as air. You cannot use air as a carrier gas.

Caution message:



Press [Clear] to remove the message. You can then reconfigure the instrument, if desired, or continue with your current configuration.

Errors mean that:

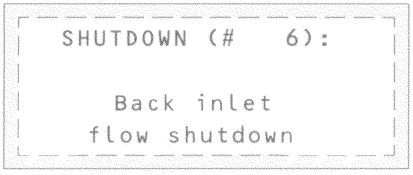
- The setpoint you've entered is out of the allowable range.
- You do not have the hardware on your instrument to support the operation you have requested.

Error message:

Press [Clear] to remove the message. You must enter a new setpoint, change that hardware, or reconfigure the instrument before continuing.

Popups appear when a Shutdown, Fault, or Warning occurs. They contain the type and number of the error and a brief description. See chapter 12, in this volume, for more information.

Popup message:



Press [Clear] to remove the message.

The status board

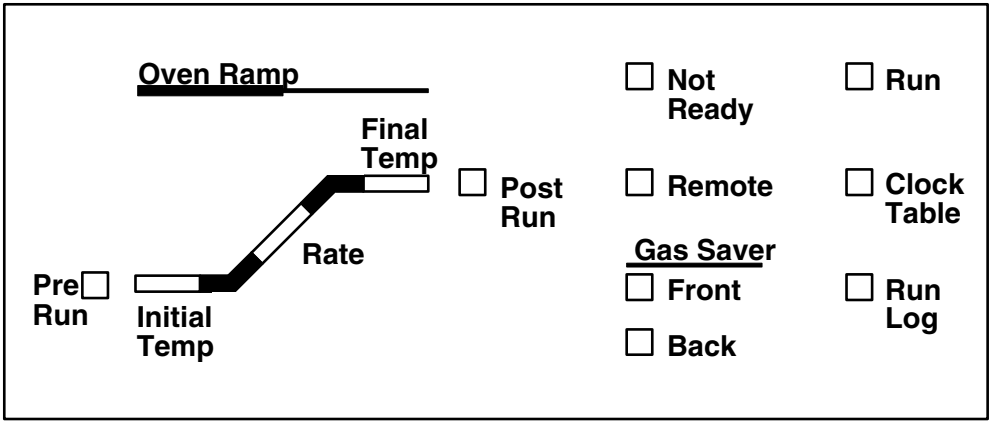


Figure 3 The status board

Table 4 The Display LEDs

LED	Description
Pre Run	Lit when the GC is in the *Pre Run* state (after [Prep Run] is pressed). See the *Inlets* volume for more information.
Oven Ramp LEDs	Show the progress of the oven temperature program. The Rate LED blinks if the oven is unable to follow the program.
Post Run	Lit when the instrument is executing a post run.
Not Ready	Lit when the GC is not yet ready to make a run. Blinks when the instrument has one or more fault conditions. Press the [Status] key to see which parameters are not ready or what faults have occurred.
Run	Lit when the instrument is executing a chromatographic run.
Remote	Indicates that communication with a remote device has been established.
Clock Table	Indicates that the clock table has entries.
Gas Saver	Indicates that the front or back gas saver is on.
Run Log	Indicates that the run log has entries. This information can be used for Good Laboratory Practice (GLP) standards.

The keyboard

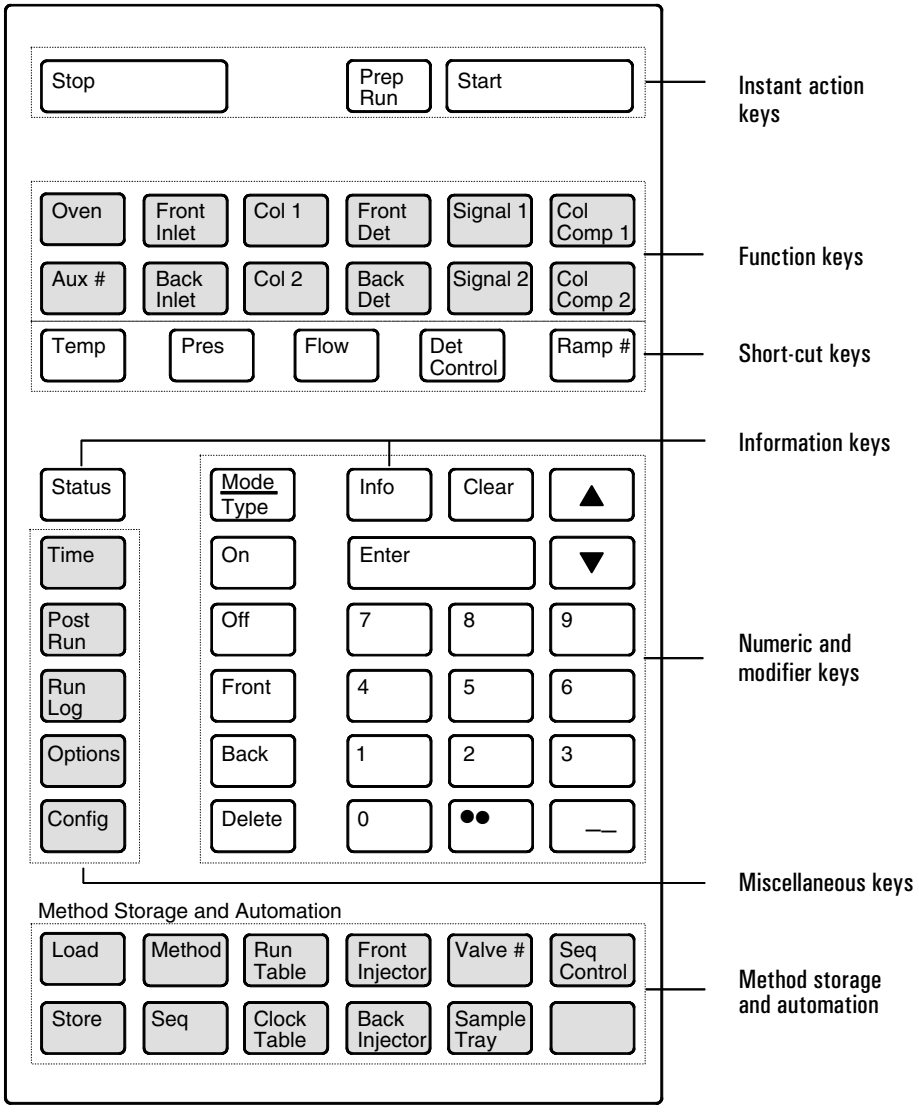


Figure 4 The keyboard

Instant action keys [Start], [Stop], and [Prep Run]

These keys cause the instrument to do something now.

[Start] and **[Stop]**

Start and stop any type of run. [Stop] cancels a Pre Run, Post Run, or power fail recovery and aborts an INET or local sequence.

[Prep Run]

If you are using one or more of the following functions, you must press [Prep Run] to prepare for a run:

- Gas saver—cancels gas saver flow and brings inlet flow to its setpoint value.
- Splitless injection—closes the purge valve.
- Pulsed split or splitless injection—increases inlet pressure to the pulse setpoint.
- Solvent vent injection—changes inlet pressure to the vent pressure setpoint and split vent flow to the vent flow setpoint.

Pressing [Prep Run] turns on the *Pre Run* LED. When the LED is blinking, the instrument is preparing for a run and waiting for instrument setpoints (other than the ones associated with Prep Run) to be reached. Once these setpoints are ready, the LED remains on and the Prep Run events occur. After a 6-second equilibration time, the instrument is ready for a run and the *Not Ready* light goes out.

If you press [Prep Run] while the *Pre Run* LED is blinking, the LED stops blinking before all setpoints are ready. At this point, the gas saver and purge valve portions of your split/splitless inlet are ready for a run.

With most automatic injection systems, you do not need to use the [Prep Run] key. If your sampler or automation controller (for example, an integrator or workstation) does not support the Prep Run function, you must set the instrument to Auto Prep Run. See the example on page 35.

Function keys

Table 5 lists the function keys, a brief description of their use, and where to find detailed information.

Table 5 The Function Keys

Key	Use to:	For more information:
[Oven]	Set oven temperatures, both isothermal and temperature programmed.	Chapter 4, The Column Oven
[Aux #] [1] and [Aux #] [2]	Control extra temperature zones such as a heated valve box, a mass selective detector, an atomic emission detector transfer line, or an "unknown" device. Can do temperature programming.	Chapter 9, Valve Control
[Aux #] [3], [Aux #] [4], and [Aux #] [5]	Provide auxiliary pneumatics to an inlet, detector, or other device. Can do pressure programming.	Chapter 3, Flow and Pressure Control, and Chapter 11, Valve Control
[Front Inlet] and [Back Inlet]	Control inlet operating parameters.	*Inlets* volume
[Col 1] and [Col 2]	Control column pressure, flow, or velocity. Can set pressure or flow ramps.	Chapter 3, Flow and Pressure Control *Inlets* and *Detectors* volumes
[Front Det] and [Back Det]	Control detector operating parameters.	*Detectors* volume
[Signal 1] and [Signal 2]	Assign a signal, usually to the front or back detector.	Chapter 6, Signal Handling
[Col Comp 1] and [Col Comp 2]	Create a column compensation profile.	Chapter 6, Signal Handling

Short-cut keys [Temp], [Pres], [Flow], [Det Control], [Ramp #]

Quickly access a setpoint from within a table.

[Temp], [Pres], and [Flow]

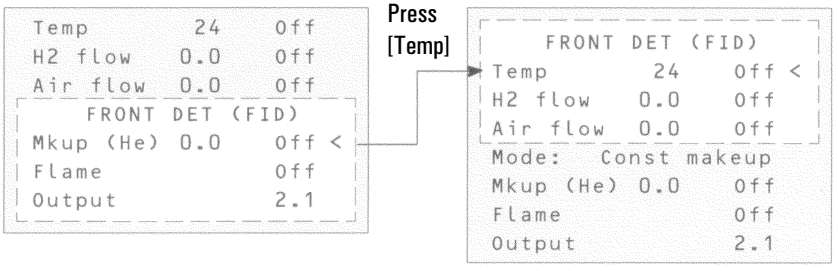
If no control table is open, pressing these keys gives you:

[Temp]	Oven temperature
[Pres]	Front inlet pressure (back or auxiliary pressure channel if front inlet is not installed)
[Flow]	Column 1 or 2 flow if EPC inlet. If not EPC, front detector or back detector flow.

If the parameter is in the open control table, the cursor jumps to that line:

[Front Det] table open, cursor on `Mkup (He)`

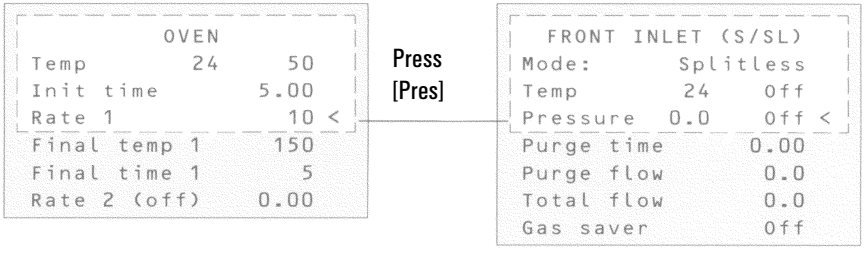
Cursor moves to `Temp`



If the parameter is not in the open table, the key opens an appropriate table. For example, if the oven control table is open and you push [Pres], the front inlet control table opens with the cursor on the `Pressure` line.

[Oven] table open

[Front inlet] table opens



[Det Control]

When viewing a detector control table, [Det Control] moves the cursor to the on/off control for that detector.

[Front Det] table open, cursor on `Temp` line

```
    FRONT DET (TCD)
Temp          24     Off <
Ref flow    0.0      Off
Mkup (He)   0.0      Off
Filament             Off
Output               2.1
Negative polarity    Off
```

Press [Det Control]

```
Temp          24     Off
Ref flow    0.0      Off
Mkup (He)   0.0      Off
    FRONT DET (TCD)
Filament             Off <
Output               2.1
Negative polarity    Off
```

Cursor on `Filament`, the On/Off line for the thermal conductivity detector

With a nondetector control table, [Det Control] opens the front detector control table (or back, if a front detector is not installed). The cursor is at the on/off control for that detector.

[Front Inlet]

```
  FRONT INLET (S/SL)
Mode:          Splitless
Temp          24     Off <
Pressure    0.0      Off
Purge time           0.00
Purge flow           0.0
Total flow           0.0
Gas saver            Off
```

[Det Control]

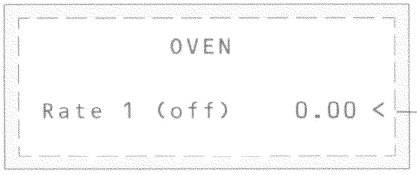
```
Temp          24     Off
H2 flow     0.0      Off
Air flow    0.0      Off
Mode:    Const makeup
Mkup (N2)   0.0      Off
    FRONT DET (FID)
Flame                Off <
Output (off)         0.0
```

Cursor on `Flame`, the on/off control for the flame ionization detector

[Ramp #]

With a control table open that has no temperature, flow, or pressure ramps, [Ramp #] plus a number opens the Oven control table. If no ramps are specified, the cursor is on the `Rate 1 (off)` line.

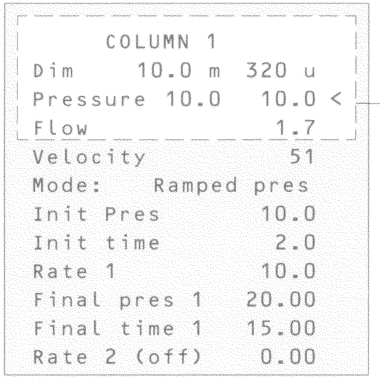
Press [Ramp # [2]



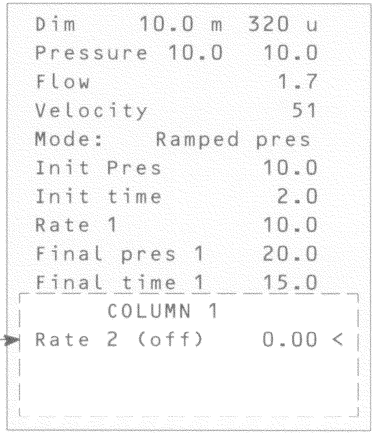
Oven control table opens. Because no temperature ramps are set on this table, cursor is on the `Rate 1 (off)` line.

With a control table that contains temperature, flow, or pressure ramps, [Ramp #] (1-6) moves the cursor to the first line of the ramp number specified. If the ramp number does not exist, the cursor goes to the highest ramp number in the control table.

[Col 1] table open, cursor on `Pressure` line.



Press [Ramp #] [2]



Cursor moves to `Rate 2` line.

[Info]

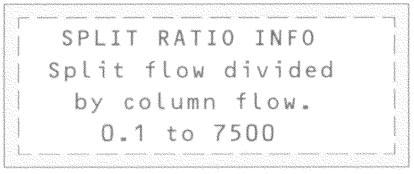
This is a context-sensitive help that provides information about an active parameter (line with the cursor).

These info messages may be in several different forms,

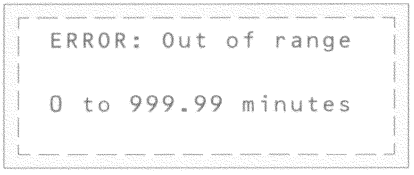
- Definitions
- Setpoint ranges
- Actions to perform

The following examples are possible, depending upon the control table you are in. Press [Info].

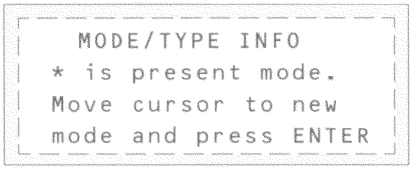
Definition:



Setpoint ranges:



Perform an action:



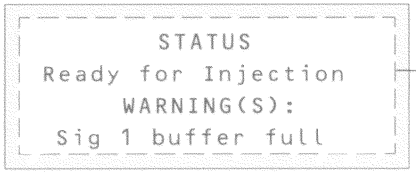
[Status]

The [Status] key has two tables associated with it. You switch between them by pressing the key.

The Ready/Not Ready status table

This table lists parameters that are *Not Ready* or gives you a *Ready for Injection* display. If there are any *faults*, *warnings*, or *method mismatches* present, they are displayed here. See chapter 12 for detailed information about the not ready, fault, and warning status displays. The method mismatch displays are discussed in chapter 8, “Methods”.

Ready for injection display



Ready display—check for warnings.

Not ready display

Not ready—items that are not ready. If you have a *not ready* display, check for faults or warnings.

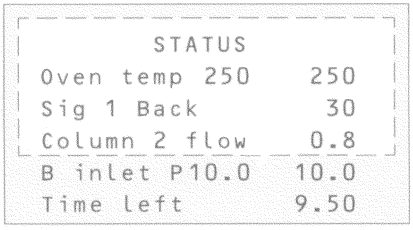
Fault—a hardware problem requiring user intervention.

Warning—problems that user should be aware of but that will not prevent instrument from executing a run.

Method mismatch—message appears if hardware or user-entered configuration has changed after loading a method or power on.

The setpoint status table

This table lists setpoints compiled from the active control tables on the instrument. This is a quick way to view active setpoints during a run without having to open multiple control tables.



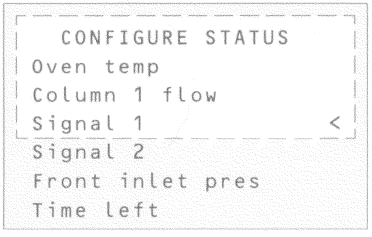
Procedure: Configuring the setpoint status table

You can change the order of the list. You might want the three most important setpoints to appear in the window when you open the table.

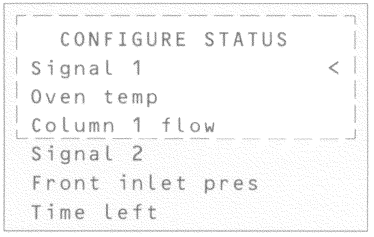
1. Press [Config] [Status].
2. Scroll to the setpoint that should appear first and press [Enter]. This setpoint will now appear at the top of the list.
3. Scroll to the setpoint that should appear second and press [Enter]. This setpoint will now be the second item on the list.
4. And so on, until the list is in the order you wish.

Press [Config][Status]

a. Scroll to Signal 1 and press [Enter].



b. Signal 1 is now the first item on the list.

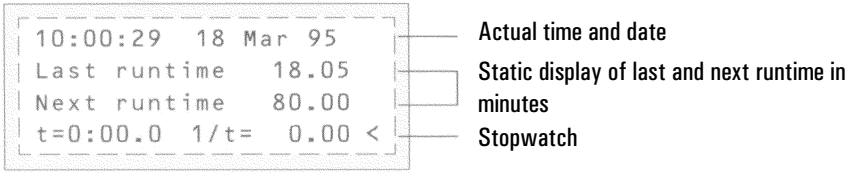


Miscellaneous keys

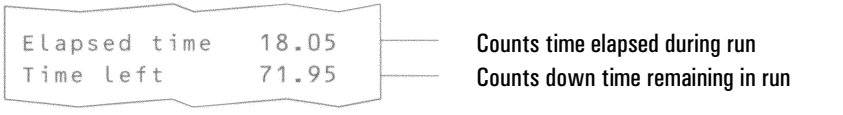
[Time]

The time control table does not have a title. The first line always displays the current date and time, and the last line always displays a stopwatch. The two middle lines vary as shown.

Time display between runs



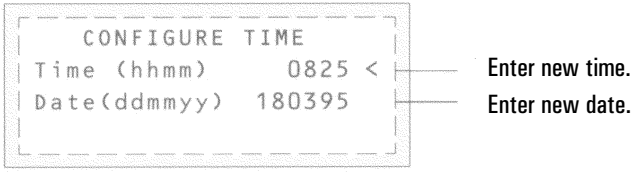
Time display during a run



Time display during Post Run

Procedure: Setting time and date

Press [Config][Time]



Procedure: Using the stopwatch

In the stopwatch mode, both the time (to 0.1 second) and reciprocal time (to 0.01 min^{-1}) are displayed. The stopwatch is useful when measuring flows with a bubble flowmeter.

1. Scroll to the stopwatch line of the Time Control Table.
2. Press [Enter] to start the stopwatch.
3. Press [Enter] again to stop.
4. Press [Clear] to set to zero.

You can access other functions while the stopwatch is running. Press [Time] again to view the stopwatch display.

Procedure: Setting up [Post Run]

Use this key to program the instrument to clean out a column after a run. To set up a Post Run:

1. Press [Post Run]

When `Time` is set at 0.00, other lines of the control table are not available.

2. Enter the post-run (column cleanout) `Time`, in minutes.

Once a setpoint for `Time` is entered, other lines of control table become available.

3. Enter `Oven temp` and `Column pres`.

The instrument is now programmed to maintain the oven temperature at 250°C for 10 minutes following a run, with column 2 head pressure at 15.0 psi.

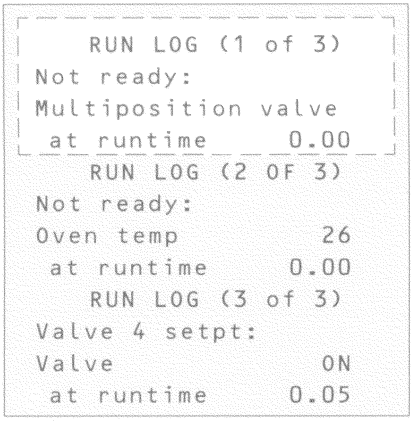
The Post Run LED on the status board is lit during a Post Run.

If you press [Time] while in a Post Run, you can view the amount of time remaining.

[Run Log]

Deviations from the planned method (including keyboard intervention) during the most recent run are listed in the run log table. Up to 50 run log entries can be stored. The run log information can be used for Good Laboratory Practice (GLP) standards. The run log can be uploaded to a workstation or printed out on an integrator.

Press [Run Log]



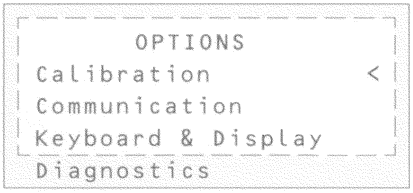
The Run Log LED is lit if there are any entries in the run log for the run in progress. The run log is cleared at the start of a new run.

If no run deviations have been logged, the display is:

[Options]

The option key accesses instrument parameter setup options.

Press [Options]



Scroll to the appropriate line and press [Enter] to access the associated control table.

Calibration

Lists the parameters that can be calibrated. The calibration displays are discussed in the *Agilent 6890 Service Manual.*

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

To activate this, select `Calibration` on the `OPTIONS` menu, then choose either Front inlet or Back inlet, and turn `Auto flow zero` on.

Communication

Allows access to the communications setpoint parameters. The communication displays are discussed in the *Agilent 6890 Site Preparation and Installation Manual.*

Diagnostics

The diagnostic parameters are for use by your Service Representative. Diagnostics are discussed in the *Agilent 6890 Service Manual*.

Keyboard and display

User interface setpoints are accessed in the keyboard and display control table. The following parameters are turned on and off by pressing the [On] or [Off] keys.

- `Keyboard lock`—the following keys and functions are operational when the keyboard lock is ON:
  [Start], [Stop], and [Prep Run]
  [Load][Method] and [Load][Seq]
  [Seq]—to edit existing sequences
  [Seq Control]-to start or stop sequences.
- `Key click`—click sound when keys are pressed, can be turned on or off.
- `Warning beep`—allows you to hear warning beeps.
- `Method mod beep`—turn [ON] for high pitched beep when method setpoint is modified.

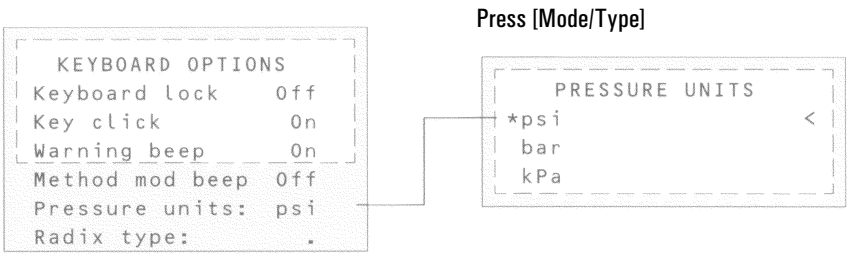
Press [Mode/Type] to change the pressure units and radix type.

- `Pressure units`

  psi—pounds per square inch, lb/in^2

  bar—absolute cgs unit of pressure, $dyne/cm^2$

  kPa—mks unit of pressure, $10^3\ N/m^2$
- `Radix type`—determines the numeric separator type—1.00 *or* 1,00

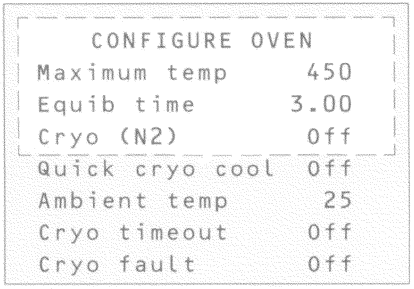


[Config]

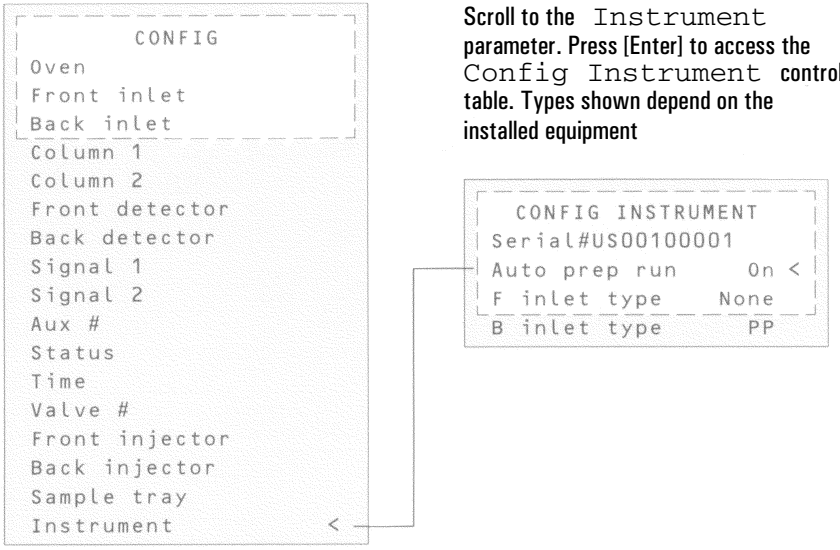
The [Config] key sets up configurations for instrument control. The column mode and dimensions, inlet, and makeup gas type configurations are critical to proper operation of EPC.

Use [Config] with other keys for infrequently changed parameters.

Press [Config] [Oven]



Press the [Config] key to obtain a listing of configurable parameters:



Scroll to the `Instrument` parameter. Press [Enter] to access the `Config Instrument` control table. Types shown depend on the installed equipment

Modifier keys

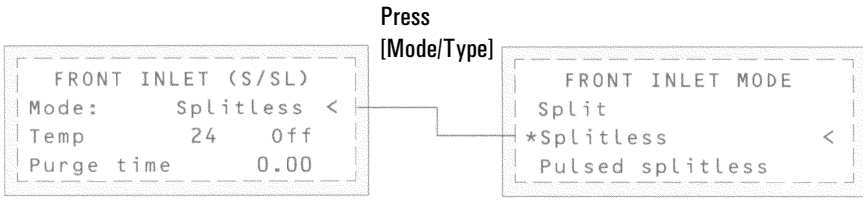
Modifier keys extend the functions of some setpoint control keys.

[Mode/Type]

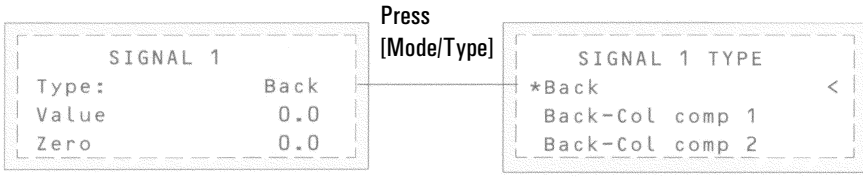
Use this key to access a list of possible modes or types associated with nonnumeric setpoints. To change mode or type, scroll to the desired line and press [Enter]. An asterisk (*) marks the present mode or type.

The following are several examples of **Mode/Type** functions.

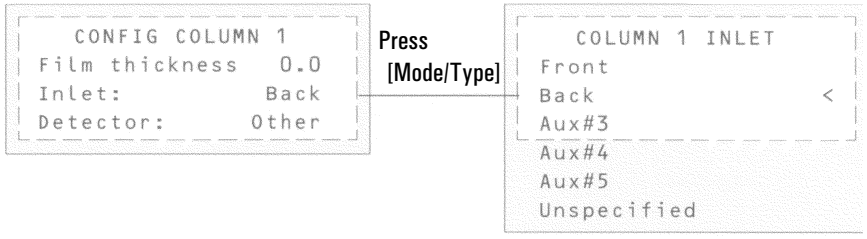
Mode:



Type:



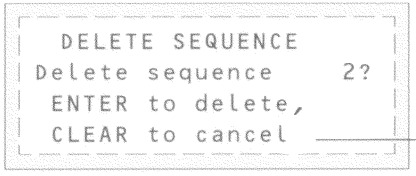
Examples of instances when the words "Mode" or "Type" do not appear. When in doubt, press [Info] to find out if [Mode/Type] is to be used.



[Clear]

The [Clear] key is used to:

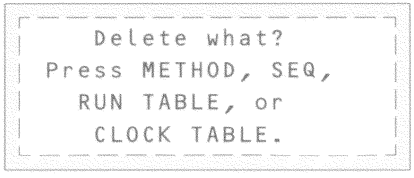
- Clear mis-entered setpoints in a control table *before* pressing [Enter] (when the * is still flashing).
- Back out of Mode/Type select before pressing [Enter].
- Return to upper level in nested control tables (config, option).
- Clear the stopwatch to zero.
- Clear info message and return to previous display.
- Clear error messages (popup messages, errors on setpoint entries, etc.).
- Cancel a function during a sequence, method, clock table, or run table and loading or storing sequences and methods.



[Delete]

Deletes methods and sequences or run table and clock table entries.

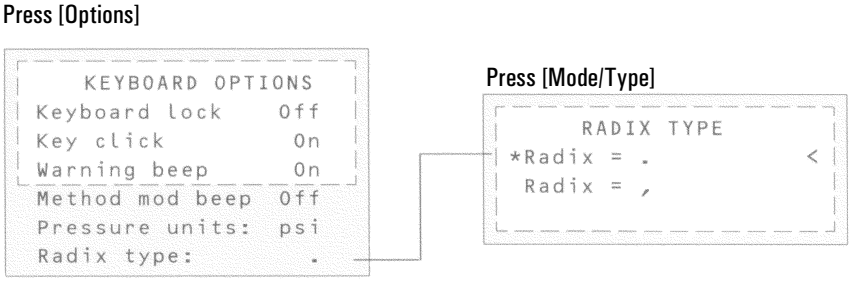
Press [Delete]



[Delete] aborts the adjust offset process for the nitrogen-phosphorus (NPD) and electron capture (ECD) detectors, without interrupting other detector parameters.

[.]

The radix is a decimal place holder. This parameter can be changed from the decimal point to the comma in the Keyboard options control table, which is nested under the Options control table.



[–]

The dash key is used to denote ranges of numbers (inclusive).

Sample range: 1 to 3, press [1] [–] [3]

Bottle # range: 1 to 10, press [1] [–] [1] [0]

This key is also used as a minus sign for negative values.

For –5, press [–] [5]

Storage and automation

Table 6 lists the storage and automation keys, a brief description of their use, and a place to find detailed information.

Table 6 Method and Sequence Storage and Automation Keys

Key	Use to:	For more information:
[Load]	Load up to five stored methods and five stored sequences.	Chapter 8, Analytical Methods Chapter 11, Analytical Sequences
[Store]	Store up to five methods and five sequences. Stored methods and sequences are labeled and dated.	Chapter 8, Analytical Methods Chapter 11, Analytical Sequences
[Method]	Review a table of stored methods. You can load, store, delete, or set default method.	Chapter 8, Analytical Methods
[Seq]	Review a table of stored sequences. The [Seq] key toggles between the stored sequences control table and sequence definition control table.	Chapter 11, Analytical Sequences
[Run Table]	View a table of events and the run time at which they occur.	Chapter 7, Instrument Automation
[Clock Table]	Display the clock time table of events in the order that they occur based on a 24-hour clock. You load, store, or delete.	Chapter 7, Instrument Automation
[Front Injector] or [Back Injector]	Edit injector control parameters such as injection volumes, sample and solvent washes, etc.	Chapter 10, Automatic Liquid Sampler
[Valve#]	Turn GSV and selection valves 1 to 8 on or off. Sets multiposition valve position.	Chapter 9, Valve Control
[Sample Tray]	Display the tray status.	Chapter 10, Automatic Liquid Sampler
[Seq Control]	Start, stop, pause or resume a sequence, and view sequence status.	Chapter 11, Analytical Sequences

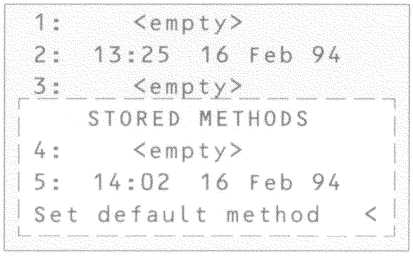
Default parameters

The GC software supplies default values for most parameters if you do not specify them. These values are reasonable operating parameters for inlets and detectors. Once you change a parameter, the default value for that parameter is erased.

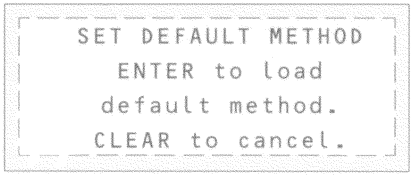
At some time, you may find it desirable to reload the default parameters. Doing this erases all current parameters except any methods you have stored and replaces them with the default set.

Procedure: Loading the default parameters

1. Press [Method]
2. Scroll to the `Set default method` line and press [Enter].



3. This message will appear:



4. Press [Enter] to load the default parameters.

3

Flow and Pressure Control

Control types. Shutdown conditions. Electronic Pneumatic Control (EPC) inlets and detectors. NonEPC inlets and detectors. Measuring flows.

Flow and Pressure Control

The 6890 Series Gas Chromatograph (the GC) has two types of gas control. Both types can be present on the same instrument.

- EPC—Electronic Pneumatic Control. Flows and pressures (inlets, detectors, and up to three auxiliary gas streams) are set at the keyboard.
- NonEPC —Conventional flow/pressure control. Inlets use flow controllers and pressure regulators in a pneumatics module on the left side of the GC. Detector controls are on top of the GC behind the detectors. Flows are measured with a bubble meter or other device.

Table 7 Detector and Inlet Controls

Module type	Control type	Control location
Inlet	EPC	Internal, via keyboard
Inlet	nonEPC	Module on left side
Detector	EPC	Internal, via keyboard
Detector	nonEPC	Top cover, behind detectors
Auxiliary	EPC	Internal, via keyboard

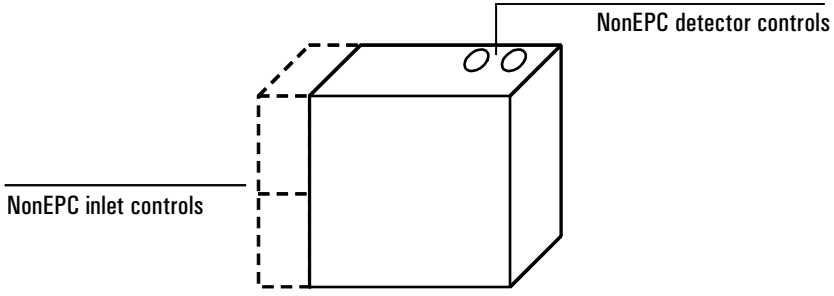


Figure 5 Control location
The pneumatics module (dashed lines) is present if a nonEPC inlet is installed.

Hydrogen shutdown

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

The GC monitors inlet and auxiliary gas streams. If a stream shuts down because it is unable to reach its flow or pressure setpoint and if that stream is configured to use hydrogen, the GC assumes that a leak has occurred and declares a *hydrogen safety shutdown*. The effects are:

- The carrier supply valve to the inlet closes and both pressure and flow controls are turned off.
- The split valves in the split/splitless and PTV inlets open.
- The oven heater and fan turn off. The flaps at the rear open fully.
- The small heated zones are turned off.

To recover from this state, fix the cause of the shutdown (tank valve closed, serious leak, others). Turn the instrument off, then back on.

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

Column shutdown

If the carrier gas source shuts down, the oven heater turns off to avoid column damage from heat without carrier gas. The flaps at the rear open halfway.

To recover from this state, fix the cause of the shutdown (tank valve closed, serious leak, others). Turn the oven and the offending inlet or auxiliary channel back on.

Turning gas flows on and off

All gas flows can be turned on or off from the keyboard without disturbing the flow or pressure settings. However, the effect of an Off command depends on whether the gas stream is EPC-controlled or not.

EPC-controlled streams

The valves in an EPC gas control module are designed for gas metering rather than On/Off operation. When this type of valve is driven to the Off state, there may still be a small flow, as much as 0.2 mL/min, through it. The display will show this flow even though `Off` also appears. Note that this is an internal leak, not a leak to the outside.

NonEPC-controlled streams

The valves in a nonEPC gas control module are designed only for On/Off action. They are gas-tight when Off.

Part 1. Electronic Pneumatic Control (EPC)

The GC can electronically control all the gas flows and pressures in the instrument. It provides:

- Flow and/or pressure control for all inlets, including flow and pressure programming for the carrier gas through the column
- Flow control via pressure regulation across fixed restrictors for all detector gases
- Pressure control for three auxiliary channels
- A gas saver mode to reduce carrier gas consumption with the split/splitless inlet, PTV inlet, and volatiles interface.
- Direct entry of split ratios, provided the column is configured

The controlling hardware is mounted internally at the top rear of the instrument. Setpoints are entered in the inlet, detector, or auxiliary control tables.

Interpreting flow and pressure readings

The EPC control board uses sensors for atmospheric pressure and the temperature of the flow pneumatics modules to eliminate local conditions as causes of retention time variability.

All flow and pressure displays are corrected to a defined set of conditions. These conditions, which we call Normal Temperature and Pressure (NTP), are 25°C and 1 atmosphere pressure. Similarly, setpoints are adjusted for the local conditions.

Thus a flow displayed on the instrument and the flow measured with a bubble meter may not agree, because the bubble meter readings represent local conditions rather than NTP conditions. However, retention times become independent of the local environment.

VERY IMPORTANT

The 6890 with EPC measures flows and pressures continuously. This has a strong effect on how the user sets up the instrument, and the rules for doing so are different from the conventional approach to gas chromatography. The differences are described in the next few pages.

Configuration

The GC identifies EPC inlets and detectors and most other devices by running presence checks during power-up. Some information must be entered manually. This is called configuration. A few things that must be configured are:

- A description of the column (optional, but extremely desirable for capillary columns)
- NonEPC inlets and detectors (configured at the factory, if installed there)
- The carrier gas in use
- Some detector gases (if there is a choice)

Configuration information is stored in a battery-powered section of memory independent of line power.

Columns and inlets

The GC, with an EPC inlet, allows you to specify gas flow through capillary columns directly. To use this feature:

1. Configure the column (supply length, inside diameter, and film thickness).
2. Configure the carrier gas. (What gas are you using?)
3. Select a column mode (constant flow or pressure, ramped flow or pressure).
4. Enter the initial flow or pressure or average linear velocity.
5. Enter a flow or pressure program (optional).
6. Enter the rest of the inlet parameters.

The rest of this chapter assumes that you have a split/splitless capillary column inlet. If you have a different inlet, the discussion still applies but some details differ. The procedures used as illustrations in the rest of this chapter are somewhat simplified, because they show the most common ways to do things but not all the alternatives. For the full details, see the *Inlets* and *Detectors* volumes.

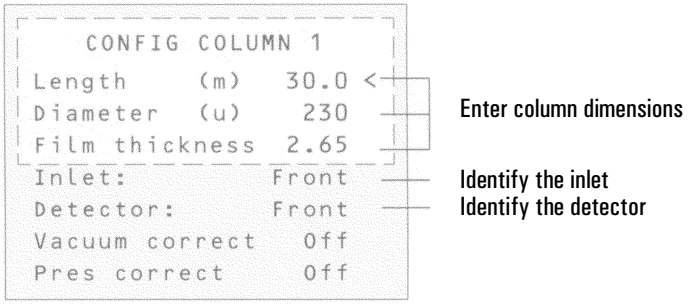
Configure the column

You define (configure) a capillary column by entering its length, diameter, and film thickness. With this information, the instrument can calculate the flow through the column. This has great advantages when using capillary columns because it becomes possible to:

- Enter split ratios directly and have the instrument calculate and set the appropriate flow rates.
- Enter flow rate or head pressure or average linear velocity. The instrument calculates the pressure needed to achieve the flow rate or velocity, sets that, and reports all three values.
- Perform splitless injections with no need to measure gas flows.
- Choose any of the four flow modes (discussed soon). If the column is not defined, your choices are limited and vary depending on the inlet.

Procedure: Configuring a capillary column

1. Press [Config] [Col 1] or [Config] [Col 2]. The column configuration screen appears.
2. If necessary, use the ▲ and ▼ keys to move (scroll) the cursor to the `Length` line.



3. Type the column length, in meters, followed by [Enter].
4. Scroll to `Diameter`, type the column inside diameter in microns, followed by [Enter].
5. Scroll to `Film thickness`, type the film thickness in microns, followed by [Enter]. The column is now *defined*.

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

6. Scroll to `Inlet` and press [Front] or [Back] to identify the inlet that the column is connected to.
7. Scroll to `Detector` and press [Front] or [Back] to identify the detector that the column is connected to.

This completes configuration for a capillary column. See the *Inlets* and *Detectors* volumes for more detail.

Additional notes on column configuration

- `Vacuum correct`—If the detector exhausts into the atmosphere, this parameter should be Off. If a column is connected directly to a mass selective detector, the parameter should be On. This allows the GC to compensate for either the local atmospheric pressure (Off) or for the reduced pressure in a mass selective detector (On).
- `Pres correct`—Some detectors, such as an atomic emission detector, operate at pressures that are neither atmospheric or vacuum. This parameter lets the user enter an appropriate pressure value.
- Packed columns should be entered as column not defined. To do this, enter `0` for either column length or column diameter.
- You should check configurations for both columns to verify that they specify separate inlets. If you are only using one column, it is still important that the second column be indicated for a different inlet, even if it is undefined. Failure to do this will lead to some very unusual flow calculations.

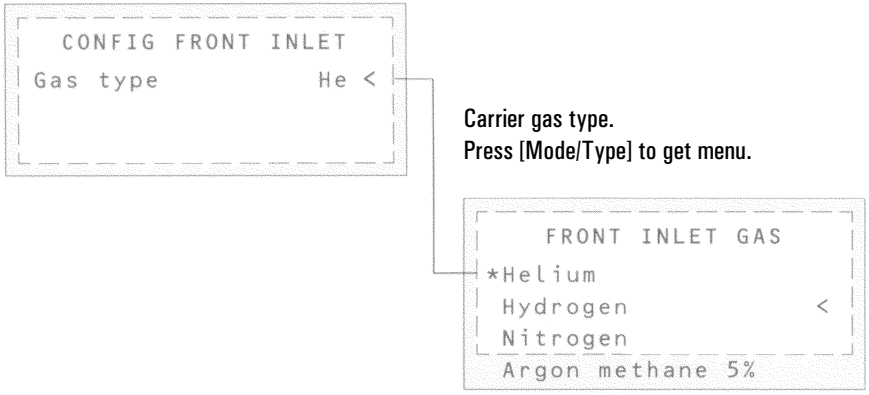
  It is possible, and sometimes appropriate, to configure both installed columns to the same inlet.
- Some pneumatic setpoints change with oven temperature because of changes in column resistance and in gas viscosity. This may confuse users who observe pneumatics setpoints changing when their oven temperature changes. However, the flow condition in the column remains as specified by the column mode (constant flow or pressure, ramped flow or pressure) and the initial setpoint values.

Configure the carrier gas

The GC needs to know what carrier gas is being used.

Procedure: Configuring the carrier gas

1. Press [Config] [Front Inlet] or [Config] [Back Inlet].
2. Press [Mode/Type] to see the carrier gas menu.



3. Scroll to the gas you will use. Press [Enter].

This completes carrier gas configuration. See the *Inlets* volume for more detail.

Select a column mode

The flow modes

Flow rates are corrected to NTP (normal temperature and pressure, 25°C and 1 atmosphere. For more detail, see pages 45 and 79.

- **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.

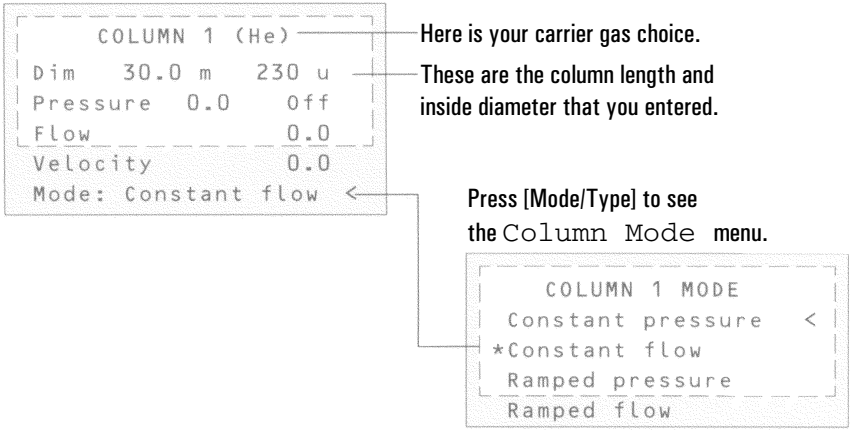
The pressure modes

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 changes, the gauge pressure does not change but the mass flow rate does.
- **Ramped pressure**—Increases the column head gauge pressure during the run according to a program you enter. A column pressure profile can have up to three ramps, each consisting of a programmed increase followed by a hold period.

Procedure: Selecting a column mode

1. Press [Col 1] or [Col 2].
2. Scroll to the `Mode` line.
3. Press [Mode/Type] to see the column mode menu.



4. Scroll to the column mode you want. Press [Enter].

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).

Enter the initial flow or pressure or average linear velocity

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.

See the following table for recommended flows for various column diameters. These are close to optimum for a wide variety of components.

Table 8 Column Size and Carrier Gas Flow Rate

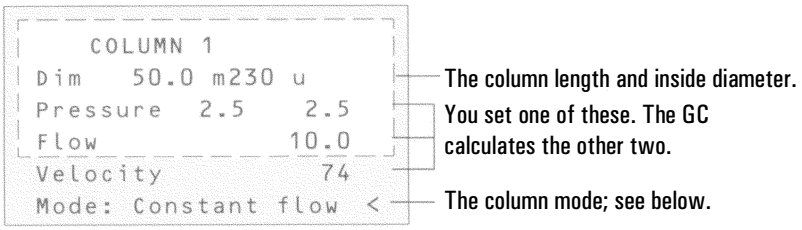
		Carrier gas flow rate	
Column type	**Column size**	**Hydrogen**	**Helium**
Packed	1/8-inch		30
	1/4-inch		60
Capillary	50 μm id	0.5	0.4
	100 μm id	1.0	0.8
	200 μm id	2.0	1.6
	250 μm id	2.5	2.0
	320 μm id	3.2	2.6
	530 μm id	5.3	4.2

These flow rates, in mL/min at normal temperature and pressure (25°C and 1 atm) are recommended for all column temperatures.

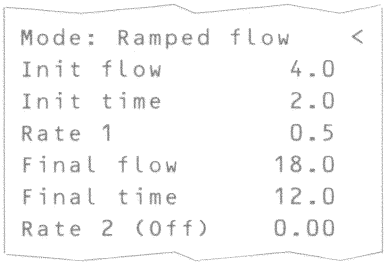
For capillary columns, flow rates are proportional to column diameter and are 20% lower for helium than for hydrogen.

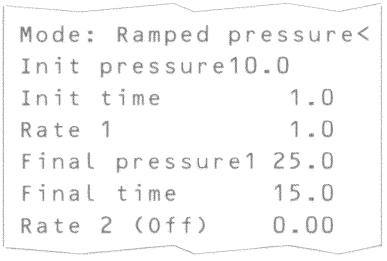
Procedure: Setting initial flow or pressure or average linear velocity

1. Press [Col 1] or [Col 2].



The control table will have one of these, depending on the column mode selected:





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 control table contains entries for setting up a ramp program.

You begin with an initial value, either `Init Pres` or `Init Flow`, and an `Init time`. At the end of that time, `Rate 1` begins and runs until it reaches `Final pres` (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`.

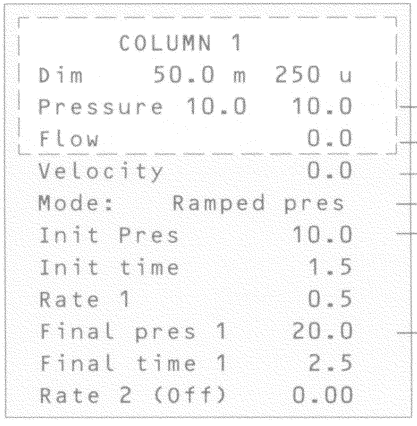
The program ends when it reaches a `Rate` that is set to 0 (off).

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.

Procedure: Programming column pressure or flow

1. Press [Col 1] or [Col 2].



2. Scroll to `Init Pres` (or `Init flow`). Type the desired value and press [Enter].
3. Similarly, enter a value for `Init time`. This completes the initial (constant pressure) 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 pres 1` (or `Final flow 1`) and `Final time 1`. This completes the first ramp.
7. To enter a second (or third) ramp, scroll to the appropriate `Rate` line and repeat steps 5 and 6.

SUMMARY

Note that, except when setting the carrier gas type, we have been concerned only with the Column tables. This is fundamental to successful operation of the 6890 with EPC inlets.

FIRST: Set up the column
THEN: Set up the rest of the instrument

Enter the rest of the inlet parameters

The split/splitless inlet has four operating modes:

- Split—The sample is divided between the column and a vent flow.
- Splitless—The sample is not divided. Most of it enters the column. A small amount is purged from the inlet to avoid excessive peak broadening and solvent tailing.
- Pulsed split—Similar to split, except that the inlet pressure is raised before and during injection and returned to normal at a user-specified time. Total flow is increased as well so that the split ratio does not change. This special kind of "programming" is independent of the three-ramp flow or pressure programming.
- Pulsed splitless—Like pulsed split, but splitless.

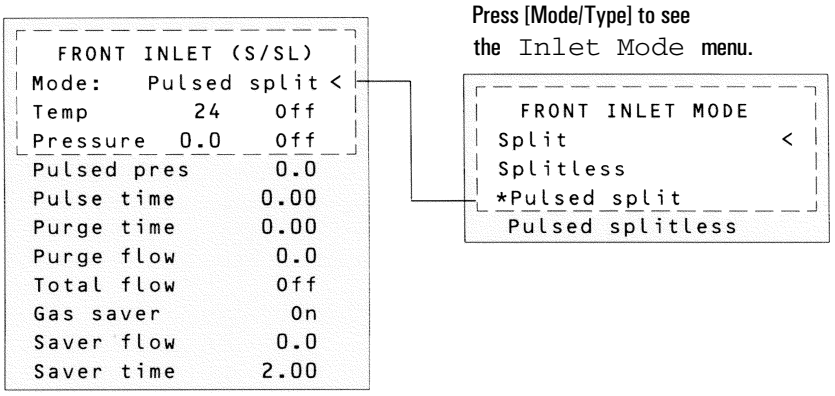
The split/splitless inlet has a gas saver feature. This reduces the flow of carrier into the inlet and out the split vent after the injection is complete. It does not alter the flow through the column.

The septum purge flow is set automatically on all EPC inlets.

And, of course, the inlet temperature can be controlled.

Procedure: Setting the rest of the inlet parameters

1. Press [Front Inlet] or [Back Inlet].
2. Scroll to the `Mode` line.
3. Press [Mode/Type] to see the inlet mode menu.



4. Move the cursor to the inlet mode you want. Press [Enter]. The inlet table may change, depending on your choice. The possibilities for the defined-column case are shown on the next page.
5. Scroll to `Temp`. Type the temperature you want. Press [Enter].
6. If you selected `Split`, and if the column is defined, you may enter the split ratio directly.

For details on the inlet parameters, see the *Inlets* volume.

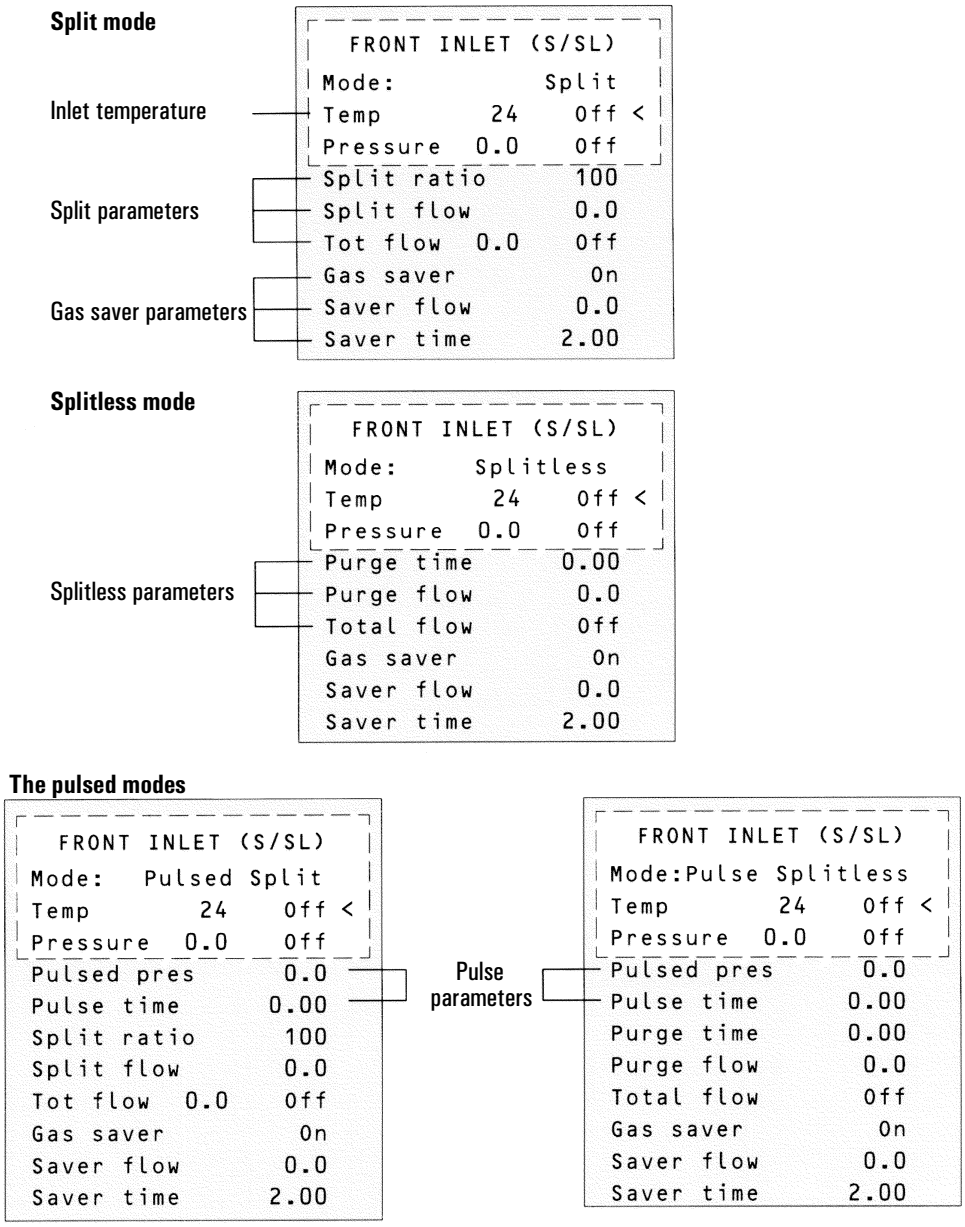


Figure 6 Control tables for the Split/Splitless inlet

Detectors

Although EPC detectors have built-in pressure regulation, you still need external regulators so that the electronic control has a stable gas supply to work with.

You may want to use traps to remove contaminants from the gas supply. If so, they should be as close to the back of the GC as possible.

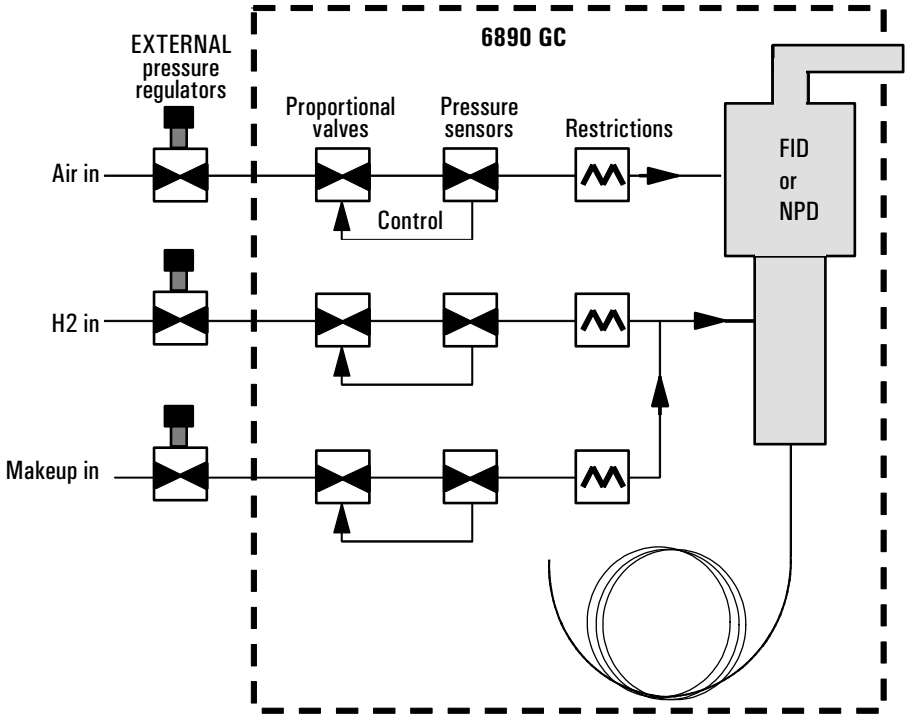


Figure 7 Internal/external plumbing: FID and NPD *with* EPC
For more detail, see the *Detectors* volume.

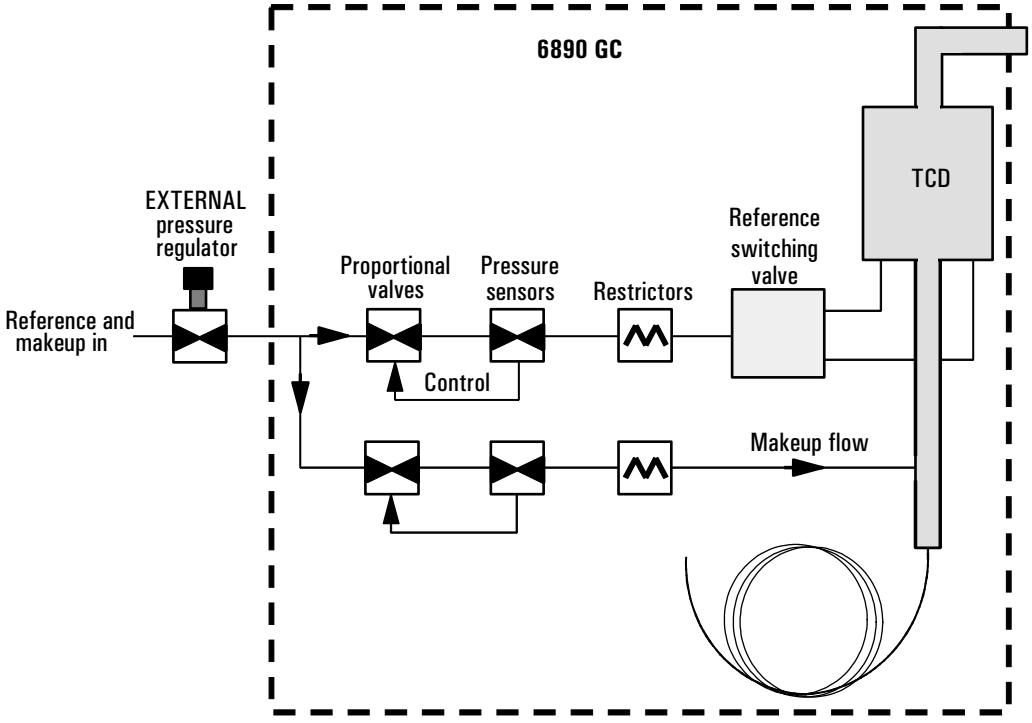


Figure 8 Internal/external plumbing: TCD *with* EPC
For more detail, see the *Detectors* volume.

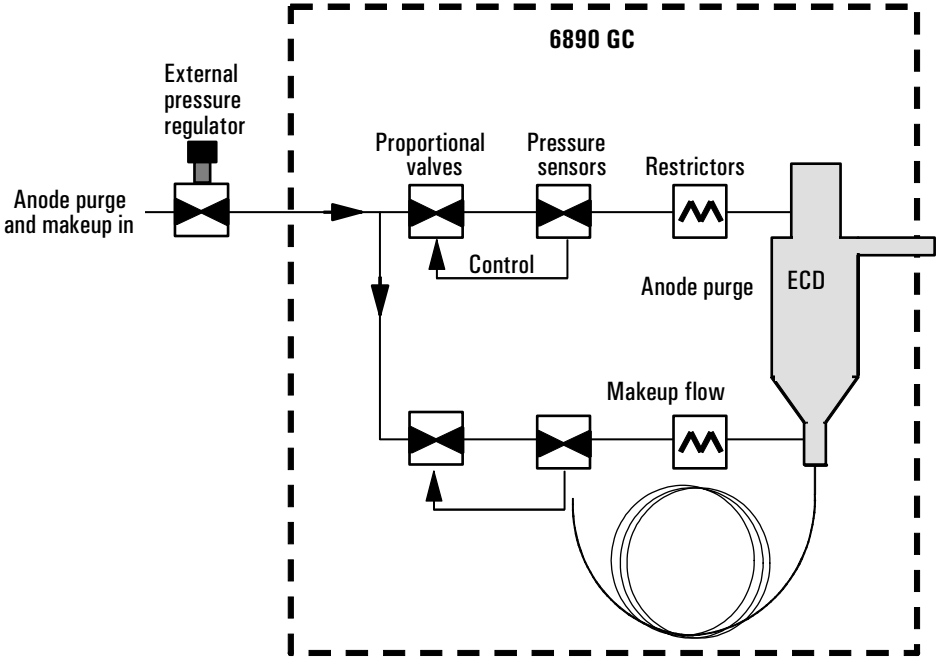


Figure 9 Internal/external plumbing: ECD *with* EPC
For more detail, see the *Detectors* volume.

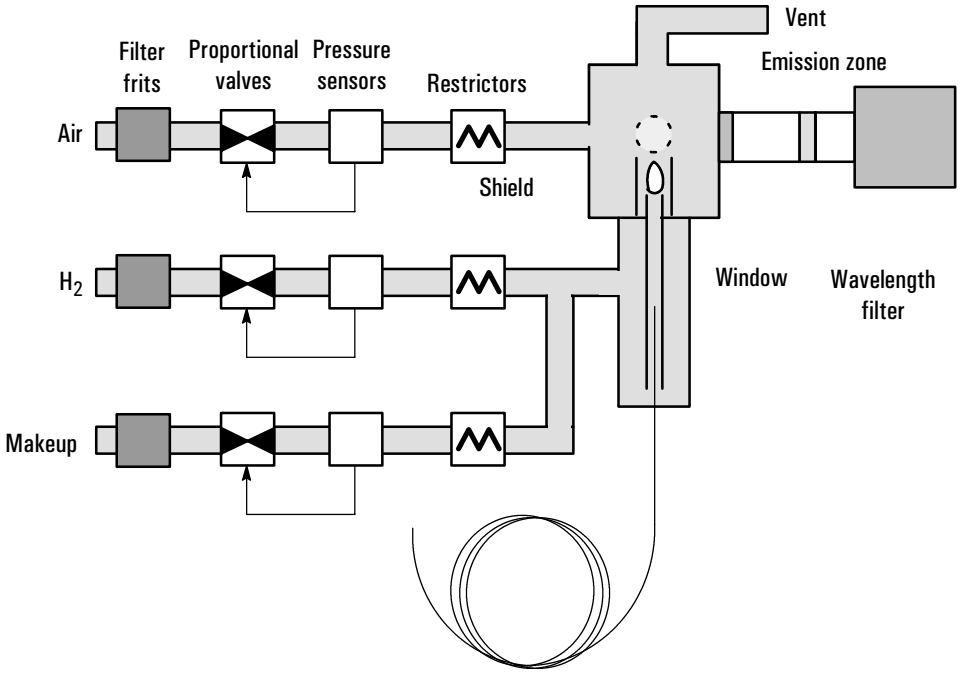


Figure 10 Internal/external plumbing: FPD *with* EPC
For more details, see the *Detectors* volume.

Gas configuration

The GC assumes that hydrogen is plumbed to the FID, FPD and NPD H_2 locations and that air is plumbed to the air locations (see the labels on the EPC gas modules).

Some locations allow a choice of gases. In these cases (mostly makeup gases), you must identify the gas using the [Config] process.

Makeup gas

You can select either constant makeup flow or constant (makeup + column) flow. See the *Detectors* volume for details, since they vary with the detector type.

Auxiliary channels

Three additional auxiliary pressure control channels are available as an option. They are controlled by the Aux 3, Aux 4, and Aux 5 tables (Aux 1 and 2 are heater controls).

If an auxiliary channel is specified as the `Inlet` during column configuration, the channel allows run time programming as well as three-ramp programming. The most common case of this is when a gas sampling valve is used.

The auxiliary channels are controlled by a pressure setpoint. To work properly, there must be adequate flow resistance downstream of the pressure sensor. The auxiliary channel pneumatics manifold provides a frit-type restrictor for each channel. Four frits are available:

Frit marking	Flow resistance	Part no.
Blue Dot	High	19234-60660
Red Dot	Medium	19231-60770
Brown Dot	Low	19231-60610
None (brass tube)	Zero	G1570-20540

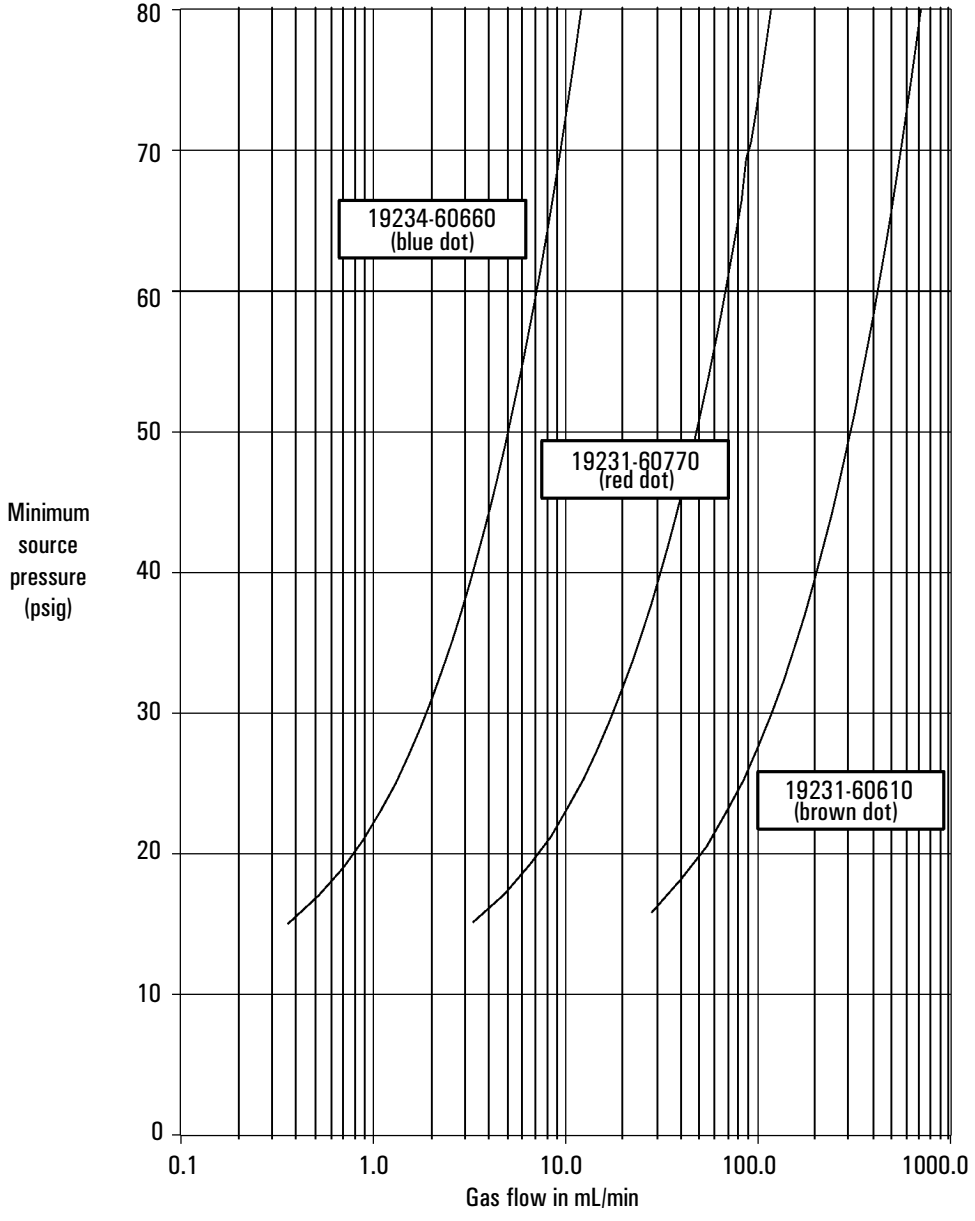
The Red Dot frit is in all three channels when the instrument is shipped.

The figures on the next two pages show approximate pressure/flow relationships for the three Dot frits, assuming there is no significant additional resistance downstream of the frits.

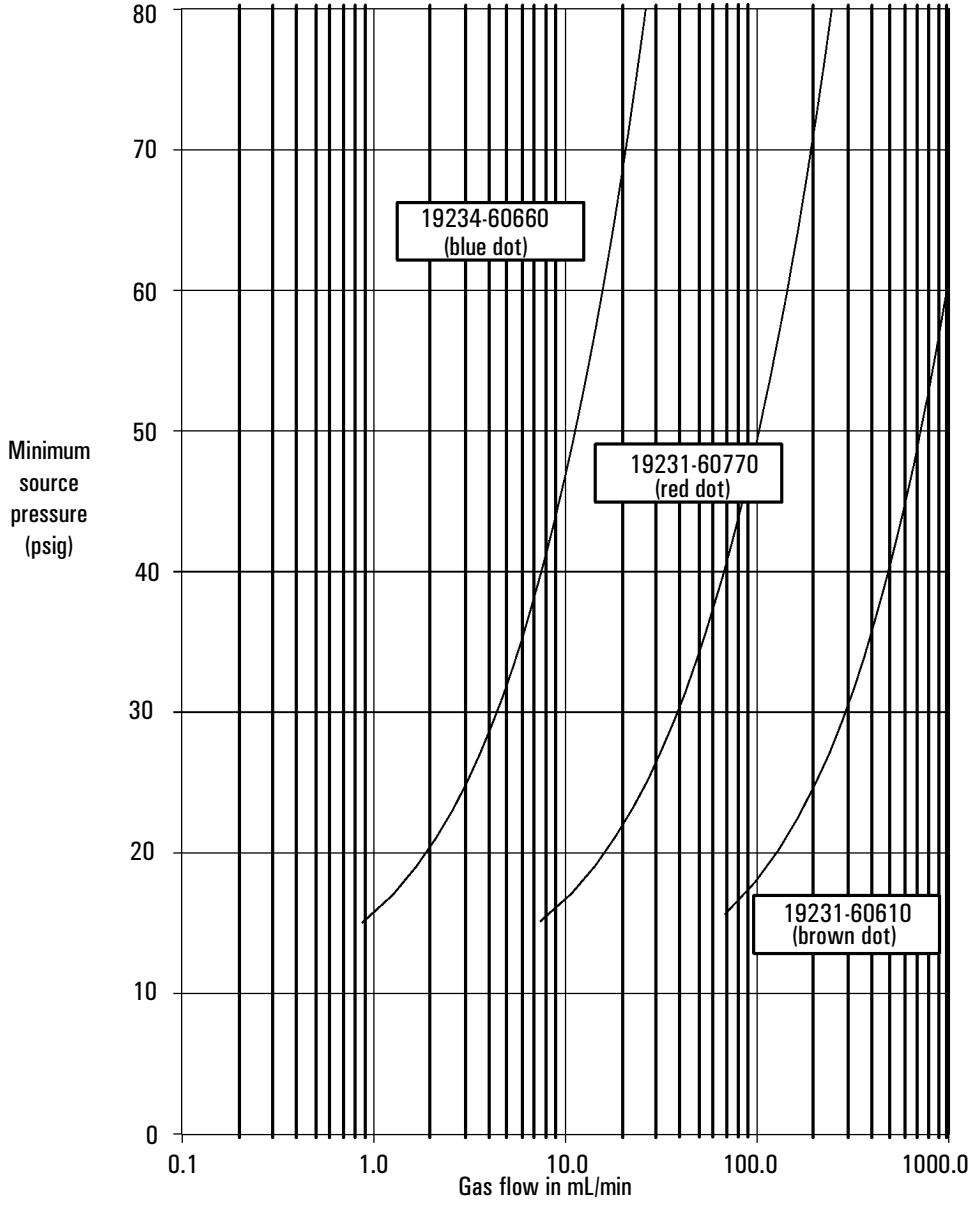
If the Zero resistance frit is installed, the user must provide flow resistance downstream and generate the pressure/flow relationships.

WARNING When hydrogen is used, dangerously high flows are possible if insufficient flow resistance is provided downstream of the supply tube. Always use either the High (Blue Dot) or Medium (Red Dot) frit with hydrogen.

Pressure requirements for AUX EPC flow restrictors
with air, nitrogen, or helium
(ambient conditions: 25°C, 14.7 psia)

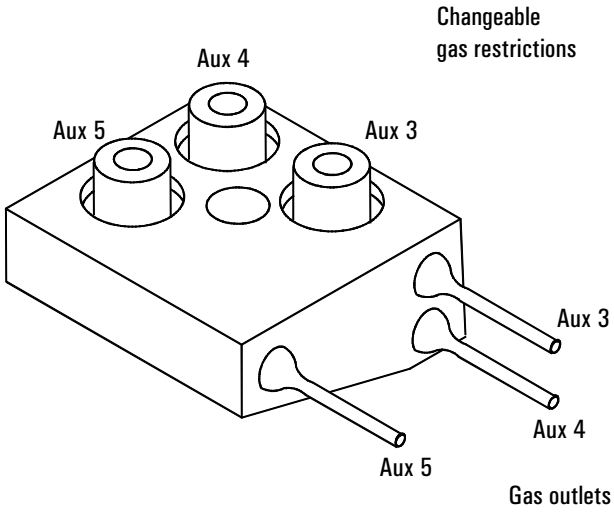


Pressure requirements for AUX EPC flow restrictors
with hydogen
(ambient conditions: 25°C, 14.7 psia)



Procedure: Changing an auxiliary channel frit

1. Locate the block that connects the three gas outlet tubes for the auxiliary channels to the pneumatics module.
2. Remove the screw that holds the block to the pneumatics module. Pull the block free of the module and rotate it so that the frits are on top.



3. Pull the frit to be changed out of the block. Also remove the O-ring that seals it.
4. Place an O-ring on the new frit. Place the O-ring/frit combination in the block.
5. Reconnect the block to the pneumatics module. Tighten the screw firmly.

Maintaining EPC calibration

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

Flow sensors

The split/splitless and purged packed inlet modules use flow sensors. If the `Auto flow zero` feature (see page 33) is on, they are zeroed automatically after each run. This is the recommended way. They can also be zeroed manually—see the next page.

Pressure sensors

All EPC control modules use pressure sensors. They can be zeroed as a group or individually. There is no automatic zero for pressure sensors.

Zero conditions

IMPORTANT Flow sensors are zeroed with the carrier gas connected and flowing. Pressure sensors are zeroed with the supply gas line disconnected from the gas control module.

Table 9 Flow and Pressure Sensor Zero Intervals

Sensor type	Module type	Zero interval
Flow	All	Use `Auto flow zero`
Pressure	Inlets	
	Packed columns	Every 12 months
	Small capillary columns (id 320 μm or less)	Every 12 months
	Large capillary columns (id > 320 μm)	At 3 months, at 6 months, then every 12 months
	Auxiliary channels	Every 12 months
	Detector gases	Every 12 months

Procedure: Zeroing flow and pressure sensors

To zero a flow or pressure sensor in a specific module

1. Press [Options], scroll to Calibration, and press [Enter]
2. Scroll to the module to be zeroed and press [Enter]

```
CALIB FRONT DETECTOR
H2 zero          0.0 <
H2 flow          0.0
Oxidizer zero    0.0
Oxidizer flow    0.0
Makeup zero      0.0
Makeup flow      0.0
Factory calibration
```

Note: After zeroing or flow calibration, the `Factory Calibration` line is replaced by the time and date of the recalibration.
To restore the Factory Calibration, select the time and date line and press [Delete]. This destroys the user calibration.

3. Scroll to a zero line and press [Info]

```
CAL FLOW ZERO INFO
 Press ON to zero.
 Will momentarily
disrupt inlet flow.
```

or

```
CAL PRES ZERO INFO
 Press ON to zero
  after applied
   pressure = 0
```

4. To cancel, press [Clear]
5. To zero flow, verify that the carrier gas is connected and is turned on.
6. Press [On] to zero or [Clear] to cancel.

4. To cancel, press [Clear]
5. To zero pressure, verify that the supply gas line is not connected.
6. Press [On] to zero or [Clear] to cancel.

To zero all pressure sensors in all modules

1. Press [Options], scroll to Diagnostics, and press [Enter]
2. Scroll to `Electronics` and press [Enter]
3. Scroll to `Pneumatics Board` and press [Enter]
4. Scroll to `Zero all p sensors` and press [Info]

```
ZERO P SENSORS INFO
 Press ON to zero
 all pres sensors,
when applied pres=0
```

5. To cancel, press [Clear]
6. To zero, verify that the supply gas lines are disconnected from all modules.
7. Press [On] to zero or [Clear] to cancel.

Part 2. NonEPC control

Control tables for nonEPC inlet and detector gases provide on/off control but do not control flow rates or pressures. These must be set manually and verified using a bubble meter or other flow meter. See page 76 for bubble meter operation.

Inlets

Pressure regulators, flow controllers, and other controls for nonEPC sample inlets are mounted in a module on the left side of the GC. See the *Inlets* volume for operating information.

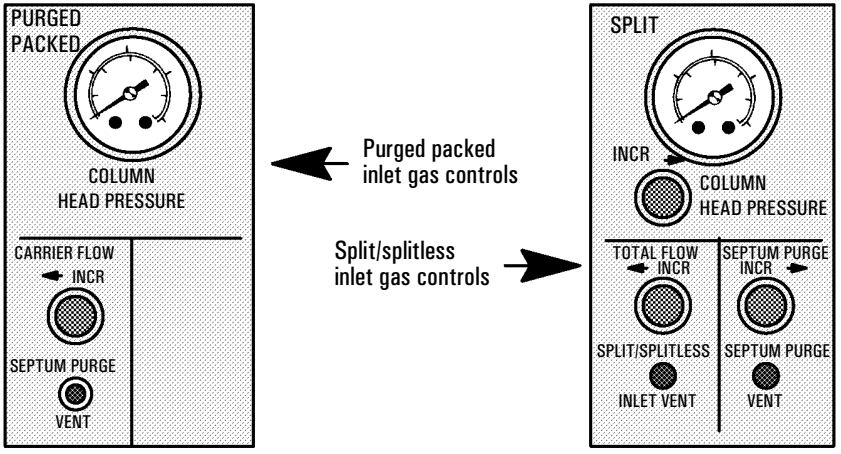


Figure 11 NonEPC inlet gas controls

Septum purge

Septum purge flow is set automatically on the nonEPC purged packed inlet; it can be measured at a vent on the front panel. Septum purge is a user adjustment on the nonEPC split/splitless inlet.

Detectors

NonEPC detectors have internal single-stage pressure regulators for some gas streams. You should still use external two-stage regulators.

You may also want to use traps to remove contaminants from the gas supply. If so, they should be as close to the back of the GC as possible.

Internal/external plumbing: FID and NPD *without* EPC

Hydrogen and air flows for these detectors are controlled by external pressure regulators and internal flow restrictors. Makeup gas is controlled by an internal pressure regulator located behind the detector positions. See the *Detectors* volume for flow rate details.

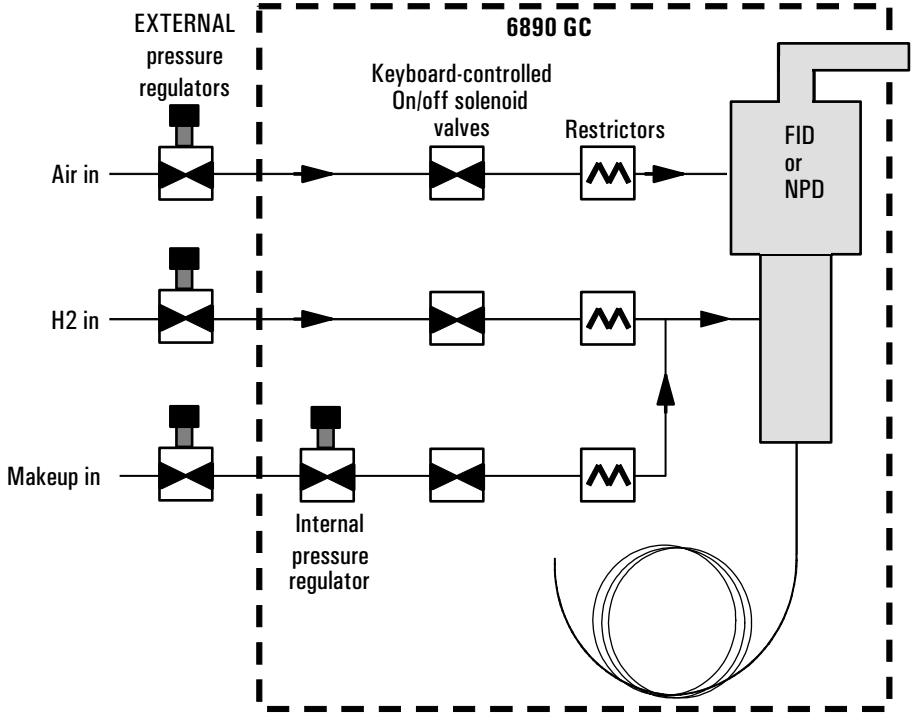


Figure 12 FID and NPD *without* EPC

Internal/external plumbing: TCD *without* EPC

This detector has two internal pressure regulators, one for reference gas and one for makeup gas, that are supplied from a single fitting. The pressure regulators are located behind the detector positions.

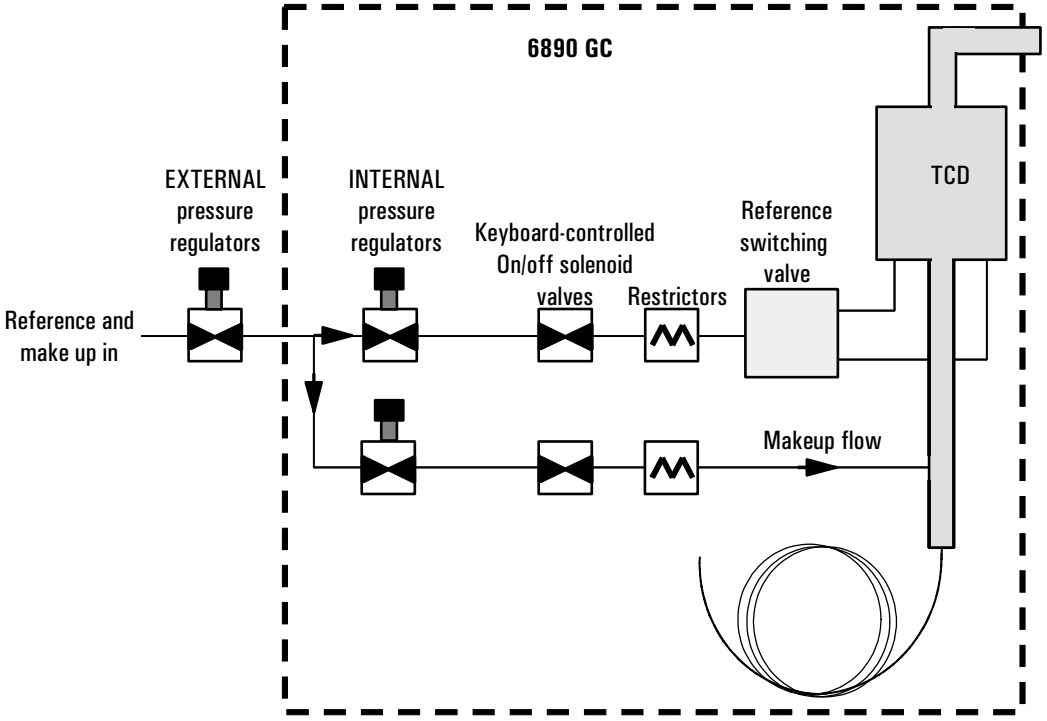


Figure 13 TCD *without* EPC
See the *Detectors* volume for flow rate details.

Internal/external plumbing: ECD *without* EPC

This detector has an internal pressure regulator that supplies both the anode purge gas and the makeup gas. The pressure regulator is located behind the detector positions.

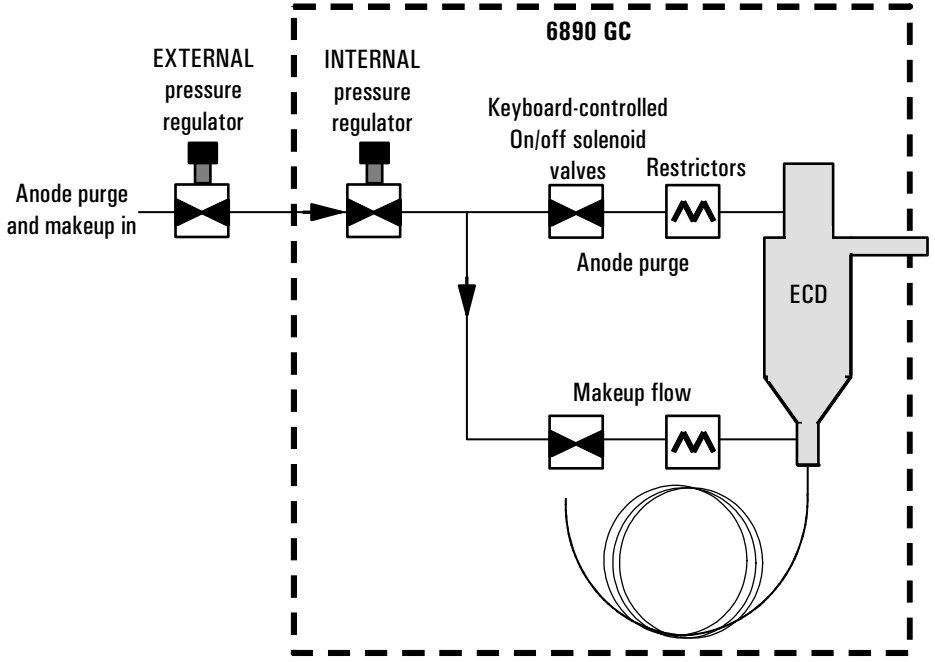


Figure 14 ECD *without* EPC
See the *Detectors* volume for flow rate details.

Part 3. Measuring flow rates

This section describes how to measure flow rates in the GC and how to convert the measurements to the conditions used by the GC. If your GC uses EPC, please note that the flow and pressure sensors in the GC are often more accurate than off the shelf, inexpensive flow meters. If you can establish a **calibrated** flow or pressure in the GC, a measurement that agrees with the GC within a few percent (after conversion to NTP; see page 79) should verify the GC's manifolds are operating properly and do not need replacement

Measuring flow rates with a bubble meter

A bubble flow meter is a very basic, reliable tool for measuring gas flow. It creates a bubble meniscus across a tube through which the gas is flowing. The meniscus acts as a barrier, and its motion reflects the speed of the gas through the tube. Most bubble flow meters have sections of different diameters so they can measure a wide range of flows conveniently.

A bubble flow meter with rate ranges of 1, 10, and 100 mL/min is suitable for measuring both low flow rates (such as carrier gases) and higher flow rates (such as air for an FID).

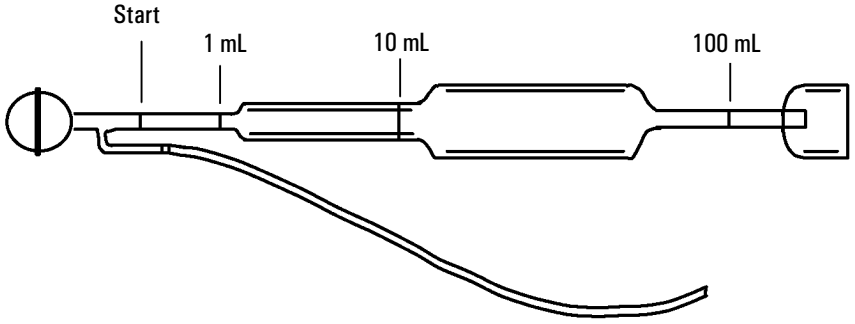
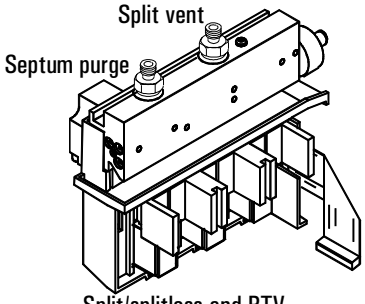


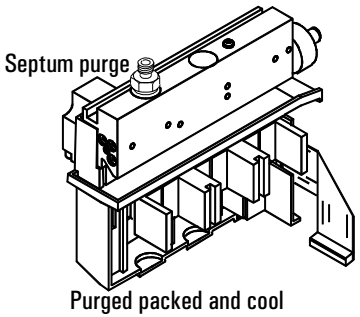
Figure 15 A three-volume bubble meter

Where to measure flows

EPC inlets—Septum purge and split vent flows exit through the pneumatic module at the top rear of the GC.



Split/splitless and PTV pneumatic module



Purged packed and cool on-column pneumatic module

EPC and nonEPC detectors—Measure all flows, including carrier, at the exit of the detector. Use the control tables to select one gas at a time.

NonEPC inlets—The flow vents are on the front panel. See page 72.

Adapters for measuring flow rates

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

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

Procedure: Measuring gas flows with a bubble meter

Tools:

- Bubble meter graduated at 1, 10, and 100 mL. Bulb half-filled with soapy water.
- Adapter for detector or vent
- GC internal stopwatch

WARNING Do not measure hydrogen together with air or oxygen. This can create explosive mixtures that may be ignited by the automatic ignitor.

To avoid this hazard:
Turn the automatic ignitor off before you begin.
Always measure gases separately.

1. Attach the inlet line of the bubble meter to the fitting where you will measure flow. Use the appropriate adapter, if needed.
2. Hold the bubble flow meter vertically—squeeze and release the bulb to create a meniscus in the bubble meter. Do this several times to wet the sides before taking measurements.
3. Press [Time] to see the stopwatch screen. Squeeze the bulb.
4. Press [Enter] to start the stopwatch when the meniscus passes the START (lowest) line in the bubble flow meter.
5. Press [Enter] again to stop the stopwatch when the meniscus passes the 1 mL, 10 mL, or 100 mL line.
6. Calculate the flow rate in mL/min from the 1/t value:
   - If you used the 1 mL line, the flow rate in mL/min = 1/t.
   - If you used the 10 mL line, the flow rate in mL/min = $10 \times 1/t$.
   - If you used the 100 mL line, the flow rate in mL/min = $100 \times 1/t$.
7. Press [Clear] to reset the stopwatch. Repeat the measurement at least once to verify the flow.

Interpreting flow meter measurements

Bubble meter measurements yield flow rates at the local temperature and local atmospheric pressure. Electronic flow meters may be calibrated for temperatures other than 25°C or for pressures other than 1 atm. However, the GC display shows values corrected to Normal Temperature and Pressure (NTP) conditions. If you do not correct your meter's flow rate to NTP, the readings will not agree with the GC.

To convert meter flow rate measurements to NTP (25°C and 1 atmosphere), you must know:

- The local atmospheric pressure or the electronic meter calibrated pressure
- The bubble meter temperature at the time of measurement or the electronic meter's calibration temperature.

The conversion is:

$$\textbf{Flow rate at NTP} = \frac{\textbf{Flow rate}_{\textbf{local}} \textbf{ x 298 x Pressure}_{\textbf{local}}}{\textbf{Temperature}_{\textbf{local}}}$$

where:

Flow rate at NTP	is the flow rate in mL/min corrected to Normal Temperature (25°) and Pressure (1 atmosphere)
$\text{Flow rate}_{\text{local}}$	is the flow rate in mL/min measured by the bubble meter
$\text{Temperature}_{\text{local}}$	is the temperature of the bubble meter at the time of measurement or the meter's calibration temperature. This number is in Kelvins (Kelvin = Centigrade + 273).
$\text{Pressure}_{\text{local}}$	is the local atmospheric pressure at the time of measurement or the meter's calibration temperature. This number is in atmospheres (1 atm = 1.01325 bars = 760 Torr = 760 mm Hg (at 0°C) = 101.325 kPa = 14.7 psi).

Part 4. Flow and pressure problems

A gas does not reach the setpoint pressure or flow

The gas cannot reach the pressure entered at the keyboard. If an EPC inlet does not reach its pressure setpoint it will shut down in an amount of time determined by the type of inlet:

Type of inlet	Time before shutdown
Purged packed, cool on-column	2 minutes
Split/splitless, PTV, volatiles inteface	5.5 minutes
Auxiliary	4 minutes

- The gas supply pressure is too low to reach the setpoint. The pressure at the supply should be at least 10 psi greater than the desired setpoint.
- A large leak is present somewhere in the system. Use an electronic leak detector to find leaks; correct them. Don't forget to check the column—a broken column is a very large leak.
- If you are using gas saver, be sure that the gas saver flow rate is high enough to maintain the highest column-head pressure used during a run.
- The flow is too low for the column in use.
- The column is plugged or misinstalled.
- The inlet or detector pressure sensor is not operating correctly. Contact your Agilent service representative.

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

- The split ratio is too low. Increase the amount of split flow.
- The inlet proportional control valve is stuck because of contamination or other fault. Contact your Agilent service representative.

If you are using a purged packed or cool on-column inlet:

- The inlet control valve is stuck closed because of contamination or other fault. Contact your Agilent service representative.

A gas exceeds the setpoint pressure or flow

- The pressure sensor for that device is not operating properly. Contact your Agilent service representative.

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

- The split ratio is too high. Decrease the split ratio.
- The proportional control valve is stuck closed. Contact your Agilent service representative.
- The trap on the split vent line is contaminated. Contact your Agilent service representative.

If you are using a purged packed or cool on-column inlet:

- The inlet proportional control valve is stuck open. Contact your Agilent service representative.

The inlet pressure or flow fluctuates

A fluctuation in inlet pressure will cause variations in the flow rate and retention times during a run.

- A small leak is present in the flow system. Use an electronic leak detector to find leaks; correct them. You should also check for leaks in the gas supply plumbing.
- Large restrictions, such as a blockage in a liner or the split vent trap, are present in the split/splitless or PTV inlets. Make sure that you are using the correct liner. Replace liners with large pressure drops caused by design or tight packaging. If the liner does not appear to be causing the problem, the split vent trap may be blocked. Contact your Agilent service representative.
- Extreme changes in room temperature during runs. Correct laboratory temperature problem or move the instrument to a more suitable location.

- Large volumes have been added to the system (this may occur if you are using a sampling valve). Decrease the sample volume. Use EPC inlets which correct for variations in temperature and pressure.

The measured flow is not equal to the displayed flow

You checked the flow at an inlet with a bubble flow meter, corrected the measurement to NTP conditions, and discovered that it does not match the calculated flow displayed on the GC.

- The column length, internal diameter, or gas type is configured incorrectly. Enter the correct information. Press [Config] [Column 1] or [Config] [Column 2] to enter the column specifications. Press [Config] [Front Inlet] or [Config] [Back Inlet] to enter the gas type. If a considerable amount has been cut off a capillary column, its actual length may no longer match its original. Configure the column with a new length.
- A new pressure setpoint was not entered after constant flow mode was selected. Enter a new pressure setpoint each time constant flow is turned on or off.
- A short (<15 m) 0.58 to 0.75 mm id WCOT column is being used with a split/splitless capillary inlet. The total flow controller is set for a high flow rate, which creates some pressure in the inlet and causes column flow even with a setpoint pressure of zero. (In these situations, an actual pressure may be shown on the display, even with a zero setpoint.) With short, 530 to 750 mm columns, keep the total flow rate as low as possible (for example, 20 to 30 mL/min). Install a longer column with higher resistance (for example, 15 to 30 m).
- The split vent line may be partly plugged, creating an actual inlet pressure higher than the setpoint pressure. Replace the split vent line.
- A Mass Selective Detector is in use and vacuum compensation is not selected.

4

The Column Oven

Controlling the oven. Making isothermal and programmed runs. Cryogenic operation.

The Column Oven

Oven capabilities

Capability	Range
Temperature range	–80°C (liquid N_2) or –60°C (CO_2) to the configured limit
Maximum temperature	450°C
Temperature programming	Up to six ramps
Maximum run time	999.99 minutes
Temperature ramp rates	0 to 120°C/min, depending on instrument configuration

The oven holds two inlets and two detectors, up to four valves in a heated box on top of the oven and two valves inside the oven, and either capillary or packed columns.

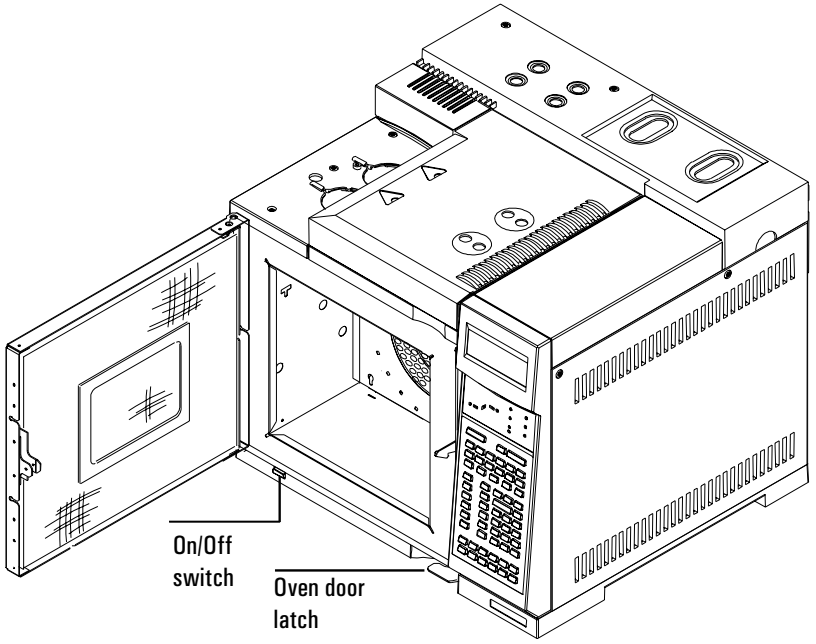


Figure 16 Column oven

Oven safety

For safety, opening the oven door turns off power to the oven heater, fan, and cryogenic valve (if installed) but maintains the setpoints in memory.

Closing the oven door returns the oven to normal operation.

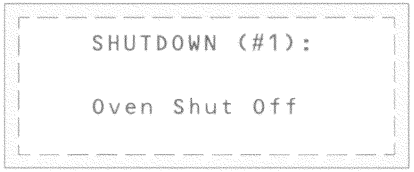
If the oven cannot attain or maintain an entered setpoint temperature during normal above-ambient operation, a problem is assumed and the oven is switched off.

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 control table blinks and the oven remains off until switched on again via [Oven] [On] or by editing the `Temp` setpoint in the oven control table.

When the oven shuts itself off, the following display appears:



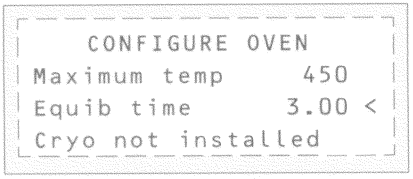
See chapter 12 in this volume for more information on shutdowns.

Configuring the oven

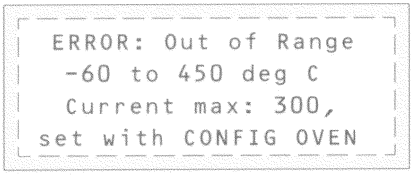
Oven configuration sets maximum temperature, equilibration time, and the cryogenic setpoints, if cryo is installed.

Press [Config] [Oven]

Maximum temp setpoint range: 70 to 450°C



`Maximum temp` Maximum allowable oven temperature setpoint. Some accessories, such as the valve box, valves and columns have specific temperature limits. When configuring `Maximum temp`, 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.

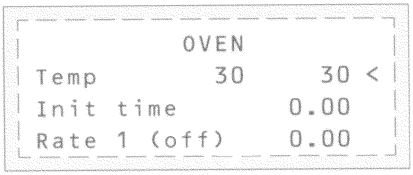


`Equib time` The time required for the oven temperature to equilibrate after temperature is modified. Equilibration time begins when the actual oven temperature comes within 1°C of the oven temperature setting. The `Equib time` setpoint can be 0 to 999.99 minutes.

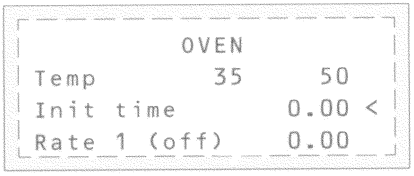
Procedure: Setting up an isothermal run

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

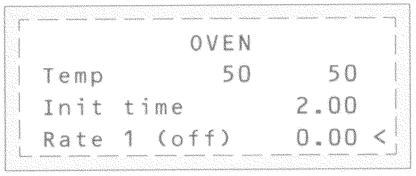
1. Press [Oven] to access the oven control table.



2. Enter the oven temperature for the isothermal run. Note that your actual and setpoint values will probably differ from the example.



3. Enter the number of minutes (`Init 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 0, enter zero for an isothermal run.

Making a temperature-programmed run

You can program the oven temperature from an initial temperature to a final temperature using up to six 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.

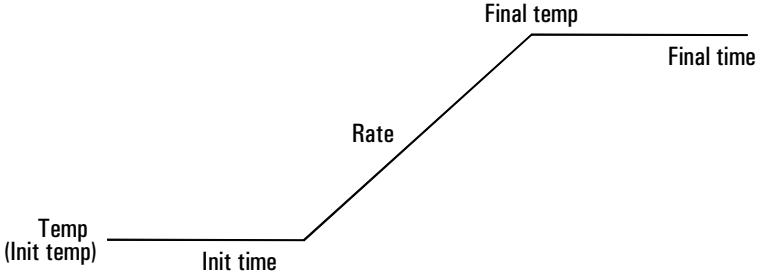


Figure 17 Single ramp

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.

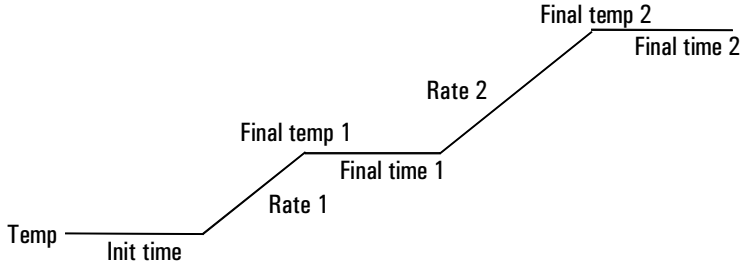


Figure 18 Multiple ramp

Oven temperature programming setpoints

`Temp` 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, `Temp` is reset to the value in `Init temp` and the oven returns to its starting temperature.

`Init temp` Equal to `Temp` except during a programmed run (`Init temp` remains constant; `Temp` changes as directed by the program). Changing `Init temp` changes the starting temperature for the next run. Changing `Temp` causes an immediate change but the value is not saved to the next run.

`Init 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 temp` 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.

Oven ramp rates

To use the fast oven ramp rates (a 240 V power option is required), your electric service must be able to supply ≥ 200V at ≥ 15 Amp.

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. The optional oven insert for fast chromatography (see page 94), increases oven ramp rates for the back column. Table 10 lists typical oven ramp rates.

Table 10 Oven Ramp Rates

	100/120 V oven ramp rate (°C/minute)		200/220/230/240 V oven ramp rate (°C/minute)	
Temperature range (°C)	Without insert	With optional insert	Without insert	With optional insert
50 to 70	75	120	120	120
70 to 115	45	95	95	120
115 to 175	40	65	65	110
175 to 300	30	45	45	80
300 to 450	20	35	35	65

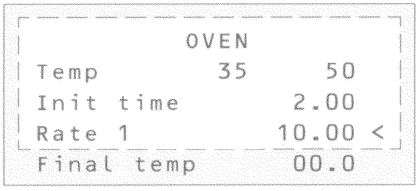
Procedure: Setting up a single-ramp program

This example increases the oven temperature from 50°C to 150°C at a rate of 10°C/minute.

1. Press [Oven] to access the oven control table.
2. Enter a starting temperature (`Temp`).

3. Enter the time (`Init time`) that you want the oven to stay at Temp.

4. Enter the rate (`Rate 1`) at which the oven temperature is to increase.



5. Enter the final temperature (`Final temp 1`).
6. Enter the time (`Final time 1`) the oven is to hold `Final temp 1`.

7. To end the oven ramp program after `Ramp 1`, set `Rate 2` to zero.

Procedure: Setting up a multiple-ramp program

Set up the first oven ramp as described on the preceding page.

In a multiple-ramp temperature program, the `Final time` for one ramp is also the `Init time` for the next ramp. Thus, there is only one `Init time` (before `Ramp 1`).

1. Enter the rate (`Rate 2`) at which you want the oven temperature to increase for the second oven ramp.
2. Enter the final temperature (`Final temp 2`).
3. Enter the number of minutes (`Final time 2`) that you want the oven to hold the final temperature.

4. To end the temperature program after `Ramp 2`, set `Rate 3` to zero.

To add additional oven ramps, repeat the steps described above.

Fast chromatography

The 6890 GC has several options for increasing throughput and cycle time. These are the fast heating oven (optional in some countries), the oven insert for fast chromatography (see page 94), and cryogenic cooling (see page 97).

Fast-heating oven

The fast-heating oven requires the following:

- A GC equipped with a fast-heating oven. A fast heating oven is standard with most 200–240 V power option GCs. A GC ordered for the United States, Canada, Switzerland, China, and Australia must be ordered with the fast heating oven option, or must be converted (contact Agilent service).
- The electric service must be capable of providing ≥ 200 V at ≥ 15 amperes.
- In the United States, the electric service must be 240 V.

Configuring the oven

GCs ordered with the fast-heating oven will be properly configured from the factory. If you convert a regular oven to a fast-heating oven, and have the correct electric service installed, you will need to configure the GC to use the new oven heater properly.

WARNING Do not perform this procedure unless your GC meets **all** of the criteria listed under "Fast-heating oven" above. Conversion from a regular to fast-heating oven (and the reverse) requires replacement of the oven heater, internal fuses, and power circuitry by qualified Agilent personnel. Changing the oven configuration at the keypad without making the proper hardware changes can damage your instrument and may present a fire hazard.

1. Press [Config], scroll to [Instrument], and press [Enter].
2. To change the oven type, press [.][.], then [Mode/Type].
3. Select the correct oven type (fast or regular), then press [Enter].

Using the oven insert for fast chromatography

The 6890 Oven Insert for Fast Chromatography reduces the oven volume so that the column and sample heat more quickly, yielding faster separation and faster chromatography. Furthermore, the smaller volume oven cools faster than a full-sized oven, reducing the overall analytical cycle time.

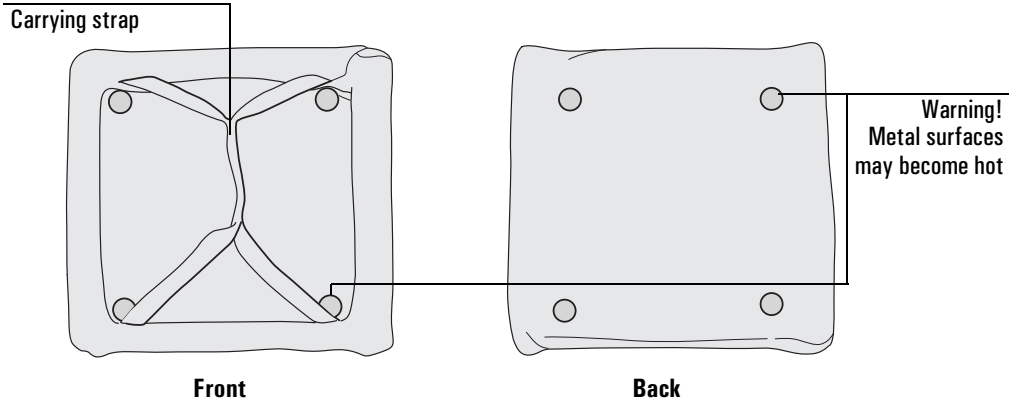


Figure 19 Oven insert

The oven insert is used with any inlet, column, and detector mounted in the **back** position. It is not compatible with any accessory which obstructs access to the back of the oven or which requires the use of either the front inlet or the front part of the oven.

To install the oven insert

1. Turn off your GC oven and allow it to cool.

Caution The cutouts in the interior oven walls may have sharp edges that can damage the oven insert fabric.

2. Orient the oven insert as shown in Figure 20 below. Tilt the upper edge away from you and insert it between the column fittings for the front inlet/detector and the back column hanger.

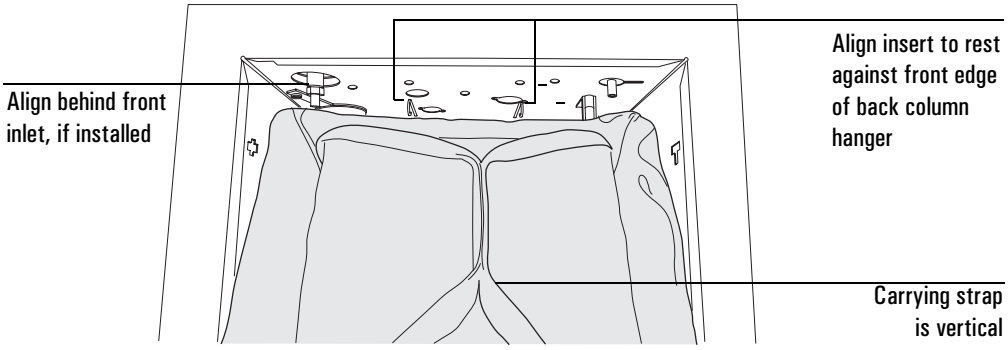


Figure 20 Installing the insert in front of the back column hanger

3. Push the bottom of the insert into place as shown in Figure 21. Keep the insert upright as shown.

4. If a TCD, ECD, µ-ECD, or NPD is installed in the front detector position, cap off the make-up adapter and establish a purge flow.

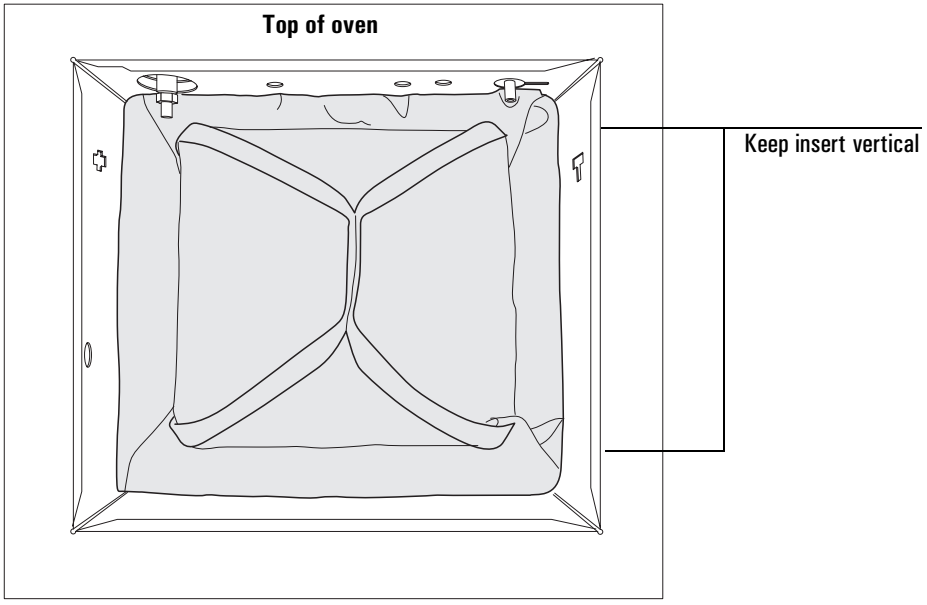


Figure 21 Oven insert installed in the oven

Removing the insert

1. To remove the insert, turn off the GC oven, inlet, and detector heated zones and allow them to cool.

WARNING The metal fasteners on the oven insert may remain hot even after the oven has cooled. Always handle the insert only by its carrying strap, or wear heat-resistant gloves.

2. Use the carry strap to remove the oven insert, pulling out the bottom edge first.

Cryogenic operation

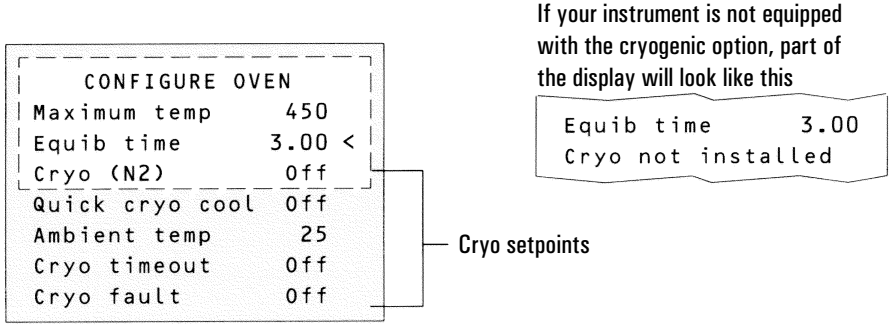
The cryogenic valve lets you operate the oven below ambient temperature. Minimum attainable oven temperature depends on the type of valve installed.

The GC senses the presence and type of cryogenic valve and disallows setpoints if no valve is installed. When cryogenic cooling is not needed or cryogenic coolant is not available, the cryogenic operation should be turned off. If this is not done, proper oven temperature control may not be possible, particularly at temperatures near ambient.

For information on installation and coolants see "Cryogenic Cooling Requirements" in the *Agilent 6890 Site Preparation and Installation Manual.*

Cryogenic control setpoints

All cryogenic setpoints are in the [Config] [Oven] control table.



`Cryo` [ON] enables cryogenic cooling, [OFF] disables it.

`Quick cryo cool` This feature is separate from `Cryo`. Quick cryo cool makes the oven cool faster after a run than it would without assistance. This feature is useful when maximum sample throughput is necessary, however it does use more coolant. Quick cryo cool turns off soon after the oven reaches its setpoint and Cryo takes over, if needed.

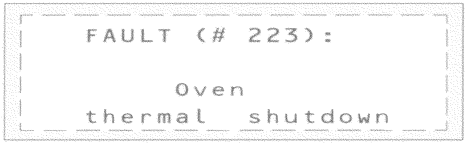
`Ambient temp` The temperature in the laboratory. This setpoint determines the temperature at which cryogenic cooling is enabled:

- Ambient temp + 25°C, for regular cryo operation
- Ambient temp + 45°C, for Quick Cryo Cool.

`Cryo timeout` Cryo timeout occurs, and the oven shuts off, when a run does not start within a specified time (10 to 120 minutes) after the oven equilibrates. Turning cryo timeout off disables this feature. We recommend that it be turned on because cryo timeout conserves coolant at the end of a sequence or if automation fails.

`Cryo fault` Shuts the oven down if it does not reach setpoint temperature after 16 minutes of continuous cryo operation. Note that this is the time to *reach* the setpoint, not the time to stabilize and become ready at the setpoint. For example, with a cool on-column inlet and cryo control in the oven track mode, it may take the oven 20 to 30 minutes to achieve readiness.

If the temperature goes below the minimum allowed temperature (–90°C for liquid nitrogen,–70°C for liquid CO_2), the oven will shut down with the following display:



5

Columns and Traps

Preparing and installing capillary columns, metal columns, and glass columns.

Columns and Traps

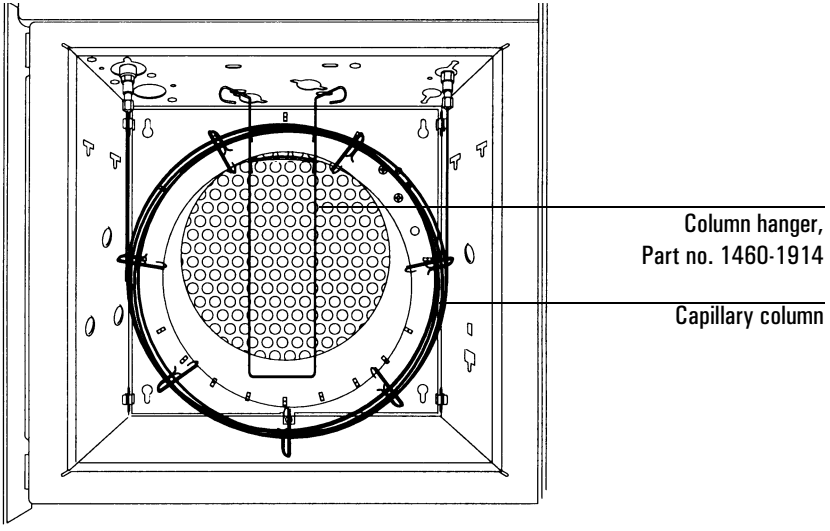
Part 1. Capillary columns

This section contains information on preparing and installing capillary columns in inlets and detectors. See page 122 for packed metal columns and page 131 for packed glass columns.

Column hanger

Agilent capillary columns are wound on wire frames that mount on a hanger connected to the top of the oven interior.

You can connect the column hanger in two positions. Use the position that best centers the column in the oven. Column ends should make smooth curves to the inlet and detector fittings. Do not let any section of the column come in contact with the oven surfaces.



Procedure: Preparing capillary columns

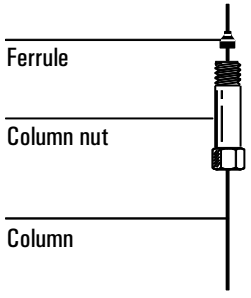
You must prepare your capillary column before installation. Proper preparation assures that the column end has no burrs or jagged edges and is not contaminated with graphite or other material.

WARNING Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.

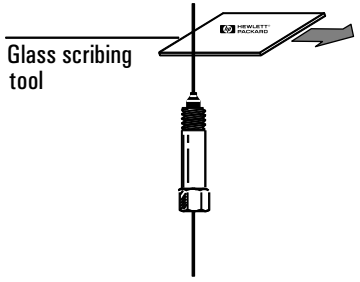
Materials required

Column nut and ferrule
Capillary column
Column cutter
Magnifying loop
Isopropanol
Tissue

1. Place a capillary column nut and ferrule on the column.

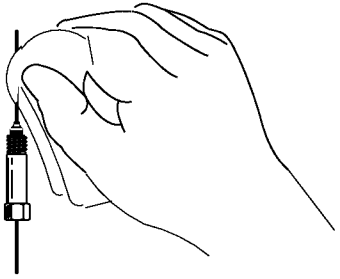


2. Score the column using a glass scribing tool. The score must be square to ensure a clean break.



3. Break off the column end by supporting it against the column cutter opposite the scribe. Inspect the end with a magnifying glass to make certain there are no burrs or jagged edges.

4. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.



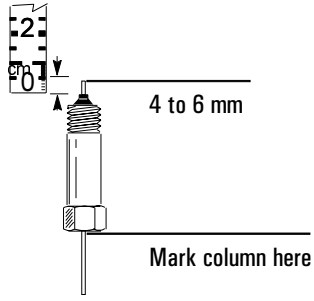
Procedure: Installing capillary columns in the split/splitless inlet

Before installing the column, be sure you have the correct glass liner installed. Instructions on choosing and installing liners are in the *Inlets* volume.

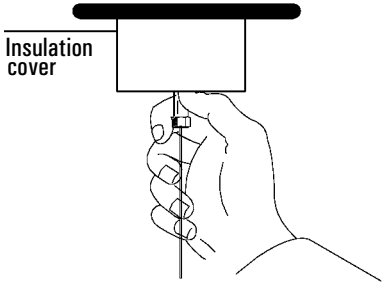
Materials required

Column nut and ferrule
Column cutter
Typewriter correction fluid
1/4-inch wrench
Metric ruler

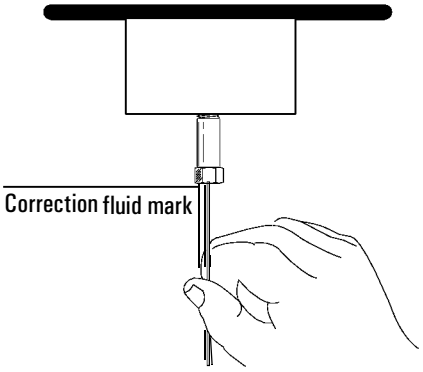
1. Prepare the column. See page 101 for instructions.
2. Position the column so it extends 4 to 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



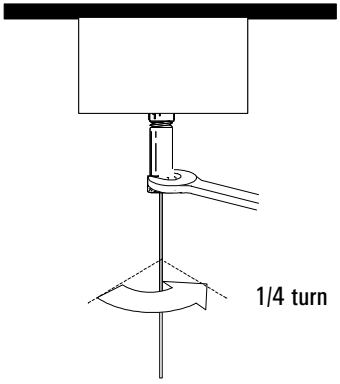
3. Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut until it starts to grab the column.



4. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.



5. Tighten the column nut an additional 1/4 to 1/2 turn so that the column cannot be pulled from the fitting with gentle pressure.



6. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperature. Allow them to cool, and then retighten the fittings.

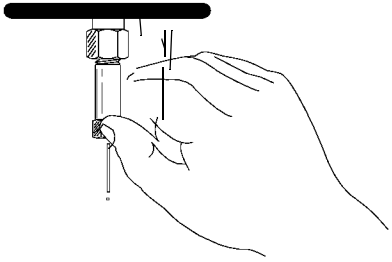
Procedure: Installing capillary columns in the cool on-column inlet

Before installing the column, be certain you have the correct hardware installed for the column and type of injection you are doing. See the *Inlets* volume for detailed information.

Materials required

Column nut and ferrule
Column cutter
1/4-inch wrench

1. Prepare the column. See page 101 for instructions.
2. Gently insert the column into the inlet until it bottoms. Insert the column nut into the inlet fitting and tighten the nut finger tight.



3. Tighten an additional 1/4-turn with a wrench or until the column does not move.
4. If you are using an automatic injection system with 250 µm or 320 µm columns, verify the installation by pushing the syringe manually into the inlet.
5. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

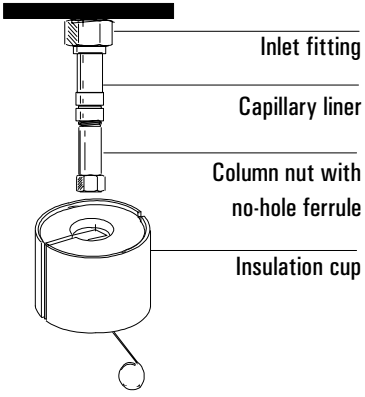
Procedure: Installing capillary columns in the purged packed inlet

Before installing a column in this inlet, be sure you have a capillary liner and glass insert installed. Instructions on choosing and installing this hardware are in the *Inlets* volume. If your insulation cup is not installed, begin with Step 1. Otherwise, begin with Step 4.

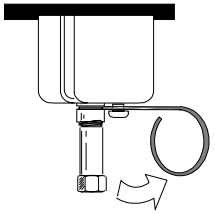
Materials required

Column nut and ferrule
Column cutter
Typewriter correction fluid
1/4-inch wrench
Metric ruler
Insulation cup
No-hole ferrule to use as a plug when installing the insulation cup

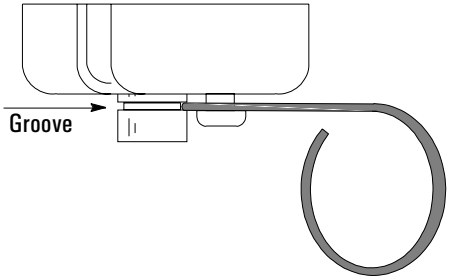
1. Install a plug in the inlet fitting.



2. Install the insulation cup, if needed. Push the cup spring to the right. Slide the cup over the inlet fitting so that the insulation at the top of the cup is flush against the oven roof.

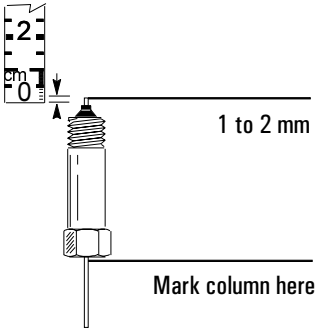


3. Place the spring into the groove in the inlet liner. Remove the column nut and put the no-hole ferrule aside.

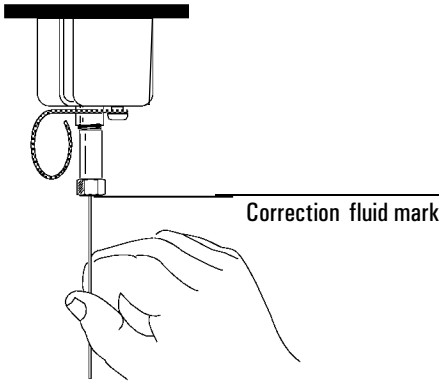


4. Prepare the column. See page 101 for instructions.

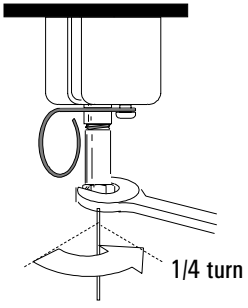
5. Position the column so it extends above the end of the column nut by 1 to 2 mm. Mark the column with typewriter correction fluid at a point even with the column nut.



6. Push the column up 1 cm and guide it into the inlet liner. Slide the nut and ferrule up the column to the inlet liner. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Finger tighten the column nut until it starts to grab the column.



7. Tighten the column nut an additional 1/4 to 1/2 turn so that the column cannot be pulled from the fitting when gentle pressure is applied.



8. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow these to cool, and then retighten the fittings.

Procedure: Installing capillary columns in the PTV inlet and Volatiles Interface

The column installation procedures for these two inlets are unique to them. Details are in the PTV and Volatiles Interface chapters of the *Inlets* volume.

Procedure: Installing capillary columns in NPD and FID detectors

Be sure you have the correct jet installed in your detector before installing a column. Details about choosing and installing detector jets are later in this chapter.

There are two types of NPD/FID detector fittings:

- *Adaptable*– for use with both packed and capillary columns
- *Capillary optimized*– for use with capillary columns only. If your adaptable fitting does not have a capillary adapter installed, begin with step 1. If you have a capillary optimized fitting or if the capillary adapter is already installed in your adaptable fitting, begin with step 5.

Materials required

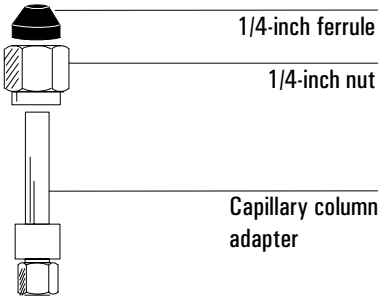
Both fitting types:

Column nut and ferrule
Column cutter
1/4-inch wrench
Metric ruler
Typewriter correction fluid

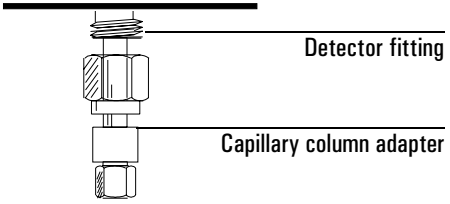
For adaptable fitting only:

1/4-inch nut and ferrule
Capillary column adapter
9/16-inch wrench

1. Assemble a brass nut and graphite/Vespel ferrule onto the adapter.

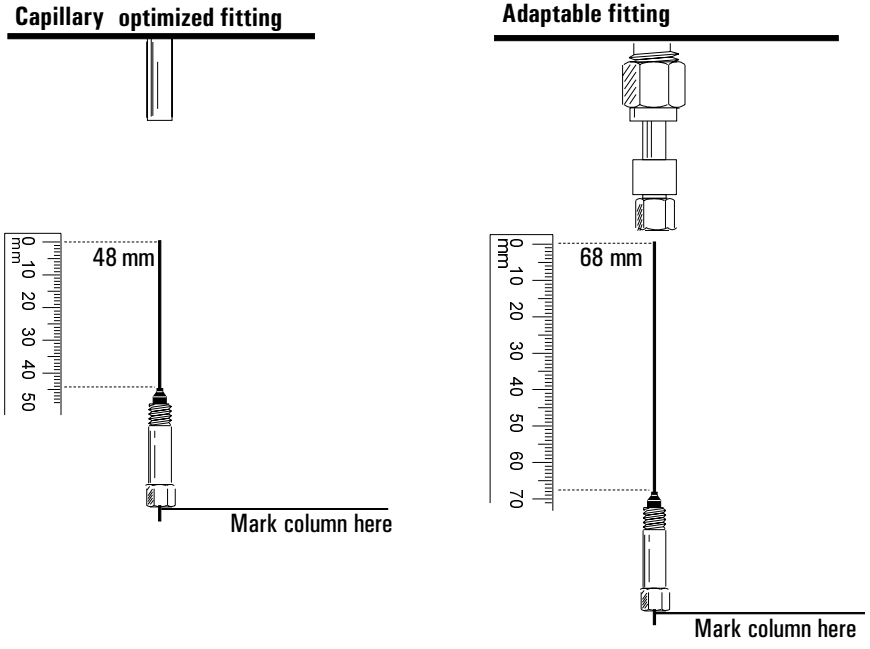


2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.



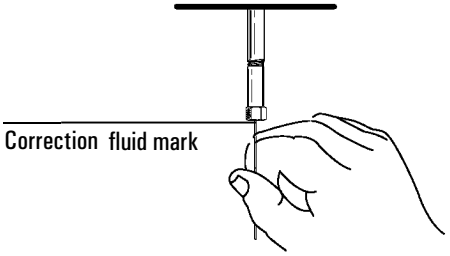
3. Prepare the column (see page 101 for instructions). If the column id is greater than 100 µm, follow steps 7 to 9. If the column id is less than 100 µm, follow steps 4 to 6, then follow steps 8 and 9.

4. If your column inside diameter is less than 100 µm:
   Position the column so it extends above the ferrule by 48 mm (*capillary optimized* fitting) or 68 mm (*adaptable* fitting). Mark the column with typewriter correction fluid even with the column nut.



5. Insert the column in the detector. Slide the nut and ferrule up the column to the detector base. Finger tighten the column nut until it starts to grab the column.

6. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Proceed to step 8.



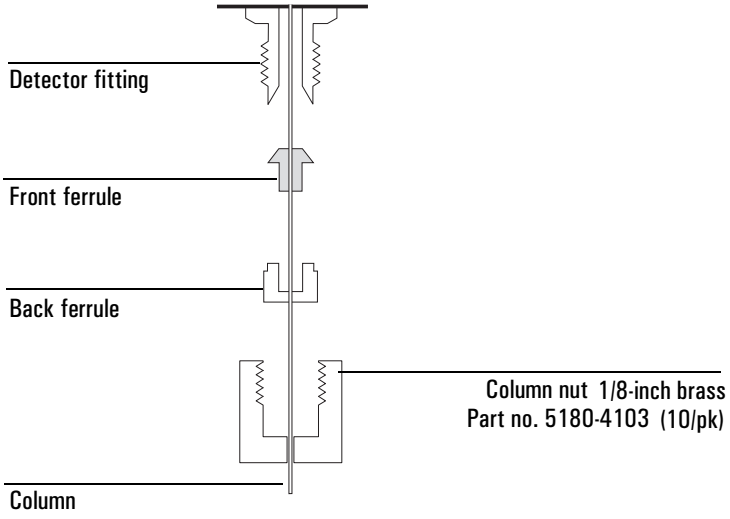
7. Gently insert the column into the detector until it bottoms; do not attempt to force it further.
8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

Procedure: Installing capillary columns in the TCD

Materials required

Capillary column adapter
Column nut and ferrule set
Column cutter
Wrenches

1. Assemble the ferrules and a 1/8-inch brass nut on the column as shown.



   See Table 11 for the proper ferrules. Trim off a short piece of column to remove any ferrule fragments inside the column.

2. Insert the column into the detector until it bottoms. Do not attempt to force it.

3. Slide the column nut and ferrule up the column to the detector and tighten the nut finger tight.

4. Pull the column out 1 mm. Use a wrench to tighten the nut an additional 1/4 turn. The column should not move.

Table 11 Ferrules for the TCD detector

Column outside diameter	Back ferrule	Front ferrule
0.8 mm	G1530-80400	G1530-80410
0.53 mm	G1530-80400	G1530-80420
0.45 mm	G1530-80400	G1530-80430
No-hole ferrule	G1530-80400	G1530-80440

Procedure: Installing capillary columns in the ECD and μ-ECD

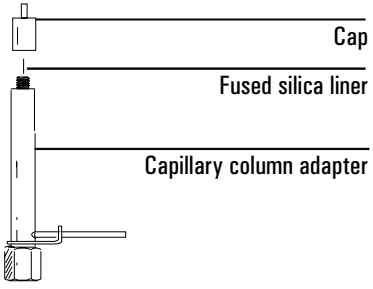
Both detectors are shipped with a capillary column adapter installed. If it has been removed, you must replace it before installing a capillary column.

There are two kinds of adapter liner. The straight liner is a simple tube and is a light brown color. The indented liner is necked down near one end and is clear. The μ-ECD requires the indented liner.

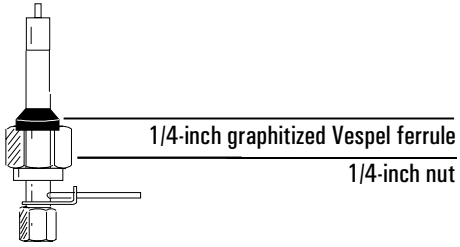
Materials required

Capillary column adapter
Fused silica liner, straight or indented (required for μ-ECD)
1/4-inch nut and 1/4-inch graphitized Vespel ferrule
Column nut and ferrule
Column cutter
1/4-inch and 9/16-inch wrenches

1. Remove the adapter cap and check the liner. Replace it if it is broken and reinstall the cap. If you use an indented liner, the indentation must be at the cap end of the adapter.



2. Install a 1/4-inch nut and graphitized-Vespel ferrule on the adapter.

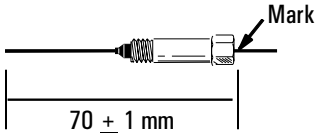


3. Prepare the column. See page 101 for instructions.

4. If you are using an indented liner (required for μ-ECD):

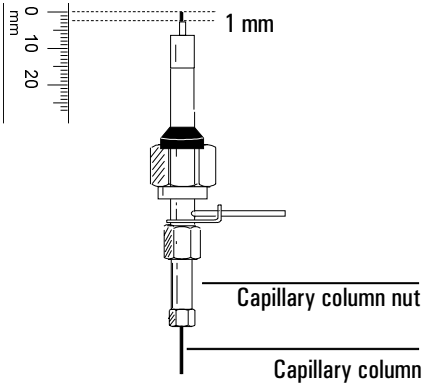
   If the column id is 200 μm or more, push the column into the adapter until it stops at the indentation. Pull it back 1 to 2 mm and tighten the column nut firmly.

   If the id is less than 200 μm, mark the column 70±1 mm from the end. Insert column and nut into the adapter with the mark at the rear of the column nut, and tighten the column nut firmly.

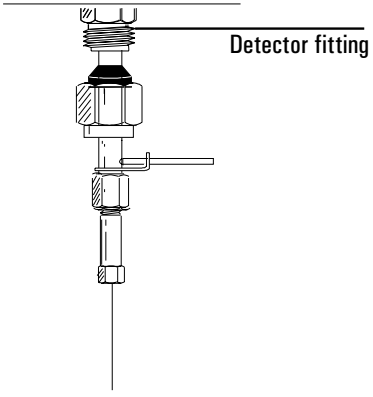


5. If you are using a straight liner:

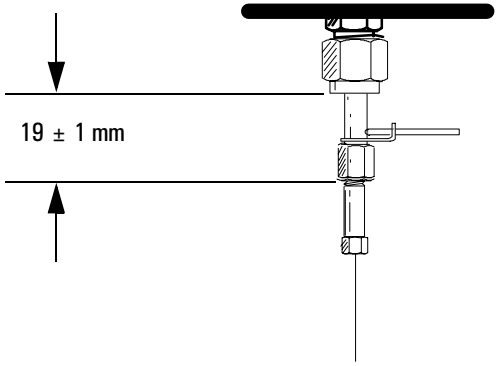
   Insert the column in the ECD adapter so that 1 mm extends above the cap extension. Tighten the capillary column nut firmly.



6. Slowly install the adapter straight into the detector fitting. Make sure that the adapter is seated all the way into the detector fitting—jiggle it if necessary. Be careful not to break the column end.



If the adapter is properly installed, the distance between the 1/4-inch nut and the bottom of the adapter will be 19 ± 1 mm. If it is 22–23 mm, reinstall the adapter into the detector fitting.

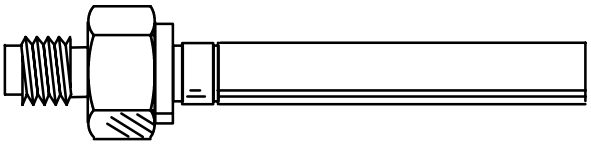


7. Slide the nut and ferrule up to the detector fitting and tighten the nut finger tight. Use a 9/16-inch wrench to tighten the nut an additional 1/4 turn.
8. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

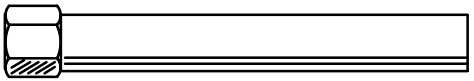
Procedure: Installing capillary columns in the FPD

The FPD uses an adaptable fitting that can use both packed and capillary columns. If your adaptable fitting does not have a capillary adapter installed, begin with step 1. If the capillary adapter is already installed in your adaptable fitting, begin with step 5.

The FPD uses a special adapter for capillary columns. The FPD Capillary Adapter, part number 19256-80570, allows fused silica columns as large as 530 mm ID to be run right to the base of the FPD flame, minimizing sample tailing or loss of chemically active sites.



19256-80590
FPD 1/8-inch adapter

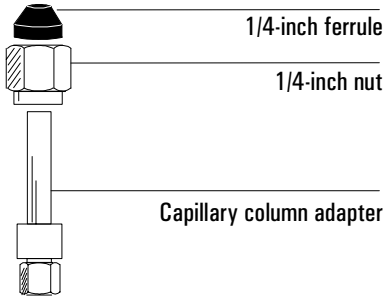


19256-80570
FPD Capillary Adapter

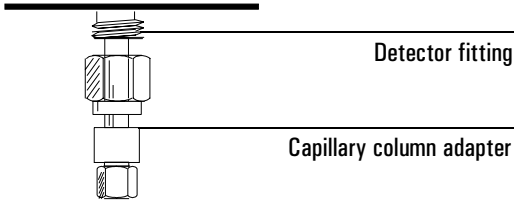
Materials required

Column nut and ferrule
FPD Capillary column adapter
1/4-inch nut and ferrule
Column cutter
1/4-inch wrench
9/16-inch wrench
Metric ruler
Typewriter correction fluid

1. Assemble a brass nut and graphite/Vespel ferrule onto the adapter.



2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.



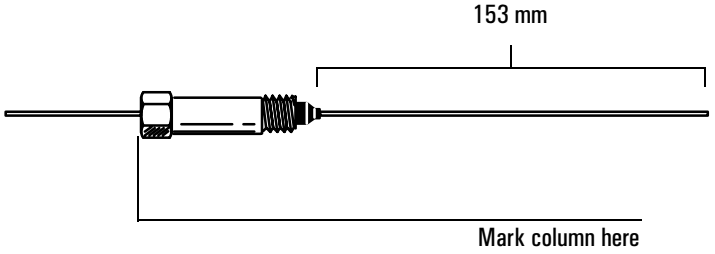
3. Install a column nut (part no. 18740-20870) and graphite ferrule (1.0 mm ID, part no. 5080-8773 or 0.5 mm ID, part no. 5080-8853) on the column.

4. After installing the nut and ferrule, prepare a fresh column end by cutting off a short piece of the column. See page 101 for instructions.

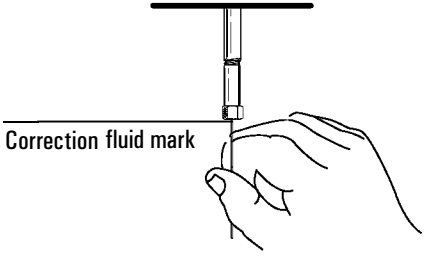
5. Position the ferrule about 153 mm from the end of the column.

   Optimum height depends on sample type and gas flow rates. If it is too high, the column end will be exposed to the flame. If too low, the sample may be exposed to hot stainless steel, causing slight tailing.

Mark the column at a point even with the bottom of the nut. Typewriter correction fluid works well.



6. Insert the column in the detector. Slide the nut and ferrule up the column to the detector base. Finger tighten the column nut until it starts to grab the column.
7. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Proceed to step 8.



8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

Ferrules for capillary columns

Table 12 lists some of the ferrules used with capillary columns and inlet and detector liners/adapters. See the Agilent catalog for consumables and supplies for a more complete listing.

Graphite and graphitized-Vespel ferrules

Place some ferrules in a petri dish in the GC oven at 250 to 300°C for 30 minutes to remove compounds absorbed by the graphite. Leave a dish of assorted ferrules in the oven to ensure a clean supply.

The ferrule should slide onto the column but not fall off from its own weight. If it fits properly, 1/4 turn from finger tight will make a good seal. If it is loose, the column nut must compress the ferrule around the column. This is not a problem with soft graphite ferrules, but hard ferrules may require so much force that the inlet fitting may, the nut, or the ferrule may be damaged. With hard ferrules, it is best to start with an undersize hole and drill it to fit the column.

Vespel ferrules

These ferrules can be more leak-tight than graphite but have a lower temperature limit. Retighten after a few oven temperature cycles.

Table 12 Hardware Used with Capillary Columns

Item*	Typical use	Part no.
1/4-inch graphitized Vespel ferrule, pkg of 10	Inlet/detector liner/adapters	5080-8774
1.0-mm graphite ferrule, pkg of 10	Capillary columns	5080-8773
0.5-mm graphite ferrule, pkg of 10	Capillary columns	5080-8853
Column nut	Connect column to inlet or detector	5181-8831
Column cutter	Cutting capillary columns	5181-8836

* Ferrule and O-ring ids

Part 2. Packed metal columns

Overview: installing packed metal columns

There are two sizes of packed metal columns, 1/4-inch and 1/8-inch, in common use. This general procedure applies to both sizes of columns, as well as PTFE columns used with the FPD.

1. Prepare your packed column (page 124).
2. See Table 13 or Table 14 for fittings required.
3. Install the adapter, if needed (page 128).
4. Install the column (page 129).
5. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

Fittings

Table 13 Fittings for 1/4-inch and 1/8-inch Packed Metal Columns

Inlet or detector	1/4-inch packed metal column		1/8-inch packed metal column	
	Where to install	**Comments**	**Where to install**	**Comments**
Purged-packed inlet	1/4-inch liner	See *Inlets* volume for instructions on installing liner.	1/8-inch liner	See *Inlets* volume for instructions on installing liner.
Adaptable NPD*, FID or FPD	1/4-inch adapter (Part no. 19231-80530)	Remove or install adapter, as desired. See page 128 for instructions on installing an adapter.	1/8-inch adapter (Part no. 19231-80520)	See page 128 for instructions on installing an adapter.
ECD	Detector fitting	Remove adapter, if necessary.	1/8-inch adapter. (Part no. 19301-80530)	See page 128 for instructions on installing an adapter.
TCD	1/4-inch adapter (Part no. G1532-20710)	See page 128 for instructions on installing an adapter.	Detector fitting	Remove adapter, if necessary.

* Do not remove the plugs from your NPD until you are ready to connect the column and operate the detector. Failure to observe this simple procedure may reduce the collector's effectiveness or slow down the bead's stabilization period the first time the detector is used.

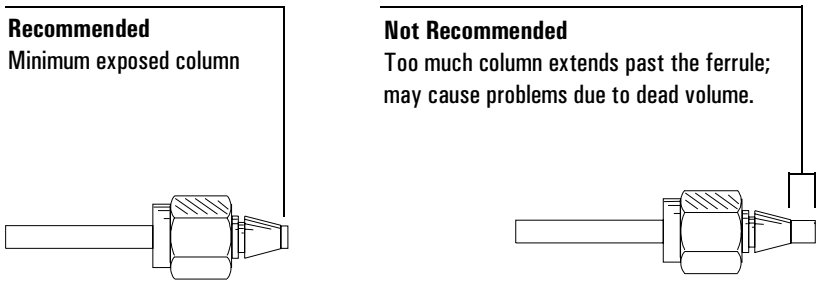
The FPD 1/8-inch OD Adapter, part number 19256-80590, allows installation of PTFE columns concentrically around the FPD fused silica liner, eliminating exposed hot stainless steel.

Table 14 Fittings for FPD with a PTFE Column

1/4-in PTFE column		1/8-in PTFE column	
Where to install	**Comments**	**Where to install**	**Comments**
1/4-inch adapter (Part no. 19231-80530)	Remove or install adapter, as desired. See page 128 for instructions on installing an adapter.	1/8-inch adapter (Part no. 19256-80590)	See page 128 for instructions on installing an adapter.

Preparing packed metal columns

Before installing these columns, a ferrule should be locked on the column end so that it is flush with the end of the column. This prevents problems caused by dead volume in the fitting.



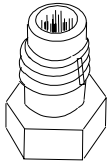
Use the following instructions to install new SWAGELOK nuts and ferrules onto 1/8-inch or 1/4-inch metal columns. If your column already has ferrules installed, proceed to the instructions on installing adapters (page 128) or installing packed metal columns (page 129).

Procedure: Making a spacer from Teflon tubing

Materials required

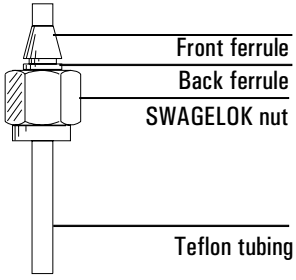
1/4-inch or 1/8-inch Teflon tubing
1/4-inch or 1/8-inch nut and ferrule set
Bench vise
Male SWAGELOK fitting
9/16-inch or 7/16-inch wrench
Razor or sharp knife

1. Secure a new male SWAGELOK fitting in a bench vise.

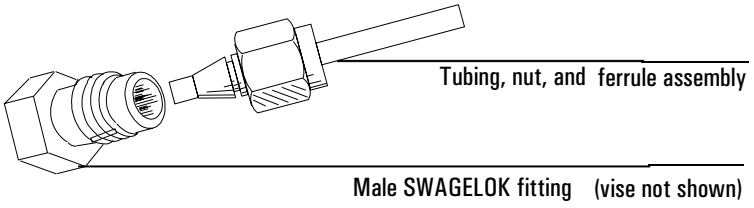


Male SWAGELOK fitting

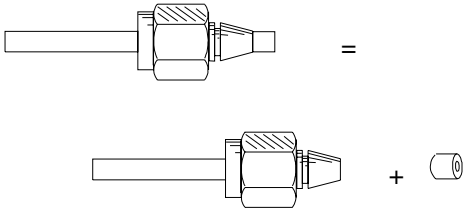
2. Slide a SWAGELOK nut, back ferrule, and front ferrule onto a piece of Teflon tubing. If the end of the tubing is not cut straight, use a razor or sharp knife to make a flat, smooth end.



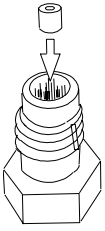
3. Insert the Teflon tubing, ferrules, and nut into the vise-held SWAGELOK fitting. Tighten the nut 3/4 turn past finger tight to set the ferrules on the tubing.



4. Loosen the nut and remove the assembly from the male SWAGELOK fitting.
5. Cut off the end of the tubing extending beyond the ferrule with a razor or sharp knife. This piece of tubing is the spacer.



6. Insert the spacer into the vise-held SWAGELOK fitting.



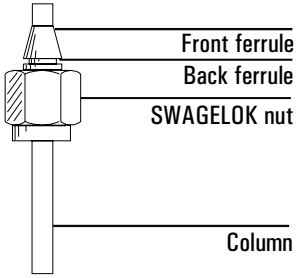
The male SWAGELOK fitting and spacer should be kept on hand to be used whenever new ferrules are being installed on a column.

Procedure: Installing ferrules on a metal column

Materials required

Male SWAGELOK fitting with Teflon tubing spacer
SWAGELOK nut and ferrule set
Wrenches

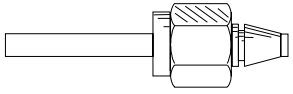
1. Install new SWAGELOK nut and ferrules on the column.



2. Install the Teflon tubing spacer in the male fitting. Fully insert the column with its nut and ferrules into the vise-held fitting. Tighten the nut finger tight.

   Use a wrench to tighten the nut an additional 1-1/4 turn for 1/4-inch columns or 3/4 turn for 1/8-inch columns.

3. Unscrew the column nut from the vise-held fitting and remove the column. Ferrules should now be set in place on the column with the column correctly positioned.



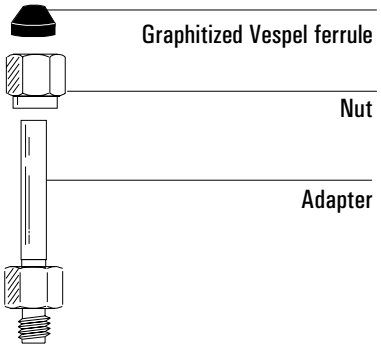
Procedure: Installing an adapter in a detector fitting

This is a general procedure for installing many types of adapters onto detector fittings. See Table 13 for adapter part numbers.

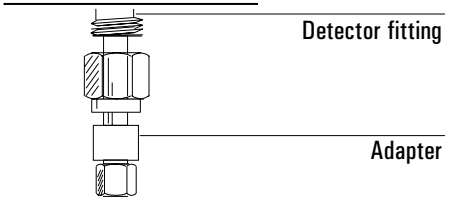
Materials required

7/16-inch or 9/16-inch wrench
Graphitized Vespel ferrule
Nut
Adapter

1. Assemble a brass nut and a graphitized Vespel ferrule onto the adapter.



2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight.



   1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.
   1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.

3. Proceed to “Installing Packed Metal Columns” on page 129.

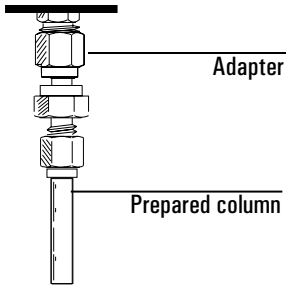
Procedure: Installing packed metal columns

Before following this procedure, make sure an adapter or liner is installed (page 128), if needed, and that your column is prepared (page 124.)

Materials required

Prepared metal column
Column adapter, if needed
Wrench

1. Insert the column into the adapter, detector, or inlet liner until it bottoms. Tighten the nut finger tight.



2. If you are installing a column directly into the detector fitting:
   1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.
   1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.

3. If you are installing a column onto an adapter:
   Tighten the column nut using two wrenches in opposition, one on the column nut and the other on the liner or adapter body. This prevents the liner or adapter from rotating while you tighten the column nut.
   1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.
   1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.

4. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

Ferrules for packed metal columns

Table 15 lists some of the nuts and ferrules used with packed metal columns. Consult the Agilent catalog for consumables and supplies for a more complete listing.

Ferrules that are prepared improperly cause leaks and contamination. Here are some hints to avoid problems.

Graphite and graphitized-Vespel ferrules. Place these ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

Vespel ferrules. These ferrules can be more leaktight than graphite, but have a lower temperature limit. They should be retightened after a few oven temperature cycles to ensure a good seal. Be sure to use the correct ferrule for the size column you are using.

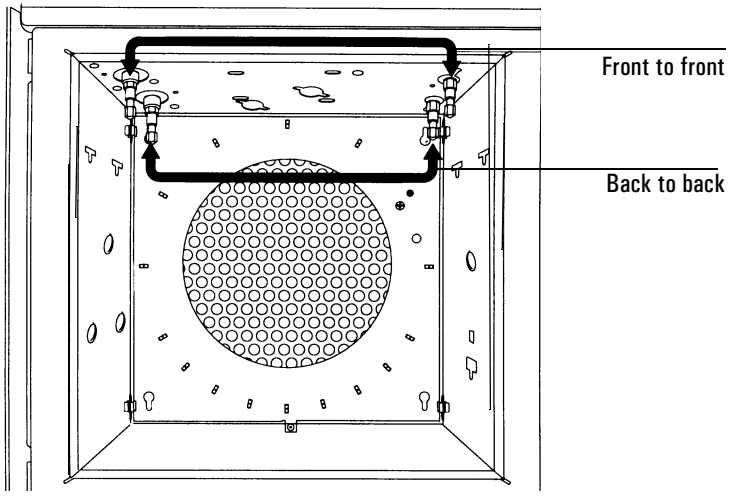
Table 15 Nuts and Ferrules Used with Packed Metal Columns

Item*	Typical use	Part no.
1/4-inch swage stainless steel, pkg of 20 (*nut, front ferrule, back ferrule*)	1/4-inch	5080-8753
1/8-inch swage stainless steel, pkg of 20 (*nut, front ferrule, back ferrule*)	1/8-inch	5080-8751
1/4-inch swage brass, pkg of 20 each (*nut, front ferrule, back ferrule*)	1/4-inch	5080-8752
1/8-inch swage brass, pkg of 20 each (*nut, front ferrule, back ferrule*)	1/8-inch	5080-8750
1/4-inch graphitized Vespel ferrule, pkg of 10	inlet/detector liner/adapters 1/4-inch columns	5080-8774
1/8-inch graphitized Vespel ferrule, pkg of 10	1/8-inch columns	0100-1107

* O-ring and ferrule ids

Part 3. Packed glass columns

Glass packed columns must be installed simultaneously at the inlet and the detector and must be installed parallel to the oven door:



You can install glass packed columns directly in the purged-packed inlet, ECD, and adaptable NPD, FID, and FPD fittings. The TCD requires an adapter.

There are three types of glass packed columns available. You must make certain that your column is compatible with the inlet fitting and detector used. Table 16 summarizes the inlet and detector fittings required and the appropriate column configuration.

Overview: Installing glass packed columns

1. See Table 16 for information on fittings and column configuration required.
2. Remove or install an adapter, if necessary (see page 128).
3. Follow the general procedure for installing glass columns on page 133.

Table 16 Installing Glass Packed Columns

Inlet or detector	Where to install	Column configuration	Comments
Purged-packed inlet	Inlet fitting (no liner installed) *or* 1/4-inch liner*	A or B, depending on the detector C (works with all detectors)	Allow at least 50 mm of empty column to prevent an inserted syringe needle from contacting either the glass wool plug or column packing.
Adaptable NPD, FID, or FPD Cannot use with *capillary optimized* detector	Detector fitting	A	Remove adapter, if installed. There must be at least 40 mm of empty column to prevent the bottom end of the jet from touching either column packing or the glass wool plug.
ECD	Detector fitting	A	Remove capillary column adapter, if installed.
TCD	1/4-inch adapter (Part no. G1532-20710)	B	Instructions for installing adapters are on page 128.

* See the *Inlets* volume for instructions on installing liners

Procedure: Installing glass packed columns

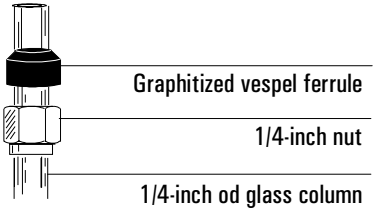
Materials required

Recommended:
Two 1/4-inch graphitized Vespel ferrules
Two 1/4-inch nuts
9/16-inch wrench

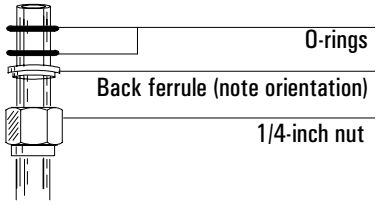
Alternative:
Four O-rings
Two back ferrules
Two 1/4-inch nuts
9/16-inch wrench

1. Assemble a brass nut and graphitized Vespel ferrule on each end of the column. Alternative method: Install a 1/4-inch nut, back ferrule, and two O-rings on each end of the column. An extra O-ring below the nut keeps the nut from dropping into the coiled portion of the column.

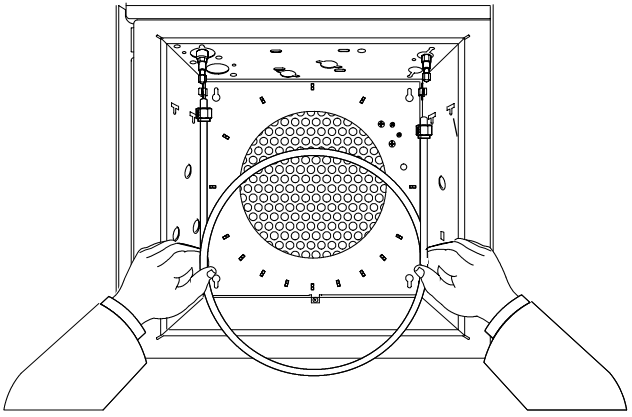
Recommended



Alternative installation method



2. Insert the column into the inlet until it bottoms. Insert the column into the detector fitting but *do not* force it. It may be necessary to start the long end of the column in the inlet at an angle to clear the oven floor.



3. Withdraw the column 1 to 2 mm from both the inlet and detector. Tighten both column nuts finger tight.

4. Tighten both column nuts 1/4 turn with a wrench. If you use graphitized Vespel ferrules, proceed to step 5. If you use O-rings, proceed to step 6.

Caution Overtightening the column nut or forcing it to bottom in both the inlet and detector may shatter the column.

5. Set flow through the column and raise the inlet, detector, and oven to operating temperature. Then set the oven to ambient and allow it to cool.

6. Use the wrench to tighten the nut an additional 1/2 turn. Tighten further, as necessary, to prevent leakage.

Ferrules and O-rings for glass packed columns

Table 17 lists ferrules and O-rings used with glass packed columns. Consult the Agilent Catalog for consumables and supplies for a more complete listing.

Ferrules that are prepared improperly cause leaks and contamination. To avoid problems, place graphitized Vespel ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

Table 17 Glass Packed Columns Consumables

Item*	Typical use	Part no.
1/4-inch graphitized Vespel ferrule, pkg of 10	Inlet/detector liners, 1/4-inch glass packed columns	5080-8774
Silicone O-ring, 6.0-mm	1/4-inch glass packed columns	0905-0322

* O-ring and ferrules ids

Part 4. Conditioning columns

Conditioning involves establishing a flow of carrier gas through a column and then heating it for one-half hour for capillary columns and overnight for packed columns. This drives off contaminants and makes the column fit for analytical use.

New packed columns should be conditioned, since they often contain volatile contaminants from the coating process. It may also be necessary to condition a used column that has been stored for some time without end caps or plugs.

Conditioning is not as important with capillary columns since they contain a minimal amount of stationary phase.

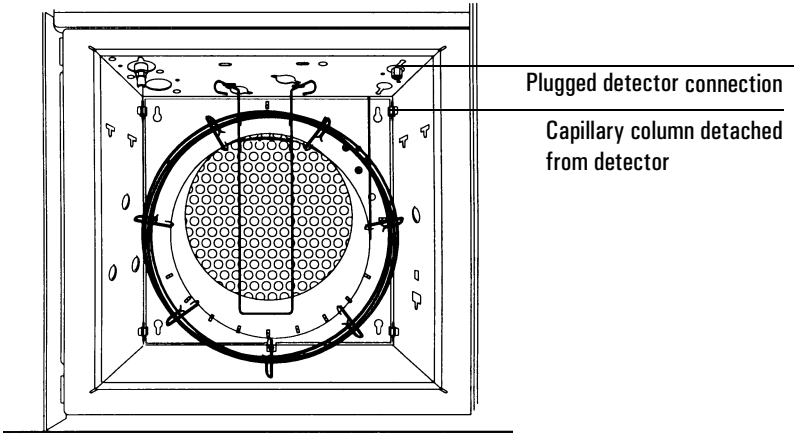
The following procedures include preliminary steps and the actual conditioning procedure, which differs for packed and capillary columns.

Procedure: Preliminary column conditioning steps

Materials required

Two 7/16-inch wrenches
No-hole ferrule and capillary nut for detector connection

1. Turn off the detectors. Shut off the detector support gases. It is especially important to shut off hydrogen!
2. If the column to be conditioned is not already installed, connect one end to an available inlet. If you are not sure how to install a column, see the instructions earlier in this chapter. DO NOT connect the remaining end to a detector!
3. If you plan to condition a capillary column in a split/splitless inlet, install the proper liner and attach the column in the normal manner, making sure about 5 to 7 mm of column extends above (in front of) the column ferrule.
4. Cap the detector(s) fittings with the no-hole ferrule and column nut.



Procedure: Conditioning a capillary column

WARNING Do not use hydrogen as the carrier for conditioning! It could vent into the oven and present an explosion hazard.

1. Select an appropriate column pressure—given as **psi (kPa)**—from this table.

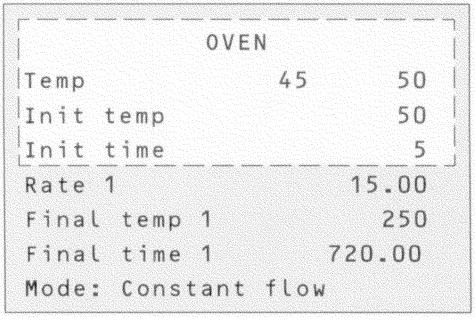
	Inside diameter				
Length, m	**0.10 mm**	**0.20 mm**	**0.25 mm**	**0.32 mm**	**0.53 mm**
10	25 (170)	6 (40)	3.7 (26)	2.3 (16)	0.9 (6.4)
15	39 (270)	9 (61)	5.6 (39)	3.4 (24)	1.4 (9.7)
25	68 (470)	15 (104)	9.5 (65)	5.7 (40)	2.3 (16)
30	83 (570)	18 (126)	12 (80)	7 (48)	2.8 (19)
50		32 (220)	20 (135)	12 (81)	4.7 (32)
60		39 (267)	24 (164)	14 (98)	5.6 (39)

2. Enter the selected pressure. Let gas flow through the column at room temperature for 15 to 30 minutes to remove air.
3. Program the oven temperature from room temperature to the maximum temperature for the column. Increase the temperature at a rate of 10 to 15°C/min and hold at the maximum temperature for 30 minutes.
4. If you will not be using the conditioned column immediately, remove it from the oven. Cap both ends to prevent air, moisture, and other contaminants from entering the column.

Procedure: Conditioning packed columns

WARNING Do not use hydrogen as the carrier for conditioning! It could vent into the oven and present an explosion hazard.

1. Press [Col 1] or [Col 2] to open the column control table.
2. Enter an appropriate column flow:
   - 20 to 30 mL/min for 2 mm ID glass or 1/8 inch OD metal columns.
   - 50 to 60 mL/min for 4 mm ID glass or 1/4 inch OD metal columns.
3. The conditioning temperature is never greater than the maximum temperature limit for the column; 30°C less than the maximum is usually sufficient. Slowly raise oven temperature to the conditioning temperature for the column.



4. Continue conditioning overnight at the final temperature. If you will not be using the conditioned column immediately, remove it from the oven. After removing the column, cap both ends to prevent air, moisture, or other contaminants from entering the column.

Part 5. Conditioning chemical traps

If your traps are preconditioned, you will not need to perform a conditioning procedure before using them. However, all traps need regeneration periodically, for example after using one to four cylinders of gas, or if gases of the highest purity were not used. You can recondition Agilent moisture and activated charcoal traps. Agilent oxygen traps cannot be reconditioned; you must replace them if they become contaminated. Follow the manufacturer's instructions for reconditioning traps.

The molecular sieve and activated charcoal traps can also be repacked. Instructions for repacking traps are shipped with each trap.

Table 18 Ordering Information for Agilent Traps

Item	Part no.
Moisture trap (packed with Molecular Sieve 5A, 45/60 mesh)	5060-9077
Conditioned moisture trap (packed with preconditioned Molecular Sieve 5A, 45/60 mesh)	5060-9084
Activated charcoal trap	5060-9096
Molecular Sieve 5A (100 grams, 45/60 mesh)	5080-6759
Activated charcoal (100 grams, 30/60 mesh)	5080-6751
Cap for ends of traps, 1/8-inch, 6 per package	5180-4124
Reducer trap fittings	5062-3502

Part 6. Calibrating your capillary column (optional)

Preparation

As you use a capillary column, you may occasionally trim off portions, changing the column length. If measuring the actual length is impractical, and if you are using EPC with a defined column, you can use an internal calibration routine to estimate the actual column length. Similarly, if you do not know the column internal diameter or believe it is inaccurate, you can estimate the diameter from related measurements.

Before you can calibrate the column, make sure that:

- You are using a capillary column
- The column is defined
- There are no oven ramps
- The column gas source (usually the inlet) is `On` and non-zero

Also note that column calibration fails if the calculated column length correction is ≥ 5 m, or if the calculated diameter correction is ≥ 20 µm.

Calibration modes

There are three ways to calibrate the column length and/or diameter:

- Calibrate using an actual measured column flow rate
- Calibrate using an unretained peak time (elution time)
- Calibrate both length **and** diameter using flow rate **and** elution time

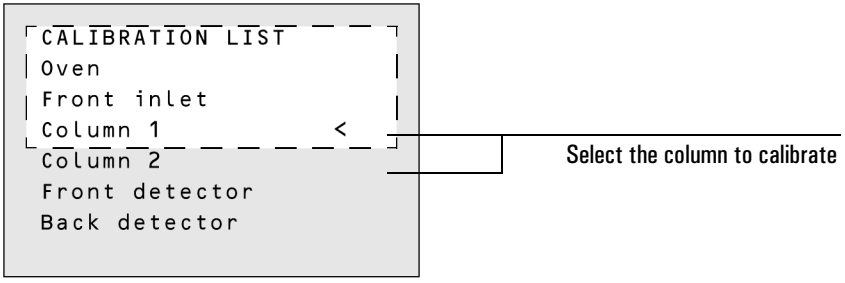
Caution When you measure the column flow rate, be sure to convert the measurement to normal temperature and pressure if your measurement device does not report data at NTP. If you enter uncorrected data, the calibration will be wrong. See "Interpreting bubble meter flow measurements" on page 79 for details.

Column calibration procedures

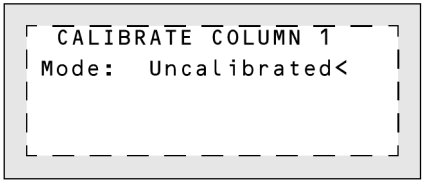
These procedures are described below using Column 1 as an example.

Procedure: Estimate the actual column length or diameter from an elution time

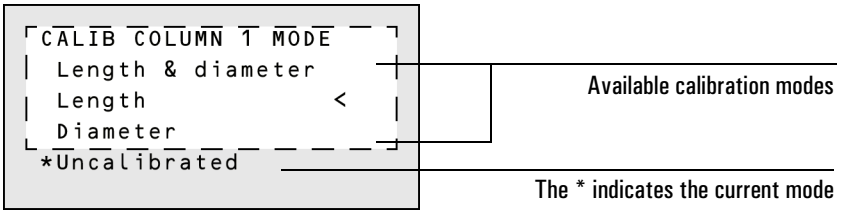
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Setting up an isothermal run" on page 87 or "Configure the column" on page 48.
2. Perform a run using an unretained compound and record the elution time.
3. Press [Options]. Scroll to `Calibration` and press [Enter].
4. From the calibration list, select `Column 1` or `Column 2` and press [Enter].



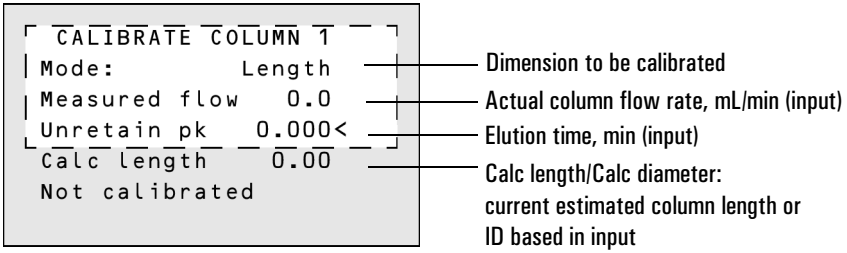
5. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



6. To recalibrate or to change calibration mode, press [Mode/Type] to see the column calibration mode menu.



7. Scroll to `Length` or `Diameter` and press [Enter]. The following menu appears:



8. Scroll to `Unretain pk` and enter the actual elution time from the run performed above.

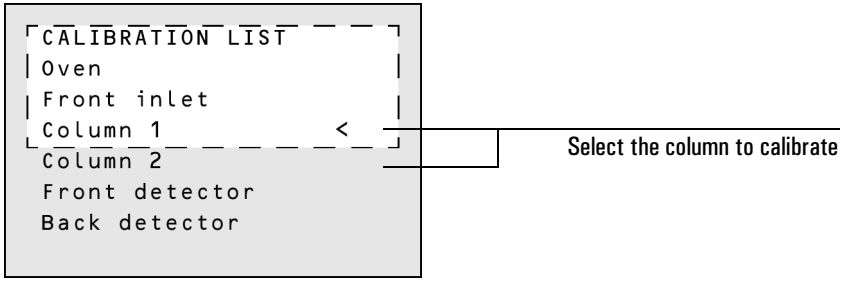
9. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

Procedure: Estimate the actual column length or diameter from the measured flow rate

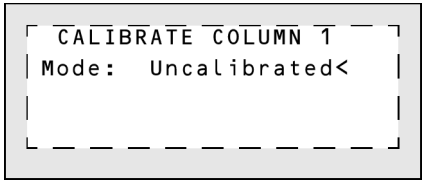
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Setting up an isothermal run" on page 87 or "Configure the column" on page 48.
2. Set the oven, inlet, and detectors temperatures to 35°C and allow them to cool to room temperature.
3. Remove the column from the detector. See chapter 5 for details.

Caution When you measure the column flow rate, be sure to convert the measurement to normal temperature and pressure if your measurement device does not report data at NTP. If you enter uncorrected data, the calibration will be wrong. See "Interpreting bubble meter flow measurements" on page 79 for details.

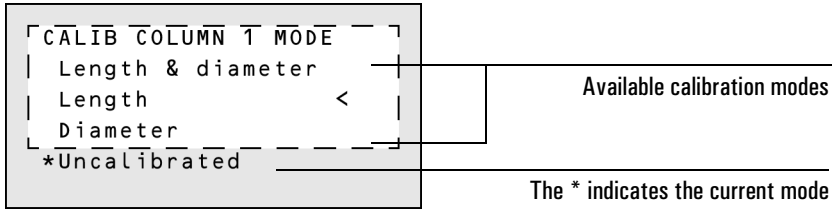
4. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
5. Press [Options]. Scroll to `Calibration` and press [Enter].
6. From the calibration list, select `Column 1` or `Column 2` and press [Enter].



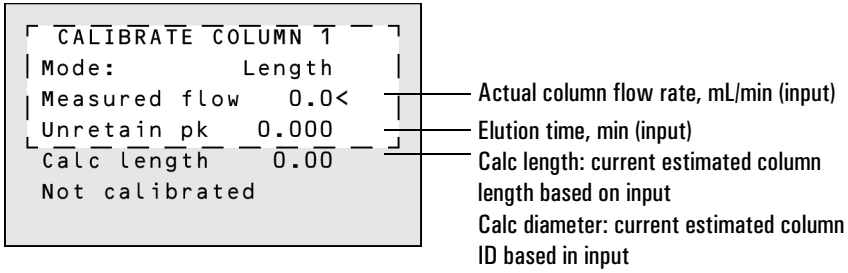
7. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



8. To recalibrate or change calibration mode, press [Mode/Type] to see the column calibration mode menu.



9. Scroll to `Length` or `Diameter` and press [Enter]. The following menu appears:



10. Scroll to `Measured flow` and enter the corrected column flow rate (in mL/min) from the run performed above.

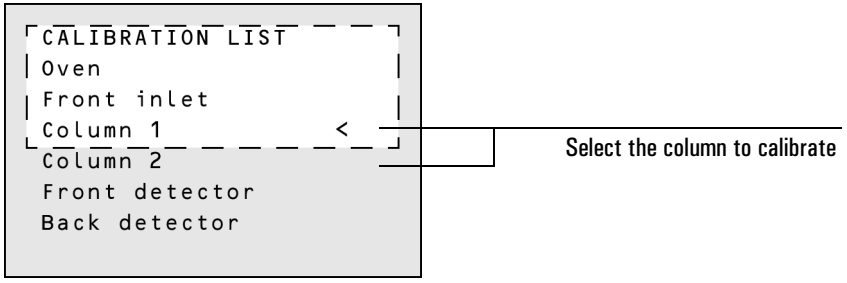
11. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

Procedure: Estimate the actual column length and diameter

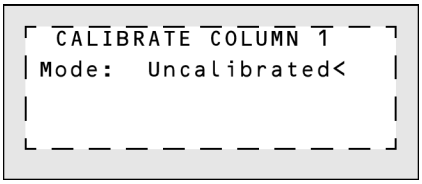
1. Set oven ramp 1 to `0.00`, then verify that the column is defined. For more information, see "Setting up an isothermal run" on page 87 or "Configure the column" on page 48.
2. Perform a run using an unretained compound and record the elution time.
3. Set the oven, inlet, and detectors temperatures to 35°C and allow them to cool to room temperature.
4. Remove the column from the detector. See chapter 5 for details.

Caution When you measure the column flow rate, be sure to convert the measurement to normal temperature and pressure if your measurement device does not report data at NTP. If you enter uncorrected data, the calibration will be wrong. See "Interpreting bubble meter flow measurements" on page 79 for details.

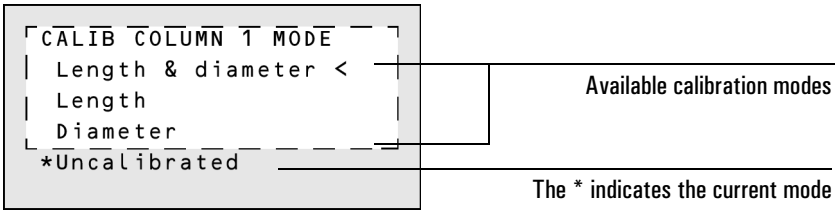
5. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
6. Press [Options]. Scroll to `Calibration` and press [Enter].
7. From the calibration list, select `Column 1` or `Column 2` and press [Enter].



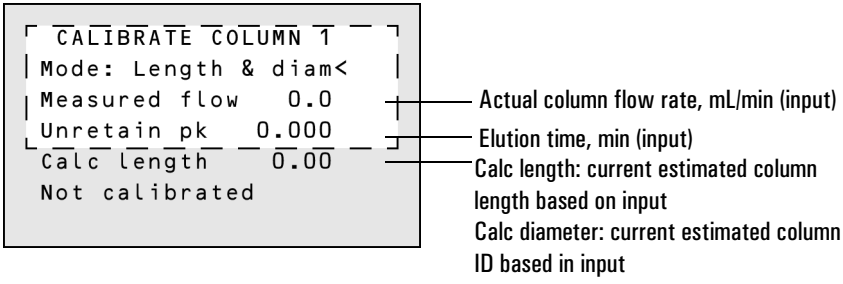
8. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



9. To recalibrate or change calibration mode, press [Mode/Type] to see the column calibration mode menu.



10. Scroll to `Length` or `Diameter` and press [Enter]. The following menu appears:



11. Scroll to `Measured flow` and enter the corrected column flow rate (in mL/min) from the run performed above.

12. Scroll to `Unretain pk` and enter the actual elution time from the run performed above.

13. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

6

Signal Handling

Specifying and controlling output signals, analog signals, digital signals, Chem Station and INET considerations

Signal Handling

Signal is the GC output to a data handling device, analog or digital. It can be a detector output or the output from temperature, flow, or pressure sensors. Two signal output channels are 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 to the ChemStation is available at 11 speeds ranging from 0.1 Hz to 200 Hz, capable of handling peaks from 0.001 to 2 minutes wide. Set this rate from your ChemStation.

Data for INET integrators is at 20 Hz, capable of handling peaks of at least 0.01 minute width.

Using the signal control tables

Signal type

When assigning detector signals, use the [Mode/Type] key and choose from the Signal Type control table, or press a key or combination of keys. [Front], [Back], [–], [Col Comp 1], and [Col Comp 2] will work — alone or in combination. For example, press [Back] for back detector or [Back] [–][Front] for back detector minus front detector.

The nondetector signals are test plot, thermal, pneumatic, and diagnostic. Access them by pressing [Mode/Type]. Diagnostic signals are for use by your service representative and are not described in detail here.

Signal type can be programmed as a run time event. See chapter 7 for details.

Value

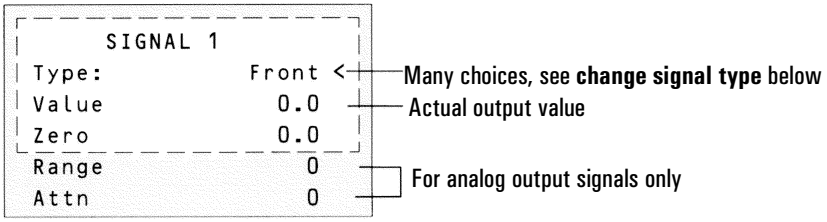
`Value` on the signal control table is the same as Output on the detector control table if your signal type is `Front` or `Back`. If you are subtracting one signal from another (as in `Front - Back`), the signal `Value` will be the difference. You cannot enter a setpoint for `Value`.

A conversion factor may be involved when interpreting `Value`—for example, one FID unit is one picoamp; one ECD unit is 5 Hz (1 Hz for the μ-ECD). The units for detector and other signals are listed below.

Table 19 Signal Conversions

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

Press [Signal 1] or [Signal 2]



To **change signal type**, press [Mode/Type]:

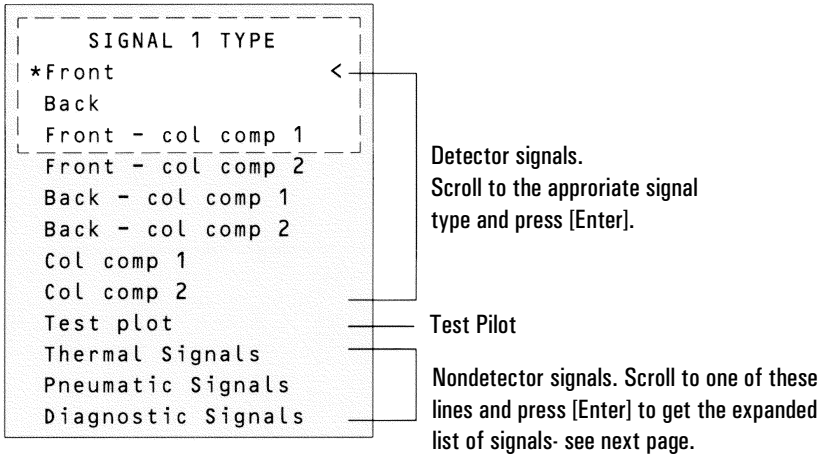
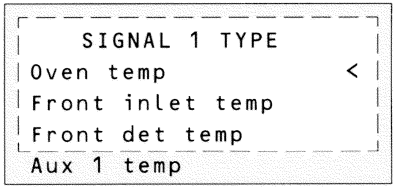
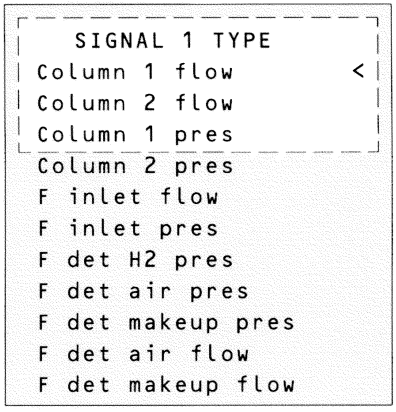


Figure 22 Signal control table

Thermal signals:

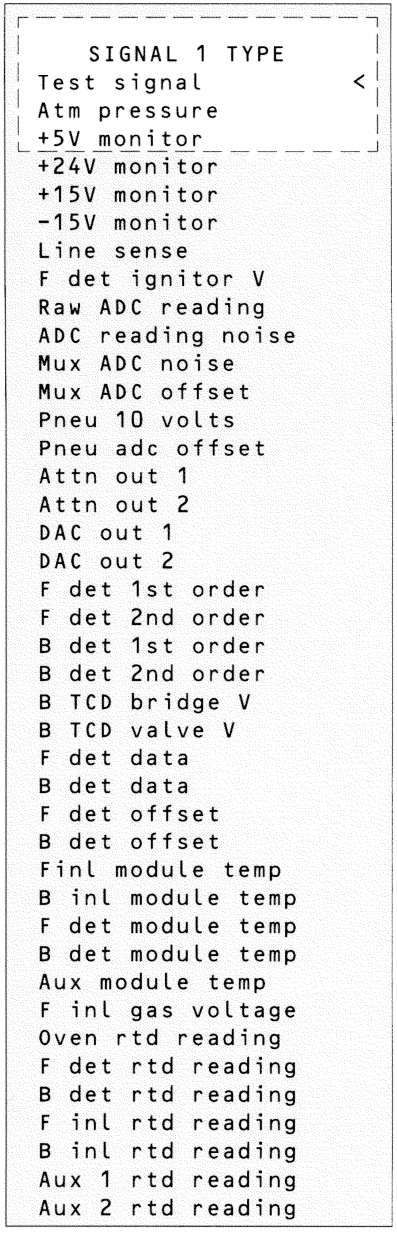


Pneumatic signals:



Only installed items are listed in submenus. Column flows and pressures are only listed when you have electronic pneumatics control (EPC).

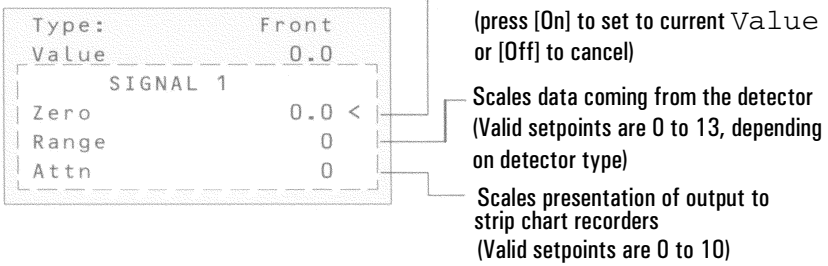
Diagnostic signals:



Analog output settings—zero, range, and attenuation

If you use an analog recorder, you may need to adjust the signal to make it more usable. `Zero`, `Range`, and `Attn` in the Signal control table do this.

Press [Signal 1] or [Signal 2].



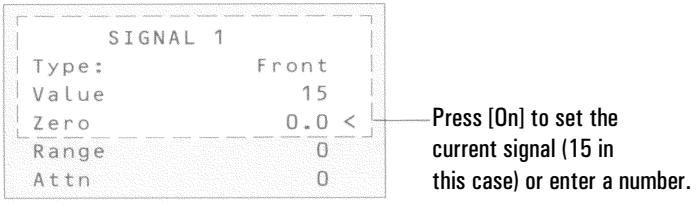
Analog zero

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 control table minus the `Zero` setpoint.

`Zero` can be programmed as a run time event. For details, see chapter 7.

Procedure: Zeroing signal output

1. Verify that the detector is on and in a ready state.
2. Press [Signal 1] or [Signal 2] to access the signal control table.



3. Scroll to `Zero`.

4. Press [On] 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.

Range—for analog outputs only

`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 (1 mV, 1 V, etc.)

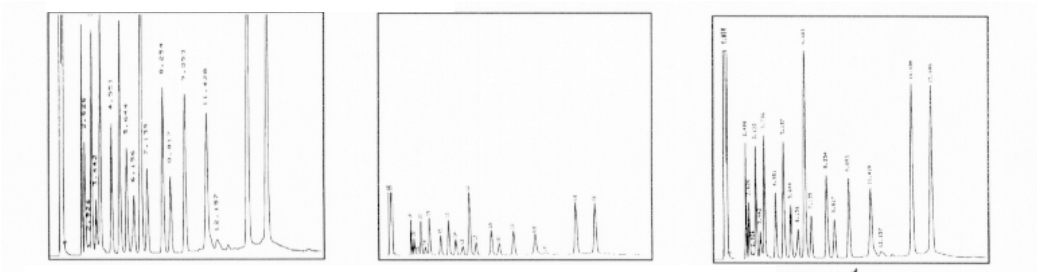
If a chromatogram looks like A or B in Figure 23, the data needs to be scaled (as in C) so that all peaks are visible on the paper.

Valid setpoints are from 0 to 13 and represent 2^0 (1) to 2^{13} (8192). Changing a setpoint by 1 changes the width of the chromatogram by a factor of 2. The chromatograms in Figure 23 illustrate this. Use the smallest possible value to minimize integration error.

See Table 20 for output scaling with different analog output devices.

Table 20 Output Scaling

Analog	One display unit =	Digital	One display count =
0 to 1 mV	$1mV/2^{Range}*2^{Attn}$	ChemStation	1 height count
0 to 1 V	$1\ mV/2^{Range}$	INET (3396B and 3396C)	10,000 height counts $2^{SIGRANGE}$ (set from 3396)
0 to 10 V	$1\ mV/2^{Range}$	INET area counts (on INET SIGRANGE 0) are approximately 10,000 * ChemStation area counts	



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

Figure 23 Range settings

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

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

Range may be run time programmed. See chapter 7 for details.

Attenuation—for analog outputs only

Attenuation(`Attn`) scales the presentation of output on 0- to 1-mV strip chart recorders. Valid setpoints are from 0 to 10 and represent 2^0 to 2^{10}. As with range, each higher setpoint value reduces the size of the chromatogram by one half, while each higher setpoint doubles the size.

Attenuation is in addition to range. Thus, the total scaling factor is:

$$2^{\text{Range}} \times 2^{\text{Attenuation}}$$

Attenuation may be run time programmed. See chapter 7 for details.

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.

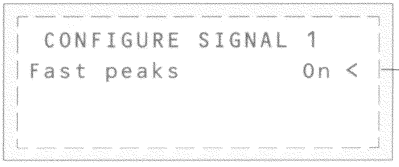
Speed is measured in terms of bandwidth. Your recorder or integrator should have a bandwidth twice that of the signal you are measuring.

The GC allows you to operate at two speeds. The faster speed—to be used only with the FID, FPD, and NPD—allows minimum peak widths of 0.004 minutes (8 Hz bandwidth), while the standard speed—which can be used with all detectors— 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.

Procedure: Selecting fast peaks

1. Press [Config][Signal 1] or [Config][Signal2].



2. Press [ON] (FID only).

Digital data handling

Digital zero

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

Baseline level shifts

Some run time operations, such as changing signal assignment or switching a valve, can produce large changes in the signal baseline position. This can complicate signal processing by external devices. The GC provides two run table commands minimize such problems—see chapter 7 for information on using the run table.

`Store signal value` Saves the value of the signal at the time of the command.

`Sig zero - value` Creates a new zero by subtracting the stored value from the current value of the signal and applies this zero to all future values.

When these commands surround a baseline-shifting command, the effect is to bring the new baseline to the previous level, as shown in Figure 24.

The `Store` event must occur before the event that shifts the baseline, and the `zero - value` event must occur after the baseline has stabilized at the shifted level.

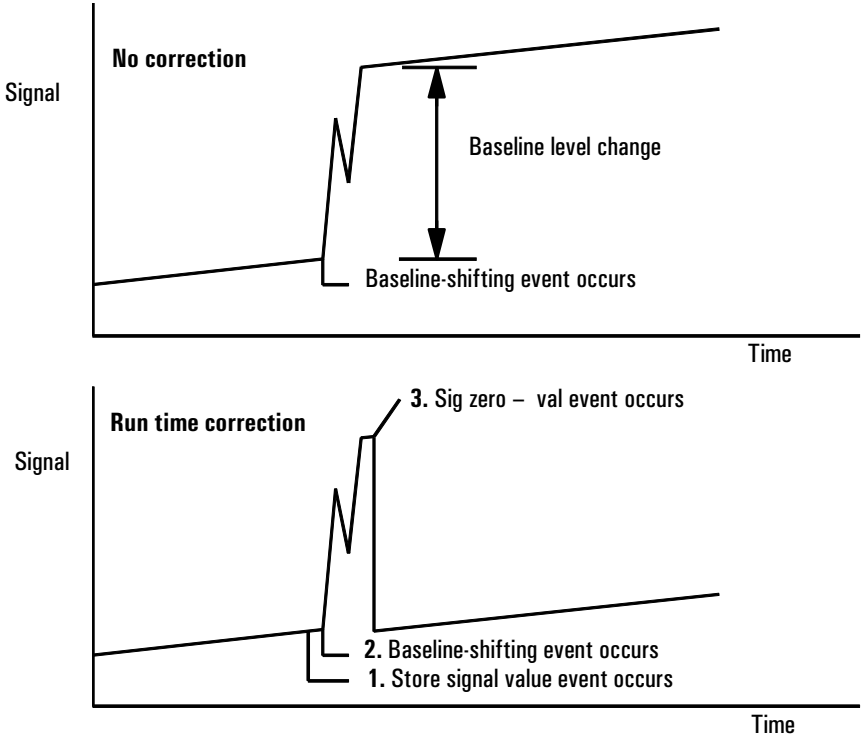


Figure 24 Correcting baseline level shifts in digital signals

ChemStation

The GC can process data at 11 different data rates, each corresponding to a minimum peak width. The table shows the effect of data rate selection.

Table 21 ChemStation Signal Processing

Data rate (Hz)	Minimum peak width (minutes)	Relative noise	Detector	Column type
200	0.001	3.1		Narrow-bore (50 µm)
100	0.002	2.2	FID/FPD/NPD only	capillary
50	0.004	1.6		
20	0.01	1		
10	0.02	0.7		
5	0.04	0.5		to
2	0.1	0.3	All types	
1	0.2	0.22		
0.5	0.4	0.16		
0.2	1.0	0.10		
0.1	2.0	0.07		Slow packed

You cannot change the data rate during a run.

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

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

Figure 25 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.

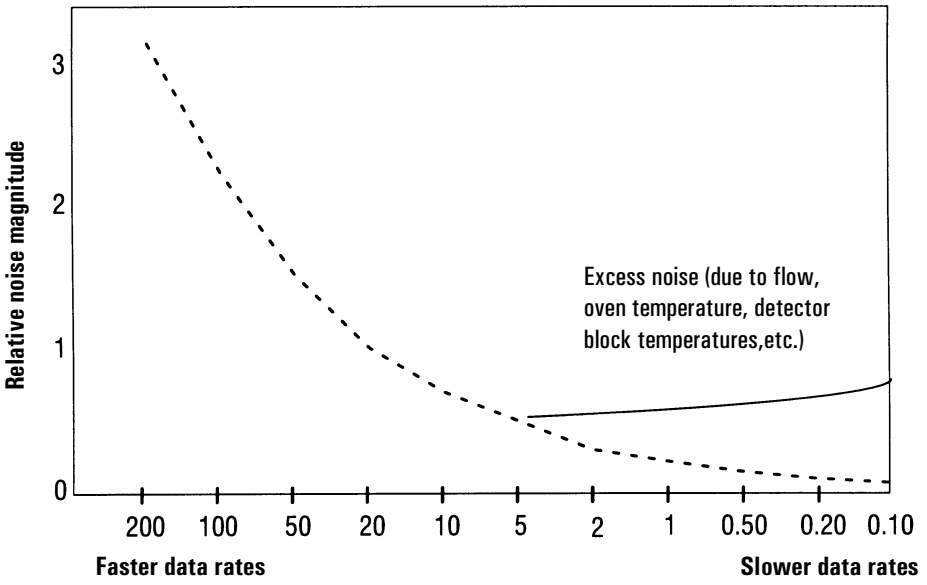


Figure 25 Relationship of noise to data rates

INET

This applies only to 3395A/B or 3396B/C integrators with an INET board installed.

INET transmits data digitally at a rate of 20 Hz and can handle peaks of at least 0.01 minute width. INET can select which of the two GC signals to use—you define what those signals are with the control table.

Column compensation

Peaks are integrated more accurately and repeatably on a flat baseline than on a rising baseline. Column compensation corrects for baseline rise during temperature programming. This is done by making a blank run—one with no

sample injected. This run is stored and subtracted from the *real* run to produce a flat baseline. Figure 26 illustrates the concept.

All conditions must be identical in the column compensation run and the *real* run. The same detector and column must be used, operating under the same temperature and gas flow conditions. Two baseline profiles may be stored (as [Col Comp 1] and [Col Comp 2]).

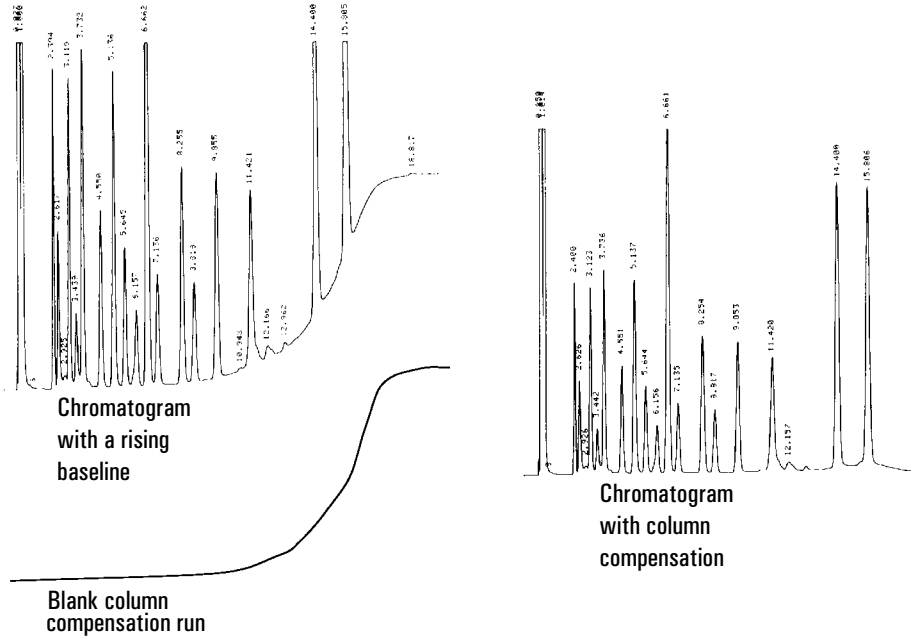
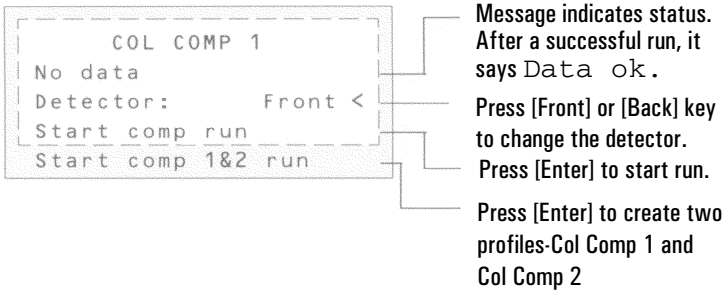


Figure 26 Column compensation

Procedure: Creating a column compensation profile

1. Set up the instrument for a run.
2. Make a blank run to verify that the baseline is clean. This is particularly important for new conditions or if the GC has been idle for several hours.
3. Press [Col Comp 1] or [Col Comp 2] to open the control table.
4. Press [Front] or [Back] depending on the detector you are using.

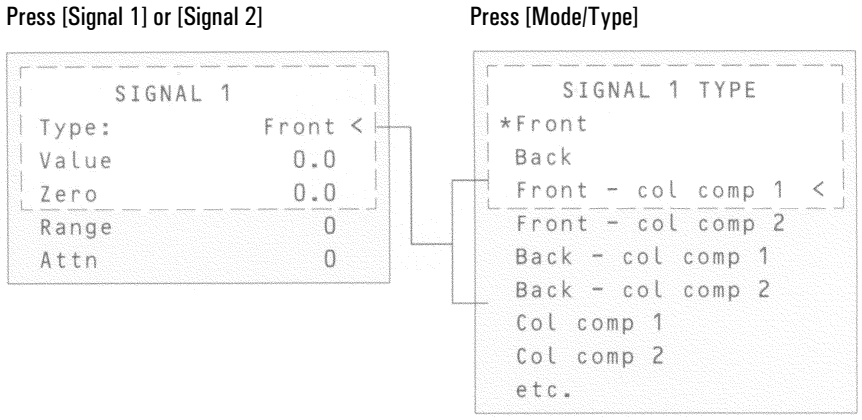


5. Select `Start comp run` or `Start comp 1&2 run`. Press [Enter].
   a. `Start comp run` creates one profile.
   b. `Start comp 1&2 run` creates two profiles (using different detectors and columns but the same oven temperature program).
6. If the run is successful, the first line of the control table will say `Data ok`, and a time and date will appear at the bottom.

Procedure: Making a run using column compensation

1. Set the up chromatographic conditions. They must be identical to those in the stored column compensation run except that `Final time` in the last ramp of the oven program can be longer or shorter.
2. Press [Signal 1] or [Signal 2] to access the signal control table.

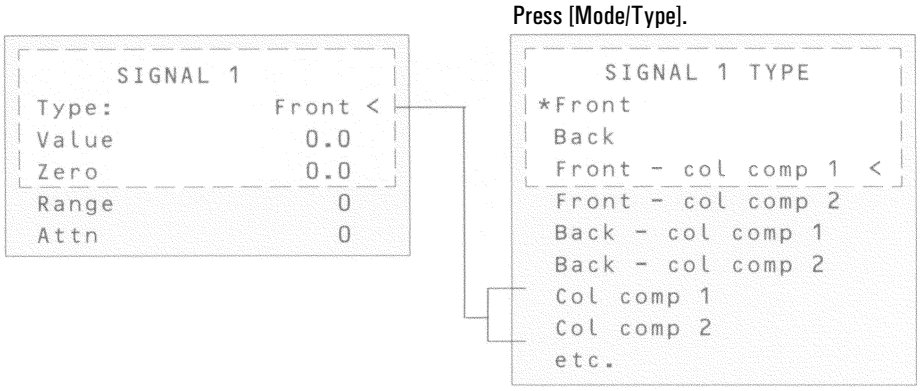
3. Scroll to `Type`: and press [Mode/Type].



4. Choose `Front - col comp 1` or one of the other three column compensation options on the list.
5. Enter setpoints for `Zero`, `Range`, and `Attn`, if applicable.
6. Start your run.

Procedure: Plotting a stored column compensation profile

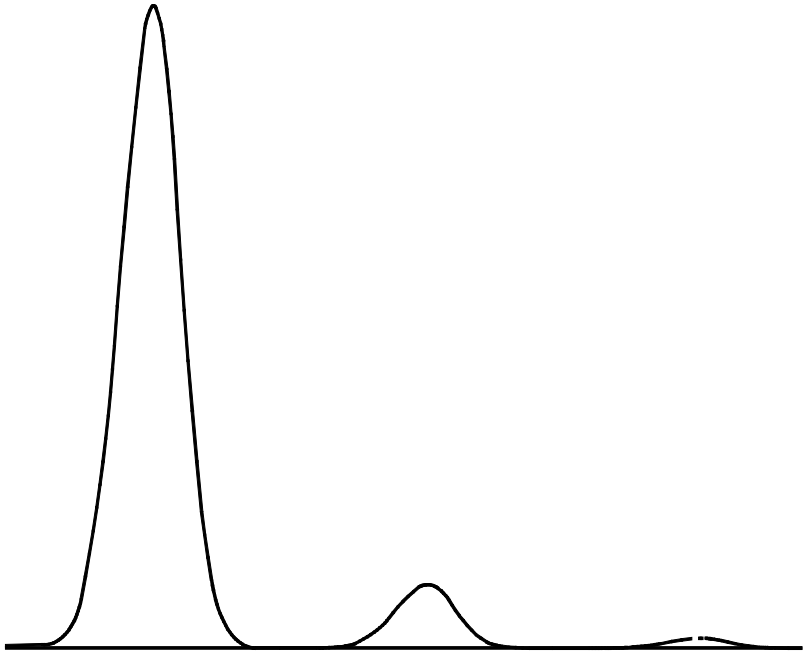
1. Press [Signal 1] or [Signal 2] to open the signal control table.
2. Scroll to `Type:` and press [Mode/Type].
3. Choose `Col comp 1` or `Col comp 2`.
4. Press [Start].



Test plot

`Test plot` is an internally generated "chromatogram" that can be assigned to a signal output channel. It consists of three baseline-resolved, repeating peaks.

The area of the largest is approximately 1 Volt-sec, the middle one is 0.1 times the largest, and the smallest is 0.01 times the largest.



`Test plot` can be used to verify the operation of external data processing devices without having to perform repeated chromatographic runs. It may also be used as a stable signal to compare the results from different data processing devices.

Test plot

7

Instrument Automation

Creating and using timed events. Executing events during a run. Using the clock table.

Instrument Automation

Executing events during the run

Instrument automation allows you to program events using run time programming via the run table or clock time programming via the clock table. Up to 25 timed events can be executed in each of these tables.

Run time programming

Run time programming 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 signal definition, zero, range, or attenuation
- 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
- Pausing (freezing) and resuming a signal value

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 chapter 9 for more information.

Using run time events

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

You can control the following events during a run.

- Valves (1-8)
- Multiposition valve
- Signal type (see page 150)
- Analog signal zero, attenuation, and range
- Digital signal zero and baseline level shifts (see page 157)
- Auxiliary pressures (3, 4, 5)
- TCD negative polarity (on/off)
- NPD H_2 flow (on/off)
- Pausing (freezing) and resuming a signal value

Procedure: Programming run time events

1. Press [Run Table] to open the run time control table. The following message will be displayed if no programmed entries presently exist.

```
  RUN TIME TABLE
No entries in table.
(Press MODE/TYPE
to select new entry)
```

2. Press [Mode/Type] to see the run time event types.

```
  RUN TIME TABLE TYPE
Valve
Multipos valve
Signal definition
Signal zero
Signal range
Detector H2O flow
Detector polarity
Store Signal value
Sig zero - value
Freeze sig. value
Resume sig. value
```

Note: Only those types that are possible with your configuration will appear.

3. Scroll to the event type to be programmed.

```
  RUN TIME TABLE TYPE
RUN TABLE (1 of 1)
Time:          0.00
Type:   Sig 1 zero
Setpoint        On
```

Run time for event to occur

Event type for entry #1

Setpoint parameter: varies with event type

4. Enter values for the `Time`: and `Setpoint`: parameters.

The run table

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

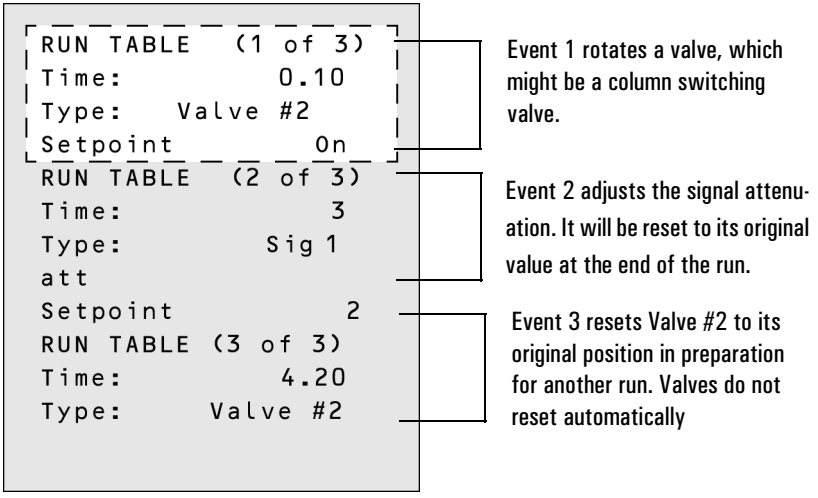


Figure 27 A run table example

Procedure: Adding events to the run table

1. To add new events to the run table, press [Mode/Type] while on the `Time:` or `Type:` line of any entry.
2. Select the event type.
3. Set appropriate `Time:` and `Setpoint:` parameters.

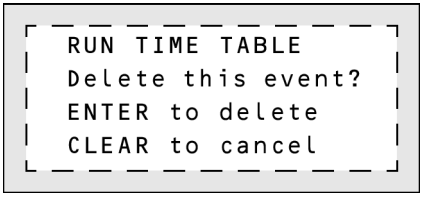
Repeat until all entries are added. Events are automatically placed in order by execution time.

Procedure: Editing events in the run table

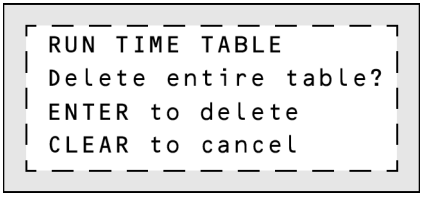
1. Press [Run Table].
2. Move the cursor to the event you want to change.
3. To edit the time for an event, move the cursor to the line labeled `Time`. Type the desired time and press [Enter].
4. To edit a setpoint value, scroll to the setpoint item and press the [On] or [Off] key or enter a numeric value for the setpoint. Press [Enter].

Procedure: Deleting run time events

1. Press [Run Table] to access the run time table.
2. From within this table press the [Delete] key to delete events from the run time table. Pressing [Delete] while in an existing time event table produces the following display.



3. Press `[Enter]` to delete the current timed event; press `[Clear]` to cancel this operation.



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

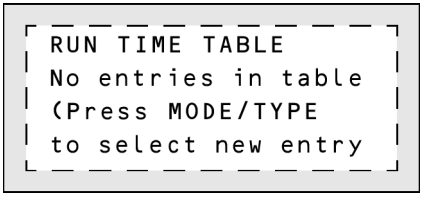
Using clock time events

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.

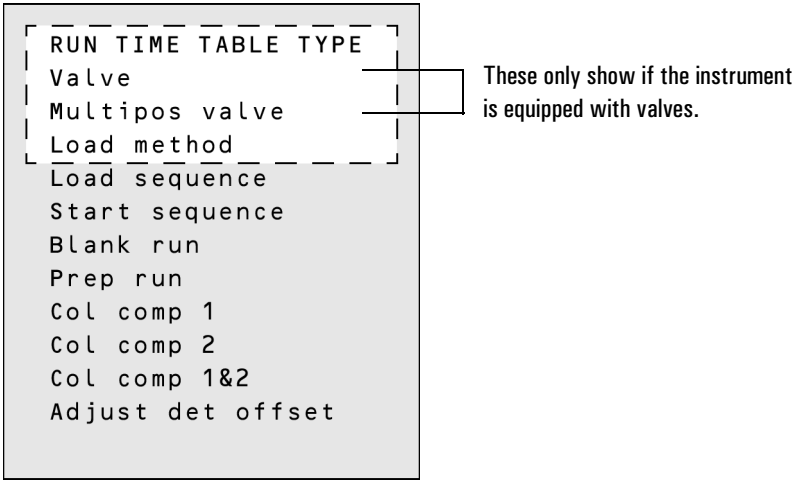
For example, the clock table could be used to start an analysis before you even get to work in the morning.

Procedure: Programming clock time events

1. Press [Clock Table] to access the clock time control table. The following message will be displayed if no events are programmed.



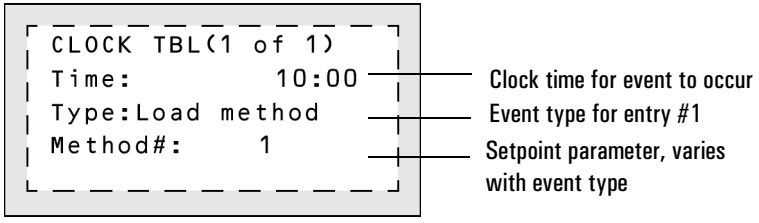
2. Press [Mode/Type] to view the clock time program types.



3. Scroll to select the parameter to be programmed.

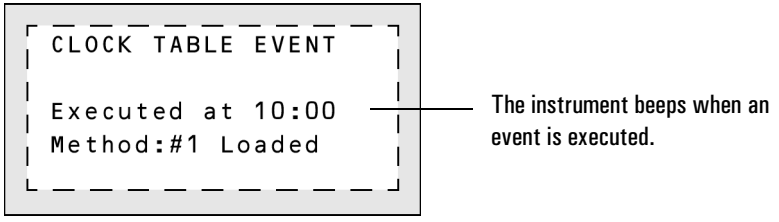
   For example, if the option "Load Method" is chosen for clock time event #1, the display would look similar to the one below.

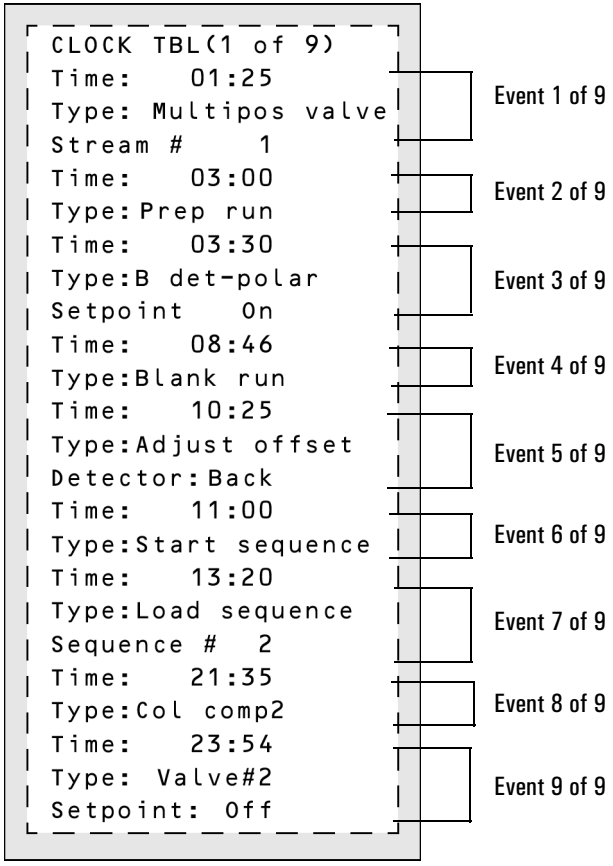
4. Edit `Time`: and `Method#`: setpoints for this event.



   This allows you to program a specific time at which your GC will load a predetermined method.

5. When the clock event is executed, the following screen appears:





The clock table will resume at 01:25 the next morning with the multiposition valve event.

Figure 28 A clock table example

Note: This is *not* a "realistic" clock table. It is intended to show the variety of events that can be programmed and to demonstrate that the size of any entry depends on the parameters required for that event

Up to 25 clock time events can be programmed.

Procedure: Adding events to the clock table

1. Press [Clock Table].
2. To add new events to the clock table, press [Mode/Type]. When entries are added, they are automatically ordered chronologically.
3. Select next event type.
4. Set appropriate parameters.

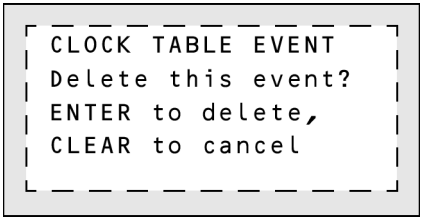
Repeat this process until all entries are added.

Procedure: Editing clock time events

1. Press [Clock Table] to view all events programmed.
2. Scroll to the event you want to change.
3. Edit the time for an event, move the cursor to the line labelled `Time`: and type the desired time.
4. Edit a setpoint value by scrolling to the setpoint item and pressing the [On] or [Off] key, or enter a numerical value for the setpoint.

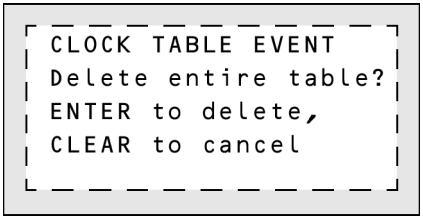
Procedure: Deleting clock time events

1. Press [Clock Table].
2. Press the [Delete] key to remove events from the clock time table. Pressing the [Delete] key while in an existing time table produces the following display:



3. Press [Enter] to delete the current timed event; press [Clear] to cancel this operation.

   To delete the entire table, press [Delete][Clock Table]. The following display appears.



8

Analytical Methods

Saving an instrument setup as a method. Restoring and using saved methods.

Analytical Methods

What is a method?

An analytical method is a collection of setpoints required to run a single sample on the 6890 Series GC. Methods make it possible to restore the instrument to a desired setup without reentering all the setpoints.

You can think of a method as a collection of completed control tables, containing information such as oven temperature programs, pressure programs, inlet temperatures, etc. Actually, there is always an active method in the GC—it is the set of conditions that are controlling the machine now. A method is created by saving these conditions as a numbered method using the [Store] key.

There are three kinds of methods:

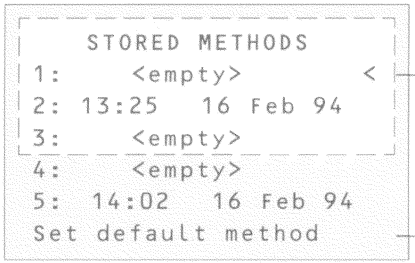
- The active method—the setup that you are presently using.
- Stored methods—one of the five methods that can be stored in the GC.
- The default method—a set of default parameters for the GC. It can be reloaded at any time.

What can you do with it?

Methods can be:

- **Created** by setting the GC up the way you want it. This is the active method.
- **Stored** by pressing [Store] and giving the method an identifying number from 1 to 5.
- **Loaded** by pressing [Load] and specifying the method number to be loaded. Loading a method overwrites the setpoints of the active method.
- **Modified** by loading, making the changes you want, and then storing using the original number. The new version replaces the old one.

Methods are viewed in a method status control table, which shows the times and dates when the methods were stored. Access this table by pressing [Method].



Method status. <empty>means that no method is stored. If a method is stored the time and date it was last stored are shown.

Set default method. Replaces the active method with the default setpoints.

Creating a method

Because a method is a set of control tables of setpoints used for analysis, it depends on instrument configuration. The following is a list of parameters for which you can store setpoints during method development:

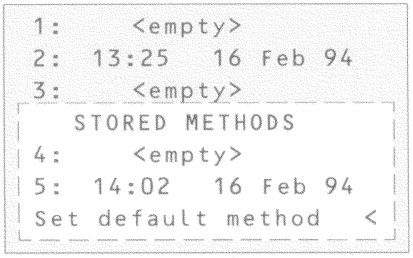
- Oven
- Front/Back inlet
- Column 1 & 2
- Front/Back detector
- Signals 1 & 2
- Aux #1-5
- Post run
- Valve # 1-8
- Run time table
- Front and back injectors
- Sample tray

These parameters are saved when the GC is turned off and reloaded automatically when you turn the instrument back on. However, if the hardware has been changed while the instrument was turned off, it may not be possible to restore all setpoints in the method.

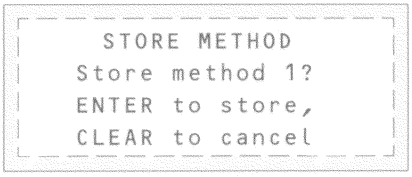
Procedure: Storing a method

To store a method:

1. Press [Method] and scroll to the method number you wish to use.

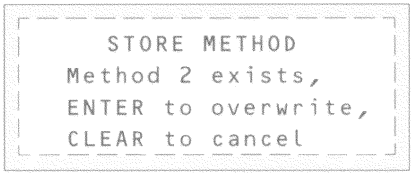


2. Press the [Store] key. You are then asked to confirm the store.



3. [Enter] stores the method using the chosen number. [Clear] returns to the STORED METHODS status table without storing the method.

4. If a method with this number already exists, this screen appears:

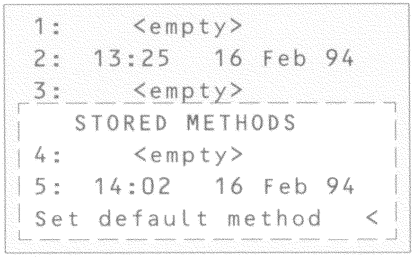


   - [Enter] to replace the existing method with the new one and return to the STORED METHODS status table.
   - [Clear] to return to the STORED METHODS status table without storing the method.

Procedure: Loading a previously stored method

To load a stored method:

1. Press [Method] to access the STORED METHODS status control table.
2. Scroll to the method you wish to load.



3. Press the [Load] key.

   You are prompted to either load the method by pressing [Enter] or to cancel this function by pressing [Clear].

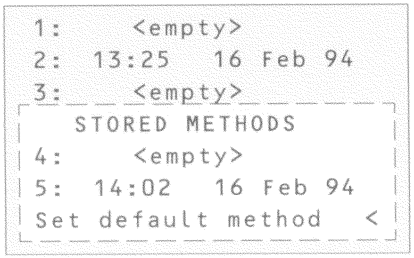
4. Press [Enter] to load the method. The selected method replaces the active method.

[Clear] exits this function and returns to the STORED METHODS status table.

Procedure: Loading the default method

The GC default parameters can be reloaded at any time.

1. Press [Method].
2. Scroll to Set default method.



3. Press [Enter].

See chapter 2, *Keyboard and Display*, for more information.

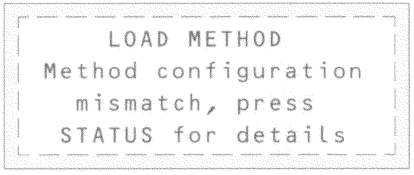
Method mismatch

Method mismatch messages appear when the method you load contains parameters that do not match the GC's current configuration. If this happens, the setpoints that do not match may be ignored.

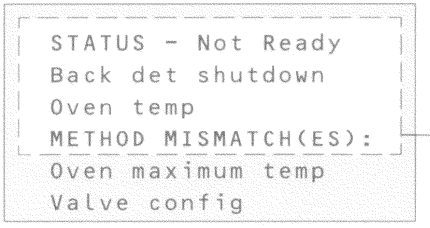
Mismatches are caused by user changes (different choice of carrier gas, etc.) or by hardware changes (replace a TCD with an FID, etc.) that are made after the method is stored.

User-entered configuration changes

You will be warned of user-entered configuration changes between the stored method and the active method. The active method will overwrite the parameter change.



Press [Status] to see which parameters are causing the method mismatch.



Method mismatch-—message will appear if hardware or user-entered configuration has changed.

Hardware configuration changes

If the hardware has changed, some parts of the method may be ignored. You are warned if this happens. For example, suppose you replace the front FID with an ECD. If you now load a method that uses the FID, the FID setpoints cannot load. They will be ignored and current ECD setpoints will be retained. All other setpoints that can be loaded, will be loaded.

Procedure: Modifying a previously stored method

When a method is loaded it replaces the active method.

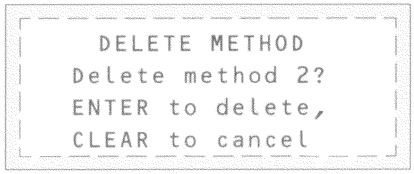
You can modify a previously stored method by:

1. Loading the desired method
2. Making the appropriate changes
3. Storing this method under the same method number (overwrite the original method) ***or*** store as a different method number

Procedure: Deleting a stored method

There are two ways to delete methods.

1. From within the method status control table, scroll to the appropriate method and press [Delete]. You will be prompted with the following:



2. To delete this method, press [Enter].
3. To change your mind and not delete this method, press [Clear].

Method listings

When the 6890 transmits a formatted method listing to an external device, the listing shows the pneumatics setpoints relative to the initial (start-of-run) oven temperature, regardless of the current temperature.

This provides consistent method listings that depend only on the method content and are not affected by the current state of the instrument.

As a result, the pneumatics setpoints listed on an integrator (or other products that use the formatted method listing) may differ from the setpoints that appear at the same time in the 6890 display.

9

Valve Control

Using valves for flow path changes,
sample selection, and sample injection

Valve Control

The 6890 Series Gas Chromatograph (the GC) holds up to four valves in a heated valve box on top of the oven.

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

The valve box

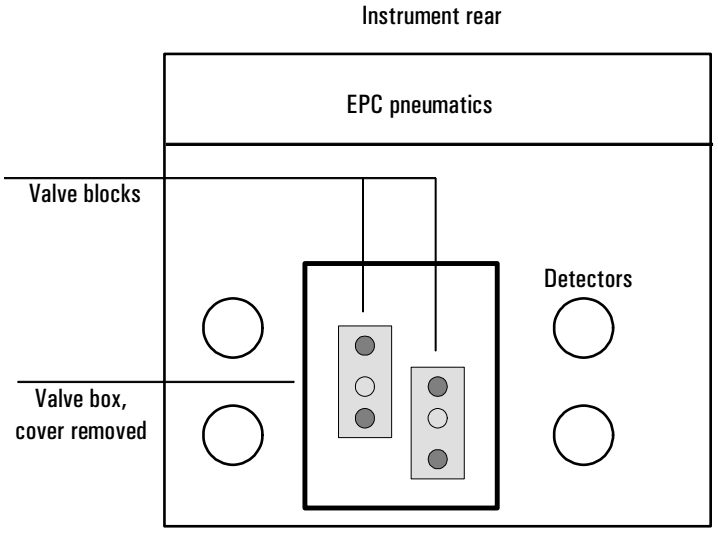


Figure 29 The valve box

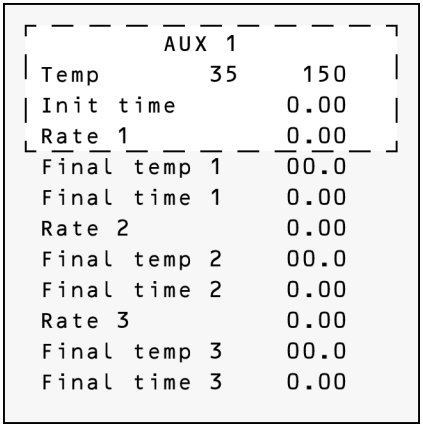
Heating the valves

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

If two valves are used, mount them on the same block. This allows them to be heated using only one control channel (Aux 1 or Aux 2, depending on how the heaters are wired). With more than two valves, both Aux 1 and Aux 2 must be used for heating the two blocks. Set them at the same temperature.

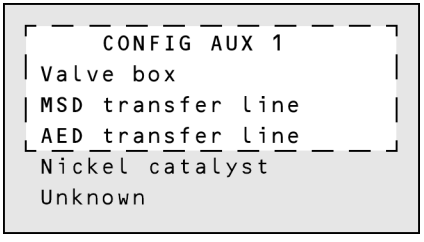
Valve temperature programming

Most valve applications are isothermal; however, you can define three temperature ramps if desired. Press [Aux #], then [1] or [2]. Program this ramp the same as an oven ramp. Refer to *Making a temperature-programmed run* on page 88 for more information.



Configuring an Aux thermal zone

To configure a thermal Aux zone (1 or 2), press [Config], then [Aux #]. Press [Mode/Type], then select the type of device to be controlled by the zone and press [Enter].



Valve control

Valves can be controlled manually from the keyboard or as part of a clock or run time program. Note that if a valve position is changed during a run, it is *not* reset automatically at the end of the run unless it is configured as a gas sampling valve. For other valve types, you must include any desired resets in the program.

The valve drivers

A valve driver is the software and circuitry in the GC that controls a valve or related function. There are eight drivers known as Valve 1 through Valve 8.

Table 22 Valve Driver Characteristics

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

The internal valve drivers

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

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

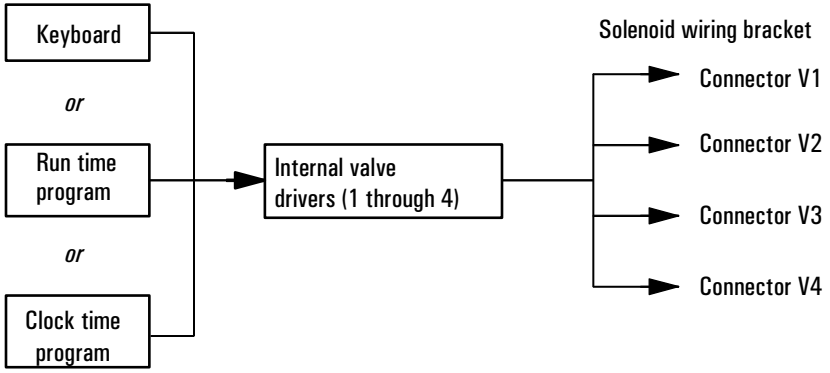


Figure 30 Internal valve drivers

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.

The external valve drivers

Valve drivers 5 and 6 control a current that may be used to drive a relay or other low-power device. Valve drivers 7 and 8 switch a current from an external source. Electrical details are in Table 22 on page 192.

These drivers, particularly Valve 7 and 8, may be used to control a motor driven multiposition valve for stream selection.

All four of these drivers appear on the External Event connector on the back of the GC.

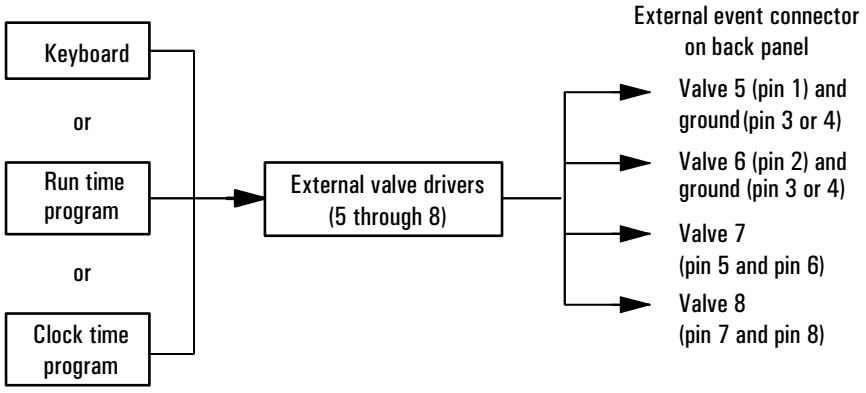


Figure 31 External valve drivers

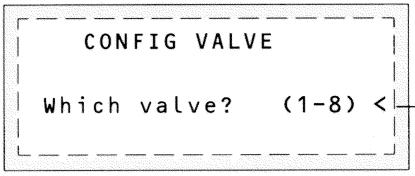
Valve configurations

There are five possible valve types:

- Gas sampling—a two-position (load and inject) valve. In load position, an external gas stream flows through an attached 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 198.
- Switching—a two-position valve with four, six, or more ports. These are general-purpose valves used for such tasks as column selection, column isolation, and many others. For an example of valve control, see page 197.
- Multiposition—also called a stream selection valve. It is usually used to select one from a number of gas streams and feed it to a sampling valve for analysis. It has a special actuator that advances the valve one position each time it is activated, or it may be motor driven. An example that combines a stream selection valve with a gas sampling valve is on page 199.
- Other—could be anything.
- Not installed—self-explanatory.

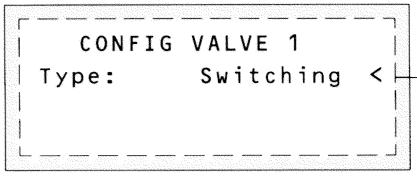
Procedure: Configuring a valve

1. Press [Config] [Valve #].

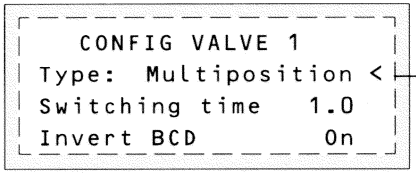


2. Specify the valve driver to be configured. Type the number (1 in this example) and press [Enter].

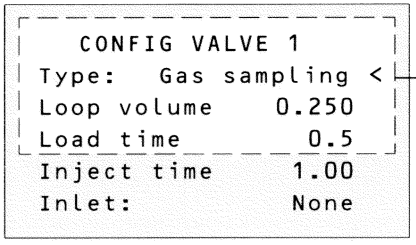
3. The present type for this driver is shown.



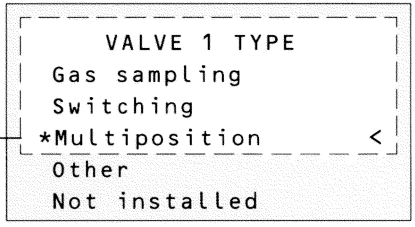
or



or



4. Press [Mode/Type] for the valve choices.



5. Select a valve type and press [Enter].

Valve control

Procedure: Controlling valves from the keyboard

Valves (except multiposition 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 chapter 7 for more information.

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.

Valve control examples

Simple valve—column selection

This is the plumbing for a single valve, configured as a switching valve, that selects one of two columns for analysis. It has no configuration parameters.

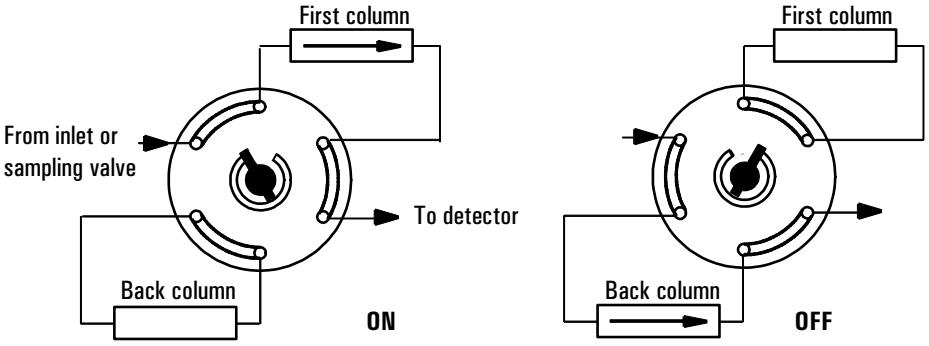


Figure 32 A column selection valve

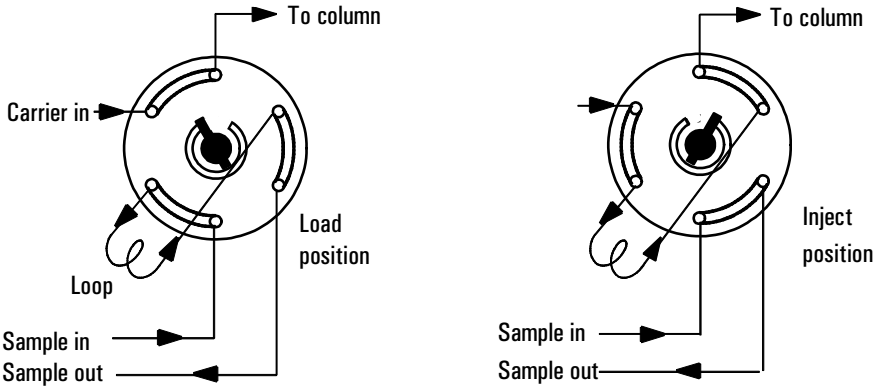
The column is selected by pressing [Valve #] <scroll to valve 2> [On] (for the front column) or [Off] (for the back column). This run table ensures that the valve is in the Off state between runs:

```
RUN TABLE ( 1 of  1)
Time:          15.00
Type:       Valve #2
Setpoint:        Off
```

Ensure that the valve is in the `Off` state between runs.

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.



Load position—the loop 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.

Figure 33 A gas sampling valve

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

```
   CONFIG VALVE 1
Type:   Gas sampling <
Loop volume     0.250
Load time         0.5
Inject time      1.00
Inlet:           None
```

`Loop volume` and `Inlet:` are information only—they do not affect operation.

Time in minutes that the valve remains in the load position before becoming ready

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 everything else 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.

Multiposition stream selection valve and sampling valve

Several manufacturers provide multiposition stream selection valves that can be driven by valve drivers 1 through 4. Only one multiposition valve can be configured. See Table 22 on page 192 for the electrical details.

If a valve is configured as a multiposition valve and has a BCD position output connected to the GC, the valve position can be selected directly.

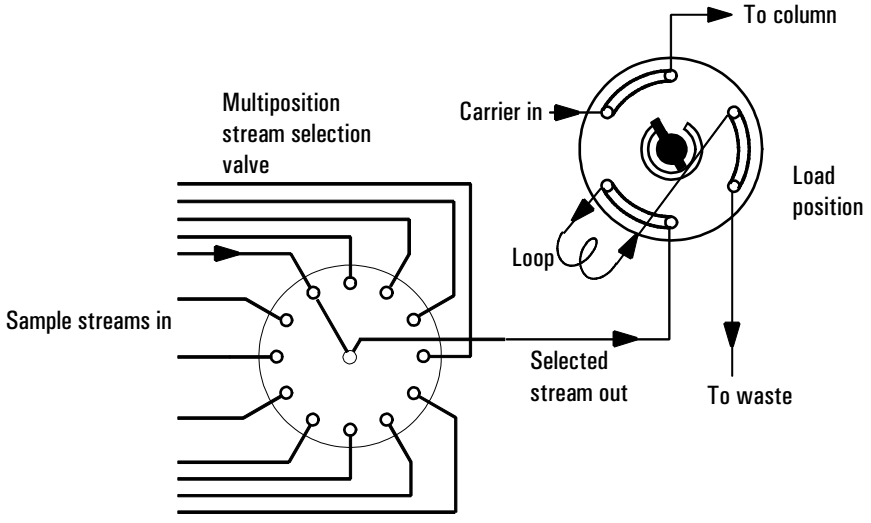
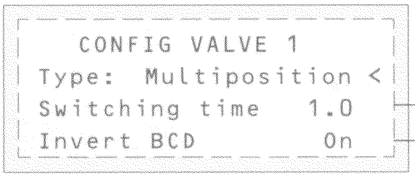


Figure 34 Multiposition valve with gas sampling valve

If the GC has one valve configured as a multiposition valve and another configured as a gas sampling valve, it assumes that they are to be used as shown in Figure 34. This “double configuration” can be used to replace an automatic

liquid sampler and sample tray in an analytical sequence. The multiposition valve becomes the sample tray; the gas sampling valve becomes the injector. See chapter 11 for details.

Two configuration parameters provide mechanical and electrical compatibility with most multiposition valve actuators.



- `Switching time`, in seconds, is a delay between successive actuator movements. It allows time for the actuator mechanism to prepare for the next movement.
- `Invert BCD` complements the BCD input—1's become 0's and 0's become 1's. This accommodates coding convention differences among manufacturers.

10

The Automatic Liquid Sampler

The injector, the sample tray, and the bar code reader

The Automatic Liquid Sampler

There are two Agilent automatic liquid sampler (ALS) systems available for the 6890 GC, the 7673 and the 7683. Refer to the ALS compatibility matrix below.

Table 23 ALS Compatibility Matrix

ALS	GC Type	GC Serial Number	G2612A ALS Interface Board installed?
7673	6890 or 6890 Plus	Any	No
7683	6890 Plus	> 20,000	Yes

Note that once a G2612A ALS Interface board is installed, that GC can use **only** the 7683 ALS.

Part 1. The 7673 ALS

This section contains information on how to configure and use your 7673 ALS.

The automatic liquid sampler system can include one or two injector towers, a bar code reader, and a tray. You use the GC keyboard to enter injector and tray setpoints and to control simple sequences. The system requires an G1512A controller module. The parts of the sampler are:

- *Injector tower*—houses a 5-µl or 10-µl syringe for sample injection. Two towers can be mounted for injection into both inlets. The tower can be lifted off the inlet and parked on posts at the back of the GC.
- *Sample tray*—holds a maximum of 100 sample vials.
- *Injector turret*—holds sample, waste, and wash vials.
- *Bar code reader*—reads and decodes several different bar codes.

For more information on the 6890 automatic liquid sampler, refer to its *Operating Manual* (Part no.07673-90187).

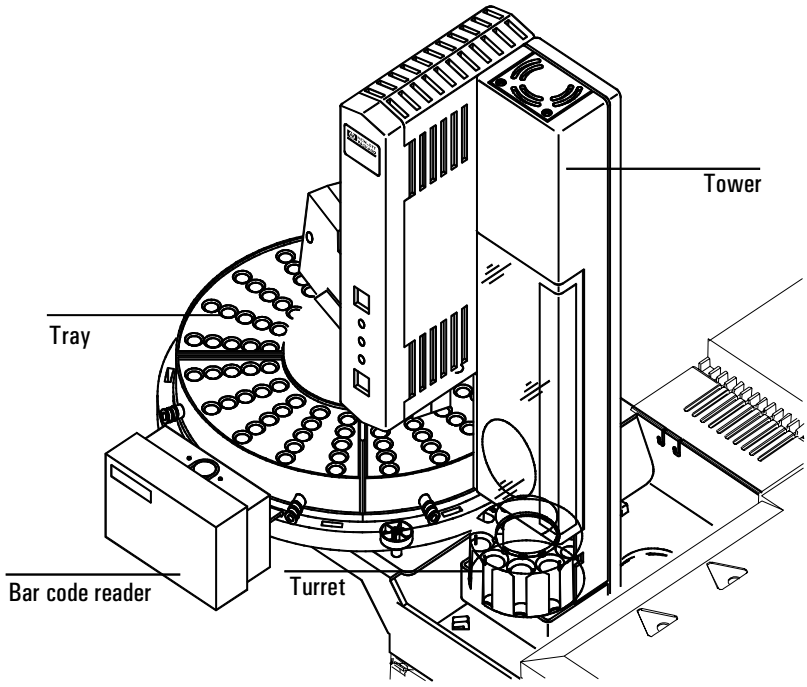
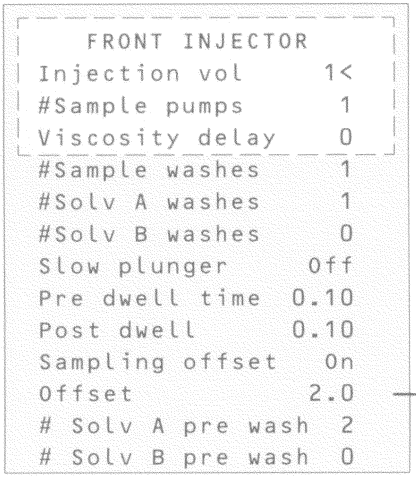


Figure 35 The 7673 automatic sampler components

Injector control table

Press [Front Injector] or [Back Injector].



`Injection volume`—Each volume unit corresponds to one-tenth the syringe capacity (enter 5 to inject half a syringe-full).

`Number of sample pumps`—How many times the syringe plunger is moved up and down with the needle in the sample to expel air bubbles and improve reproducibility.

`Viscosity delay`—How many seconds the plunger pauses at the top of the pump and injection strokes. For viscous samples, the pause allows the sample to flow into the vacuum created in the syringe.

`Number of sample washes`—How many times the syringe is rinsed with sample before the injection. The injector lowers the needle into the sample vial, fills the syringe to eight-tenths its full volume, and empties it into one of the waste bottles.

`Number of solvent A washes`—How many times the syringe is rinsed with solvent from the solvent A bottle.

`Number of solvent B washes`—How many times the syringe is rinsed with solvent from the solvent B bottle.

`Slow Plunger`—The speed of the syringe plunger during injection. Enables you to reduce the average speed of the plunger and hold the syringe needle in the inlet for 4 seconds after the injection. Turning the setpoint `On` pushes at a rate of about 5 µL/sec (with a 10 µL syringe)—`Off` is about 20 times faster. The plunger speed during the pump and waste dispensing does not change.

Procedure: Editing injector setpoints

Accessing either of the injector keys allows you to edit injector control setpoints, such as injection volumes, sample and solvent washes, etc.

To edit the injector setpoints:

1. Press [Front Injector] or [Back Injector].
2. Scroll to the desired setpoint.
3. Enter a setpoint value or turn the setpoint On or Off.

Configuring the injector

Tower position

Injector cables are connected to either the `INJ1` or `INJ2` port on the controller. This setpoint indicates which tower is on which inlet. With only one injector, you do not have to move cables when you move the tower, merely reconfigure the tower position.

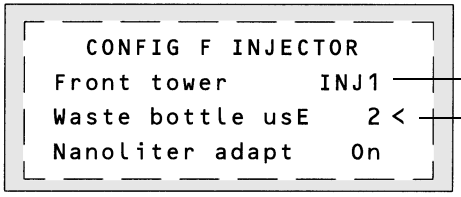
Waste bottle position

The turret waste bottle positions are controlled by entering a value of:

- 0 for the A position only
- 1 for the B position only
- 2 to alternate between the two waste bottles

Procedure: Configuring the injector

1. Press [Config][Front Injector] or [Config][Back Injector].



2. With the cursor on a tower line, use the [On] or [Off] key to set the tower position to either `INJ1` or `INJ2`.
3. Enter a value for `Waste bottle use`.

Sample tray setpoints

The sample tray delivers sample vials to the front and rear injectors according to the defined sequence parameters. There are a separate set of sequence parameters for each injector. The sample tray delivers vials to the front injector before the rear injector. Stored sequences and bar code configurations can be used to tell the sample tray where to deliver and retrieve sample vials.

`Enable tray`—turn `On` for a tray sequence, `Off` for sample bottles in the injector turret.

`Enable bar code`—turns the bar code reader on or off.

Procedure: Editing the sample tray setpoints

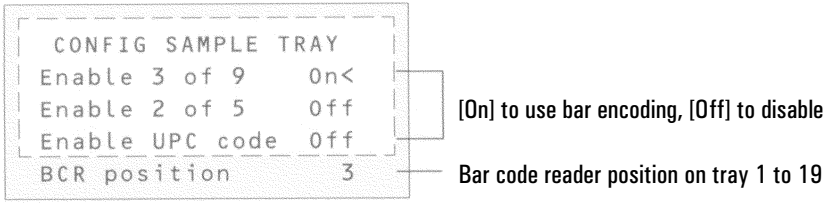
1. Press [Sample tray] to access the sample tray and bar code reader setpoints.

2. Press [On] or [Off] to enable or disable the tray.
3. Press [On] or [Off] to enable or disable the bar code reader.

Procedure: Configuring the bar code reader

The bar code reader can be configured to read specific codes on the bar code label.

1. To edit the bar code reader setpoints, press [Config][Sample Tray].



2. Press [On] or [Off] to control the following bar code setpoints.

   `Enable 3 of 9`—the 3 of 9 code offers the greatest versatility for laboratory use. It can encode both letters and numbers, plus a few punctuation marks, and message length can be varied to suit both the amount of data to be encoded and the space available.

   `Enable 2 of 5`—the 2 of 5 code is restricted to numbers but does allow variable message length.

   `Enable UPC code`—the Universal Product Code (UPC) is probably the most well-known code in use today. UPC codes are numbers-only and have fixed message length.

3. Enter a tray location to define the bar code reader position. The range is 1 to 19. Position 3 is the front of the tray.

For more information on the 18587A bar code reader, refer to its Operating Manual (Part no.18587-90107) or Installation and Service Manual (Part no. 18587-90127).

Storing injector setpoints

After setting up injector setpoints, sample tray setpoints and bar code reader configurations, store them as part of a method by following the procedures in chapter 8, “Analytical Methods.” This method becomes a part of the sequence used to run the samples.

For more information on injector sequences and sequence control, see chapter 11, *Analytical Sequences*.

Part 2. The 7683 ALS

This section describes how to configure and use your 7683 ALS.

The automatic liquid sampler system can include one or two injector towers, a bar code reader, and a tray. You use the GC keyboard to enter injector and tray setpoints and to control simple sequences. The system requires an accessory board (G2612A) installed in the GC. The parts of the sampler are:

- *Injector tower*—houses a syringe for sample injection. Two towers can be mounted for injection into both inlets. The tower can be lifted off the inlet and parked on posts at the back of the GC.
- *Sample tray*—holds a maximum of 100 sample vials.
- *Injector turret*—holds sample, waste, and wash vials.
- *Bar code reader*—reads and decodes several different bar codes.

For more information on the 6890 automatic liquid sampler, refer to its Operation Guide (Part no. G2612-90117).

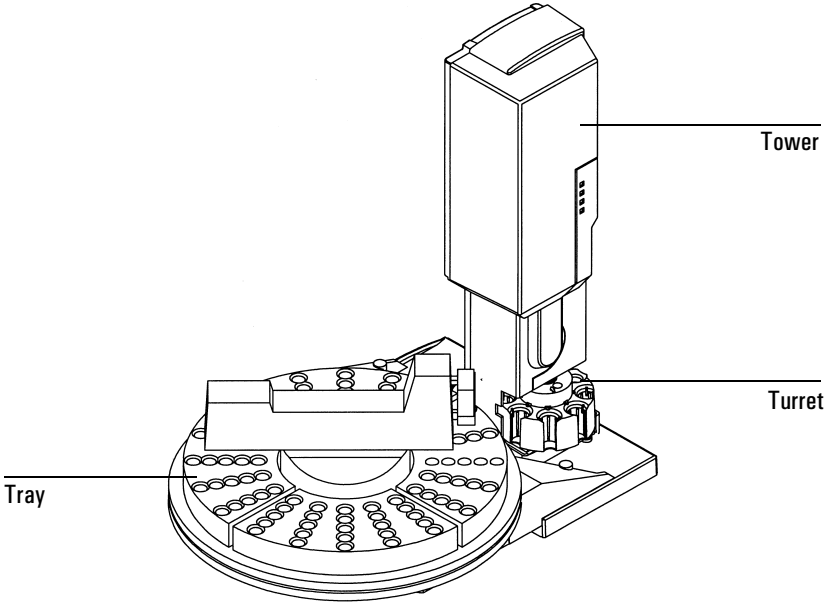
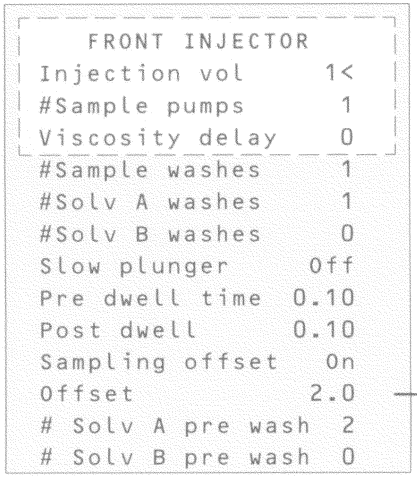


Figure 36 7683 automatic sampler components

Injector control table

Press [Front Injector] or [Back Injector].



`Injection volume`—Each volume unit corresponds to an injection volume as shown below.

	Injection volume as percent of syringe size	
Volume unit	**Nanoliter adapter On**	**Nanoliter adapter Off**
1	2%	10%
2	10%	20%
3	20%	30%
4	30%	40%
5	40%	50%

`Number of sample pumps`—How many times the syringe plunger is moved up and down with the needle in the sample to expel air bubbles and improve reproducibility.

`Viscosity delay`—How many seconds the plunger pauses at the top of the pump and injection strokes. For viscous samples, the pause allows the sample to flow into the vacuum created in the syringe.

`Number of sample washes`—How many times the syringe is rinsed with sample before the injection. The injector lowers the needle into the sample vial, fills the syringe to eight-tenths its full volume, and empties it into one of the waste bottles.

`Number of solvent A washes`—How many times the syringe is rinsed with solvent from the solvent A bottle.

`Number of solvent B washes`—How many times the syringe is rinsed with solvent from the solvent B bottle.

`Slow plunger`—The speed of the syringe plunger during injection. Enables you to reduce the average speed of the plunger and hold the syringe needle in the inlet for 4 seconds after the injection. Turning the setpoint `On` pushes at a rate of about 5 µL/sec (with a 10 µL syringe)—`Off` is about 20 times faster. The plunger speed during the pump and waste dispensing does not change.

`Dwell times`—How long, in minutes, the needle remains in the inlet before or after the injection.

`Sampling offset`—Enables variable sampling depth.

Procedure: Editing injector setpoints

Accessing either of the injector keys allows you to edit injector control setpoints, such as injection volumes, sample and solvent washes, etc.

To edit the injector setpoints:

1. Press [Front Injector] or [Back Injector].
2. Scroll to the desired setpoint.
3. Enter a setpoint value or turn the setpoint on or off.

Configuring the injector

Tower position

Injector cables are connected to either the `INJ1` or `INJ2` port on the controller. This setpoint indicates which tower is on which inlet. With only one injector, you do not have to move cables when you move the tower, merely reconfigure the tower position.

Waste bottle position

The turret waste bottle positions are controlled by entering a value of:

- 0 for the A position only
- 1 for the B position only
- 2 to alternate between the two waste bottles

Nanoliter adapter enable

On the 6890 Plus, you can use this option to vary the injection volumes available with your automatic liquid sampler. When On, injection volumes range from 2% through 40% of syringe volume. When Off, the volume range is 10% through 50%.

Use B2 wash

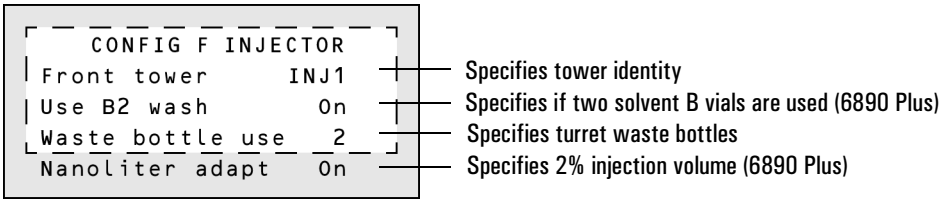
On the 6890 Plus, you can enable this option to use two 4 mL vials of solvent B, increasing the number of runs you can make before refilling solvent vials.

- Use the same solvent in position B and in position B2. (This option does not enable you to use a third solvent.)
- Use the three sample vial position turret.
- Because your solvent capacity is now 6 mL (2 mL each for solvent vials A, B, and B2), you **must** use two waste vials. See *Waste bottle position* above.
- Configure each injector separately.

Note that the number of solvent B washes for each injection does not change. The injector simply alternates use between the two solvent B vials.

Procedure: Configuring the injector

1. Press [Config][Front Injector] or [Config][Back Injector].



2. With the cursor on a tower line, use the [On] or [Off] key to set the tower position to either `INJ1` or `INJ2`.
3. Enter a value for `Waste bottle use`.

Sample tray setpoints

The sample tray delivers sample vials to the front and rear injectors according to the defined sequence parameters. There are a separate set of sequence parameters for each injector. The sample tray delivers vials to the front injector before the rear injector. Stored sequences and bar code configurations can be used to tell the sample tray where to deliver and retrieve sample vials.

`Enable tray`—turn `On` for a tray sequence, `Off` for sample bottles in the injector turret.

`Enable bar code`—turns the bar code reader on or off.

Procedure: Editing the sample tray setpoints

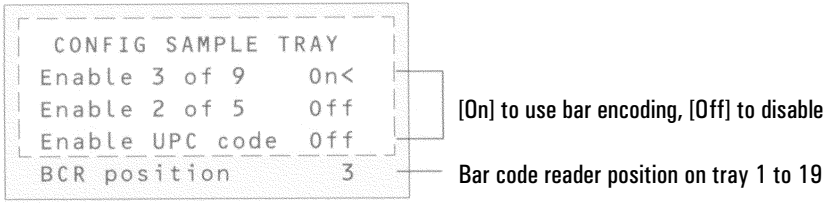
1. Press [Sample tray] to access the sample tray and bar code reader setpoints.

2. Press [On] or [Off] to enable or disable the tray.
3. Press [On] or [Off] to enable or disable the bar code reader.

Procedure: Configuring the bar code reader

The bar code reader can be configured to read specific codes on the bar code label.

1. To edit the bar code reader setpoints, press [Config][Sample Tray].



2. Press [On] or [Off] to control the following bar code setpoints.

   `Enable 3 of 9`—the 3 of 9 code offers the greatest versatility for laboratory use. It can encode both letters and numbers, plus a few punctuation marks, and message length can be varied to suit both the amount of data to be encoded and the space available.

   `Enable 2 of 5`—the 2 of 5 code is restricted to numbers but does allow variable message length.

   `Enable UPC code`—the Universal Product Code (UPC) is probably the most well-known code in use today. UPC codes are numbers-only and have fixed message length.

3. Enter 3 as the bar code reader position when it is installed in the front of the tray.

For more information on the bar code reader, refer to its Operating Manual or Installation Manual.

Storing injector setpoints

After setting up injector setpoints, sample tray setpoints and bar code reader configurations, store them as part of a method by following the procedures in chapter 8, “Analytical Methods.” This method becomes a part of the sequence used to run the samples.

For more information on injector sequences and sequence control, see chapter 11, *Analytical Sequences*.

11

Analytical Sequences

Performing multiple analyses

Analytical Sequences

What is a sequence?

A sequence specifies the samples to be run and the stored method to be used for each. It is divided into subsequences, each of which uses a single method, plus a priority sequence and post-sequence events.

A sequence can contain one to five subsequences, and can be either automatic liquid sampler- or valve-driven.

What can you do with it?

Sequences can be:

- **Created** by entering the sample and method information through the keyboard.
- **Stored** by pressing [Store] [Seq] and giving the sequence an identifying number from 1 through 5.
- **Loaded** by pressing [Load] [Seq] and specifying the sequence number.
- **Modified** by loading, making the changes you want, and then storing using the same number. The new version replaces the old one.

The stored sequence control table, Figure 37, shows the times and dates that the sequences were stored. This table is accessed by pressing [Seq]. The [Seq] key toggles between the stored sequence control table and the sequence definition control table, Figure 38.

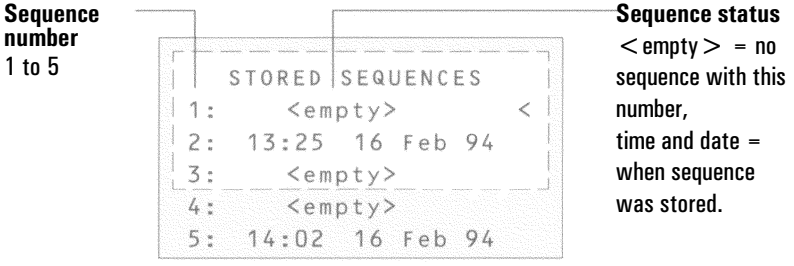


Figure 37 Stored sequence control table

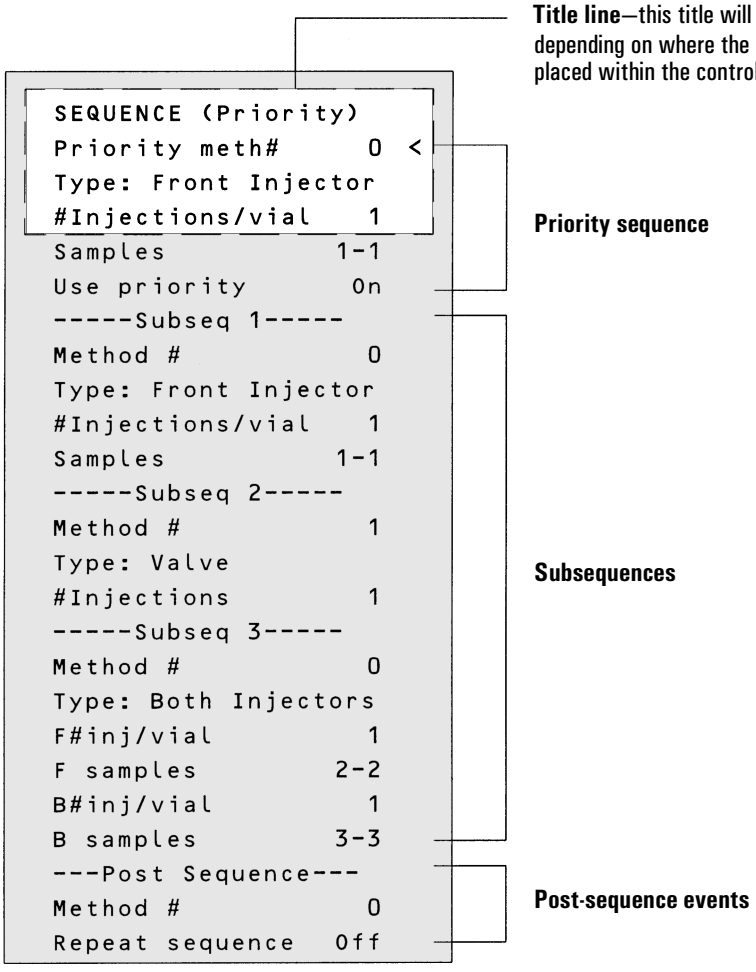


Figure 38 Sequence definition control table

When in the sequence control table, you will find the [Info] key useful if an explanation of sequence parameters is needed.

Defining a sequence

A sequence may consist of the following parts (all three are optional):

- Priority sequence—this is a special provision that allows you to interrupt a sequence to analyze urgent samples.
- Subsequences—each subsequence consists of the number of a stored method and information that defines a set of samples and calibrators to be analyzed using the method. There may be up to five subsequences.
- Post Sequence—names a method to be loaded and run after the last run in the last subsequence. Specifies whether the sequence is to be repeated indefinitely or halted.

Priority sequence

A priority sequence is a way to interrupt a running sequence to analyze one or more urgent samples. It consists of a single subsequence, either sampler or valve type, and a special `Use priority` parameter.

- If `Use priority` is `Off`, the Priority Sequence does nothing. It can be activated at any time, even when the sequence is running, by opening the sequence and changing the value to Yes.
- If `Use priority` is `On`, then:
  1. The sequence pauses when the current run ends.
  2. The priority method is loaded. The priority samples are run, as specified in the priority sequence.
  3. The `Use priority` parameter is turned `Off`.
  4. The main sequence resumes where it paused.

Sampler

```
SEQUENCE (Priority)
Priority meth #    0
Type: Front injector <
#Injections/vial     1
Samples          1-1
Use priority      On
```

Valve

```
  SEQUENCE (Priority)
Priority meth #    0
Type:          Valve<
#inj/position        1
Position rng     3-15
Times thru range    1
Use priority      On
```

Figure 39 Priority sequences

Subsequences

A subsequence can use either an automatic liquid sampler or a sampling valve for injection. It uses one method to analyze a group of samples.

Post Sequence

`Post Sequence` is a pair of events that may be applied after the last subsequence. Post Sequence may load a method—usually to shut down gases and lower temperatures—and may repeat the set of subsequences.

Procedure: Creating a sequence

1. Press [Seq] to open the sequence control table.
2. Create a Priority Sequence, if desired. This is either a valve or sampler subsequence, with two differences. The method line is labeled `Priority meth #`. An additional line, labeled `Use priority`, may be set either `On` or `Off`.
3. Create one to five subsequences. Subsequences may be either valve subsequences (page 223) or sampler subsequences (page 222). Both types can be used in the same sequence.
4. Change the Post Sequence events, if desired.
5. Store the completed sequence.

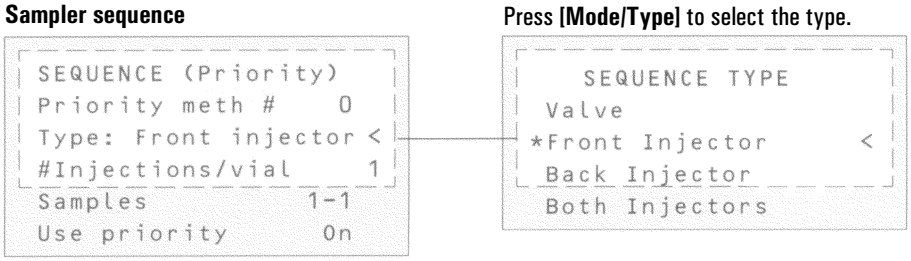
Procedure: Creating a sampler subsequence

To create a sampler subsequence:

1. Press [Seq] to open the sequence control table.
2. Scroll to a subsequence `Method #` line. If this is the Priority Sequence, the line is labeled `Priority meth #`.
3. Enter a method number. Use 0 for the currently active method, 1 to 5 for the stored methods, or Off to end the sequence.

   The active method, 0, will change during the sequence if the subsequences use stored methods. Therefore method 0 should be chosen for the Priority Sequence only if *all* subsequences use method 0.

4. Press [Mode/Type] and select one of the three injector types.



5. Supply the rest of the subsequence parameters. If you are using both injectors, there will be two sets of parameters.

   - `#Injections/vial`—the number of repeat runs from each vial. Enter 0 if no samples are to be injected.
   - `Samples`—the range (first-last) of sample vials to be analyzed.

6. If this is the Priority Sequence, set `Use priority` to `On`.

7. Proceed to the next subsequence or to the `Post Sequence`.

Procedure: Creating a valve subsequence

If your GC is equipped with a gas sampling valve and an (optional) multiposition valve, a valve subsequence can be created.

1. Press [Seq] to open the sequence control table.

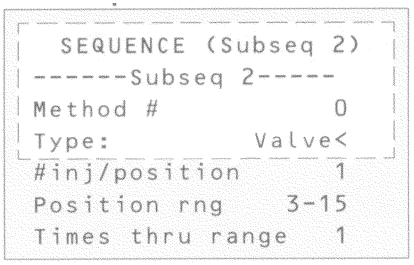
2. Scroll to a subsequence `Method # line`. If this is the Priority Sequence, the line is labeled `Priority meth #`.

3. Enter a method number. Use 0 for the currently active method, 1 to 5 for the stored methods, or Off to end the sequence.

   The active method, 0, will change during the sequence if the subsequences use stored methods. Therefore, method 0 should be chosen for the Priority Sequence only if *all* subsequences use method 0.

4. Press [Mode/Type] and select `Valve`.

With multiposition valve



Without multiposition valve

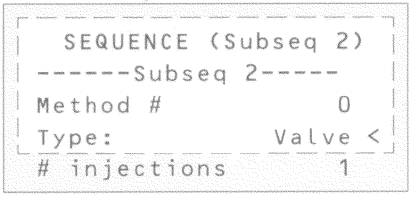


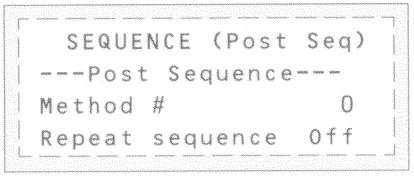
Figure 40 Valve subsequences

5. Enter the valve sequence parameters (the first three appear only if a multi-position valve is configured):

`#inj/position`	number of injections at each position, (0-99)
`Position rng`	first-last valve positions to sample, (1-32)
`Times thru range`	number of times to repeat the range, (1-99)
`# injections`	number of injections for each sample

Procedure: Setting the Post Sequence events

1. Scroll to the `Post Sequence` title section.

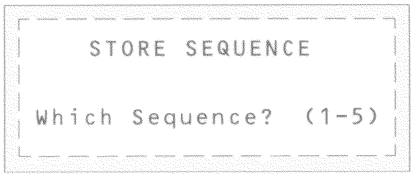


2. `Method #` is the method to be loaded and run once at the end of a sequence. Enter 1 through 5 for stored methods. If there is no method to be loaded, enter 0.

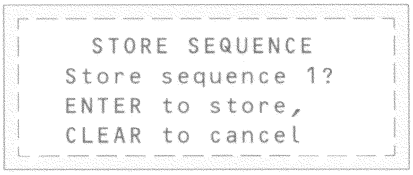
3. `Repeat sequence`—`On` keeps repeating the sequence. This function is useful for valve sequences. `Off` halts the sequence at the end. Turn `Repeat sequence` On or Off.

Procedure: Storing a sequence

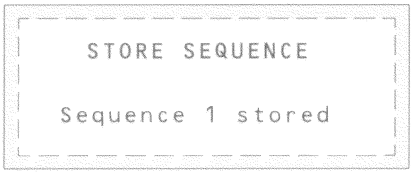
1. Press [Store][Seq] to open the Store Sequence control table.



2. Enter an identifying number for the sequence.



3. Press [Enter] to store the sequence.



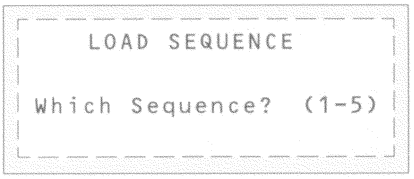
If the sequence number you specified already exists, you will be prompted to either:

- Overwrite the existing sequence, which will replace the existing sequence with the new sequence
- Cancel the store and return to the `STORED SEQUENCES` status table.

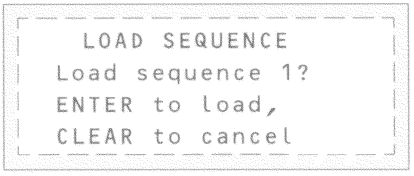
Sequences can also be stored from within the `STORED SEQUENCES` status setpoint table by scrolling the cursor to the appropriate sequence number and pressing the [Store] key.

Procedure: Loading a previously stored sequence

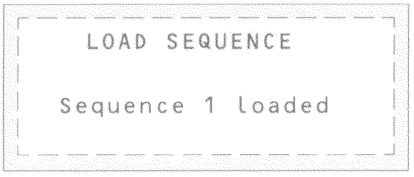
1. Sequences can be loaded by pressing [Load][Seq].



2. Press a number key to select the sequence to be loaded.



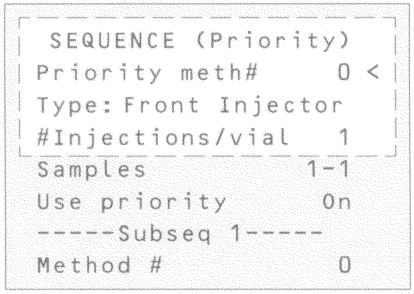
3. Press [Enter] to load the sequence or cancel this by pressing [Clear].
4. If you press [Enter], the load is confirmed. This is now the active sequence.



If the sequence number you specified has not been stored previously, you will be informed by an `ERROR:` message.

Procedure: Modifying a previously stored sequence

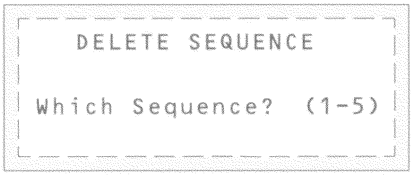
1. Load the sequence you wish to edit.
2. Open the sequence control table. Scroll to the parameter within the subsequence or Post Sequence you wish to edit.



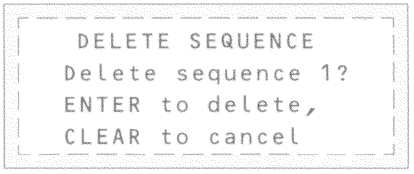
3. Make the changes.
4. To save the new values, store the sequence under its original number.

Procedure: Deleting a sequence

1. To delete a sequence, press [Delete] [Seq]. You will be prompted with:



2. Press one of the indicated number keys to select one of the five possible sequences to be deleted.



3. To delete the sequence, press [Enter]. You will see this display:

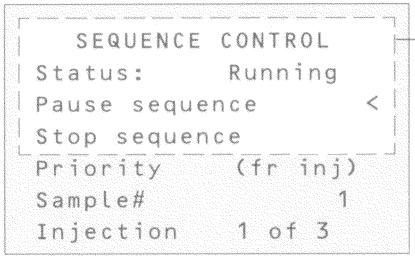
Sequence control

To access the Sequence Control table, press the [Seq control] key. This displays the current status of the active sequence.

Sequence status

There are six possible sequence status modes:

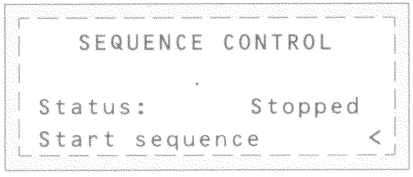
- Start/running
- Ready wait
- Paused/resume
- Stopped
- Aborted
- No sequence



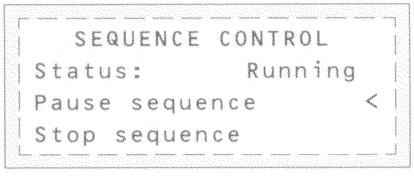
Sequence Control—diplays the current status of the sequence, shows which subsequence is currently active, current sample # or valve position, and which injection number of multiple runs is currently executing.

Procedure: Starting/running a sequence

To start a sequence, scroll to the `Start sequence` line and press [Enter].



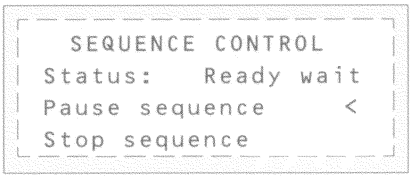
Pressing [Enter] changes the sequence status to `Running`.



The sequence continues to run until all subsequences are executed, or until one of the events described on page 231 occurs.

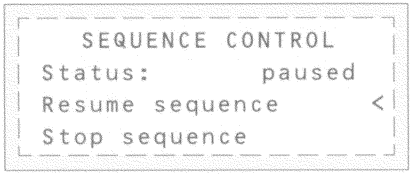
Ready wait

If a sequence is started and the instrument is not ready (due to oven temp, equib times, etc), the sequence will not start until all instrument setpoints are ready.



Procedure: Pausing and resuming a sequence

1. A running sequence can be paused by scrolling to `Pause sequence` and pressing [Enter]. Pressing [Enter] changes sequence status to `paused`, and you are given the option to resume or stop the paused sequence.



   When a sequence is paused, it stops when the current sample run is complete.

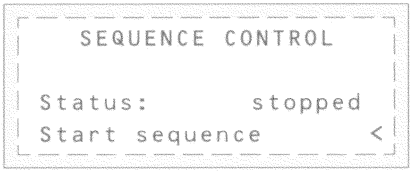
2. To continue the paused sequence, scroll to `Resume sequence` and press [Enter].

   When a sequence is resumed, it starts with the next sample.

Procedure: Stopping a sequence

To halt a sequence, scroll to `Stop sequence` and press [Enter].

When a sequence is stopped, it can only be restarted from the beginning and the sampler tray is halted immediately.

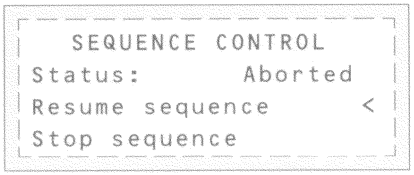


A sequence stops at the end of the last active subsequence unless `Repeat sequence` is `On` in the Post Sequence events.

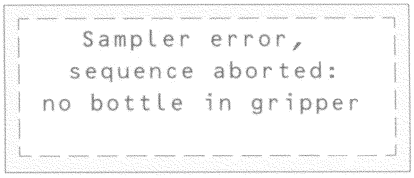
Aborting a sequence

When a sequence is aborted, it stops immediately without waiting for the current run to finish. These will cause a sequence to abort:

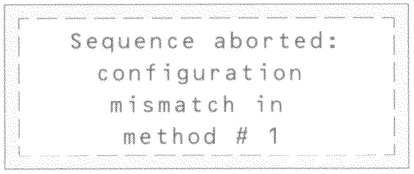
A run is stopped by pressing the [Stop] key.



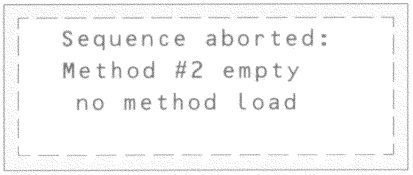
A sampler error occurs producing an error message.



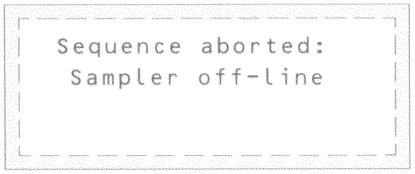
The GC detects a configuration mismatch during a method load (see chapter 8, "Analytical Methods").



A running sequence tries to load an empty method.



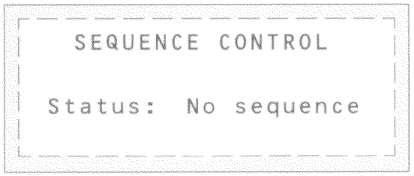
The sampler is turned off.



You can correct the problem and then resume the sequence. The aborted sample run will be repeated.

No sequence

If the sequence is off or not defined, the sequence control status will state no sequence.



To correct this, use the [Seq] key to define a sequence or turn the sequence parameters on.

Special considerations when using an integrator

The definitions of sequence are not the same in the 6890 Series GC and in the 3396 integrator. The following points must be considered when sequences are used with this GC/integrator combination:

- The integrator has only one subsequence plus the priority sequence. Only one GC method can be used within an integrator sequence.
- The ALS method parameters are prepared using the [Front injector] and [Back injector] keys on the 6890 keyboard.
- The sample information table is prepared on the integrator.
- The injection sequence parameters can be prepared either with the [Seq] key on the 6890 keyboard or in the [PREP][SEQ] dialog of the 3396. Setting sequence parameters on either instrument changes the sequence in both places.
- The Start sequence function on the GC is inactive.
- A sequence must be started from the integrator using [SEQ][START].
- If you use INET, pressing the stop key on either instrument stops the run and aborts the sequence.
- If you use cable # 03396-61010 instead of INET, the two stop keys have different effects. The [STOP] key on the integrator stops the current run and aborts the sequence. Stop on the GC stops the current run, but the sequence continues as soon as the GC becomes Ready.

12

Messages

What a message means,
and what to do about it.

Messages

The GC regularly monitors the state of its detectors, pneumatics, oven, PC boards, and other components. If a problem exists, the GC displays a message, beeps or activates an LED, and puts itself in a “safe state” if the problem could be dangerous to the user.

In addition to the information in this chapter, there is device-specific troubleshooting and maintenance information in many of the chapters in this and the *Inlets* and *Detectors* volumes.

There are six message types:

Not Ready

A Not Ready message means that some component of the GC is not ready to begin a run. When the GC is not ready, the Not Ready LED lights but there is no popup message on the display. Press [STATUS] to see a message that explains why the GC is not ready. Not ready messages are recorded in the run log.

Method Mismatches

These messages appear if you load a method that contains parameters that do not match the current GC configuration. One of two things occurs if the method and configuration do not match:

- If the parameter that does not match is set from the keyboard, the method overwrites the current parameter; the message states that the current parameter has been replaced. For example, if the gas type currently configured differs from the one in the method, the current gas type is overwritten with that of the method.
- If the parameter that does not match is hardware dependent, the method is ignored and the current setpoints remain; the message states that the method parameter is being ignored. For example, if the method indicates that the front detector is an NPD but you have replaced it with an FID, the method NPD information is ignored and the current FID parameters remain.

Warning

A Warning message means that a problem exists but that the problem will not prevent the instrument from executing the run. The GC emits one beep and a Warning message appears on the display. The GC can start the run and the warning disappears when a run starts. The warning is not recorded in the run log.

Shutdown

Shutdown occurs when there is a hardware problem that could compromise the safety of the user or damage the instrument. Before shutdown occurs, the GC emits a series of warning beeps. After a length of time specific for the component elapses, the component with the problem shuts down, the GC emits one beep, and a warning message appears. The GC is still in a ready state. No additional information appears under the [STATUS] key and the error is not recorded in the run log.

Faults

Fault messages indicate hardware problems that require user intervention. Depending on the type of error, the GC emits no beep or a single beep. The Not Ready LED lights because the GC is unable to begin a run and an error message appears. Press [STATUS] for more information. The error is recorded in the run log.

Two faults can occur that shut down the entire GC; they are a pneumatics problem for an inlet configured for hydrogen gas and a thermal runaway condition for the GC oven. In these cases, the GC beeps continuously until you press [Clear].

Bad mainboard and Fatal error messages

These messages almost always indicate that the mainboard is malfunctioning and must be replaced. These messages are not numbered and usually appear when the instrument is first turned on. See Table 24 for a list of messages. With a few exceptions which are listed in the table, if you get a Bad mainboard or Fatal error message, you will need to contact your Agilent service representative to replace the board.

Table 24 Bad Mainboard and Fatal Error Messages

Popup message	Comments
BAD MAINBOARD	
Main FPGA Failure Static RAM Failure Boot ROM Checksum	Contact your Agilent service representative.
ROM #2 Checksum ROM #3 Checksum	Either EEPROM 2 or 3 is malfunctioning. Contact your Agilent service representative.
Incorrect ROM #2 Incorrect ROM #3	EEPROM 2 or 3 is installed in the wrong position. Change the position the EEPROM is installed in; contact your Agilent service representative for more information.
ROM #2 wrong version ROM #3 wrong version	The version of either EEPROM 2 or 3 is different than that of the other EEPROMs. Contact your Agilent service representative.
DMA FPGA Failure DRAM Failure	Contact your Agilent service representative.
FATAL ERROR	
Exception Vector Bus Error Address Error Illegal Instruction Divide by Zero No 512Hz Interrupt	Contact your Agilent service representative.

Part 1. Not Ready messages

A listing of the Not Ready messages is given in Table 25 below.

Table 25 Not Ready Messages

Status message	Run log entry	Comments
Temperature zone not ready messages		
Oven temp	Not ready: Oven temp ####	See page 240.
Front inlet temp	Not ready: F inlet temp ####	
Back inlet temp	Not ready: B inlet temp ####	
Front det temp	Not ready: Front det temp ####	See page 240.
Back det temp	Not ready: Back det temp ####	
Aux 1 temp	Not ready: Aux 1 temp ####	
Aux 2 temp	Not ready: Aux 2 temp ####	
Pressure or flow not ready messages		
Front inlet pressure	Not ready:	
Front inlet flow	Not ready: F inlet flow ##.#	
Back inlet pressure	Not ready:	
Back inlet flow	Not ready: B inlet flow ##.#	See page 241.
Front det H2 flow	Not ready:	
Front det air flow	Not ready:	
F det makeup gas	Not ready:	
Back det H2 flow	Not ready:	

Table 25, continued

Status message	Run log entry	Comments
Back det air flow	Not ready:	
B det makeup gas	Not ready:	
Aux 3 pressure	Not ready:	See page 241.
Aux 4 pressure	Not ready:	
Aux 5 pressure	Not ready:	
Detector not ready messages		
Front det waiting	Not ready: Front det on wait	See page 241.
Back det waiting	Not ready: Back det on wait	See page 241.
Front det igniting	Not ready: Front det ignite	See page 242.
Back det igniting	Not ready: Back det ignite	See page 242.
Front det adjusting	Not ready: Front det adjust	See page 242.
Back det adjusting	Not ready: Back det adjust	See page 242.
Front det equib time	Not ready: Front det equib	See page 242.
Back det equib time	Not ready: Back det equib time	See page 242.
Front det shutdown	Not ready: Front det shutdown	See page 242.
Back det shutdown	Not ready: Back det shutdown	See page 242.
F NPDBead slewing	Not ready: Front NPD slewing	See page 243.
F NPDBead slewing	Not ready: Back NPD slewing	See page 243.

Table 25, continued

Status message	Run log entry	Comments
Inlet not ready messages		
Gas saver	Not ready: Gas saver active	The inlet is in Gas Saver mode. Press [Prep Run].
Front inlet purging	Not ready: F inlet purge	Inlet in split mode is purging. Press [Prep Run]. See page 244.
Back inlet purging	Not ready: B inlet purge	Inlet in split mode is purging. Press [Prep Run]. See page 244.
F inl pulse inactive	Not ready: F inlet pres pulse	Press [Prep Run].
B inl pulse inactive	Not ready: B inlet pres pulse	Press [Prep Run].
F inl VI flow idle	Not ready: F inlet VI flow	Press [Prep Run].
B inl VI flow idle	Not ready: B inlet VI flow	Press [Prep Run].
Need F inl Solv vent	Not ready: F inlet Solv. vent	Press [Prep Run].
Need B inl Solv vent	Not ready: B inlet Solv. vent	Press [Prep Run].
Valve not ready messages		
24V pneu valve drive	Not ready: 24V pneu valve drive	See page 243.
Multiposition valve	Not ready: Multiposition valve	See page 243.
Gas sampling valve 1	Not ready: Gas sampling valve 1	See page 243.
Gas sampling valve 2	Not ready: Gas sampling valve 2	See page 243.

Table 25, continued

Status message	Run log entry	Comments
Other not ready messages		
Diagnostics mode	Not ready: Diagnostics active	See page 244.
Test in progress	Not ready: Test in progress	A diagnostic test is in progress. Wait until it is completed.
Front injector	Not ready: Front injector	
Back injector	Not ready: Back injector	
Host system	Not ready: Host system	See page 244.
External device		An device connected to the Remote Start connector is not ready.
Power on in progress	Power-on restart: Blank run	See page 245.

Temperature zone not ready

Oven temp

The GC is not ready to begin a run until the oven temperature is within ±1 degree of the setpoint for the equilibration time. The GC is not ready if the oven is not turned on.

If the oven is unable to reach the setpoint, the GC remains not ready indefinitely unless the oven temperature is out of the oven range, which will cause a shutdown.

Other heated zones

The GC has a number of heated zones in addition to the oven. These are inlets, detectors, and auxiliary, or “aux,” zones. The GC is not ready to start a run until

all the zones are within ± 1°C of the setpoint *and* have maintained the setpoint temperature for 30 seconds. A zone that is turned off is considered ready.

If a temperature zone is unable to reach the setpoint, the GC remains not ready indefinitely. The GC does not shut down unless a temperature is out of the range for the zone.

Pressure and/or flow not ready

The GC will not start a run until all pressurized areas have reached their setpoints and maintained them for 6 seconds. The acceptable pressure range of an area is between 0.05 and 0.5 psi, depending on its sensor type.

Likewise, the GC is not ready to begin a run until flows are within 1 mL/min of the setpoint and remain in the range for 6 seconds. Pressure zones that are turned off are considered ready.

If the zone does not become ready within a specified time, the GC goes into shutdown mode. See the Shutdown messages for more information.

When a pressure or flow cannot become ready, check that the gas supply is on and has enough gas.

Detector not ready

Front det waiting
Back det waiting

To prevent condensation, FID and NPD temperatures must be at least 150°C before they can ignite. The FPD must be at 120°C or higher before it can ignite. The TCD must be at 100°C or higher before the filament current turns on. If temperatures are below the minimum, the GC is not ready.

If a detector is unable to reach its minimum temperature, the GC remains not ready indefinitely.

- Verify that the detector temperature setpoint is high enough for operation. Raise it if it is too low.

- If the temperature setpoint is high enough but the detector is unable to reach it, the heater may have failed or the sensor or mainboard may be bad. Contact your Agilent service representative.

Front det igniting
Back det igniting

The GC is not ready while the FID or FPD is going through the flame ignition sequence. The messages clear if the detector is turned off.

If the FID or FPD is unable to ignite, the detector may eventually shutdown. See the FID or FPD section of the *Detectors* volume.

Front det adjusting
Back det adjusting

The GC is not ready because the NPD or ECD is adjusting its baseline to reach the offset (FID) or output (ECD) setpoint. The ECD adjustment is usually complete in 30 seconds. The NPD may require an hour to adjust.

The NPD may be unable to reach the setpoint if there is contamination in the system (for example, if the gas is not pure enough or the bead is damp) or if the bead is worn out. If it cannot reach the setpoint, you will not receive an error message; the GC simply does not become ready.

Turning the detector off clears the message.

Front det equib time
Back det equib time

The NPD has completed adjusting the offset and is waiting for the value to remain at the setpoint for the equilibration time.

The NPD may not be able to equilibrate if the system is contaminated or the bead is worn out. In addition, changes in the room temperature could prevent equilibration. The GC becomes ready if the detector is turned off.

You can change the equilibration time from the Detector control menu.

Front det shutdown
Back det shutdown

The FID, FPD, NPD, or TCD shut down if they experience a pneumatics failure or if the TCD experiences a filament failure.

The GC remains not ready until the detector with the failure is turned off. Turning off the FID or FPD turns off the igniter, hydrogen flow, and air flow. Turning off the NPD turns off bead voltage, hydrogen flow, and air flow. Turning off the TCD turns off filament voltage and reference flow.

F NPD bead slewing
B NPD bead slewing

The NPD bead voltage is adjusting to a new setpoint.

Valve not ready

24V pneu valve drive

This Not Ready state means that the +24 V supply to the pneumatics valves is actually less than +16.5 V. All valves are disabled to prevent improper operation. When full voltage is restored, the GC becomes ready.

This Not Ready state could indicate a hardware problem.

Gas sampling valve 1
Gas sampling valve 2

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

Multiposition valve

The multiposition valve is causing the GC to be in a not ready state for one of the following reasons:

- The multiposition valve is not at the setpoint position. The GC remains not ready until the valve reaches the setpoint.
- The BCD cable is missing or not plugged into the receptacle. If the cable is missing, the valve will never become ready.
- The BCD setpoint is incorrect for the valve BCD output polarity. The valve will most likely shutdown with Illegal Position or Not Switching shutdown errors.

- If the valve is plugged or the sample is viscous, the switching time may be too short for the valve to switch. Increase the switching time.

Other not ready messages

Diagnostics mode

The GC is not ready when it is in diagnostics mode. The instrument is in diagnostics mode whenever a Diagnostics control table has been accessed through the [Options] key.

Exit the Diagnostics section of the keyboard for the GC to become ready.

External device

An instrument that is part of the start/stop bus is not ready. For example, the automatic liquid sampler is not ready to begin injecting. The GC becomes ready when the other instruments on the bus are ready.

Host system

The GC is not ready if the integrator, Agilent ChemStation, or other controller is not ready to begin a run. It becomes ready when the host does.

Front inlet purging
Back inlet purging

This applies only if you have a split/splitless inlet. The message appears if you try to start a run while the inlet purge valve is still in the split mode.

The inlet remains not ready and purging continues until you press the [Prep Run] key. Pressing [Prep Run] closes the valve (it also turns off the gas saver mode and increases pressure for a pressure pulse, if selected).

Power on in progress

This message appears when:

- Power is restored after a power failure during a run or while the oven was turned on and the GC was not performing a run.
- Power is turned on again after a user turned it off while the oven was turned on.

The GC heats all the other thermal zones and then heats the oven. When the oven temperature reaches the setpoint for equilibration time, the GC becomes ready.

If the power failure occurred during a run, upon power restoration the GC heats all the thermal zones and the oven and automatically performs a blank run. When the blank run is completed, the GC becomes ready.

Part 2. Shutdown messages

When the GC encounters a Shutdown condition, a popup message appears on the display. The popup message is numbered and briefly explains the problem. This chapter provides more thorough information about the problems that cause the GC or a component of the GC to shut down.

Table 26 Shutdown Messages

Shutdown no.	Popup message	Comments
1	Oven shut off	See page 247.
2	Oven cryo shutdown	See page 247.
3	Front inlet pressure shutdown	See page 248.
4	Front inlet flow shutdown	See page 248.
5	Back inlet pressure shutdown	See page 248.
6	Back inlet flow shutdown	See page 248.
7	Front detector fuel gas shutdown	See page 248.
8	Front detector air/ref shutdown	See page 248.
9	Front detector makeup shutdown	See page 248.
10	Back detector fuel gas shutdown	See page 249.
11	Back detector air/ref shutdown	See page 249.
12	Back detector makeup shutdown	See page 249.
13	Pres aux 3 shutdown	See page 249.
14	Pres aux 4 shutdown	See page 249.
15	Pres aux 5 shutdown	See page 249.
16	Multiposition valve is not switching	See page 249.
17	Can't reach setpoint of multipos valve	See page 250.
18	Front inlet cryo shutdown	See page 250.
19	Back inlet cryo shutdown	See page 250.
20	Aux 1 cryo shutdown	See page 251.
21	Aux 2 cryo shutdown	See page 251.
22	Front inlet heating too slowly: temperature shut off	See page 251.
23	Back inlet heating too slowly: temperature shut off	See page 251.

Shutdown 1—Oven shut off

The power required to keep the oven at setpoint exceeds the expected power for that temperature. The GC becomes not ready. The oven flaps open half-way (if they are operating correctly). Turn the GC off and then on again or change the oven temperature to restore operation. Possible causes include:

- Malfunctioning oven flap. Check the oven flap on the back of the GC. It should be open when cooling (for temperatures between 50 and 250°C) or closed completely to reach temperature setpoints. If the flap is stuck completely or partially open, it is not operating correctly. Contact your Agilent service representative.
- Look for thermal leaks in the oven (for example, missing insulation around an inlet or detector location or a leak in the door).
- Check for excessive load in the oven (for example, a very large packed column).
- The oven heater or the heater electronics are not operating correctly. Contact your Agilent service representative.

Shutdown 2—Oven cryo shutdown

The GC oven has shut down. Cryogenic shutdowns conserve liquid coolant when the GC is unable to start a run. A cryo shutdown does not mean that the cryogenic cooling system is malfunctioning. Instead, one of the following could be the cause:

- A “cryo timeout” has occurred. This happens if the GC oven has reached its temperature setpoint but the amount of time you specified for the cryo time-out setpoint has elapsed without a run beginning.

  Turn the oven off and then on again or change the setpoint to restore normal operation. Then turn the timeout option off to prevent another shutdown or lengthen the timeout period.

- A “cryo fault” has occurred. Cryogenic cooling has been on for over 16 minutes but the oven has not reached its temperature setpoint.

  Check the level of the cryogenic fluid and replace the supply if it is too low for proper cooling. The cryo valve may be stuck open or closed. If your fluid supply is adequate, the valve may be broken or the electronics driving it may

be malfunctioning (this is a less likely cause). Contact your Agilent service representative.

Shutdown 3—Front inlet pressure shutdown

The front inlet failed to reach its setpoint in the allotted time. The time varies with the type of inlet; it is 2 minutes for purged packed and cool on-column inlets and 5.5 minutes for the split/splitless inlet. The GC is not ready until the problem is corrected and the inlet reaches the setpoint.

Shutdown 4—Front inlet flow shutdown

The front inlet failed to reach its flow setpoint in the allotted time. In flow-control mode, the inlet has 2 minutes to reach the setpoint before shutdown. The GC is not ready until the problem is corrected and the inlet reaches the flow setpoint.

Shutdown 5—Back inlet pressure shutdown

The back inlet can not reach or maintain the pressure setpoint. See Shutdown 3.

Shutdown 6—Back inlet flow shutdown

The back inlet can not reach or maintain the flow setpoint. See Shutdown 4.

Shutdown 7—Front detector fuel gas shutdown

The front detector (EPC version) fuel gas is unable to reach or maintain the pressure setpoint in the allotted 2 minutes. The GC is not ready until the problem is corrected and the detector reaches the setpoint.

Shutdown 8—Front detector air/ref shutdown

The front detector (EPC version) air or reference gas is unable to reach or maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 7.

Shutdown 9—Front detector makeup shutdown

The front detector (EPC version) makeup gas is unable to reach or maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 7.

Shutdown 10—Back detector fuel gas shutdown

The back detector (EPC version) fuel gas is unable to reach or maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 7.

Shutdown 11—Back detector air/ref shutdown

The back detector (EPC version) air or reference gas is unable to reach or maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 7.

Shutdown 12—Back detector makeup shutdown

The back detector (EPC version) makeup gas is unable to reach or maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 7.

Shutdown 13—Pres aux 3 shutdown

The pneumatics aux 3 module can not maintain the pressure setpoint. All the detector gases are shut off and the GC is not ready. See Shutdown 3.

Shutdown 14—Pres aux 4 shutdown

The aux 4 module can not maintain the pressure setpoint. See Shutdown 3.

Shutdown 15—Pres aux 5 shutdown

The aux 5 module can not maintain the pressure setpoint. See Shutdown 3.

Shutdown 16—Multiposition valve is not switching

The multiposition valve has tried to switch twice without success. The valve shuts down and reports that it is not ready (not at setpoint). Clear the shutdown by entering a new setpoint. Possible causes include:

- The valve is not connected to the correct valve driver or is not connected at all. Connect the valve to the correct valve driver.
- The valve is stuck.
- The switching time is too short for the speed of the valve. The valve could be switching more slowly than usual because it is sticking slightly or the sample is viscous. Increase the switching time.

Shutdown 17—Can't reach setpoint of multipos valve

The valve is switching to the wrong position or is unable to switch to the setpoint position. The valve will shut down. Clear the shutdown by entering a new setpoint. Possible causes include:

- The valve position is incorrect. A setpoint was entered that the valve is unable to reach. For example, position ten was entered for an eight-port valve. Enter a correct valve position setpoint.
- The Invert BCD setpoint is incorrect. With most valves, the invert should be On. If the BCD setpoint is already On and you experience a shutdown, set it to Off.

Shutdown 18—Front inlet cryo shutdown
Shutdown 19—Back inlet cryo shutdown

The inlet is shut down. A cryogenic shutdown conserves liquid coolant when the GC is unable to start a run. A cryo shutdown does not mean that the cryogenic cooling system is malfunctioning. Instead, one of the following could be the cause:

- A "cryo timeout" has occurred. This happens if the GC inlet has reached its temperature setpoint but the amount of time you specified for the cryo timeout setpoint has elapsed without a run beginning.

  Turn the inlet off and then on again or change the setpoint to restore normal operation. Then turn the timeout option off to prevent another shutdown or lengthen the timeout period.

- A "cryo fault" has occurred. Cryogenic cooling has been on for over 16 minutes but the inlet has not reached its temperature setpoint.

  Check the level of the cryogenic fluid and replace the supply if it is too low for proper cooling. The cryo valve may be stuck open or closed. If your fluid supply is adequate, the valve may be broken or the electronics driving it may be malfunctioning (this is a less likely cause). Contact your Agilent service representative.

Shutdown 20—Aux 1 cryo shutdown
Shutdown 21—Aux 2 cryo shutdown

The Auxiliary temperature zone equipped with cryo cooling has shut down. A cryogenic shutdown conserves liquid coolant when the GC is unable to start a run. A cryo shutdown does not mean that the cryogenic cooling system is malfunctioning. Instead, one of the following could be the cause:

- A "cryo timeout" has occurred. This happens if the GC Aux zone has reached its temperature setpoint but the amount of time you specified for the cryo timeout setpoint has elapsed without a run beginning.

  Turn the zone off and then on again or change the setpoint to restore normal operation. Then turn the timeout option off to prevent another shutdown or lengthen the timeout period.

- A "cryo fault" has occurred. Cryogenic cooling has been on for over 16 minutes but the Aux zone has not reached its temperature setpoint.

  Check the level of the cryogenic fluid and replace the supply if it is too low for proper cooling. The cryo valve may be stuck open or closed. If your fluid supply is adequate, the valve may be broken or the electronics driving it may be malfunctioning (this is a less likely cause). Contact your Agilent service representative.

Shutdown 22—Front inlet heating too slowly: temperature shut off

Shutdown 23—Back inlet heating too slowly: temperature shut off

The inlet heater has been full on for a long time but the inlet temperature is not at setpoint. Either the temperature sensor for the zone has failed, or the zone's heater is defective.

Part 3. Warning messages

Table 27 lists the Warning messages for the GC. Most require Agilent service intervention. Those that users can correct are indicated along with the corrective procedures.

Table 27 Warning Messages

Warning no.	Status message	Popup message	Run log entry	Comments
100	Oven sensor missing	Oven sensor missing		
101	Invalid heater power	Invalid heater power for front detector, inlet, and aux 1		If using an MSD, make sure the Aux zone is configured for an MSD transfer line..
102	Invalid heater power	Invalid heater power for front detector, inlet, and aux 2		
103	Sig 1 buffer full	Sig 1 buffer full	Possible data loss: Sig 1 buffer full	See page 255.
104	Sig 2 buffer full	Sig 2 buffer full	Possible data loss: Sig 2 buffer full	See page 255.
105	Analog out data loss	Analog out data loss	Possible data loss: Analog out data loss	Contact Agilent service.
106	Signal data loss	Non-recoverable data loss. Data corrupt.	Possible data loss: Signal data loss	Contact Agilent service.
107	F det config changed	Front det: config changed, method defaulted		Correct the method to match your hardware.
108	B det config changed	Back det: config changed, method defaulted		Correct the method to match your hardware.
109	F inl config changed	Front inlet: config changed, method defaulted		Correct the method to match your hardware.
110	B inl config changed	Back inlet: config changed, method defaulted		Correct method to match your hardware.
111	Col 1 config changed	Column 1: config changed, method defaulted		Correct method to match your hardware.

Table 27, continued

Warning no.	Status message	Popup message	Run log entry	Comments
112	Col 2 config changed	Column 2: config changed, method defaulted		Correct method to match your hardware.
113	Aux 3 method changed	Aux 3 config changed Method defaulted		Correct method to match your hardware.
114	Aux 4 method changed	Aux 4 config changed Method defaulted		Correct method to match your hardware.
115	Aux 5 method changed	Aux 5 config changed Method defaulted		Correct method to match your hardware.
116			Log overflow	Run log capacity is 50 entries.
117	F inl calib deleted	F inl calib deleted		Inlet module is returned to default calibration.
118	B inl calib deleted	B inl calib deleted		
119	F det calib deleted	F det calib deleted		Detector module is returned to default calibration.
120	B det calib deleted	B det calib deleted		
121	P aux calib deleted	P aux calib deleted		Module is returned to default calibration.
122	Comm data overrun	Host communications: data overrun	Possible data loss: Comm data overrun	Contact Agilent service.
123	Comm data error	Host communications: data error	Possible data loss: Comm data error	Contact Agilent service.
124	Comm abnormal break	Host communications: abnormal break	Possible data loss: Comm abnormal break	Check connection.
125	Sampler data overrun	Sampler communications: data overrun	Possible data loss: Sampler data overrun	Check your sampler settings. Contact Agilent service.
126	Sampler data error	Sampler communications: data error	Possible data loss: Sampler data error	Check your sampler settings. Contact Agilent service.
127	Sampler abnormal com	Sampler communications: abnormal break	Possible data loss: Sampler abnormal com	Check connection.

Table 27, continued

Warning no.	Status message	Popup message	Run log entry	Comments
128	F inl flow cal fail	Front inlet flow sensor auto zero calib failed.		Contact Agilent service.
129	B inl flow cal fail	Back inlet flow sensor auto zero calib failed.		Contact Agilent service.
130	Aux 1 cryo disabled	Aux 1 & front inlet on same cryo valve drive: aux1 disabled		Reconfigure aux or inlet cryo drive.
131	Aux 2 cryo disabled	Aux 2 & back inlet on same cryo valve drive: aux2 disabled		Reconfigure Aux or inlet cryo drive.
132		Chgd Col 1 Init time to ###.## ; avoids Sampling End problem		
133		Chgd Col 2 Init time to ###.## ; avoids Sampling End problem		
134		Chgd FI Saver time to ###.## ; avoids Sampling End problem		For Volatiles interface, a setpoint conflicted with the Sampling End time parameter. Check your method. See the *Inlets* volume for more information.
135		Chgd BI Saver time to ###.## ; avoids Sampling End problem		
136		Chgd FI Purge time to ###.## ; avoids Sampling End problem		
137		Chgd BI Purge time to ###.## ; avoids Sampling End problem		

Warning 103–Sig 1 buffer full
Warning 104–Sig 1 buffer full

Usually, this error occurs when your data collection device (for example, a PC running an Agilent ChemStation) goes off-line while the GC is still collecting data.

Possible causes and solutions:

- There is a problem with the PC, the cabling to the PC, or the local network that links the GC to the PC. Check the PC, cabling, and network.
- The PC was turned off without closing the Agilent ChemStation instrument session. The GC collects and stores real-time plot data until the buffer overflows and the warning appears. Next time, close the instrument session before turning off the PC so that the GC stops collecting data.
- The PC entered power saver mode. When the PC enters power saver mode, its processor slows down and cannot collect data fast enough for normal communications, eventually causing the warning to appear. If the PC stays in power saver mode overnight, for example, there will be an error on the GC but the Agilent ChemStation will show a Ready status. Close and restart the instrument session, and disable the PC's power saver feature.
- There was a software problem on the PC that stops data collection.
- There is a hardware problem in the GC. If the problem persists, contact Agilent for service.

Part 4. Fault messages

Table 28 lists the Fault messages for the GC. Most require Agilent service intervention. Those that users can correct have a page reference for the corrective procedures.

Table 28 Fault Messages

Fault no.	Status message	Popup message	Run log entry	Comments
200	Pneu board FPGA	Pneumatics shutdown: faulty pneumatics board	Not ready: Pneu board FPGA	
201	Pneumatics board	Pneumatics shutdown: faulty pneumatics board	Not ready: Pneumatics board	
202	Hydrogen shutdown	Hydrogen safety shutdown	Not ready: Hydrogen shutdown	See page 262.
203	Signal DSP faulty	Signal DSP faulty	Not ready: Signal DSP faulty	
204	Sig DSP ROM broken	Sig DSP ROM broke	Not ready: Sig DSP ROM broken	
205	Sig DSP RAM broken	Sig DSP RAM broken	Not ready: Sig DSP RAM broken	
206	Sig DSP registers	Sig DSP registers	Not ready: Sig DSP registers	
207	Sig DSP data corrupt	Sig DSP data corrupt	Not ready: Sig DSP data corrupt	
208	0-1 mV out #1	Signal path test failed	Not ready: 0-1 mV out #1	
209	0-1 mV out #2	Signal path test failed	Not ready: 0-1 mV out #2	
210	Analog out #1	Signal path test failed	Not ready: Analog out #1	

Table 28, continued

Fault no.	Status message	Popup message	Run log	Comments
211	Analog out #2	Signal path test failed	Not ready: Analog out #2	
212	F det electrometer	Front detector electrometer out of specification	Not ready: F det electrometer	
213	B det electrometer	Back detector electrometer out of specification	Not ready: B det electrometer	
214	Front det flame out	Front detector flame out	Not ready: Front det flame out	See page 263.
215	Back det flame out	Back detector flame out	Not ready: Back det flame out	See page 263.
216	F TCD filament open	Front TCD filament open	Not ready: F TCD filament open	See page 263.
217	B TCD filament open	Back TCD filament open	Not ready: B TCD filament open	See page 263.
218	F TCD filament short	Front TCD filament shorted	Not ready: F TCD filament short	See page 264.
219	B TCD filament short	Back TCD filament shorted	Not ready: B TCD filament short	See page 264.
220	Heater overcurrent	Heater overcurrent. Thermal shutdown.		
221	Thermal shutdown		Not ready:	See page 264.
222	Oven temp too hot	Oven thermal shutdown	Not ready: Thermal shutdown	See page 264.
223	Oven temp too cool	Oven thermal shutdown	Not ready: Thermal shutdown	See page 264.
224	Oven temp sensor	Oven thermal shutdown	Not ready: Thermal shutdown	See page 264.
225	F det temp too hot	Front detector thermal shutdown	Not ready: Thermal shutdown	See page 264.

Table 28, continued

Fault no.	Status message	Popup message	Run log	Comments
226	F det temp sensor	Front detector thermal shutdown	Not ready: Thermal shutdown	See page 264.
227	B det temp too hot	Back detector thermal shutdown	Not ready: Thermal shutdown	See page 264.
228	B det temp sensor	Back detector thermal shutdown	Not ready: Thermal shutdown	See page 264.
229	F inl temp too hot	Front inlet thermal shutdown	Not ready: Thermal shutdown	See page 264.
230	F inl temp sensor	Front inlet thermal shutdown	Not ready: Thermal shutdown	See page 264.
231	B inl temp too hot	Back inlet thermal shutdown	Not ready: Thermal shutdown	See page 264.
232	B inl temp sensor	Back inlet thermal shutdown	Not ready: Thermal shutdown	See page 264.
233	Aux I temp too hot	Aux 1 thermal shutdown	Not ready: Thermal shutdown	See page 264.
234	Aux I temp sensor	Aux 1 thermal shutdown	Not ready: Thermal shutdown	See page 264.
235	Aux 2 temp too hot	Aux 2 thermal shutdown	Not ready: Thermal shutdown	See page 264.
236	Aux 2 temp sensor	Aux 2 thermal shutdown	Not ready: Thermal shutdown	See page 264.
237	No line interrupt	No line interrupt thermal shutdown	Not ready: Thermal shutdown	
238	Line interrupt	Faulty line interrupt thermal shutdown	Not ready: Thermal shutdown	
239	No mux ADC response	Mux ADC thermal shutdown	Not ready: Thermal shutdown	
240	Mux ADC offset value	Mux ADC thermal shutdown	Not ready: Thermal shutdown	

Table 28, continued

Fault no.	Status message	Popup message	Run log	Comments
241	Invalid line sense	Line sense reading thermal shutdown	Not ready: Thermal shutdown	
242	Aux3 faulty fact cal	Pneu aux module invalid constants from factory calibration	Not ready: Aux3 faulty fact cal	
243	Aux4 faulty fact cal	Pneu aux module invalid constants from factory calibration	Not ready: Aux4 faulty fact cal	
244	Aux5 faulty fact cal	Pneu aux module invalid constants from factory calibration	Not ready: Aux5 faulty fact cal	
245	F det module rev	Front det module: obsolete EEPROM	Not ready: F det module rev	
246	B det module rev	Back det module: obsolete EEPROM	Not ready: B det module rev	
247	F inlet module rev	Front inlet module: obsolete EEPROM	Not ready: F inlet module rev	
248	B inlet module rev	Back inlet module: obsolete EEPROM	Not ready: B inlet module rev	
249	Aux module rev	Pres aux module: obsolete EEPROM	Not ready: Aux module rev	
250	F det wrong module	Front det: non-det module	Not ready: F det wrong module	
251	B det wrong module	Back det: non-det module	Not ready: B det wrong module	
252	F inlet wrong module	Front inlet: non-inlet module	Not ready: F inlet wrong module	
253	B inlet wrong module	Back inlet: non-inlet module	Not ready: B inlet wrong module	
254	Aux wrong module	Non-aux module in pneu aux position	Not ready: Aux wrong module	

Table 28, continued

Fault no.	Status message	Popup message	Run log	Comments
255	F det invalid type	Front detector: invalid det module	Not ready: F det invalid type	
256	B det invalid type	Back detector: invalid det module	Not ready: B det invalid type	
257	F inlet invalid type	Front inlet: invalid inlet module	Not ready: F inlet invalid type	
258	B inlet invalid type	Back inlet: invalid inlet module	Not ready: B inlet invalid type	
259	F det type mismatch	Front detector: det board not the same as module	Not ready: F det type mismatch	If you installed a new detector, check that the new detector's electronics board and module are installed in the proper locations.
260	B det type mismatch	Back detector: det board not the same as module	Not ready: B det type mismatch	
261	MIO board defective	Host communications: MIO board defective	Not ready: MIO board defective	
262	RS232 defective	Host communications: RS232 defective	Not ready: RS232 defective	
263	GPIB defective	Host communications: GPIB defective	Not ready: GPIB defective	
264	Sampler RS232 defect	Sampler communications: RS232 defective	Not ready: Sampler RS232 defect	
265	F inlet invalid pid	Front inlet: invalid pids		
266	B inlet invalid pid	Back inlet: invalid pids		
267	F det invalid pid	Front detector: invalid pids		
268	B det invalid pid	Back detector: invalid pids		
269	Pneu aux invalid pid	Pneu aux module: invalid pids		
270	F inlet bad cksum	Front inlet: invalid module checksum		

Table 28, continued

Fault no.	Status message	Popup message	Run log	Comments
271	B inlet bad cksum	Back inlet: invalid module checksum		
272	F det bad cksum	Front detector: invalid module checksum		
273	B det bad cksum	Back detector: invalid module checksum		
274	Pneu aux bad cksum	Pneu aux module: invalid module checksum		
275	F inlet bad fact cal	Front inlet: invalid constants from factory calibration		
276	B inlet bad fact cal	Back inlet: invalid constants from factory calibration		
277	F det bad fact cal	Front detector: invalid constants from factory calibration		
278	B det bad fact cal	Back detector: invalid constants from factory calibration		
279	P aux bad fact cal	Pneumatics aux invalid constants from factory calibration		
280	F inlet i/o failure			
281	B inlet i/o failure			
282	F det i/o failure			
283	B det i/o failure			
284	Pneu aux i/o failure			
285	F det adjust failure	Front detector offset adjustment failed	Not ready: F det adjust failure	
286	B det adjust failure	Back detector offset adjustment failed	Not ready: B det adjust failure	

Fault 202—Hydrogen safety shutdown

An inlet configured for hydrogen gas did not reach the pressure setpoint within 2 minutes. Because hydrogen presents an explosion hazard, the following occurred:

- The GC oven fan and heaters are turned off.
- The oven flaps are fully opened.
- Both pressure and flow controls are turned off and the control parameters are flashing when viewed.
- The small zone heaters for inlets and detectors are turned off and the control parameter are flashing when viewed.
- The warning beep continues until the [Clear] key on the keypad is depressed.
- The oven cannot be turned on unless the instrument is power failed. Turn the GC power off and on again to restore operation.

The sequence would continue until the fault is fixed. To find the fault, check for the following possible causes:

- Check the gas supply pressure. Increase the pressure at the initial supply if it is too low to reach the setpoint.
- Check for a leak somewhere in the system. Leak test the gas supply tubing, the inlet, and the inlet column fittings. Leak test procedures are found with each inlet section.
- The column may be broken. Use the leak detector to check the column for leaks and replace the broken column or break off the cracked portion.
- An inlet proportional control valve may be stuck open or closed because of contamination or other fault. Contact your Agilent service representative.

Fault 214—Front detector flame out
Fault 215—Back detector flame out

This message appears when the FID or FPD is not able to ignite or if the flame goes out during a run. During the ignition process or the run, the detector will try to ignite the flame twice; if both attempts fail, the hydrogen, air, and ignitor will shut off, and the error message will appear. The detector will be in a not ready state.

- Make sure the hydrogen and air are turned on and that the flow rates are high enough for the flame to ignite.
- Use an electronic leak detector to search for and correct leaks around the detector column fitting.
- See the discussion of your detector in the *Detectors* volume to make sure that you are using the correct jet for your column.
- Change the Lit Offset to 0.5 for the FID or 0.2 for the FPD (the default value).
- If problem persists, contact your Agilent service representative.

Fault 216—Front TCD filament open
Fault 217—Back TCD filament open

The TCD filament bridge voltage indicates that the filament resistance is too high (or "open," in the electrical sense). The resistance may be too high because the filament is broken or worn thin from use, or the wires from the TCD are not connected on the detector board, or if the cell temperature sensor (ΔPRT) is shorted.

The detector will not be ready until the condition is corrected.

- Check that the wires from the detector are connected on the detector board.
- Check the cell temperature sensor (ΔPRT).
- The TCD cell must be replaced. Contact your Agilent service representative.

Fault 218—F TCD filament shorted
Fault 219—B TCD filament shorted

The TCD filament bridge voltage indicates that the resistance of the filament is too low, indicating a shorted filament. This could be caused by a worn or sagging filament or if the wires from the TCD (including the cell temperature sensor wires) are not connected properly to the detector board or are touching each other.

The detector will not be ready until the condition is corrected.

- Check that the wires from the cell are connected on the detector board properly.
- The TCD cell must be replaced. Contact your Agilent service representative.

Faults 221 to 236—Thermal shutdown

These faults cause the GC to shut down entirely. A thermal fault is detected if the oven or another heated zone is not within its allowable temperature range (lower than minimum temperature or greater than maximum temperature by 25°C). Several things could cause this error:

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

No power reaches the oven and other heated zones. The GC is not ready.

Any of the following components can experience a thermal shutdown: the oven, the inlets, the detectors, and the aux zones. In addition, problems with electronics on the main PC board can cause a thermal shutdown.

- If you see any thermal shutdown message, turn the GC off and on. If the error was caused by a power supply problem, the error will disappear and the instrument will become ready. If the error reappears, the main board or one or more of the heater/sensor assemblies must be replaced. Contact your Agilent service representative.

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